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"PREPARACION DE UN COMPOSITO DE MATRIZ TERMOPLASTICA DE ACIDO POLILACTICO (PLA) REFORZADO CON FIBRA DE YUTE"

TESIS PRESENTADA POR LOS BACHILLERES:

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RESUMEN

Las aplicaciones de materiales compuestos cada año van en aumento, así como también las exigencias en dichos materiales. Esto permite realizar estudios para mejorar sus propiedades y acoplarse a las exigencias del mundo actual. Los polímeros biodegradables se pueden combinar con fibras de plantas para producir materiales compuestos biodegradables. En este trabajo se presenta el desarrollo de materiales compuestos, obtenidos por moldeo por compresión, con matriz de PLA comercial reforzado con fibras de yute cortas y yute tejido. Se prepararon tres mezclas de compuestos usando cargas de fibra de yute cortas o picadas de 2.5 % P/P y 3.75 % P/P, la tercera mezcla es hecha con fibra de yute tejido usando una carga en peso del 11.6 % P/P. El comportamiento de estos materiales fue comparado con la respuesta del PLA sin reforzamiento. La caracterización fue hecha midiendo resistencia a la tracción, flexión, Impacto tipo Charpy y dureza así como el índice de fluidez y densidad. Las fibras no fueron sometidas a ningún tratamiento y se utilizaron diferentes cantidades y tamaños.

Los resultados mostraron que el PLA reforzado con yute, presentó un decremento en la resistencia a la tracción con respecto al PLA sin carga hasta en un 37.96 %. Sin embargo, hay un incremento de la elongación del material compuesto en un 157 %. Las muestras compuestas fallaron de forma frágil bajo carga de tracción. Las otras propiedades evaluadas nos han mostrado que el yute mejora las propiedades como la flexión la cual aumento hasta en un 22.45 %. La Dureza también incremento hasta en un 6.22%; y en Charpy aumento la energía absorbida hasta en un 145.22 % comparándolo con el PLA puro.

ABSTRACT

The composite applications are increasing each year, as well as the demands on the materials. This allows to realize studies to improve their properties and engage the demands of today's world. Biodegradable polymers can be combined with plant fibers to produce biodegradable composites. This work presents the development of composite materials obtained by compression molding, commercial PLA matrix reinforced with short fibers woven jute and jute. Three mixtures of compounds were prepared using fiber fillers short jute or chopped to 2.5% w / w and 3.75% w / w, the third mixture is made with fiber woven jute using a load weight of 11.6% w / w. The behavior of these materials was compared with the response of PLA without reinforcement. The characterization was done by measuring tensile strength, bending strength and Charpy impact type and the melt index and density. The fibers were not subjected to any treatment and were used different amounts and sizes.

The results showed that the jute reinforced PLA presented a decrease in tensile strength compared to PLA unloaded up 37.96%. However, there is an increase in elongation of the composite material 157%. Composite samples failed so fragile under tensile load. The other evaluated properties have shown that jute improves properties such as bending which increased up to 22.45%. Hardness also increase up to 6.22%; and Charpy absorbed energy increase up to 145.22% compared to the pure PLA.

DEDICATORIA

Con todo mi cariño y mi amor para las personas que hicieron todo en la vida para que yo pudiera lograr mis sueños, por motivarme y darme la mano cuando más lo necesitaba, a ustedes que por siempre me han enseñado a que nada es imposible y que grandes retos ameritan grandes sacrificios. Gracias por todo mamá Delia y papá Octavio.

Jorge Rodríguez

Dedicado a mis Padres, Carmen J. y Edwin que gracias a ellos tuve todo para salir adelante, por su confianza y apoyo en todo momento, siendo el motivo de esforzarme cada día más y ser el mejor en todo lo que me proponga.

Edwin Tejeda

Gracias a los ingenieros que nos apoyaron a lo largo de nuestra carrera, y en especial a los ingenieros que nos guiaron para la culminación de nuestra tesis.

Jorge Rodríguez / Edwin Tejeda

INDICE GENERAL

CAPÍTULO 2. MATERIALES Y METODOLOGÍA EXPERIMENTAL

CAPÍTULO 3. ANÁLISIS Y DISCUSIÓN DE RESULTADOS

ÍNDICE DE FIGURAS

INDICE DE TABLAS

INDICE DE CUADROS

INDICE DE GRAFICAS

INTRODUCCION

Los polímeros biodegradables han sido objeto de investigación y desarrollo desde principios de 1970 y la creciente sobre explotación de los recursos del mundo, así como las preocupaciones acerca de la eliminación de los plásticos derivados del petróleo ha llevado a aumentar el interés y las investigaciones [1]. Polímeros biodegradables o biopolímeros, en la actualidad están capturando el interés de una amplia gama de aplicaciones de alto rendimiento en especial en productos y aplicaciones de la medicina a través de los productos básicos: películas y envases [2].

Un número de polímeros biodegradables disponibles comercialmente, tales como polihidroxialcanoato (PHA), poli (ácido L-láctico) (PLLA), policaprolactona (PCL), poliesteramida (PEA), etc. se utilizan con frecuencia como matrices de materiales compuestos de fibra naturales. De estos polímeros biodegradables, el PLA tiene el mayor potencial en las industrias de compuestos debido a su fácil disponibilidad, buena biodegradabilidad y propiedades mecánicas [3]. Aunque PLA tiene propiedades superiores a las de polietileno de tereftalato (PET) en diversas aplicaciones, algunos inconvenientes tales como la fragilidad han limitado su uso extensivo [4]. Sin embargo, por la incorporación de fibra natural en la matriz de PLA se puede mejorar las propiedades generales de los materiales compuestos.

El Ácido Poliláctico o PLA es posiblemente el polímero biodegradable que tiene el mayor potencial comercial. PLA es un polímero de ácido láctico y es producida comúnmente por polimerización de apertura de anillo del dímero cíclico láctida [5].

PLA también puede ser producido por policondensación directa de ácido láctico y aunque esta ruta generalmente produce especies de bajo peso molecular, los procedimientos especiales pueden superar este problema [6]

Los Compuestos de Fibra corta natural / PLA se han investigado en varios estudios [7, 8, 9, 10] el promedio de resistencia a la tracción encontrado fue de aproximadamente 50 MPa, utilizando moldeo por compresión y la técnica de moldeo por inyección.

Los compuestos de fibra larga de cáñamo / PLA fueron estudiados por Graupner et al. (2009). Por otro lado, los tejidos están siendo estudiados como refuerzos, ya que proporcionan propiedades mecánicas aceptables, procesamiento y conformabilidad simple para aplicaciones estructurales avanzados [11]. Compuestos de tela tejida poseen propiedades más equilibradas que las láminas unidireccionales. El tejido de la fibra aumenta la fuerza de unión fibra-matriz. Puesto que las fibras se unen juntas firmemente en tejidos, por lo tanto se necesita más fuerza para la descomposición del compuesto reforzados con tejido [12].

Por lo tanto, tejidos de yute son utilizados en esta investigación para fabricar compuesto por moldeo por compresión. Con el fin de preparar y caracterizar los materiales compuestos de matriz de Ácido Poliláctico (PLA) reforzado con fibra vegetal de Yute para la aplicación en la ingeniería.

OBJETIVO PRINCIPAL

Preparar y caracterizar los materiales compuestos de matriz de Ácido Poliláctico (PLA) reforzado con fibra vegetal de Yute para la aplicación en diferentes componentes utilizados en la ingeniería.

OBJETIVOS ESPECÍFICOS

1. Evaluar la mejora que produce en las propiedades mecánicas de tracción la incorporación de fibras vegetales de yute.

2. Determinar la variación que produce la incorporacion de fibras de yute en la energía de impacto.

3. Analizar la interfase generada en la unión de la matriz de Ácido Poliláctico (PLA) con la fibra vegetal de yute.

JUSTIFICACIÓN

1. En la actualidad el consumo de las piezas de plásticos en las diferentes industrias se ha incrementado, como alternativa a la contaminación del medio ambiente, se propone el uso de los polímeros biodegradables, cuya demanda ha crecido significativamente.

2. Los materiales compuestos en la actualidad son muy utilizados por su versatilidad y por tener propiedades mecánicas, químicas, etc. muy buenas.

3. En la Universidad Católica de Santa María se ha realizado la tesis de "OBTENCIÓN Y CARACTERIZACIÓN MECÁNICA DE COMPÓSITOS CON MATRIZ DE BIOPOLÍMERO TERMOPLÁSTICO DE PLA" en la cual se trabajo con fibra de soga. Ambos componentes utilizados fueron biodegradables.

4. El objetivo de este proyecto es preparar un material compuesto en base a PLA y fibra vegetal de yute, ambos componentes son biodegradables. La tesis estará centrada en mejorar las propiedades mecánicas del PLA mediante el refuerzo con fibras yute.

5. El proyecto de tesis demostrará la sinergia que existe entre un material polimérico biodegradable con un material natural.

CAPÍTULO 1

MARCO TEÓRICO

1.1 Historia del Plástico

Los materiales plásticos en la actualidad ya alcanzaron un siglo de existencia pesar que las investigaciones que permitieron su desarrollo y producción datan de mucho tiempo atrás. Hoy en día a nivel mundial casi todos los productos y objetos que tenemos en casa contienen plásticos debido a su gran versatilidad.

Desde la antigüedad los Hombres han usado polímeros de origen natural para satisfacer algunas de sus necesidades. El asfalto, por ejemplo, era utilizado en el medio oriente en tiempos bíblicos y el algodón, en México, era conocido antes de la llegada de Colón. Además en épocas precolombinas el látex o caucho era conocido por algunos pueblos americanos y los Mayas lo emplearon para fabricar balones para jugar. Colón y otros exploradores que visitaron este continente quedaron fascinados con este material y llevaron a Europa muestras de este material. Algunos importantes desarrollos del caucho y plástico sucedieron en Massachusetts empezando con Charles Goodyear que en 1839 descubrió el proceso de vulcanización del caucho natural cerca de Weburn. La UMASS LOWELL (Lowel Technological Institute) fue la primera universidad en Estados Unidos de Norteamérica en ofrecer la carrera de Ingeniería en plásticos. [13].

Charles Goodyear descubrió que aumentando azufre al caucho natural este mejoraba su elasticidad y su resistencia. Posteriormente a este caucho “sulfurado” se lo conoció como caucho “vulcanizado” y siendo el material con el que en la actualidad se producen las llantas.

Después de unos pocos años Alfred Critchlow aplicó el moldeo por compresión y en 1847 fundó la compañía "Pro Molding Corporation" y fue esta la primera compañía de moldeo de plásticos en los Estados Unidos.

Los usos que tenía el caucho cada día iban aumentando a pasos agigantados, lo cual creo el interés en Alexander Parkes para crear otro material que pudiera ser utilizado en vez del caucho, es así que estudiando el nitrato de celulosa, Parkes obtiene un nuevo material y en 1862 en Londres, Inglaterra mostró el nuevo plástico semi.-sintético el cual era un material orgánico que contenía Nitrato de Celulosa y un solvente, a este nuevo plástico se lo conoció como Parkesine, en honor a su apellido [13]. Parkesine podía ser moldeado o tallado para obtener productos como botones, peinillas, marcos de cuadros de pintura y mangos de cuchillo, Sin embargo por su relativo alto costo no tuvo mucha acogida.

John Wesley Hyatt en 1869 obtuvo un compuesto a base de nitrocelulosa con camphor (savia que proviene del árbol de laurel) se lo conoció como Celuloide (también conocido como Pyroxylin). El celuloide fue el primer plástico semi-sintético comercializado a gran escala para ser utilizado en la fabricación de bolas de billar, marcos de lentes, collares y películas cinematográficas.

En 1907 el Dr. Leo H. Baekeland obtuvo el primer plástico sintético que resultó mediante la reacción del formaldehido y fenol. Al resultado de esta reacción se lo llamó Bakelite. Entre sus propiedades esta de ser resistente a altas temperaturas, químicamente estable, resistente a la electricidad, rígido y resistente a la humedad.

Luego entre 1914-1918 durante la Primera Guerra Mundial se aumentó el uso del celuloide y sus derivados, el fin de la guerra permitió sacar como resultado de las investigaciones la llamada seda artificial, o rayón.

En la década de los 30, químicos ingleses descubrieron que el gas etileno polimerizaba bajo la acción de la presión y el calor, formando un termoplástico al que llamaron polietileno (PE).

Luego vino la comercialización de Policloruro de vinilo PVC que fue gracias al trabajo de investigación que dirigió Waldo Semon, químico que trabajaba en B.F. la Goodrich Rubber Company.

A pesar que en 1829 el poliestireno (PS) se descubrió accidentalmente por un alemán, fue hasta 1930 que un científico de la Corporación de BASF desarrolló un proceso comercial para la fabricación de PS.

Después en 1937 se descubrió el polimetil metacrilato (PMMA) o bien conocido como el acrílico (Plexiglas).

Posteriormente se desarrolló una nueva fibra sintética que podía reemplazar a la seda siendo esta conocido como " nilón 66 " el cual fue comercializado en 1938, fabricándose cepillos dentales que anteriormente eran hechos de pelo de animal (el jabalí chino). Durante la Segunda Guerra Mundial, el nilón se usó por ejemplo para neumáticos para los bombarderos, sogas de remolque, los paracaídas, toldos para evitar los mosquitos, y ropa usada en la selva.

Los plásticos se dividen en dos grandes grupos: el de los termoestables y el de los termoplásticos. Los primeros, también llamados termorígidos, son los que fraguan y todo otro proceso de calentamiento solamente los arruina o destruye los segundos se caracterizan porque todo nuevo calentamiento los retorna a un estado de plasticidad tal que se los puede reconstruir, reelaborar.

En 1941 apareció el polietileno tereftalato (PET) un termo-plástico hecho por la reacción de la condensación de etileno glicol y el ácido tereftálico. El PET se usó inicialmente para la producción de Dacron (fibra textil sintética). Sin embargo, actualmente es usado para la fabricación de las botellas.

El científico Roy Plunkett que trabaja en DuPont, descubrió en 1938 accidentalmente el politetrafluoretileno teflón (PTFE), un plástico químicamente resistente y resbaladizo, pero la introducción en mercado no ocurrió hasta 1946.

En 1953 General Motors introdujo el corvette que usaba el plástico reforzado con fibra de vidrio en todo su cuerpo.

En el mismo año Hermann Schnell de Bayer A.G. en Alemania y Daniel Fox de la Compañía Eléctrica General en Estados Unidos descubrieron en forma separada el Policarbonato. Este es un termoplástico transparente con buenas propiedades ópticas ofrece un equilibrio entre tenacidad y dureza, resistencia al calor y por ser aislantes eléctrico.

Durante la II Guerra Mundial, por la reducción de suministros de materias primas. La industria del plástico fue una fuente inagotable de sustitutos aceptables. Alemania, por ejemplo inició un gran programa que llevó al desarrollo de un caucho sintético utilizable.

En 1955 gracias a los trabajos combinados de Karl Ziegler y Guilio Natta se obtuvo un polipropileno cristalino y surgió el concepto de estéreo regularidad que les valió la concesión del premio Nobel en 1955. Más adelante las investigaciones de otro destacado científico, Paul J. Flory, también le hicieron acreedor del premio Nobel en 1974.

A partir de entonces el desarrollo de nuevas tecnologías, materiales y aplicaciones de los polímeros ha sido explosivo.

1.2. Clasificación de los Polímeros

1.2.1 Polímeros Sintéticos

Los polímeros sintéticos se clasifican de acuerdo a su mecanismo de obtención o también llamado mecanismo de polimerización en [14]: ·

- **Polímeros de adición**, se obtienen por adición rápida de una molécula (Monómero) a la vez a una cadena creciente del polímero (macromolécula); por lo general, a través de un reactivo intermediario (catión, radical o anión) en el extremo creciente de la cadena. Se emplea para obtener polímeros a partir de monómeros vinílicos, es decir, pequeñas moléculas conteniendo dobles enlaces carbono-carbono. Entre los polímeros obtenidos por polimerización por radicales libres tenemos el poliestireno, el poli (metacrilato de metilo), el poli (acetato de vinilo) y el polietileno ramificado.
- **Polímeros de condensación**, Los polímeros lineales se forman también por reacciones de condensación (también llamada por etapas), Normalmente el calor, la presión o un catalizador causan que proceda la reacción. En este caso la formación de polímeros, implica más de una especie monomérica y generalmente se origina un subproducto (o producto secundario) de bajo peso molecular, como por ejemplo agua o alcoholes, que

se eliminan. Las sustancias reactivas tiene fórmulas químicas diferentes a la unidad que se repite y la reacción intermolecular ocurre cada vez que se repite la unidad repetitiva. Los tiempos de reacción para la polimerización por condensación son generalmente mayores que los de la polimerización por adición.

1.2.2 Polímeros Naturales

Cuando los encontramos en la naturaleza (celulosa, caucho, resinas vegetales, etc.) y se obtienen por biosíntesis como los polisacáridos, proteínas, cauchos naturales, celulosa, lignina, etc.

1.3 Características generales de los polímeros

1.3.1 Estado Amorfo o Estado Cristalino

La mayoría de los polímeros muestran al mismo tiempo las características de solidos cristalinos y amorfos, los análisis de MEB y DRX muestran señales características de solidos cristalinos y señales anchas, correspondientes a los sólidos amorfos. Los términos cristalino y amorfo son usados para el ordenamiento y desordenamiento de las regiones del polímero, un mismo polímero muestra diferentes grados de cristalinidad. El término semicristalino es usado para referirse a polímeros que son parcialmente cristalinos. Los polímeros termoplásticos, pueden ser [15]

1.3.1.1 Estado Amorfo.

El estado amorfo se caracteriza por la presencia de irregularidad en la estructura polimérica. El estado amorfo se presenta principalmente en polímeros ramificados, polímeros atácticos y copolímeros con cantidades significativas de dos o más constituyentes monoméricos muy

diferentes. Son de cadena larga con baja cristalinidad y se hacen elásticos cuando se calientan por arriba de la temperatura de transición vítrea “Tg”.

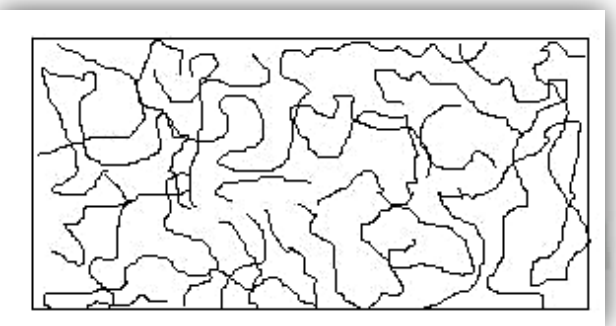


Figura 1. Modelo esquemático del estado Amorfo.

1.3.1.2 Estado cristalino

El estado cristalino lo muestran moléculas química y geométricamente regulares en su estructura, con la capacidad de cristalizar. Limitan la cristalización, pero no evitan que ocurra, las irregularidades como las ramificaciones de la cadena o la copolimerización. Será cristalino aquel polímero muy regular que se empaca bien en una red cristalina y por lo general, con mayor densidad, más fuerte y rígido que un polímero semejante con menor grado de cristalinidad. Sin embargo, el estado cristalino en los polímeros es en realidad de doble fase, en donde coexiste la fase cristalina junto con la amorfa por los que estos materiales deberían ser llamados como semi-cristalinos.

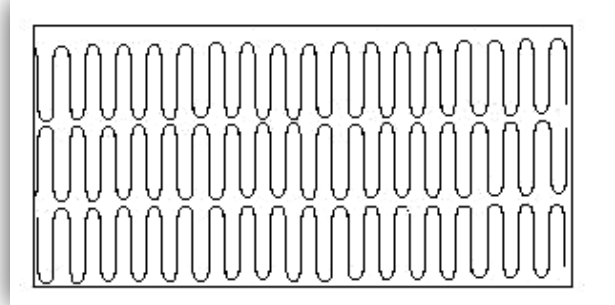


Figura 2. Modelo esquemático del estado cristalino.

Figura 3. Modelo esquemático del estado semi-cristalino.

1.3.2. Procedimientos de formación de las macromoléculas.

Existen diversos procesos para juntar moléculas pequeñas con otras para formar moléculas grandes. Para clasificarlos nos basamos en el mecanismo por el cual se unen estructuras monómeras o en las condiciones experimentales de reacción.

1.3.2.1. Polimerización por adicción.

En las reacciones de adición, varias unidades monoméricas o meros se unen, en presencia de un catalizador, como resultado de la reorganización de los enlaces “Carbono-doble enlace-Carbono”(C=C) de cada una de ellas.

- Adición de moléculas pequeñas (monómeros) de un mismo tipo unas a otras por apertura del doble enlace sin eliminar ninguna parte de la molécula:

- **Polimerización de tipo vinilo:** Los polímeros vinílicos son polímeros obtenidos a partir de monómeros vinílicos; es decir, pequeñas moléculas conteniendo dobles enlaces carbono-carbono. Por ejemplo:

  El polietileno se obtiene a partir del monómero etileno. Cuando polimeriza, las moléculas de etileno se unen por medio de sus dobles enlaces, formando una larga cadena de varios miles de átomos de carbono conteniendo sólo enlaces simples entre sí.

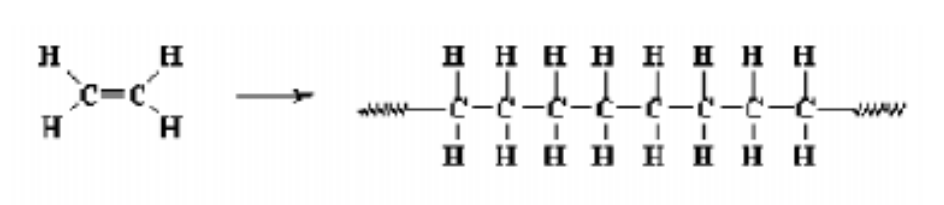


Figura 4. Polimerización del monómero de etileno

- Polimerización tipo epóxido.

- Adición de pequeñas moléculas de un mismo tipo unas a otras por apertura de un doble enlace con eliminación de una parte de la molécula

- Polimerización alifática del tipo diazo
- Polimerización del tipo aminocarboxianhidro

- Adición de birradicales formados por deshidrogenación

- Polimerización tipo p-xileno

1.3.2 2. Polimerización por condensación.

Los polímeros lineales se forman también por reacciones de condensación (llamada también por etapas), proporcionando estructuras y propiedades que se asemejan a las de polímeros lineales por adición. Normalmente el calor, la presión o un catalizador causan que proceda la reacción.

En este caso la formación de polímeros, implica más de una especie monomérica y generalmente se origina un subproducto (o producto secundario) de bajo peso molecular, como el agua o alcoholes, que se eliminan.

Las sustancias reactivas tiene fórmulas químicas diferentes a la unidad que se repite y la reacción intermolecular ocurre cada vez que se repite la unidad repetitiva.

Los tiempos de reacción para la polimerización por condensación son generalmente mayores que los de la polimerización por adición.

Las reacciones de condensación forman a menudo meros trifuncionales capaces de generar polímeros reticulados o entrecruzados.

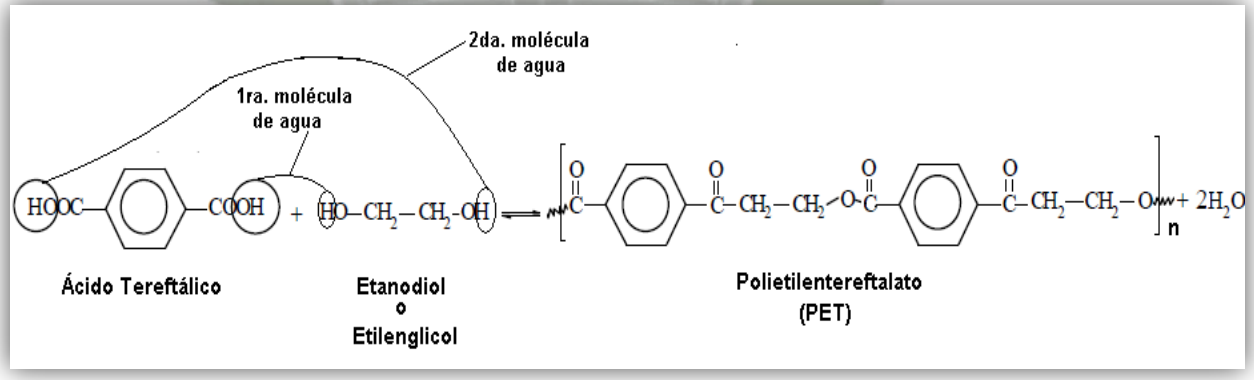


Figura 5. Obtención del PET por polimerización por condensación

- Formación de poliamidas, poliésteres, poliésteres, polianhidros, etc., por eliminación de agua o alcoholes, con moléculas bifuncionales, como ácidos o glicoles, diaminas, diésteres.
- Formación de polihidrocarburos, por eliminación de halógenos o haluros de hidrógeno, con ayuda de catalizadores metálicos o de haluros metálicos.
- Formación de polisulfuros o poli-polisulfuros, por eliminación de cloruro de sodio, con haluros bifuncionales de alquilo o arilo y sulfuros alcalinos o polisulfuros alcalinos.

1.3.3. Monómeros y polímeros

Los materiales están formados por moléculas que pueden ser de tamaño normal o moléculas gigantes (macromoléculas) llamadas polímeros.

La unidad estructural de bajo peso molecular es el monómero: Molécula generalmente orgánica, con la capacidad de combinarse con otras moléculas de su misma naturaleza para formar un producto de elevado peso molecular, denominado polímero.

Los polímeros, se producen por la unión de varios de cientos de miles de moléculas pequeñas (monómeros) que forman enormes cadenas de diferentes formas. Pueden pertenecer a la química orgánica (grasas, propilenos, proteínas, etc.) o a la química inorgánica (porcelana, cemento, vidrio, etc.).

Si el polímero es bastante uniforme en estructura molecular y peso molecular, su grado de polimerización es indicado por un numeral griego, según el número de unidades de monómero que contiene; así, hablamos de dímeros, trímeros, tetrámero, pentámero y sucesivos. El Grado de polimerización (n o GP) indica cuantas unidades repetitivas o meros

se encuentran en un polímero, se suele indicar con una "n" al final de los corchetes que indican la unidad monomérica.

1.3.4. Mecanismo de la polimerización

La polimerización implica la adición de radicales libres al doble enlace del monómero y se lleva a cabo mediante tres etapas bien diferenciadas: iniciación, propagación y terminación.

a) Iniciación:

Esta fase involucra la creación del "centro activo" del radical libre y normalmente tiene lugar en dos pasos. El primero es la formación de radicales libres a partir del iniciador y el segundo es la unión de uno de estos radicales libres a una molécula de monómero:

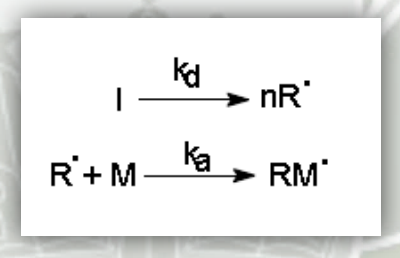


Figura 6. Iniciación

Donde "I" representa el iniciador, "R"· al radical libre formado en la descomposición del primero, "M" el monómero, kd y ka las constantes de descomposición del iniciador y de iniciación respectivamente.

Los radicales se pueden generar mediante la descomposición térmica o fotoquímica de sustancias como peróxido de benzoílo (PBO) o del azobisisobutironitrilo (AIBN) tal como se muestra a continuación:

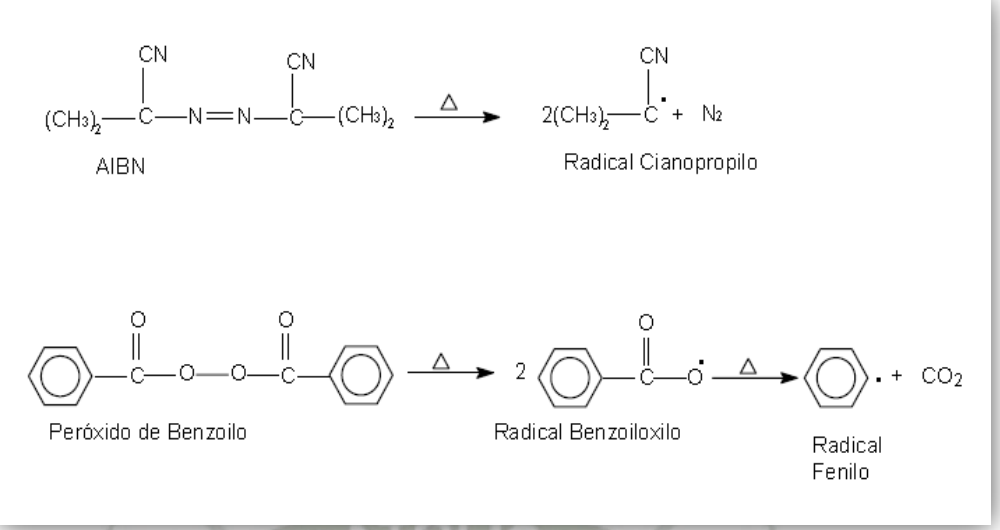


Figura 7. Iniciación del AIBN y PBO

b) Propagación

En esta etapa se van añadiendo moléculas de monómero al monómero radical formado en la etapa de la iniciación y la cadena va creciendo tal como se indica siendo kp la constante de propagación:

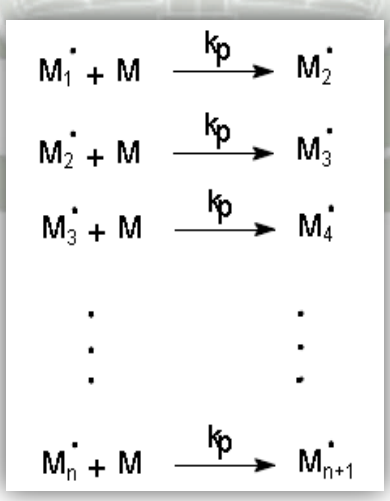


Figura 8. Reacciones de propagación

Si se examina atentamente la estructura de un polímero generado por un monómero vinílico, como los aquí descritos, se pueden evidenciar varias posibilidades de arreglos configuracionales. Estos arreglos son la consecuencia de que las unidades monoméricas pueden incorporarse a la cadena mediante uniones cabeza-cola o formando uniones cabeza-cabeza y cola-cola tal como se muestra a continuación:

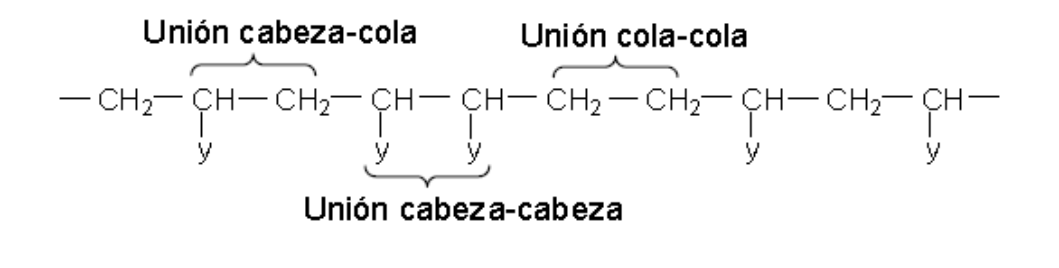


Figura 9. Uniones cabeza-cabeza y cola-cola

Es de notar que la mayoría de las polimerizaciones incorporan al monómero con una orientación cabeza-cola, debido a aspectos estéricos y electrónicos.

Sin embargo, una unión cabeza a cabeza algunas veces puede esperarse. El tiempo requerido comúnmente para cada adición de monómero es del orden de un milisegundo, así varias miles de adiciones pueden tener lugar dentro de un segundo.

c) Terminación

En esta etapa se termina el crecimiento de la cadena del polímero. Los dos mecanismos más comunes de la terminación implican la reacción biomolecular de las cadenas crecientes del polímero. La combinación involucra el acoplamiento de dos cadenas crecientes para formar una sola molécula de polímero.

Alternativamente un átomo de hidrógeno puede ser abstraído de una cadena creciente por otra en una reacción conocida como desproporción.

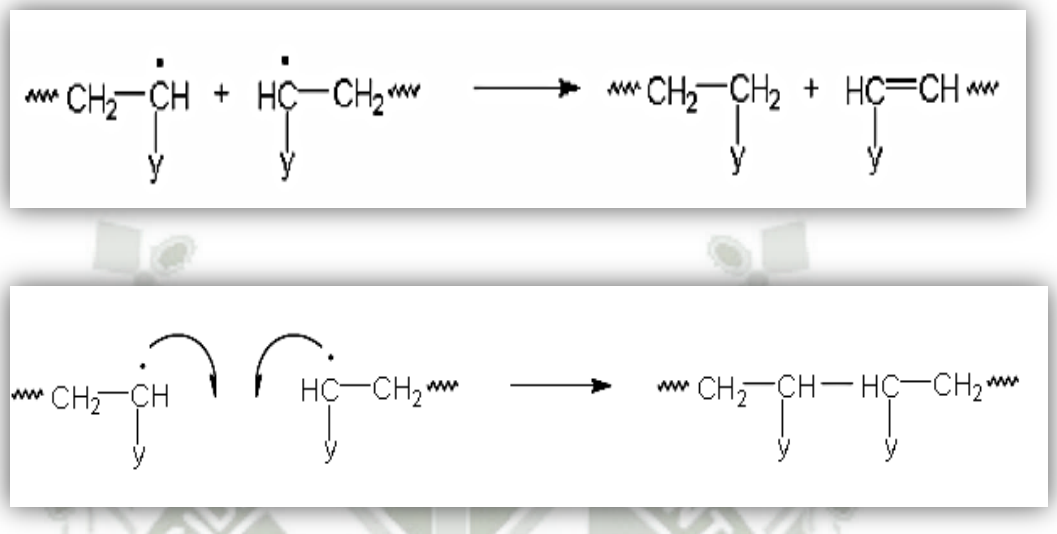


Figura 10. Reacciones de terminación

1.4. Propiedades de los polímeros

A pesar de la gran variedad en la estructura y composición que pueden presentar los distintos polímeros, hay muchas propiedades comunes que poseen los polímeros y que los distinguen de los metales y cerámicos.

1.4.1. Propiedades Térmicas de los Polímeros

Los ensayos de los materiales resistentes a la temperatura pueden clasificarse en:

- Ensayos de corto tiempo realizados a elevada temperatura.
- Ensayos de corto tiempo corridos a temperatura ambiente después de que el material haya sido sometido a elevada temperatura por largo tiempo.

- Ensayos largos corridos a alta temperatura, los cuales a su vez pueden clasificarse en:
  - ❖ A carga constante.
  - ❖ Deflexión constante.
  - ❖ Corrosión en caliente.

La temperatura de fusión Tm de los plásticos cristalinos y la temperatura de transición vítrea Tg de un plástico amorfo o semicristalino termoplástico evidencian su desempeño térmico. Los plásticos semicristalinos y amorfos son frágiles y rígidos cerca de Tg y retienen la mayor parte de su resistencia. Por encima de Tg sus propiedades mecánicas se deterioran significativamente debido al ablandamiento. A diferencia, los plásticos cristalinos tienen una resistencia y rigidez útiles por encima de Tg. Los plásticos amorfos como el PC o PES se utilizan solamente por debajo de Tg. Debido al alto grado de entrecruzamiento los termoestables como la epoxi y resinas fenólicas presentan un Tg más alto, pero aun así tienden a degradarse por debajo de Tg.

Como consecuencia los compuestos avanzados de grafito/epoxy se diseñan para trabajar a una temperatura máxima de 30°C, mientras que los compuestos avanzados de matriz termoplástica pueden trabajar cerca de Tg.

Sin embargo ni Tm ni Tg toman en cuenta como se deforman los plásticos bajo carga, por lo que se requieren de ensayos de deflexión bajo carga a cierta temperatura para comparar diversos plásticos. La DTUL es aquella temperatura en la que un plástico bajo una carga flectora de 0,45 a 1,8 MPa se deforma 0,25 mm. Añadiendo fibra de vidrio o carbono,

minerales o cerámicos de refuerzo se puede incrementar significativamente la DTUL de los termoplásticos cristalinos, termoplásticos poliésteres, PPS y fluoro plásticos.

Muchos plásticos pueden trabajar a una cierta temperatura por tiempos cortos, sin embargo, la prolongación en esas condiciones pueden afectar significativamente sus propiedades mecánicas degradándose térmicamente. Por esta razón los ensayos largos a alta temperatura son necesarios para medir la resistencia, flexión, impacto y propiedades eléctricas de los plásticos.

1.4.1.1 Temperatura de Transición Vítrea (Tg)

Un polímero puede ser amorfo, cristalino o semi-cristalino. A menores temperaturas los polímeros de cadena larga son sólidos, vidriosos e inelásticos. Cuando se aumenta la temperatura, el polímero pasa por una transición térmica conocida como temperatura de transición vítrea (Tg) que a nivel molecular representa el inicio del movimiento de los segmentos de cadenas cortas. Por arriba de su Tg un polímero muy cristalino se vuelve flexible y moldeable.

La presencia de heteroátomos en las cadenas poliméricas o con menos grupos funcionales voluminosos, favorecen que se alcance la Tg a bajas temperaturas [16]

1.4.1.2 Temperatura de Fusión (Tm)

Los polímeros termoplásticos, a la temperatura de fusión (Tm), se encuentran en un estado líquido o fundido donde la energía es suficiente para que las moléculas puedan moverse y los átomos de las cadenas puedan girar o rotar alrededor de los ángulos de enlace pasando

rápidamente de una conformación a otra. Aunque, la capacidad de rotar de los enlaces de cadena está limitada por los impedimentos estéricos [17].

Los polímeros de cadena larga con baja cristalinidad (amorfos) se hacen elásticos cuando se calientan por encima de su Tg y a mayor temperatura son pegajosos y menos sólidos, hasta que pasan a ser líquidos viscosos sin punto de fusión (Tm) definido.

Por otro lado, los polímeros entrecruzados pueden permanecer elásticos y no fundir hasta que la temperatura sea tan alta que el polímero comienza a descomponerse. Los polímeros cristalinos y amorfos, de cadena larga, muestran propiedades físicas diferentes al calentarlos

1.4.2. Propiedades mecánicas de los Polímeros

Los polímeros y elastómeros tienen módulos muy bajos. Esto ocasiona que su deflexión elástica es muy grande. Sus propiedades dependen de la temperatura y del tiempo (viscoelasticidad). Casi ninguno de ellos tiene utilidad por encima de los 200ºC. Por otra parte son más fáciles de conformar que los metales, son resistentes a la corrosión y tienen coeficientes de fricción bajos.

Las propiedades mecánicas de los polímeros tienen una delgada relación con la temperatura. Al aumentarse la temperatura, las resistencias disminuyen. Los termoplásticos, se reblandecen a altas temperaturas y al enfriarse se endurecen y vuelven más rígidos. Los polímeros laminados y reforzados con base termoestable son menos afectados ya que están estabilizados por el material de refuerzo o reforzante. El porcentaje de carga afecta a la resistencia.

Los polímeros pueden fluir, esto es, deformarse continuamente bajo tensión. Lo cual puede ser apreciable, de acuerdo al nivel de esfuerzo y d1e temperatura. A altos niveles de esfuerzo, la fluencia al principio es también alta. Después durante un tiempo disminuye, pero al final empieza un incremento de velocidad, terminando por fallar. Estos altos niveles de esfuerzo deben evitarse. Los termoplásticos son más sensibles a la velocidad de carga ya la fluencia que los termoestables, laminados y plásticos reforzados.

Los procesos de fabricación también pueden tener una gran influencia en la resistencia. En los termoplásticos extruidos tal como en tuberías, por ejemplo, las moléculas están mayormente orientadas en la dirección de la extrusión, y la resistencia es, por tanto, mayor en esta dirección que en la perpendicular.

Los polímeros varían considerablemente sus propiedades mecánicas dependiendo del grado de cristalinidad, entrecruzamiento o una alta temperatura de transición vítrea.

Los plástico con elevada resistencia mecánica tienen elevados grados de cristalinidad, entrecruzamiento o una alta temperatura de transición vítrea; mientras que los polímeros “estirables" y con poca resistencia mecánica, tienen características contrarias.

La resistencia mecánica se pierde por arriba de la T_g en el caso de los polímeros amorfos y sobre la T_m en el caso de los cristalinos.

1.4.2.1. Resistencia a la tracción

La resistencia es una propiedad mecánica que se puede relacionar acertadamente, pero no se sabría con exactitud qué significa la palabra "resistencia" cuando se trata de polímeros.

En primer lugar, existen varios tipos de resistencia. Está la resistencia a la tracción. Un polímero tiene resistencia a la tracción si soporta cargas axiales que tienden a alargarlo.

La resistencia a la tracción es importante para un material que va a ser estirado o a estar bajo tensión. Las fibras necesitan tener buena resistencia a la tracción. En los plásticos la resistencia a tracción (varía entre 350 y 550 Kg/cm^2) es muy inferior a la resistencia a compresión, aunque en algunos casos, para filamentos extruidos en frio se puede llegar a cifras del orden de 4.500 Kg/cm^2. Influye en este tipo de resistencia el sistema de moldeo del plástico, así como la temperatura ambiente y la humedad.

1.4.2.2. Resistencia a la flexión

Un polímero tiene resistencia a la flexión si es capaz de soportar cargas que provoquen momentos flectores en su sección transversal.

Los materiales menos dúctiles se pueden ensayarse en flexión, adicionalmente a los de tracción o compresión axial.

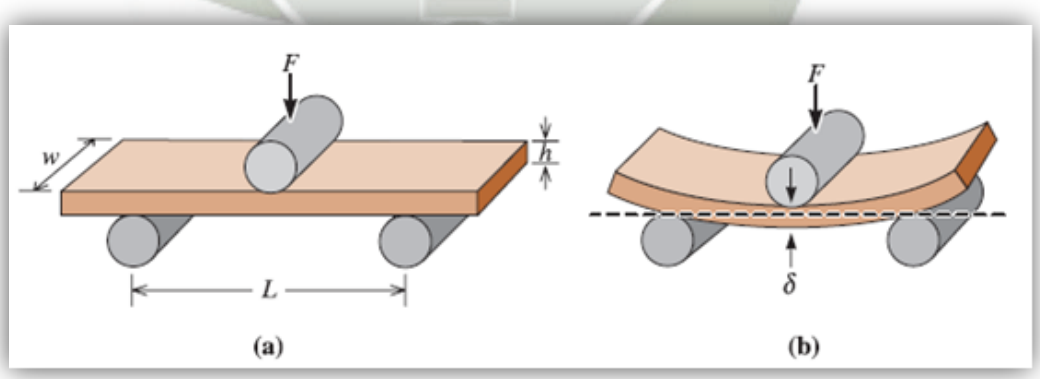


Figura 11: (a) Ensayo de flexión en 3 puntos utilizado para medir la resistencia de materiales frágiles. (b)La deflexión δ generada por el ensayo

1.4.2.3. Resistencia a la compresión

Un polímero tendrá resistencia a la compresión si soporta cargas axiales que tienden a compactarlo. El concreto es un ejemplo de material con buena resistencia a la compresión. Cualquier cosa que deba soportar un peso encima, debe poseer buena resistencia a la compresión. Según el tipo de plástico, la resistencia a compresión puede variar de 500 a 2.500 Kg/cm^2.

1.4.2.4. Tenacidad

Es la propiedad que tiene un material de absorber energía antes de fracturarse, su medida está dada por la cantidad de energía absorbida. En los ensayos para medir la tenacidad, el esfuerzo puede aplicarse lentamente, mediante un ensayo esfuerzo-deformación o súbitamente mediante un ensayo de impacto. La mayoría de los ensayos utilizan probetas entalladas y miden la tenacidad de entalla mediante el impacto o la tenacidad de fractura mediante un ensayo de baja velocidad de deformación.

1.4.2.4.1. Tenacidad de Impacto

Se mide ensayando probetas a determinadas temperaturas utilizando ensayos de impactos ya sea charpy o Izod .La energía absorbida de fractura se mide como el cambio de energía potencial del péndulo antes y después de la fractura.

Para el caso de los plásticos el ensayo de impacto por caída de un peso o de Gardner, es el más adecuado, puesto que mide la energía requerida para iniciarse la fractura.

Hoy en día la instrumentación electrónica ha incrementado el valor y la utilidad de los ensayos de impacto, pues proveen mucha información, como la capacidad de almacenar la

energía y el espectro del tiempo durante el cual se realiza el ensayo, ya se ha baja o alta velocidad.

La tenacidad de impacto obtenida por estos métodos depende mucho de la temperatura, de factores medioambientales como la humedad y del método de obtención de la probeta. Resulta difícil aplicar la mecánica de la fractura lineal elástica a los materiales plásticos, debido a su viscoelasticidad, orientación de la cadena molecular y grado de entrecruzamiento. Sin embargo, las técnicas de la mecánica de la fractura y los ensayos de caída de peso instrumentados se utilizan para evaluar la tenacidad de compuestos de matriz polimérica, tales como grafito/epoxi.

La temperatura de transición dúctil-frágil se relaciona con la temperatura de transición vítrea en los polímeros y para propósitos prácticos se considera el mismo fenómeno.

A) Método Izod

La probeta es empotrada por una de sus mitades, se golpea en el extremo del voladizo por la cara de la entalla

B) Método Charpy

La probeta se apoya en sus extremos y se golpea en el centro por la cara contraria a la entalla. El péndulo de charpy está construido por un martillo. Su arista de choque la forman dos caras inclinadas 30º, unidas por un radio de 2 mm

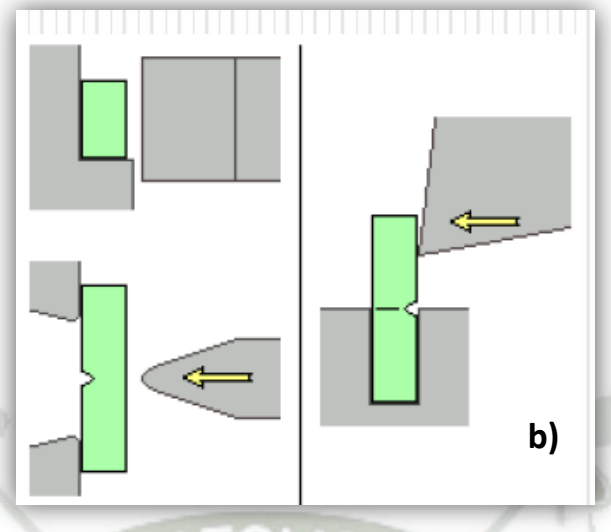


Figura 12. a) Disposición de la probeta en el método Charpy, b) Disposición de la probeta en el método Izod.

1.4.2.5. Dureza

Es la propiedad que tienen los materiales de resistirse a ser rayados o penetrados. Esta propiedad no constituye una característica específica de los materiales sino que está íntimamente ligada a las características elásticas y plásticas.

Existe una variedad en lo que respecta a durómetros porque los hay para probar polímeros, cerámicos, metales y materiales compuestos

1.4.2.6. Índice de Fluidez de los Polímeros

La industria de los materiales termoplásticos utiliza el índice de fluidez (MFI) como una herramienta básica para el control de la calidad y la aceptación de los productos entrantes. La medida de las propiedades de flujo está contenida en la tarjeta de identidad, y se utiliza como una verificación para comprobar si el grado de plástico está dentro del rango de

fluidez requerida. Esto se utiliza comúnmente para poliolefinas (polietileno HDPE, LDPE, LLDPE, y polipropileno PP). De acuerdo con la norma ISO 1133 y ASTM D1238, el MFI es el peso del polímero fundido a través de una boquilla estándar (2.095 x 8 mm) a una temperatura dada y con un peso estándar aplicado al pistón, que empuja la muestra.

La ISO 1133 especifica dos métodos para la prueba: El procedimiento A es un método manual de medición de masa, en el que segmentos cronometrados de la pieza extruida se pesan con una balanza después de la prueba para determinar el ratio de masa fundida (MFR). Esto se expresa en g/10 min. El procedimiento B es un método de medición de desplazamiento, basado en la medida del desplazamiento del pistón por medio de un encoder. El resultado es el ratio de volumen fundido (MVR). Expresado en cm/10 min, es la media de los diferentes datos obtenidos por el encoder. La densidad de masa fundida se mide también y se utiliza para calcular el MFR relacionado. Sugerimos la medición del MVR cuando se comparan termoplásticos con o sin aditivitos, así como cuando se comparan los materiales con aditivos diferentes.

1.5. Materiales biodegradables poliméricos

1.5.1. Introducción a los polímeros biodegradables

Se define un material biodegradable como aquel material que es capaz de ser descompuesta con cierta rapidez por organismos vivos (microorganismos), los más importantes de los cuales son bacterias aerobias.

La degradación de un polímero puede definirse como una variación en su estructura química que conlleva una modificación apreciable de sus propiedades. Actualmente, se

aceptan cinco mecanismos básicos de degradación que pueden interactuar entre sí, produciendo un efecto sinergético:

- Degradación térmica
- Degradación mecánica
- Fotodegradación
- Oxidación mediante aditivos químicos
- Degradación mediante microorganismos (hongos, bacterias, etc)

1.5.2. Clasificación de polímeros biodegradables

La mayor parte de los polímeros sintéticos biodegradables contienen enlaces hidrolizables a lo largo de la cadena, los enlaces más frecuentes son del tipo anhídrido, uretano, éster o carbonato (Figura 13):

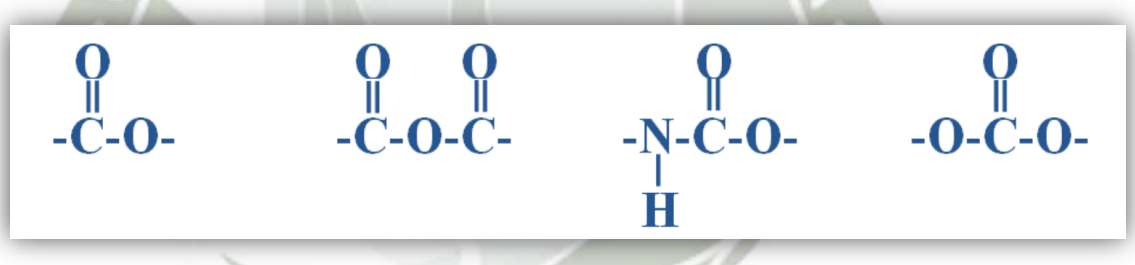


Figura 13. Enlaces hidrolizables tipo éster, anhídrido, uretano y carbonato (de izquierda a derecha).

De acuerdo a la naturaleza de los polímeros biodegradables, podemos decir que existen dos grandes grupos:

- Los polímeros de origen sintético, como por ejemplo las polilactidas o los polihidroxibutiratos.
- Los de origen natural, como el colágeno o el quitosano

La tendencia actual en el mercado muestra un descenso en el empleo de materiales naturales y un incremento importante de los polímeros de origen sintético. Sin embargo, debe mencionarse que los polímeros de origen natural que han sido sometidos a algún tipo de modificación en industrias y laboratorios han tenido también un desarrollo muy significativo.

La Tabla 1 muestra los principales polímeros degradables y el tiempo necesario para efectuar su degradación en condiciones fisiológicas.

Tabla 1: Tiempos de degradación requeridos por los polímeros biodegradables más utilizados en la actualidad.

Polímero	**Tiempo de degradación (meses)**
Poliácido D-Láctico	12-16
Poliácido L-Láctico	18-24
Copolímero de ácido glicólico y láctico	6-12
Policaprolactona	18-24
Poliglicolida	2-4
Polihidroxibutirato	18-24
Poliésteres de fosfato	12-24
Poliortoésteres	12-24
Polianhidridos de alcanos	0.2-4
Polianhidridos aromáticos	6-12
Gelatina	0.2-1
Celulosa oxidada	0.2-1
Colágeno	0.2-1
Pseudopoliaminoácidos	2-24
Poliminocarbonatos	4-12
Polifosfacenos	6-18
Polipropilenfumarato	12-24

Ref: "Incorporación de micro/nanoesferas en poliésteres degradables y su influencia en procesos de cristalización", Lopez G.. UPC. Barcelona. 2012

1.5.2.1. Polímeros biodegradables naturales

- **De naturaleza proteica,** Como la albúmina y el colágeno
- **Polisacáridos,** como los glucosaminoglucanos, Carboxicelulosa, quitina, quitosano, etc.

1.5.2.2. Polímeros sintéticos

Los polímeros sintéticos degradables más comúnmente utilizados, son los obtenidos a partir del ácido poliglicólico (PGA) y del ácido poliláctico (PLA), los cuales han encontrado una multitud de usos en la industria médica. La aplicación comercial más representativa son las suturas bioabsorbibles que fueron aprobadas en 1960.

Desde entonces, han sido desarrollados numerosos dispositivos basados en PGA y PLA. Otros poliésteres que se utilizan para la elaboración de artículos de uso biomédicos son la poli(ε-caprolactona), o polímeros relacionados como el poli(trimetilen-carbonato) y la polidioxanona, en forma de homopolímeros y copolímeros.

1.6. Síntesis, propiedades y aplicaciones del Ácido Poliláctico (PLA)

Entre los materiales plásticos biodegradables, el PLA es uno de los de mayor potencial para sustituir a los plásticos convencionales por sus excelentes propiedades físicas y mecánicas y porque puede procesarse utilizando las maquinarias existentes con solo ajustes menores. El PLA es también un material altamente versátil que puede elaborarse con distintas formulaciones para satisfacer la mayor parte de las especificaciones de los productos. [19] Mezclado con distintos polímeros naturales permite desarrollar materiales con mejores propiedades de resistencia al agua. [19]

El PLA cumple o excede las especificaciones de los materiales para empaque en cuanto a disposición de desechos y es la mejor alternativa entre los plásticos comunes para disminuir los residuos que se envían a los basureros municipales, ya que ninguna otra vía es económicamente viable o segura para la salud. [18]

Los polímeros de PLA son totalmente reutilizables. Con equipamiento apropiado, se puede convertir de nuevo en monómero, que puede ser convertido nuevamente en polímero.

Como alternativa, puede biodegradarse en agua, dióxido de carbono y material orgánico. Al final del ciclo de vida de un producto basado en PLA, éste puede descomponerse en sus partes más simples de manera que no quede ninguna señal del producto original. [20]

1.6.1. Síntesis y composición del Ácido Poliláctico (PLA)

La polimerización del ácido láctico da lugar a polímeros de ácido poliláctico (PLA) que, con otras moléculas poliméricas naturales, permiten la obtención de productos reabsorbibles y biodegradables. [21]

El ácido láctico puede ser obtenido por vía química o biotecnológica. La producción química se basa en la reacción de acetaldehído con ácido cianhídrico para formar lactonitrilo, que es hidrolizado a ácido láctico.

La producción biotecnológica se basa en la fermentación por microorganismos, de sustratos ricos en carbohidratos y tiene la ventaja de formar enantiómeros D(+) o L(−) ópticamente activos.

Industrialmente se utilizan como sustratos, sacarosa procedente de azúcar de caña, lactosa proveniente de lactosuero y dextrosa proveniente de almidón hidrolizado. El Lactobacillus delbrueckii es el microorganismo utilizado en la producción industrial, por consumir eficientemente glucosa y en medio lactosado la bacteria recomendada es Lactobacillus bulgaricus [22, 23 ,24].

El PLA es un material plástico que está comenzando a producirse comercialmente en grandes cantidades. [25] Este se fabrica por polimerización del monómero de ácido láctico. La síntesis del ácido láctico a moléculas de alto peso molecular de ácido poliláctico puede seguir tres rutas: [18]

• Polimerización por condensación de bulto para dar un polímero frágil, de aspecto vidrioso, de bajo peso molecular, que es de poco uso, a menos que se adicionen agentes enlazantes para aumentar el peso molecular.

• Polimerización por condensación en solvente para dar un PLA de alto peso molecular promedio.

• Colección, purificación y polimerización del lacturo por apertura del anillo para obtener un PLA de alto peso molecular promedio (>100 000).

1.6.2. Propiedades mecánicas del Ácido Poliláctico (PLA)

Las propiedades del PLA como punto de fusión, resistencia mecánica y cristalinidad están determinadas por la arquitectura del polímero y la masa molecular y, para el uso final, también dependerán de su estructura y las condiciones del proceso. La proporción de D y L-lacturos determina la morfología del polímero que puede producirse totalmente amorfo o hasta un 40 % cristalino.

Resinas de PLA que contienen más de 93 % de L-ácido láctico son semicristalinas mientras que con 50-93 % son estrictamente amorfas. La presencia de meso y D-ácido láctico produce imperfecciones en la estructura cristalina reduciendo el por ciento de cristalinidad. [26]

Además de su capacidad para biodegradarse, el PLA tiene propiedades que comparan favorablemente con las de los plásticos comúnmente empleados, por ejemplo, para envolturas. Este es un factor importante pues permite sustituir por PLA a polímeros de la petroquímica sin necesidad de rediseñar productos o ejecutar grandes inversiones en nuevos equipos de proceso. [18]

El PLA se puede formular para ser tanto rígido como flexible y también puede copolimerizarse con otros materiales; además puede producirse con propiedades mecánicas apropiadas para procesos de fabricación específicos como moldeo por inyección, extrusión de lámina, moldeo por soplado, termoformado, formación de películas e hilado, con la mayoría de las técnicas y equipos convencionales.

Tabla 2. Propiedades mecánicas del PLA

Propiedad	**Rango**	**Unidad**
El módulo de Young	3.45-3.83	GPa
Módulo de compresión	3.45-3.83	GPa
Módulo flexural	3.45-3.8	GPa
El módulo de esfuerzo al corte	1.23-1.37	GPa
Límite de elasticidad (el límite de elasticidad)	48 – 60	MPa
Resistencia a la tracción	48 – 60	MPa
Resistencia a la compresión	48 – 60	MPa
Resistencia a la flexión	81.5 – 99.2	MPa
Elongación	2 – 6	%
Dureza Vickers	14 – 18	HV
Dureza Rocwell M	67 – 87	
Dureza Rocwell R	110 – 120	
Resistencia al impacto	1.29-2.59	KJ/m2

Fuente: CES EduPack 2012

1.6.3. Biodegradación del Ácido Poliláctico (PLA)

La degradación del PLA se produce a través de dos procesos:

1) Primero los microorganismos (hongos y bacterias) crecen y colonizan la superficie del polímero y segregan enzimas, los grupos hidrófilos de las enzimas (carboxi −COOH, hidroxi −OH, amina $-NH_2$) atacan los grupos éster (- COO-R) de las cadenas del polímero mediante reacciones de oxidación de esta manera reducen el poliéster de alto peso molecular a oligómeros de bajo peso molecular.

2) En el segundo proceso, cuando el poliéster tiene un peso molecular promedio (Mn) de 10,000 a 40,000 g/mol los microorganismos continúan la biodegradación al transformar los componentes de bajo peso molecular a dióxido de carbono, agua y humus [27].

1.6.4. Aplicaciones del Ácido Poliláctico (PLA)

Por ser biodegradable y reabsorbible el PLA tiene múltiples aplicaciones en la medicina y en las industrias como: alimentaria, textil, cosméticos y otras.

1.6.4.1 Médicos

Al poder ser asimilado por el organismo, ha encontrado múltiples aplicaciones en cirugía, ortopedia, ortodoncia, oftalmología, traumatología y otras ramas de la medicina y como soporte para el suministro controlado de numerosos medicamentos. [20] Los siguientes son algunos de los usos en este campo.

- Estructuras biodegradables para la ingeniería de tejido [28]
- Implantes reconstructivos y bioabsorbibles.
- Equipos e instrumental para cirujanos

- Implantes para fijación de fracturas
- Tratamiento de la lipoatrofia de la cara [29]
- Placas absorbibles para fijación interna en fracturas de cara, cirugía ortognática y cráneofacial [30]
- Preparación de microesferas biodegradables [31]
- Dispositivos bioabsorbibles de fijación en reconstrucciones orbitarias [32]
- Administración intravítrea de antivíricos [33]

1.6.4.2 Industriales

El PLA también encuentra aplicación en otras ramas industriales como la alimentaria, textil, la de producción de envases, envolturas de distintos tipos, embalajes y otras. Sobre estos usos se han encontrado las siguientes referencias.

- Empaques para alimentos
- Fabricación de tejidos sin tejer
- Envolturas, materiales de empaque, envases y otros

1.6.4.3. Ingenieriles

Una de las industrias que ha mostrado mayor interés en la elaboración de materiales compuestos reforzados con fibras naturales es la automotriz, dado que se busca materiales más livianos con buenas propiedades mecánicas.

Mercedes-Benz en 1996 en vehículos clase E incorporó fibra de coco y de yute a sus vehículos.

En la figura se muestra un auto Mercedes Benz, con un rango de componentes que tienen incorporadas fibras naturales. [34]

Figura 14. Componentes que tienen incorporadas fibras naturales Mercedes-Benz en 1996 en vehículos clase E.

Fabricación de filtros de aire, aceite y combustible para equipos Pesados, en el rubro de la minería (excavadoras, cargadores frontales, Camiones, Palas, etc.), construcción (motoniveladoras, rodillos compactadores, retroexcavadoras, pavimentadoras, etc.), marinos (motores de embarcaciones marinas) y energía (grupos electrógenos).

Figura 15. Filtros para maquinaria pesada

Fabricación de sellos utilizados en sistemas hidráulicos y neumáticos.

Figura 16. Sellos hidráulicos

Fabricación de consolas o tableros eléctricos de equipos Mecánicos y Eléctricos.

Figura 17. Tableros Eléctricos

Fabricación de carcasas de equipos Electrónicos (Laptops, computadoras de escritorio, Monitores, Televisores, celulares, Equipos de sonido, etc.).

Figura 18. Tablero de automóviles

Figura 19. Carcaza de televisores

1.7. Fibras naturales

Fibras son todos aquellos materiales, de origen natural, que se pueden hilar, trenzar o utilizar para fabricar telas mediante operaciones de trenzado, fieltrado o tejido. Una fibra es un sólido relativamente flexible, microscópicamente homogéneo, con una pequeña sección transversal y una elevada relación longitud-anchura.

Fibra es cada uno de los filamentos que, dispuestos en haces, entran en la composición de los hilos y tejidos, ya sean minerales, artificiales, vegetales o animales; fibra textil es la unidad de materia de todo textil. Las características de una fibra textil se concretan en su: finura y gran longitud [35]

1.7.1. Clases de fibra.

Según su procedencia pueden ser:

- Fibras de origen mineral, como la fibra de vidrio y algunos metales (Especialmente el oro y la plata)
- Fibras de origen vegetal; como el algodón, lino, yute, esparto, etc
- Fibras de origen animal; como la **fibra de los camélidos (Llama, Alpaca, Guanaco y Vicuña) y de caprinos (Cashmere y Mohair)**, seda y cuero.
- Fibras sintéticas; como poliamidas, poliéster, acrílicas, polivinílicas, etc.

1.7.2. Fibras Vegetales

Las fibras vegetales comprenden aquellas fibras naturales extraídas del reino vegetal en sus más variadas formas: semillas, tallos, hojas, frutos y raíces y procesadas de forma tal que se obtienen productos de aplicación textil. Ya en los tiempos cuando el hombre cultivaba la tierra, su preocupación era el alimento y la vestimenta como fuente de abrigo. Los vegetales les permitieron alcanzar sus expectativas.

1.7.2.1 Clasificación de las fibras vegetales

Las fibras vegetales forman junto a las fibras animales el gran mundo de las fibras naturales, ya que las fibras minerales son de menor importancia relativa. Y las fibras naturales comienzan a revalorizarse luego del "boom" de la introducción de las fibras sintéticas, y en todo el mundo se está haciendo esfuerzos para impulsar éste desarrollo. Las fibras vegetales tienen en común una misma estructura química: la celulosa.

De acuerdo a la parte de la planta de donde se extraen se clasifican en:

A) Fibras vegetales de semilla

Hay solo dos especies vegetales cuyas semillas vienen acompañadas por una fibra que es de interés para su explotación comercial, y son:

- **Algodón**
- **Ceiba**

B) Fibras vegetales de tallo

La oferta de especies es mucho más amplia que en el caso anterior, pero la importancia comercial es de unas pocas, cuyos motivos analizaremos en cada caso en particular. Las especies vegetales de las que se extraen fibras del tallo son:

- **Lino**
- **Bambú**
- **Cáñamo**

C) Otras fibras de tallo

Mencionamos las siguientes fibras, cuyo interés es meramente decorativo y no tienen como uso final la industria de la indumentaria:

- **Banana**
- **Kenaf**
- **Yute**

D) Fibras vegetales de hoja

En este grupo, ya no se encuentran especies de difusión e importancia comercial masiva sino que constituyen vegetales de interés regional y con un desarrollo limitado a ese contexto. Estos son las siete variedades de hoja más conocidas.

- **Abacá**
- **Cabuya**
- **Esparto**

E) Fibras vegetales de fruto

Esta clase de fibras poseen un único ejemplar que tiene alguna importancia para mencionar. Se trata de la fibra del coco.

F) Fibras vegetales de otras partes de la planta

En este grupo reunimos a todas aquellas fibras que son extraídas de otras partes de la planta que no hayan entrado en los grupos mencionados anteriormente y pueden considerarse de una importancia similar a la de los dos grupos anteriores. A continuación las mencionamos: caucho, hilo de papel, fibra de turba, hierba de algodón.

1.7.3. YUTE

La fibra del yute es extraída de la corteza del yute blanco (corchorus capsularis) y en menor medida del yute rojo, que son arbustos que alcanzan hasta 4 mts. de altura, de tronco rígido y fibroso. La planta crece en áreas de tierras bajas tropicales con alto contenido de

humedad. Es una de las fibras vegetales más fuertes con propiedades antiestáticas y de baja conductividad térmica. Bangladesh e India son los mayores productores de yute junto a Myanmar y Nepal. Los hilos de fibra de yute se utilizan principalmente para tejer arpilleras destinadas a sacos, embalajes, cinchas y cordelería; además de esteras, tapices y alfombras.

Yute es una fibra lignocelulósica que posee grupo-OH (grupo polar) lo que causa susceptibilidad a la humedad y directamente afecta las propiedades del compuesto yute; especialmente la estabilidad dimensional. Debido a este grupo polar, el yute también no se adhiere de manera eficiente a las matrices no polares. Para superar esta dificultad esta fibra debe ser modificada químicamente. Para mejorar la adhesión interfaz entre las matrices no polares y fibra hidrófila, deben utilizarse agente de acoplamiento o compatibilizador.

Algunas investigaciones se realizaron por cianoetilación y la acetilación de fibra de yute para reducir el contenido de OH. Los procesos son eficaces tanto para la estabilidad dimensional. La Cianoetilación también mejora la unión entre el yute y matriz no polar como resina de poliéster insaturado.

Figura20. Imagen de (a) yute unidireccional, (b) de yute tejido liso

Tabla 3: Nombre científico del yute

Nombre científico:	Corchorus capsularis
Familia:	Malváceas
Nombre común:	Yute

Fuente: http://www.redtextilargentina.com.ar/

1.7.3.1. Parámetros de control

Los parámetros de control son las características físicas, químicas y mecánicas de las fibras textiles, que se toman como referencia para determinar los estándares de calidad de cada una de ellas.

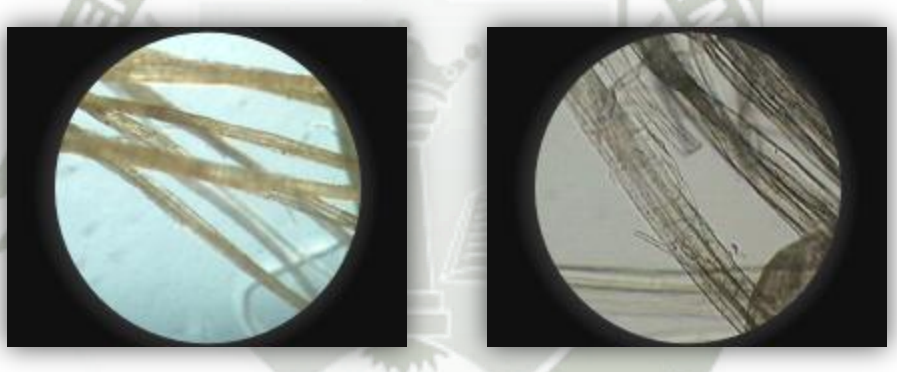


Figura 21. MICROGRAFÍA: Fibra de yute al microscopio (vista longitudinal)

1.7.3.2 Parámetros básicos generales

Tabla 4. Parámetros básicos generales de la fibra de yute

Exposición a la llama		**Tenacidad**	
Características	funde y arde	Seco (gr/den)	s/d
Olor	papel quemado	Húmedo (gr/den)	s/d
Residuo	ninguno		
Humo	s/d	**Elongación**	
		Seco (%)	1,7
Exposición al calor		Húmedo (%)	s/d
Estabilidad al calor seco	120-130ºC		
Amarillamiento	120-150ºC	**Módulo**	
Descomposición	> 240ºC	(cN/tex)	1764
Indice de Oxígeno Límite	20,1%		
		Resiliencia (60% HRA)	
Exposición a la luz solar		Para x% de extensión	s/d
(PÉRDIDA DE TENACIDAD)		Para x% de extensión	s/d
48 hrs de exposición	sensible		
480 hrs de exposición	pierde resistencia	**Acción de los ácidos**	
		Diluidos en caliente	sensible
Dimensiones		Diluidos en frío	resistente
Longitud en milímetros	1400-4000	Concentrados en caliente	s/d
Diámetro en micrones	16-20	Concentrados en frío	sensible
Solubilidad		**Acción de los álcalis**	
Ácido Sulfúrico al 75%	soluble	Diluidos en caliente	resistente
Soda Cáustica al 5%	insoluble	Diluidos en frío	resistente
Solventes Orgánicos	insoluble	Concentrados en caliente	resistente
		Concentrados en frío	resistente
Absorción de humedad			
A 21ºC con 65% HRA	s/d	**Acción agentes redox**	
Regain (%)	17%	Oxidantes	resistente
		Reductores	resistente
Biodegradabilidad			
Mohos	atacada	**Peso específico**	
Bacterias	atacada	(gr/cm3)	1,50

Fuente: http://www.redtextilargentina.com.ar/

Varios estudios de la composición y la morfología de la fibra han encontrado que el contenido de celulosa y el ángulo de las microfibrillas tienden a controlar las propiedades mecánicas de las fibras celulósicas.

1.7.3.3. Composición química de yute

Yute se compone de un polímero conocido como ligno-celulosa. Como el componente principal es la celulosa, la estructura química de yute está representada por la de la celulosa, a saber $(C_6H_{10}O_5)$ n

Tabla 5: Composición de la fibra de yute

Componente	**Porcentaje**
Celulosa	64,4%
Hemi de celulosa	12%
La lignina	11,6%
Humedad	10%
Las grasas, ceras y pectinas	11.8%

Ref. Manufacturing & applications of jute fibre composites. **T Myvizhirajeswari y K Saravanan.**

Tabla 6: Propiedades mecánicas de diferentes tipos de fibras

Propiedades mecánicas de las fibras naturales comparadas con fibras reforzadas convencionales					
Fibra	**Densidad (g/cm3)**	**Diámetro (μ m)**	**Elongación (%)**	**Esfuerzo de tensión (MPa)**	**Módulo de Young (GPa)**
Algodón	1.5-1.6	-	7.0-8.0	287-800	5.5-12.6
Yute	1.3-1.45	25-200	1.16-1.8	393-773	13-26.5
Lino	1.5	-	2.7-3.2	345-1100	27.6
Hemp	-	-	1.6	690	-
Ramie	1.5	50-200	1.2-3.8	400-938	61.4-128
Sisal (Cabuya)	1.45-1.5	20-80	3.0-7.0	468-635	9.4-22
Coco	1.15-1.2	-	15-40	131-175	4.0-6.0
Fibras convencionales					
Fibra de vidrio – E	2.5	-	2.5	2000-3500	70
Fibra de vidrio – S	2.5	-	2.8	4570	86

Ref. "Estudio comparativo de las propiedades mecánicas y geológicas de compuestos de polietileno de alta densidad con cascarilla de arroz y bagazo de caña". Mesias J. Ecuador. 2008

1.7.3.4. Yute usado como reforzante

Trabajos de investigación de compuestos con reforzados yute se han llevado a cabo desde principios de los años 80. El primer trabajo fue llevado a cabo en colaboración con AE RE, Harwell, Reino Unido usando como matriz epoxi, poliéster, etc para comparar los compuestos obtenidos con el yute con los obtenidos con fibra de vidrio.

Se utilizaron películas de polietileno de baja densidad con yute no tejido y la tela para la fabricación de compuestos de yute.

A continuación se presentan algunos resultados:

Tabla 7: Las propiedades de flexión de material compuesto no tejido de yute y baja polietileno de densidad como matriz

·	**Muestras**	**Resistencia a la flexión (MPa)**	**Módulo de flexión (MPa)**	**Elongación %**
1.	Yute no tejido * + film LDPE	31.84	1433	8.013
* Yute non woven- unidireccional y 400 gsm (nominal) * LDPE Film- 50 gsm				

Ref: "Los estudios sobre el yute compuesta de material no tejido de yute", 16 ª Tecnológico conferencia, IJIRA, 11 º - 12 º de Febrero de 1993

Tabla 8: Propiedades de material compuesto no tejido de yute y de polietileno de baja densidad como matriz para el envasado. (IIP- Kolkata)

Materiales	**Valor medio de prueba**				
	Gram / m ^{2}	**Resistencia a la perforación oz pulgadas lágrima**	**Absorción de agua (superficial) 24 horas a 30 ° C, g / m^2**	**Resistencia a la tracción. (MPa)**	**Módulo de elasticidad (MPa)**
Yute no tejido + film LDPE	1470	577.1	20.7	31.36	1756

Ref: "Estudios sobre compuesta de yute no tejido de yute", 16 ª Tecnológico conferencia, IJIRA, 11 º - 12 º de Febrero de 1993

1.8 Conformado de los plásticos

1.8.1 Moldeo por compresión

El moldeo por compresión es uno de los procesos de transformación de plásticos más antiguo que existe, figura 16. Aparece descrito en bibliografía de principios del siglo XIX, aunque no comenzó a desarrollarse a escala industrial hasta 1908, cuando Leo Baeckeland desarrollo las resinas fenol-formaldehido, que siguen empleándose aún hoy en día.

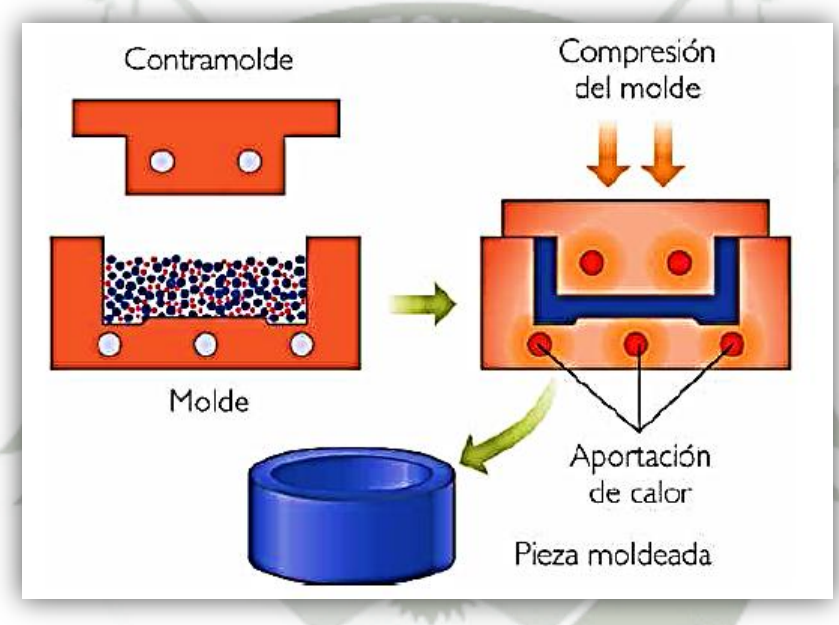


Figura 22. Moldeo por compresión

El moldeo por compresión es un método de moldeo en el que el material de moldeo, en generalmente precalentado, es puesto en la cavidad del molde abierto. El molde se cierra, se aplica calor y presión para forzar al material a entrar en contacto con todas las áreas del molde, mientras que el calor y la presión se mantiene hasta que el material de moldeo se ha curado. El proceso se usa en resinas termoestables en un estado parcialmente curado, ya sea

en forma de pellets, masilla, o preformas. El moldeo por compresión es un método de alta presión, adecuado para el moldeo de piezas complejas, de alta resistencia con refuerzos de fibra de vidrio. Los compuestos termoplásticos, aunque en menor medida, también pueden ser moldeados por compresión con refuerzos de cintas unidireccionales, tejidos, fibras orientadas al azar o de hilos cortados.

La ventaja de moldeo por compresión es su capacidad para moldear piezas grandes, bastante intrincadas o complejas. Además, es uno de los métodos de más bajo costo en comparación con el moldeo por otros métodos tales como moldeo por transferencia y moldeo por inyección, por otro lado se desperdicia poco material, dándole una ventaja cuando se trabaja con compuestos caros. Sin embargo, el moldeo por compresión a menudo proporciona productos de pobre consistencia y dificultad en el acabado, y no es adecuado para algunos tipos de piezas. En este proceso se produce una menor degradación de la longitud de la fibra en comparación con el moldeo por inyección. Materiales que normalmente se fabrican mediante moldeo por compresión incluyen: sistemas de resina poliéster con fibra de vidrio, (SMC / BMC), Torlon (Poliamidaimida: PAI), Vespel (Poliamida: PA), Polifenilen sulfuro (PPS), y muchos grados de PEEK.

Materiales compuestos reforzados con fibras termoplásticas se distinguen de los compuestos termoestables principalmente por un alto alargamiento, y la posibilidad de reciclaje. El moldeo por compresión ha demostrado ser adecuado para la producción de perfiles con cualquier material preimpregnado termoplástico.

Compuestos reforzados con fibra de lino [36], fibra de madera [37] y yute; también han sido preparados por el proceso de moldeo por compresión. Iannace y sus compañeros de

trabajo [38] fabricaron biocomposites preparados a base de fibras de algas marinas y una matriz biodegradable termoplástica e investigó los efectos de procesamiento, tales como moldeo por compresión y calandrado sobre las propiedades mecánicas de la materiales.

1.8.1.1. Definición del proceso

El moldeo por compresión es un proceso de conformación en que se coloca un material plástico directamente en un molde de metal se calienta y luego se ablanda por el calor, y es obligado a conformarse con la forma del molde en el molde cerrado por la presión y el calor.

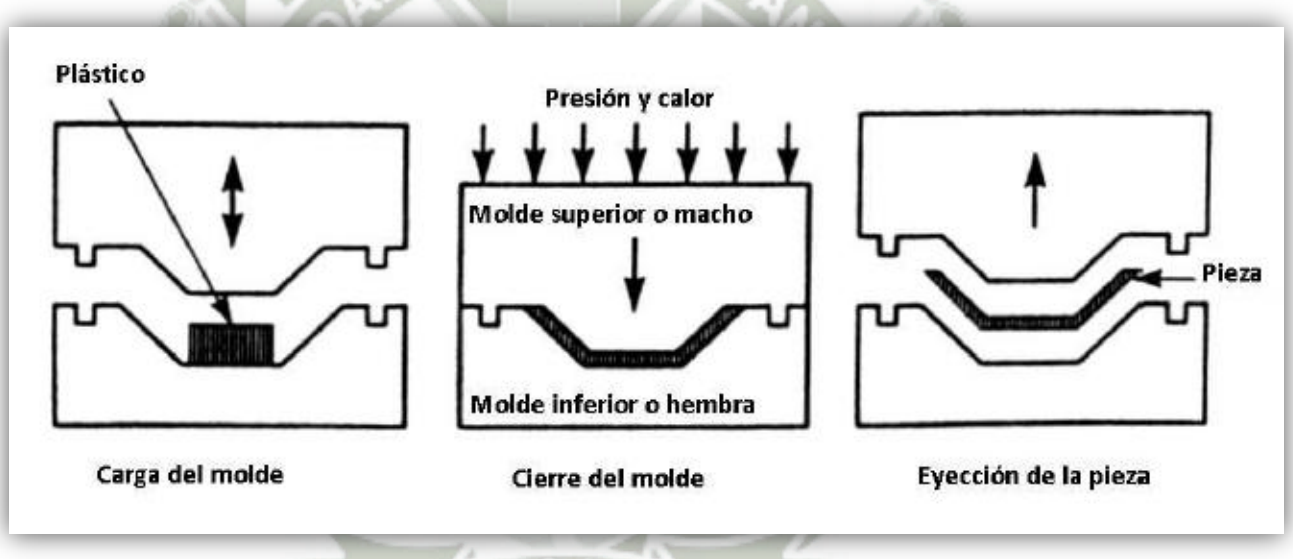


Figura 23. Esquema del proceso

1.8.2. Termoformado

El termoformado es un proceso secundario el cual consiste en dar forma a una lámina de un termoplástico. Consta de dos pasos principales: calentamiento y formado. El calentamiento se realiza por medio de calentadores eléctricos en ambos lados de la lámina. La duración

del ciclo de calentamiento debe ser suficiente para ablandar la lámina dependiendo el tipo de plástico, el espesor y su color. Los métodos de formado se pueden clasificar en tres categorías:

- Termoformado mecánico (Figura 24): usa un par de moldes (positivo y negativo también llamado hembra y macho) que se aplican contra la lámina de plástico caliente forzando a adquirir la forma. En este método no se usa vacío ni presión de aire.

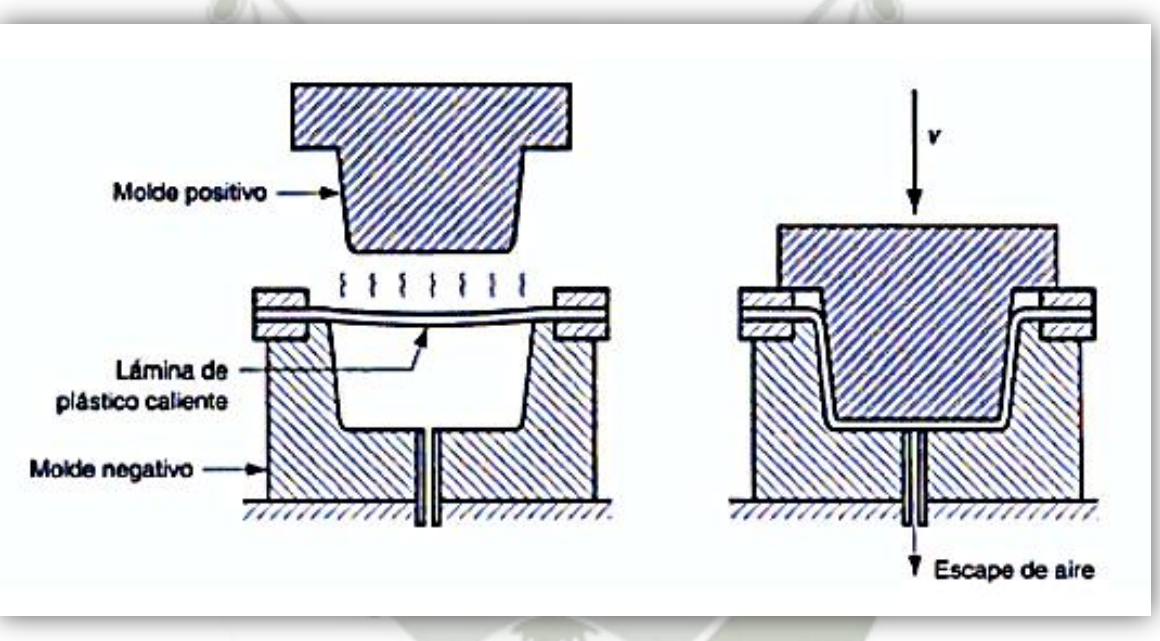


Figura 24. Termoformado mecánico.

- Termoformado al vacío (figura 25): en el cual se usa una presión de vacío para adherir la lámina precalentada a la cavidad del molde. 1. Se ablanda la lámina por medio de calentamiento, 2. Se introduce y coloca sobre la cavidad de un molde, 3. El vacío atrae la lámina hacia la cavidad, 4. El plástico se endurece al contacto con la superficie fría del molde, la parte se retira y luego se recorta de la hoja.

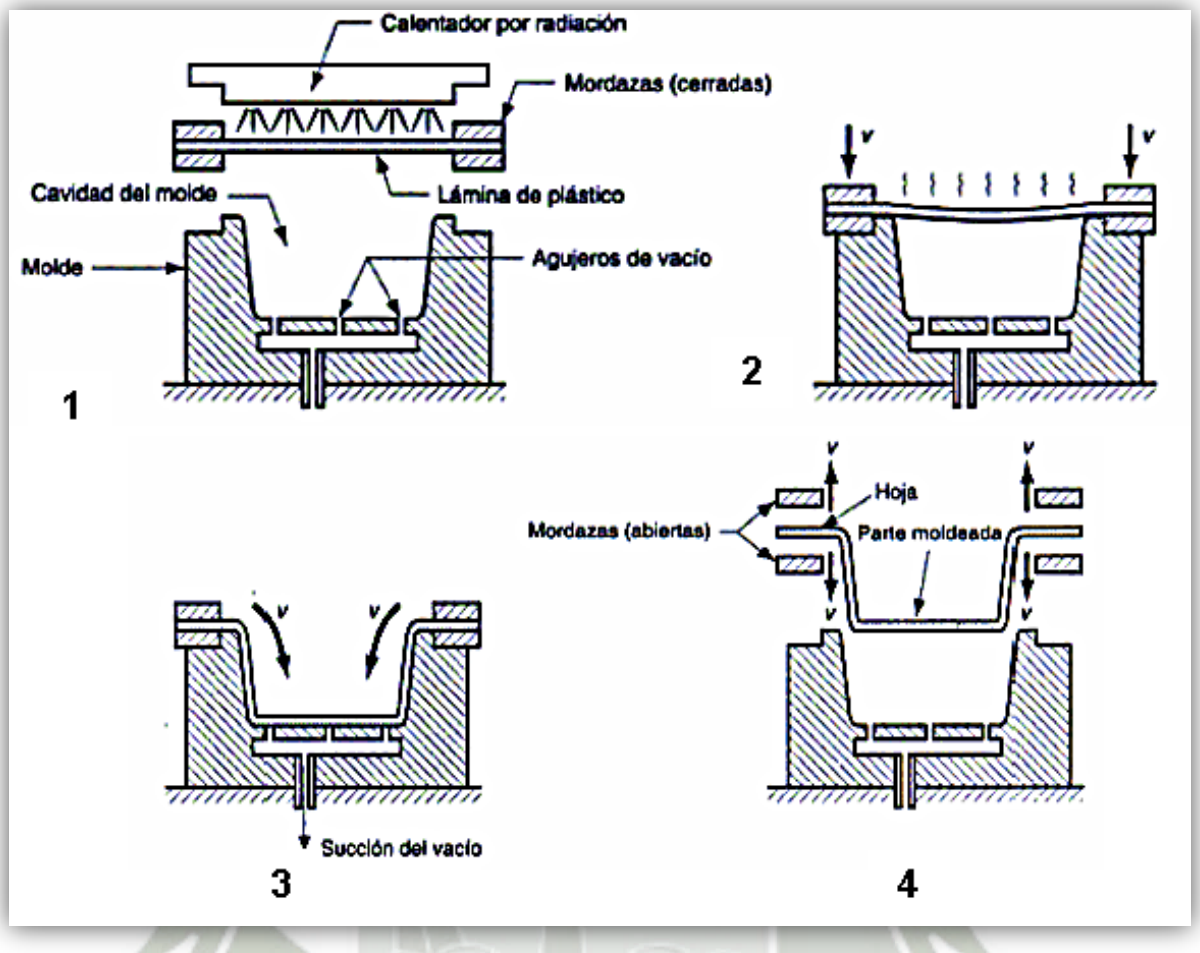


Figura 25. Se muestran los pasos principales del termoformado al vacío. 1. Calentamiento.2 acoplamiento. 3. Succión de la lámina. 4. Enfriamiento y corte.

- Termoformado a presión (figura 26): este utiliza tanto el vacío como presión de aire positiva para forzar a la lámina precalentada dentro de la cavidad del molde. El proceso es similar al termoformado al vacío la diferencia es que se aplica una presión de aire para que la lámina ya suavizada entre en contacto con la cavidad del molde.

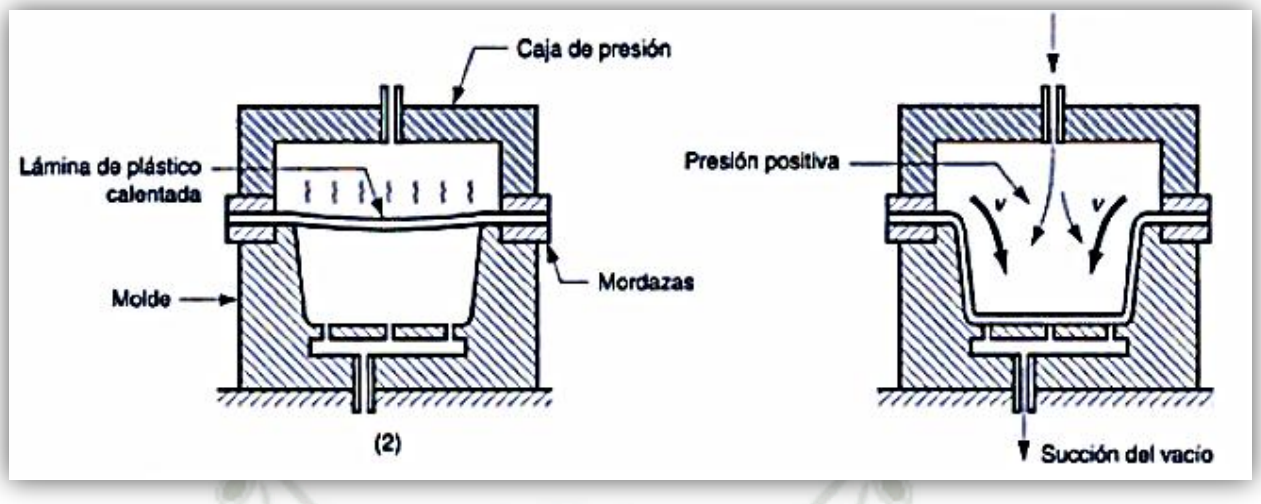


Figura 26. Termoformado a presión.

1.9 MATERIALES COMPUESTOS

1.9.1 Definición

Se entiende por materiales compuestos aquellos formados por dos o más materiales distintos sin que se produzca reacción química entre ellos.

En todo material compuesto se distinguen dos componentes:

- la MATRIZ, componente que se presenta en fase continua, actuando como ligante
- el REFUERZO, en fase discontinua, que es el elemento resistente.

Ejemplos sencillos y conocidos por todos de materiales compuestos son el hormigón y los neumáticos. Aquí, sin embargo, nos centraremos en el estudio de los llamados MATERIALES COMPUESTOS AVANZADOS, que son los que se utilizan para la fabricación de elementos estructurales.

1.9.2 Clasificación

Una primera clasificación es la que analiza el tipo de matriz, distinguiéndose los siguientes tipos:

- Materiales compuestos de matriz METÁLICA o MMC (METAL MATRIX COMPOSITES).
- Materiales compuestos de matriz CERÁMICA o CMC (CERAMIC MATRIX COMPOSITES).
- Materiales compuestos de matriz de CARBON.
- Materiales compuestos de matriz ORGÁNICA o RP (REINFORCED PLASTICS) y dentro de estos, son los más utilizados:
    - Los CFRP (CABON FIBER REINFORCED PLASTICS) o materiales compuestos de fibra de carbono con matriz orgánica.
    - Los GFRP (GLASS FIBER REINFORCED PLASTICS) o materiales compuestos de fibra de vidrio con matriz orgánica.

- En lo que a los refuerzos se refiere, los hay de dos tipos:

- FIBRAS, elementos en forma de hilo en las que la relación L/D > 100,
- CARGAS, el resto, utilizadas en elementos de poca responsabilidad estructural.

Tal y como se han resaltado, los materiales compuestos más utilizados son los de matriz orgánica y refuerzos en forma de fibras.

1.9.3 Matrices orgánicas

Antes de describir los distintos tipos de matrices orgánicas, conviene repasar cuales son las funciones que debe cumplir la matriz. Estas son:

- ❖ Dar estabilidad al conjunto, transfiriendo las cargas al refuerzo.
- ❖ Proteger al refuerzo del deterioro mecánico y químico.
- ❖ Evitar la propagación de grietas.

Para todo ello, se debe dar una buena adherencia entre la matriz y el refuerzo.

Las matrices orgánicas (más vulgarmente conocidas como plásticos) pueden ser:

- TERMOPLÁSTICOS, usadas en aplicaciones de bajos requisitos, aunque se están empezando a emplear termoplásticos avanzados para altas prestaciones.
- ELASTOMEROS, utilizadas en neumáticos y cintas transportadoras,
- DUROPLASTICOS o TERMQESTABLES, las más empleadas en materiales compuestos de altas prestaciones.

1.9.4. Fibras

Los principales tipos de fibras utilizados como refuerzo, en lo que al material que las compone se refiere, son:

- FIBRAS DE VIDRIO, de gran resistencia a tracción, duras, resistentes al ataque químico y son flexibles. Se elaboran a partir de la sílice (del 50% al 70% de su composición) y se le añaden otros componentes en función de las propiedades deseadas, distinguiéndose:
- VIDRIO-E, para aplicaciones generales.
- FIBRAS DE CARBONO, de muy alta resistencia y rigidez, por la estructura cristalográfica del grafito.

CAPÍTULO 2

MATERIALES Y METODOLOGÍA EXPERIMENTAL

2.1. Materiales y equipos de ensayo

2.1.1. Materiales

- Ácido Poliláctico (PLA)

Figura 27. Ácido Poliláctico (PLA)

- Fibra de Yute

Figura 28. Yute

- Alcohol Polivinilico

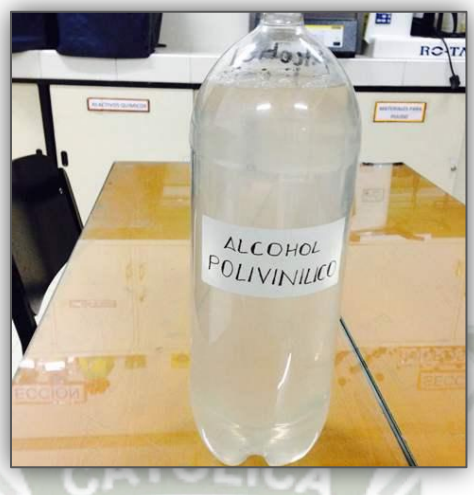


Figura 29. Alcohol Polivinilico

- Cera desmoldante

Figura 30. Cera desmoldante

2.1.2. Equipos

Los equipos utilizados para realizar las láminas muestras de PLA y con su refuerzo son los siguientes:

- ✓ Equipo Brabender

Figura 31. Brabender

- ✓ Prensa Hidráulica

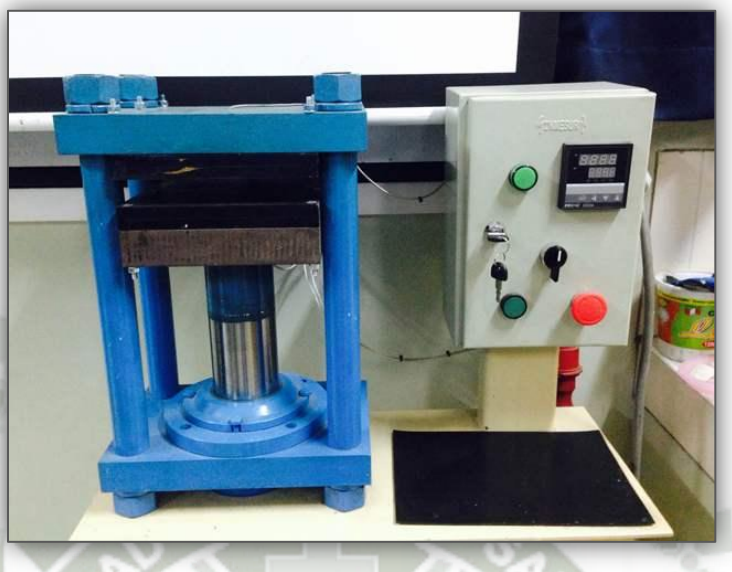


Figura 32. Prensa Hidráulica

- ✓ Planchas de acero inoxidable empleadas para realizar láminas de PLA y compuesto.

Figura 33. Planchas de acero inoxidable

- ✓ Herramientas utilizadas para desmoldar y limpiar las placas de PLA y agregados

Figura 34. Herramientas Utilizadas

- ✓ Máquina utilizada para realizar pruebas de tracción de marca Liyi-Tech

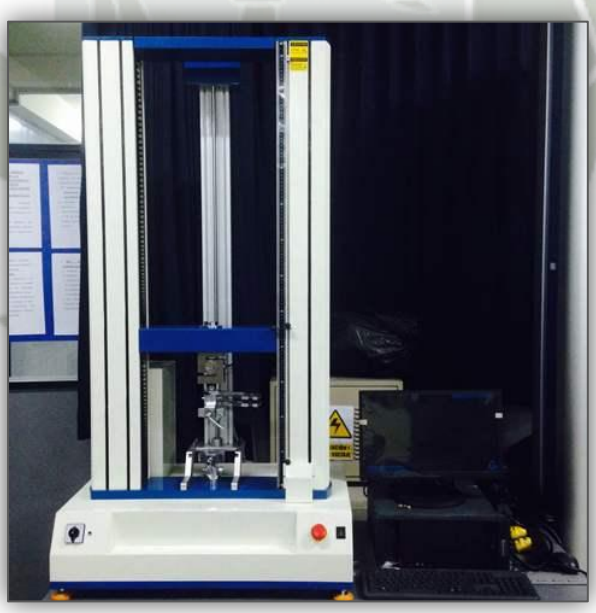


Figura 35. Maquina Liyi-Tech para pruebas de tracción

- ✓ Balanza digital

Figura 36. Balanza digital

- ✓ Equipo para medir dureza (Durómetro)

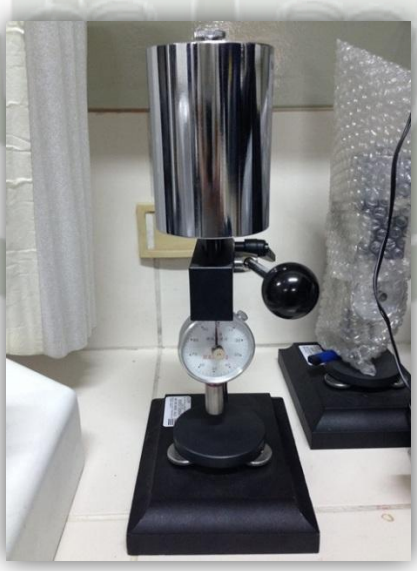


Figura 37. Durómetro

- ✓ Máquina de ensayo de impacto (Máquina de Charpy)

Figura 38. Máquina de Impacto Charpy

- ✓ Máquina para obtener el índice de fluidez (Plastómetro)

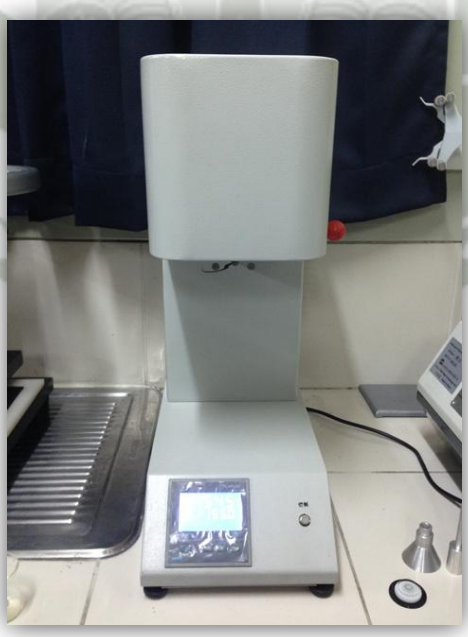


Figura 39. Plastómetro

2.2. Estrategia metodológica

2.2.1. Ensayos preliminares

2.2.1.1. Parámetros de moldeo por compresión de láminas de ácido Poliláctico puro (PLA)

La elaboración de los laminados de PLA fueron desarrollados según los siguientes pasos:

- Paso 1.-

  Se realiza el pesaje del Ácido Poliláctico (PLA) en la balanza digital, la cantidad necesaria para realizar la lámina en este caso es de 20 gramos.

- Paso 2.-

  Debemos asegurarnos que las planchas de acero inoxidable se encuentren completamente limpias, luego a dichas placas se les pasa una capa de cera desmoldante y seguidamente se le pasa otra capa de alcohol polivinilico (no deben quedar burbujas u otras partículas extrañas ya que esto generaría que nuestras placas de PLA salgan defectuosas).

- Paso 3.-

  Considerar la siguiente temperatura de operación de los equipos, según Cuadro 2.1

Cuadro 2.1: Descripción de la temperatura de trabajo

Máquina Utilizada	Temperatura de Operación
Brabender	150°C
Prensa Hidráulica	180°C

Luego de asegurarnos que los equipos se encuentren a las temperaturas indicadas según Cuadro 2.1, se procede a verter el PLA pesado a la cámara de procesamiento del Brabender (ver Figura 40), el tiempo de procesamiento es de unos 6 a 7 minutos (revisar por la mirilla superior del Brabender, ver Figura 41). Una vez que el PLA se convierta en una pasta, se procede a colocarla en una plancha de acero inoxidable (ver Figura 42).

Figura 40. Ingreso de PLA al Brabender

Figura 41. Mirilla del Brabender

Figura 42. Pasta de PLA en Plancha de acero Inoxidable

- Paso 4.-

Colocar las planchas de acero inoxidable con el PLA en la prensa Hidráulica (ver Figura 43). El tiempo de prensado es de 8 minutos, luego de ello de se debe retirar las planchas para que sean enfriadas a temperatura ambiente.

Figura 43. Colocación de Placas con PLA en prensa Hidráulica

- Paso 5.-

Luego que las planchas de acero inoxidable se encuentren completamente frías (puedan ser palpadas), se procede a desmoldar el PLA de las planchas (ver Figura 44). Con la ayuda de una espátula se retira la lámina de PLA (ver Figura 45).

Figura 44. Plancha de Acero Inox y PLA

Figura 45. Placa de PLA (foto referencial)

2.2.1.2. Elaboración de las láminas de material compuesto

a) Elaboración de lámina de 20 gr. de PLA más 0.5gr. de Yute Picado

Para la obtención de esta lámina se requiere de 0.5gr. de Yute Picado luego se tendrán que realizar los pasos 1 y 2 del item **2.2.1.1**. Se procede a verter los 20gr. de PLA junto con los 0.5gr. de Yute Picado en el Brabender (tener en cuenta las temperaturas del cuadro 2.1), el tiempo aproximado de la mezcla es de 6 a 7 minutos. Verificamos por la mirilla que la mezcla se haya convertido en un compuesto pastoso.

Retiramos el compuesto dándole una forma redonda el cual se coloca en la plancha de acero inoxidable, a su vez estas planchas se colocarán en la prensa por un tiempo aproximado de 8 minutos. Luego se retiran las planchas para que enfríen a temperatura ambiente, una vez que estas se encuentren completamente frías se procede a separarlas y finalmente retiramos la lámina.

b) Elaboración de lámina de 20 gr. de PLA más 0.75gr. de Yute Picado

Para la obtención de esta lámina se requiere de 0.75gr. de Yute Picado luego se tendrán que realizar los pasos 1 y 2 del item 2.2.1.1. Se procede a verter los 20gr. de PLA junto con los 0.75gr. de Yute Picado en el Brabender (tener en cuenta las temperaturas del cuadro 2.1), el tiempo aproximado de la mezcla es de 6 a 7 minutos. Verificamos por la mirilla que la mezcla se haya convertido en un compuesto pastoso.

Retiramos el compuesto dándole una forma redonda el cual se coloca en la plancha de acero inoxidable, a su vez estas planchas se colocarán en la prensa por un tiempo aproximado de 8 minutos. Luego se retiran las planchas para que enfríen a temperatura ambiente, una vez que estas se encuentren completamente frías se procede a separarlas y finalmente retiramos la lámina.

c) Elaboración de lámina de 20 gr. de PLA más Malla de Yute

Para la obtención de esta lámina se tendrán que realizar los pasos 1, 2 y 3 del punto 2.2.1.1. Preparamos una malla de Yute de 15 cm x 15 cm por lado (ver figura 46), la cual se colocará en una de las planchas sujetada con cinta adhesiva (ver figura 47), tener en cuenta las temperaturas del cuadro 2.1.

Retiramos el PLA dándole una forma redonda el cual se coloca en la plancha que lleva la Malla de Yute, a su vez estas planchas se colocarán en la prensa por un tiempo aproximado de 8 minutos. Luego se retiran las planchas para que enfríen a temperatura ambiente, una vez que estas se encuentren completamente frías se procede a separarlas y finalmente retiramos la lámina (ver figura 48).

Figura 46. Malla de Yute

Figura 47. Malla de Yute en plancha

Figura 48. Lámina con Malla

2.3. Caracterización mecánica de las probetas fabricadas

Para llevar a cabo los ensayos tendremos presente el siguiente cuadro:

Cuadro 2.2. Matriz (PLA) – Reforzante (YUTE)

Código	Mezcla Yute con PLA	Matriz PLA		Reforzante YUTE	
		(g)	%	(g)	%
YP-0.50	Yute Picado	20	97.50	0.50	2.50
YP-0.75	Yute Picado	20	96.25	0.75	3.75
YM-2.32	Yute Tejido	20	88.40	2.32	11.6

2.3.1. ENSAYOS DE TRACCIÓN SEGÚN NORMA ASTM D 882

Se procede a realizar la fabricación de probetas, cortando las láminas en tiras de 10 mm de ancho, se tiene que cuidar los bordes en el momento del corte, se trabajó 4 probetas por mezcla elaborada (ver figura 49).

Figura 49. Probetas (tracción)

Para la elaboración del ensayo se procedió a medir el espesor en diferentes puntos de la probeta como se registran en los cuadros siguientes:

Cuadro 2.3. Medida de espesores de YP – 0.5

Probeta	Espesor (mm)	Ancho (mm)	Longitud Inicial (mm)
1	1.001	9.7	100
2	0.992	9.3	100
3	1.123	9.1	89
4	1.127	9.2	94

Cuadro 2.4. Medida de espesores de YP – 0.75

Probeta	Espesor (mm)	Ancho (mm)	Longitud Inicial (mm)
1	0.931	10.04	80
2	0.878	9.01	110
3	0.921	9.11	100

Cuadro 2.5. Medida de espesores de YM – 2.32

Probeta	Espesor (mm)	Ancho (mm)	Longitud Inicial (mm)
1	0.99	9.89	90
2	1.01	9.98	80
3	0.93	9.79	89
4	0.98	10	100

El espesor y ancho mostrado para cada mezcla polimérica resulto del promedio del cálculo del muestreo realizado en 3 puntos. Luego cada probeta se coloca en la máquina de

pruebas de tracción marca Liyi-Tech (ver figura 50), asegurándolas en las mordazas. Tener en cuenta la norma ASTM D882 la cual nos indica que este método de ensayo cubre la determinación de la resistencia a la tracción y las propiedades de los plásticos en forma de lámina delgada.

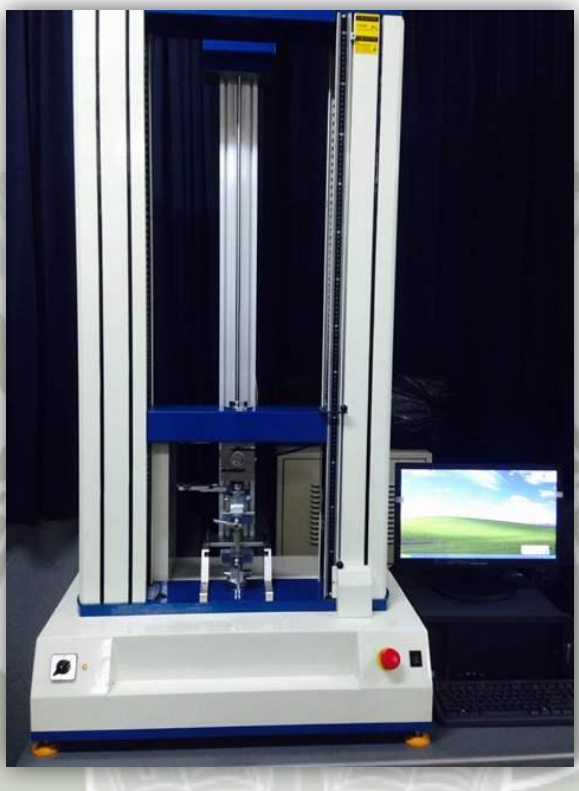


Figura 50. Máquina para Ensayo de Tracción Liyi-Tech

Figura 51. Montaje de probetas

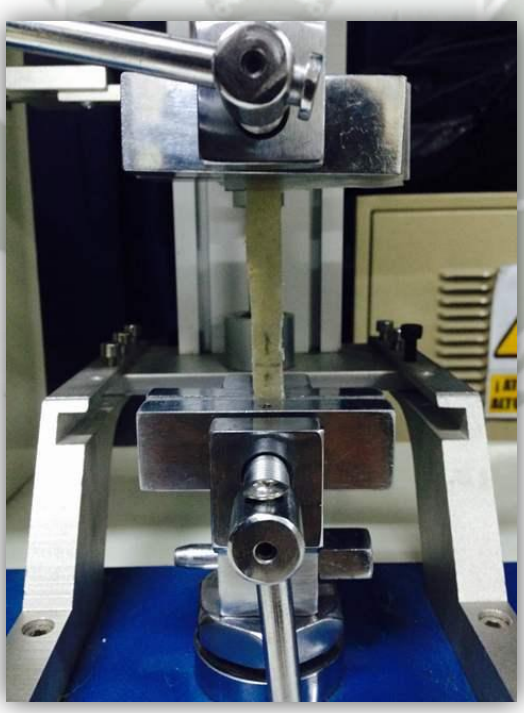


Figura 52. Probeta en ensayo YP – 0.75

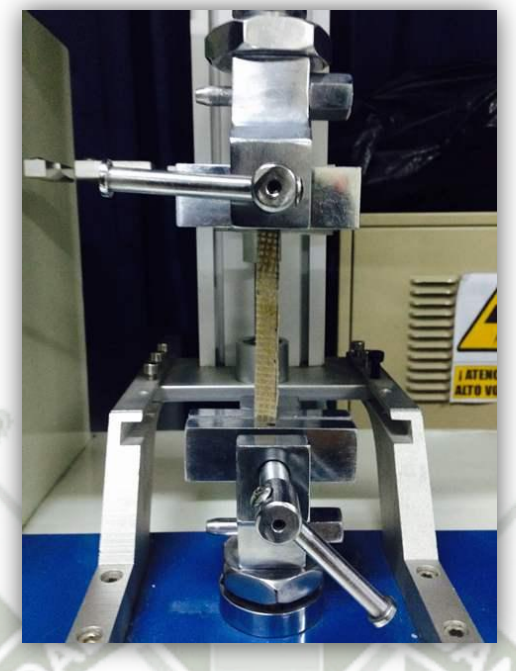


Figura 53. Probeta en ensayo YM – 2.32

También se realizó el ensayo de tracción a un hilo de yute (ver figura 54). Se tomaron medidas del hilo los cuales se muestran en el **Cuadro 2.6.**

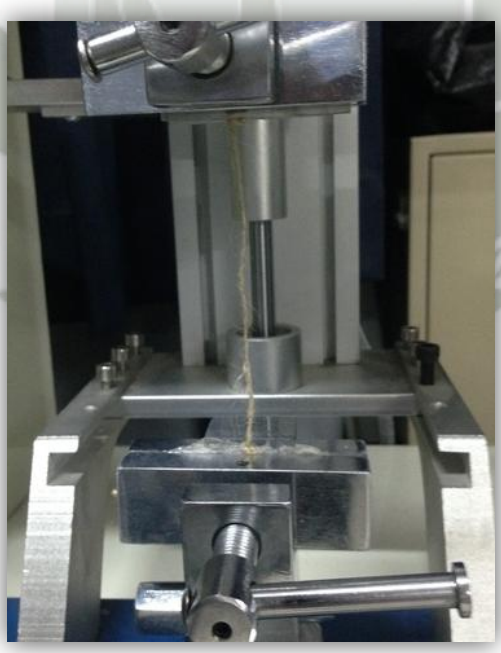


Figura 54. Hilo de Yute

Cuadro 2.6: Medidas del Hilo

Diámetro (mm)	0.48
Longitud Inicial (mm)	100

2.3.2. ENSAYO DE FLEXIÓN SEGÚN NORMA ASTM D 7264-07

Para este ensayo contamos con los tres tipos de láminas compuestas que hemos preparado, de las cuales cortamos tiras de 15 mm de ancho y las apilamos para obtener tres probetas con espesores mayores a los 3 mm (ver Cuadro 2.7) de cada una de las láminas (ver figura 49.). Una vez que se tienen las probetas procedemos a realizar el ensayo el cual consiste en apoyar la probeta en dos soportes (ver figura 56.) y ésta es cargada por el medio (ver Figura 57.), la fuerza aplicada a la muestra se mide y se registra hasta el error, la cual se produce en cualquiera de las superficies exteriores, o hasta que la deformación alcance algún valor pre-determinado (ver Figura 58 y 59.). La velocidad de compresión es de 20 mm/mín.

Cuadro 2.7. Datos de las probetas

PROBETA	CODIGO	E1 (mm)	E2 (mm)	E3 (mm)	E4 (mm)	$\bar{E}$ (mm)
1	YP-0.50	4.80	5.23	5.32	5.53	5.22
2	YP-0.75	3.56	3.36	3.39	3.60	3.48
3	YM-2.32	4.43	4.56	4.65	4.44	4.52

Figura 55. Probetas por cada lámina (flexión)

Figura 56. Montaje de la probeta (flexión)

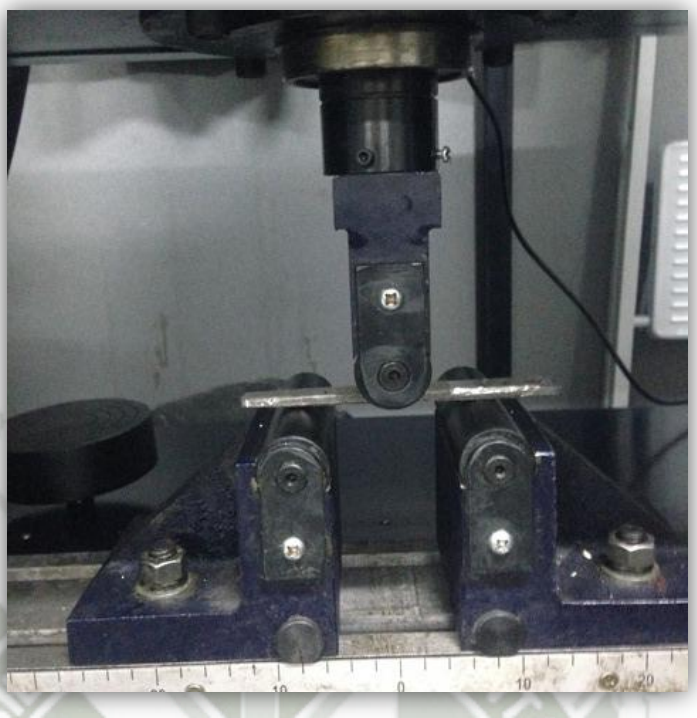


Figura 57. Carga aplicada al medio

Figura 58. Deflexión de la probeta

Figura 59. Deflexión de la probeta al final del ensayo

2.3.3. ENSAYO DE RESISTENCIA AL IMPACTO CHARPY SEGÚN NORMA ASTM D 6110-04

Para este ensayo utilizaremos las probetas que obtuvimos del ensayo de índice de fluidez las cuales tendremos que cortar a una longitud adecuada (ver Figuras 60, 61, 62) para colocarlas en la Máquina de Impacto Charpy (ver Figura 63.). En el equipo debemos ingresar las medidas de nuestras probetas, la velocidad, la carga con la que vamos a trabajar el impacto y en el martillo colocamos el peso para la carga que se requiere (ver Cuadro 2.8 y 2.9). Una vez que ingresamos los valores al equipo procedemos a colocar la probetas sobre las mordazas (ver Figura 64.), finalmente el martillo impactará en el medio de la probeta (ver Figura 65). Esto se repetirá para todas las probetas y finalmente obtendremos la energía absorbida.

Figura 60. Probeta de PLA puro

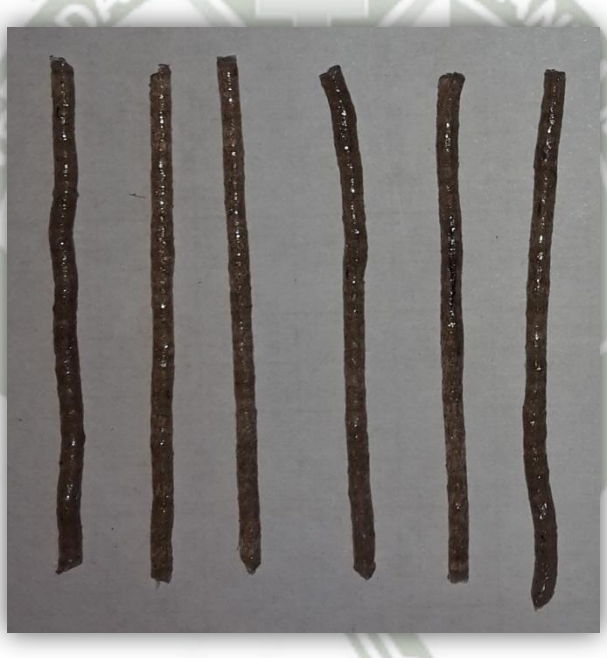


Figura 61. Probetas compuestas YP-0.5 y YP-0.75

Figura 62. Probetas compuestas Yute con malla

Figura 63. Máquina de Impacto CHARPY

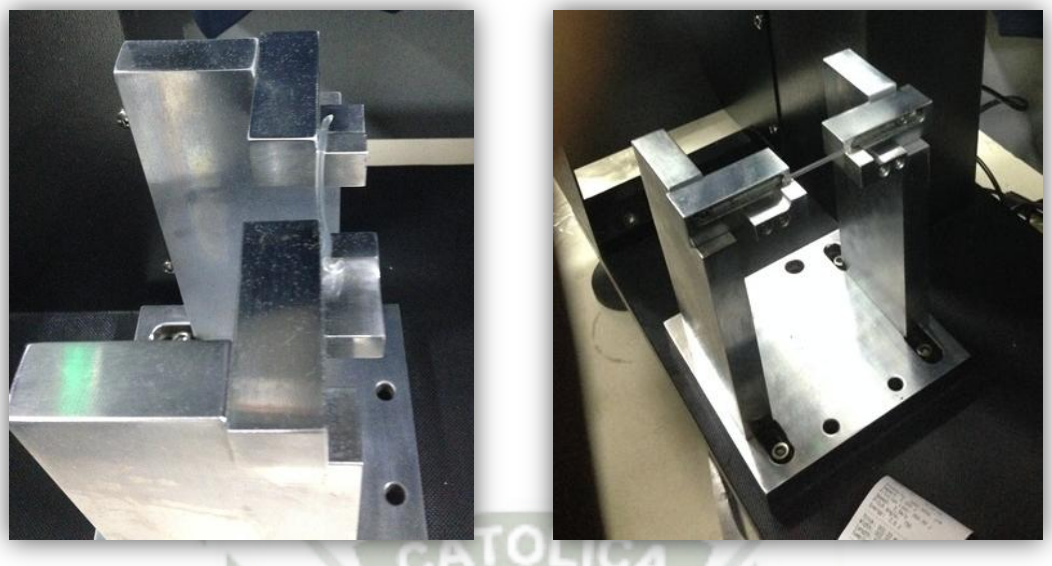


Figura 64. Montaje de la Probeta

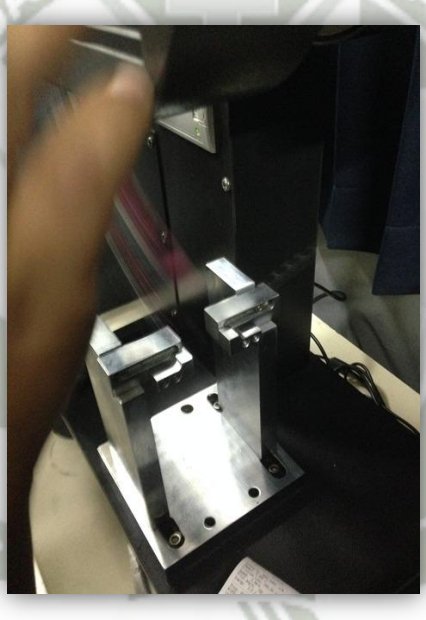


Figura 65. Impacto del martillo con la probeta

Cuadro 2.8. Datos para Probeta PLA Puro y Probetas YP-0,50 y YP-0,75 (Yute Picado)

Longitud (mm)	50
Ancho (mm)	2.33
Espesor (mm)	2.33
Velocidad (m/seg)	3.5
Carga (J)	2.75

Cuadro 2.9. Datos para Probeta YM-2.32 (Yute con Malla)

Longitud (mm)	50
Ancho (mm)	4.798
Espesor (mm)	1.40
Velocidad (m/seg)	3.5
Carga (J)	2.75

2.3.4. ENSAYO DE DUREZA SEGÚN NORMA ASTM D 2240

Este ensayo se utiliza para determinar la dureza relativa de los materiales blandos, generalmente de plástico o de goma. La prueba mide la penetración de un indentador especificado en el material bajo condiciones específicas de fuerza y tiempo. El valor de dureza se utiliza a menudo para identificar o especificar una dureza particular de elastómeros o como una medida de control de calidad en los lotes de material. En este caso utilizaremos la escala SHORE D la cual mide la dureza de materiales duros.

Para realizar el ensayo necesitamos de cada tipo de lámina por lo menos 4 tiras para lograr un espesor mínimo de 6 mm (ver Figura 66.), una vez que tenemos nuestras probetas armadas procedemos a realizar la prueba con el durómetro (ver Figura 67.).

Colocamos cada una de nuestras muestras en primer lugar en una superficie plana y dura (ver Figura 68.), luego presionamos con el indentador cada una de las muestras en diferentes puntos para obtener los datos necesarios. Una vez que se presiona la muestra inmediatamente se lee la dureza y anotamos (ver Figura 69.).

Figura 66. Probetas preparadas (dureza)

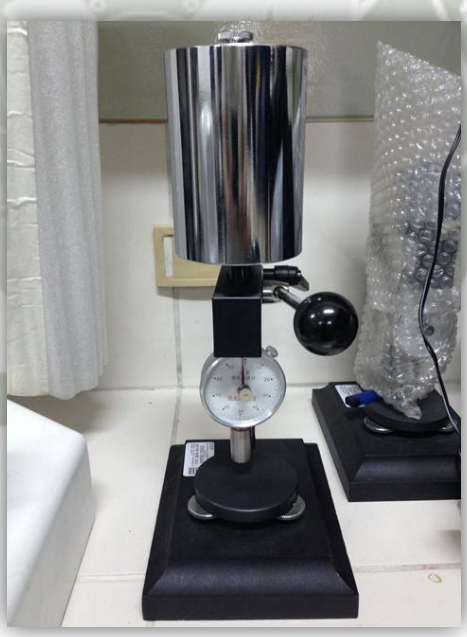


Figura 67. Durómetro

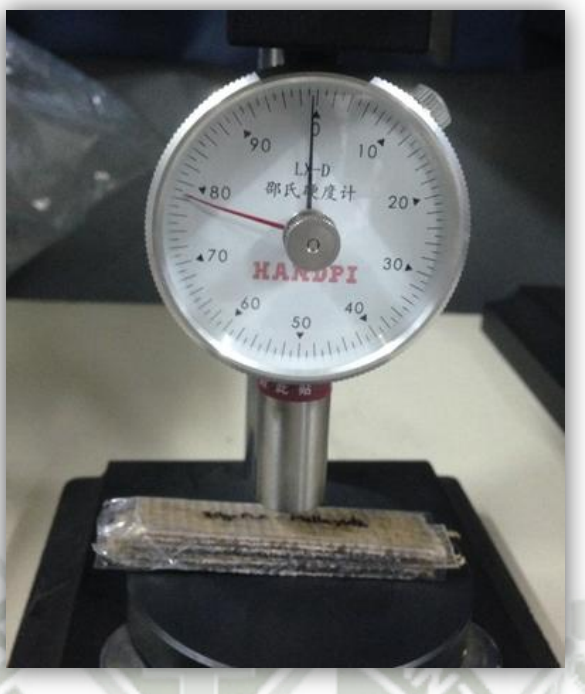


Figura 68. Probeta sobre superficie plana y dura

Figura 69. Indentador presionando la muestra

2.3.5. ENSAYO DEL INDICE DE FLUIDEZ SEGÚN NORMA ASTM 1238

Para esta prueba necesitamos cortar en trozos muy pequeños nuestras probetas (ver figura 70.) debido a que ingresarán al Plastómetro (equipo de medición del índice de fluidez ver Figura 71.), El equipo se programa con una temperatura de trabajo (ver Figura 72.) y un tiempo de corte (ver Cuadro 2.10) , el tiempo de permanencia dentro de la cámara será de 10 mín. Debemos tener cuidado con la limpieza del orificio por donde correrá el compuesto (ver Figura 73.) y a su vez debemos fijarnos que el dado se encuentre debidamente colocado en su posición de trabajo (vertical ver Figuras 74). Una vez que tenemos preparadas nuestras probetas (5g de cada una de ellas ver Figura 75.), la vertemos con ayuda de un pequeño embudo al equipo (ver Figura 76.), retiramos el embudo y colocamos el peso adecuado para el ensayo (ver Figura 77.), el cual empujará al pistón una vez que el compuesto se haya calentado lo suficiente para que pueda purgar (ver Figura 78.). Una vez que obtenemos nuestro compuesto extraído lo pesamos en la balanza digital (ver Figura 79.). Con el promedio que se obtiene del peso de cada una de nuestras probetas calcularemos nuestro índice de fluidez para cada uno de nuestros compuestos.

Figura 70. Probetas cortadas

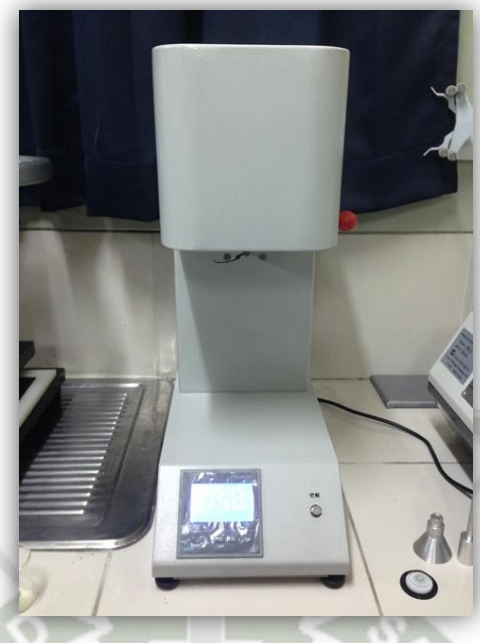


Figura 71. Plastómetro

Figura 72. Temperatura de trabajo

Figura 73. Limpieza de orificio

Figura 74. Montaje del dado

Figura 75. Pesaje de probetas

Figura 76. Colocación de trozos del compuesto

Figura 77. Montaje del peso

Figura 78. Purga del compuesto

Figura 79. Compuesto obtenido

Cuadro 2.10. Datos para el ensayo

Peso del Compuesto (gr)	5
Peso Aplicado al pistón (Kg)	7.160
Temperatura de Trabajo (°C)	158
Tiempo de Corte (seg)	90
Tiempo de Referencia (seg)	600

2.3.6. DETERMINACIÓN DE LA DENSIDAD

Para esta prueba cortamos una muestra en forma de cuadrado de cada una de las tres láminas que tenemos preparadas (ver Figura 80.). Una vez que tenemos las muestras las pesamos y tomamos medidas como el espesor, el largo y ancho de cada una de ellas (ver Cuadro 2.8) para que finalmente podamos obtener su densidad.

Figura 80. Probetas cuadradas (densidad)

Cuadro 2.11. Datos de muestras cuadradas

PROBETA	Código	Masa (g)	Espesor (mm)	Largo (mm)	Ancho (mm)
1	PLA Puro *	1.55	0.65	45.81	42.92
2	YP-0.50	0.27	0.57	20.00	20.00
3	YP-0.75	0.19	0.37	20.00	20.00
4	YM-2.32	0.56	1.32	20.00	20.00

* Dato obtenido de la tesis de Erick Ponce y Edson Espezúa

CAPÍTULO 3

ANÁLISIS Y DISCUSIÓN DE RESULTADOS

3.1 Descripción de las Láminas obtenidas

A. YP-0.50 que corresponde a una carga de 2.5 % de yute picado

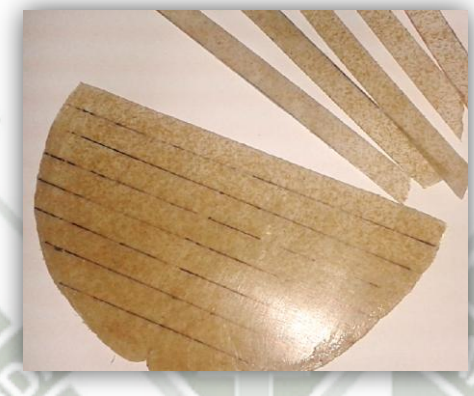


Figura 81: Imagen de la lámina **YP-0.50**

Descripción: Se obtiene una lámina translúcida de color café claro donde la fibra se encuentra uniformemente distribuido, con superficie lisa, de espesor uniforme, con presencia de algunas burbujas en los bordes y escasas en el centro. No se presentaron inconvenientes al mezclado en caliente en el Brabender.

B. YP-0.75 que corresponde a una carga de 3.75 % de yute picado

Figura 82: Imagen de la lámina **YP-0.75**

Descripción: Se obtiene una lámina translúcida de color heterogéneo café en algunas zonas, café claro en otras, la heterogeneidad en el color se debe a la mala dispersión de la fibra de yute en la matriz de PLA, esta lámina posee una superficie lisa, de espesor uniforme, con presencia de algunas burbujas en los bordes y escasas en el centro. Para la fabricación de la placa, al mezclar en caliente en el Brabender, la fibra presenta dificultades para dispersarse; por este motivo se requiere de más tiempo de permanencia en el Brabender.

C. YM-2.32 que corresponde a una carga de 11.6 % de yute tejido

Figura 83: Imagen de la lámina **YM-2.32**

Descripción: Se obtiene una lámina translúcida de color café claro, por ser un tejido el yute se encuentra uniformemente distribuido por toda la lámina, presenta algo de rugosidad en la superficie de la lámina sobre todo en los bordes, de espesor uniforme, con presencia de algunas burbujas en los bordes y escasas en el centro.

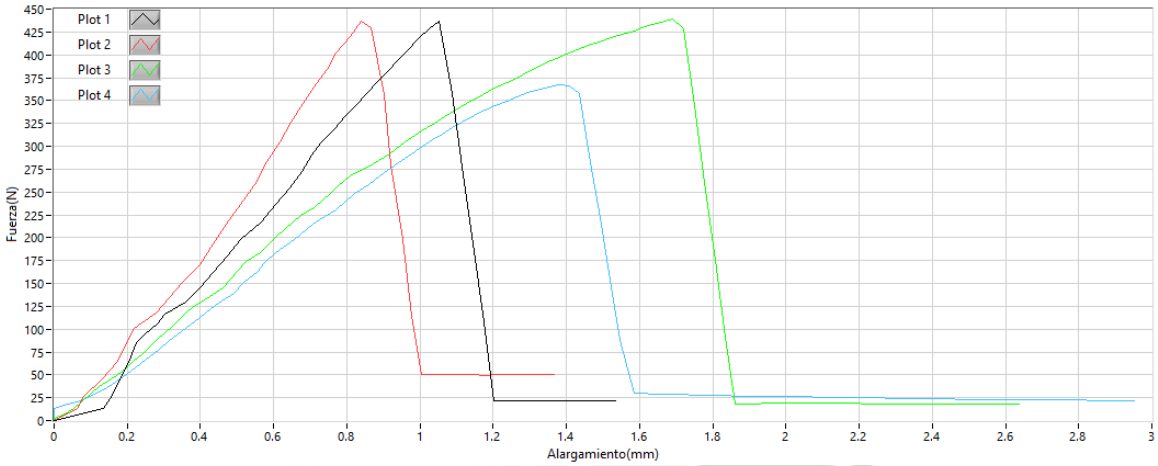
3.2 Resultados Propiedades Mecánicas

3.2.1 ANÁLISIS MECÁNICO POR RESISTENCIA A LA TRACCIÓN

Cuadro 3.1. Resultados de la probeta YP-0.50 (PLA con Yute picado)

N°	Carga Máxima (N)	Longitud Final (mm)	Resistencia a la tracción (MPa)	% de Deformación
1	437.328	100.9	45.04	0.90
2	436.298	101.1	47.29	1.10
3	438.652	90.1	42.92	1.24
4	367.014	95.2	35.40	1.28

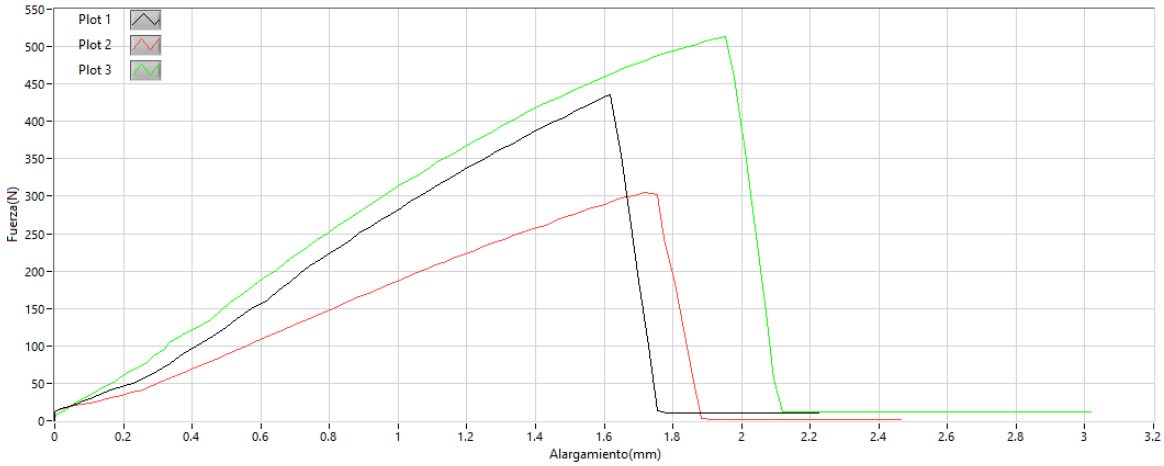
Gráfica 3.1. Esfuerzo-Deformación de YP-0.50 (PLA con Yute picado)



Cuadro 3.2. Resultados de la probeta YP-0.75 (PLA con Yute picado)

N°	Carga Máxima (N)	Longitud Final (mm)	Resistencia a la tracción (MPa)	% de Deformación
1	436.01	81.5	46.65	1.88
2	305.18	111.2	38.58	1.09
3	513.58	101.2	61.21	1.20

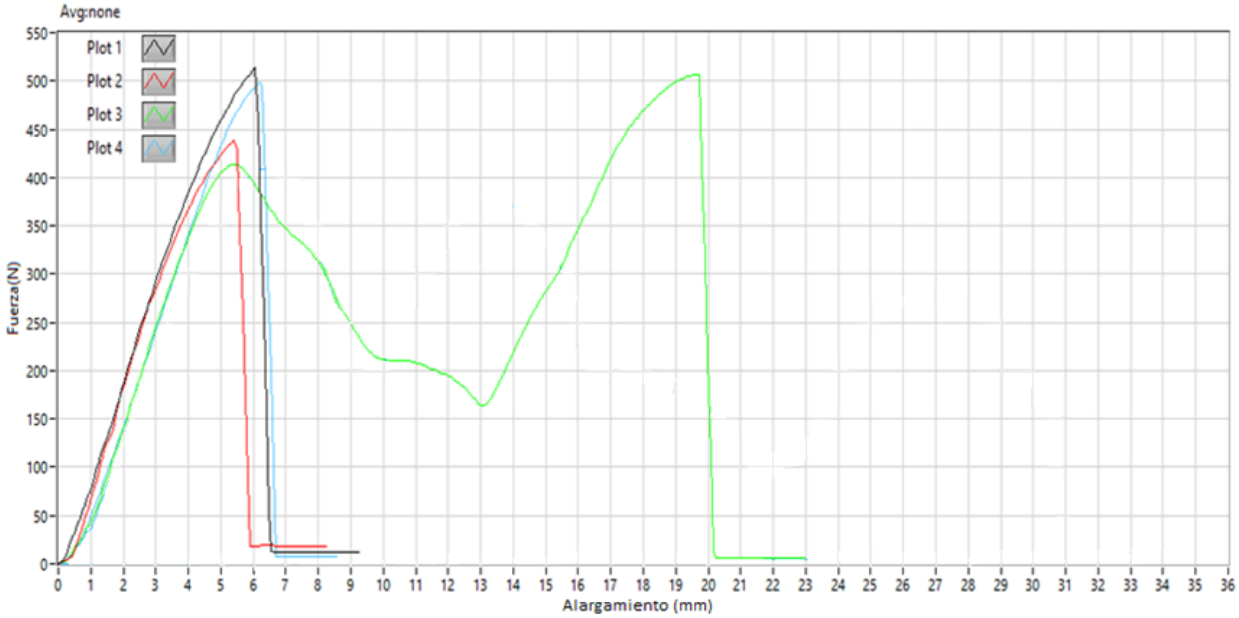
Gráfica 3.2. Esfuerzo-Deformación de YP-0.75 (PLA con Yute picado)



Cuadro 3.3. Resultados de la probeta YM-2.32 (PLA con Yute Tejido)

N°	Carga Máxima (N)	Longitud Final (mm)	Resistencia a la tracción (MPa)	% de Deformación
1	513.575	91.2	52.45	1.33
2	438.652	81.5	43.52	1.88
3	506.808	89.75	55.66	0.84
4	498.571	101.1	50.87	1.10

Gráfica 3.3. Esfuerzo-Deformación YM-2.32 (PLA con Yute Tejido)

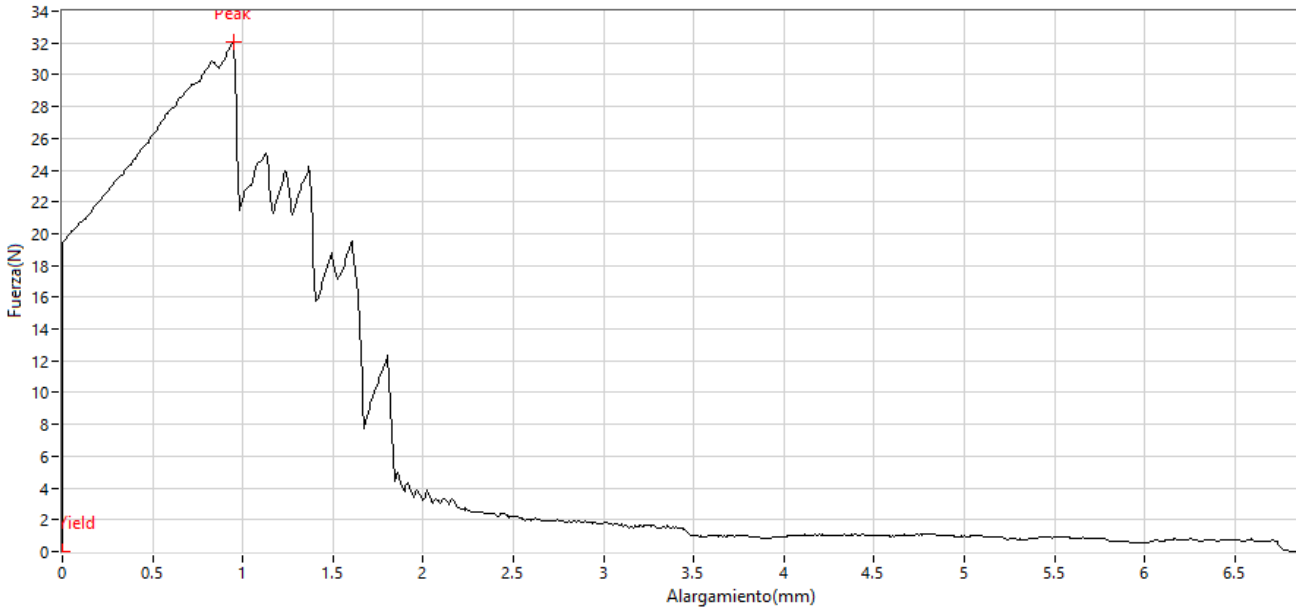


Cuadro 3.4. Resumen de resultados del ensayo de tracción

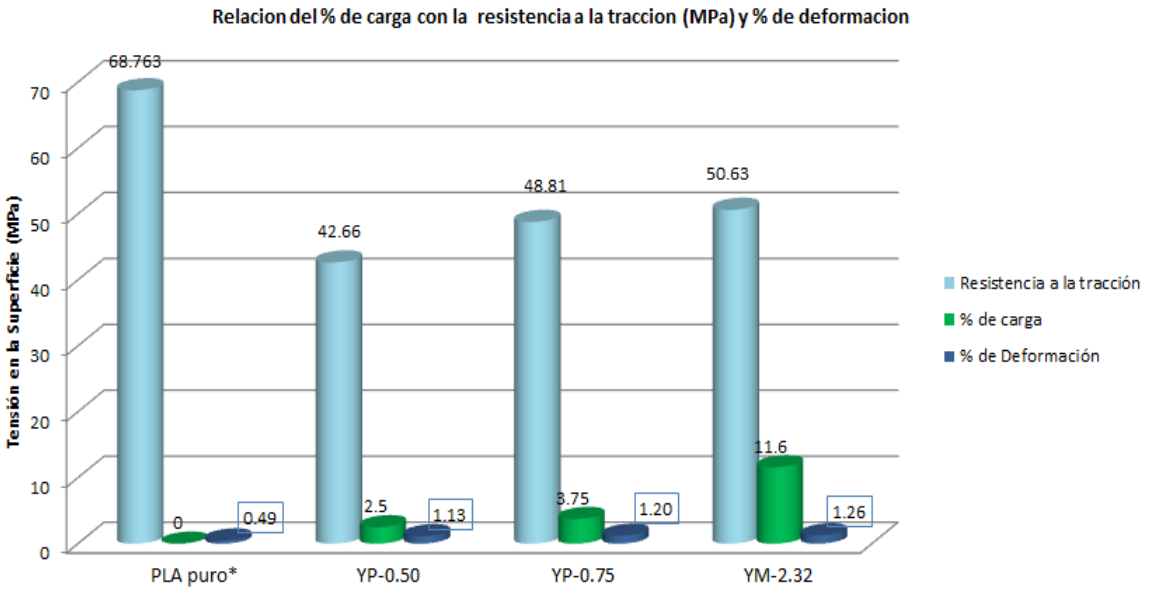
Código	% de carga	Mezcla Yute con PLA	Resistencia a la tracción (MPa)	% de Deformación
PLA puro*	0.00	Sin yute	68.763	0.494
Hilo de yute	Yute	Yute	139.425	1.21
YP-0.50	2.50	Yute Picado	42.66	1.13
YP-0.75	3.75	Yute Picado	48.81	1.20
YM-2.32	11.60	Yute Tejido	50.63	1.26

* Dato obtenido de la tesis de Erick Ponce y Edson Espezúa

Gráfica 3.4. Carga (N) – Alargamiento (mm) del Hilo de Yute



Gráfica 3.5. Comparación del % de carga de yute con la resistencia a la tracción y % de deformación



De acuerdo a la gráfica 3.5 se puede observar que existe una disminución de la resistencia a la tracción, se aprecia que de 68.763 MPa del PLA puro baja a 42.66, 48.81 y 50.63 MPa para las cargas de 2.5, 3.75 y 11.6% de yute respectivamente. El % de disminución de la resistencia a la tracción es de 37.96 % para una carga de 2.5 % de yute picado, 29.02 % para una carga de 3.75 % de yute picado y 26.37 % para una carga de 11.6 % de yute tejido. Se aprecia que la disminución de la resistencia a la tracción es menor para la mayor carga de yute bajo la forma de tejido. Por otro lado en cuanto al % de deformación se observa un aumento para todas las mezclas de los compuestos respecto al PLA puro, dándose la siguiente relación: Al aumentar la carga del reforzante el % de deformación aumenta.

En el cuadro 3.4 se muestra que el valor de la resistencia a la tracción obtenido para el hilo de yute es de 139.425 MPa (reforzante) y el valor para el PLA puro es de 68.763 MPa (matriz); analizando estos valores el compuesto final obtenido teóricamente debería tener un valor de resistencia a la tracción ubicado entre los valores de la matriz y el reforzante, sin embargo experimentalmente no sucede; incluso se observa la disminución de los valores la cual podría atribuirse a la mala interacción entre las superficies de la matriz y el reforzante siendo necesario

realizar más estudios que analicen el comportamiento de ambas superficies y mejoren su interacción.

3.2.2 ANÁLISIS MECÁNICO POR FLEXIÓN

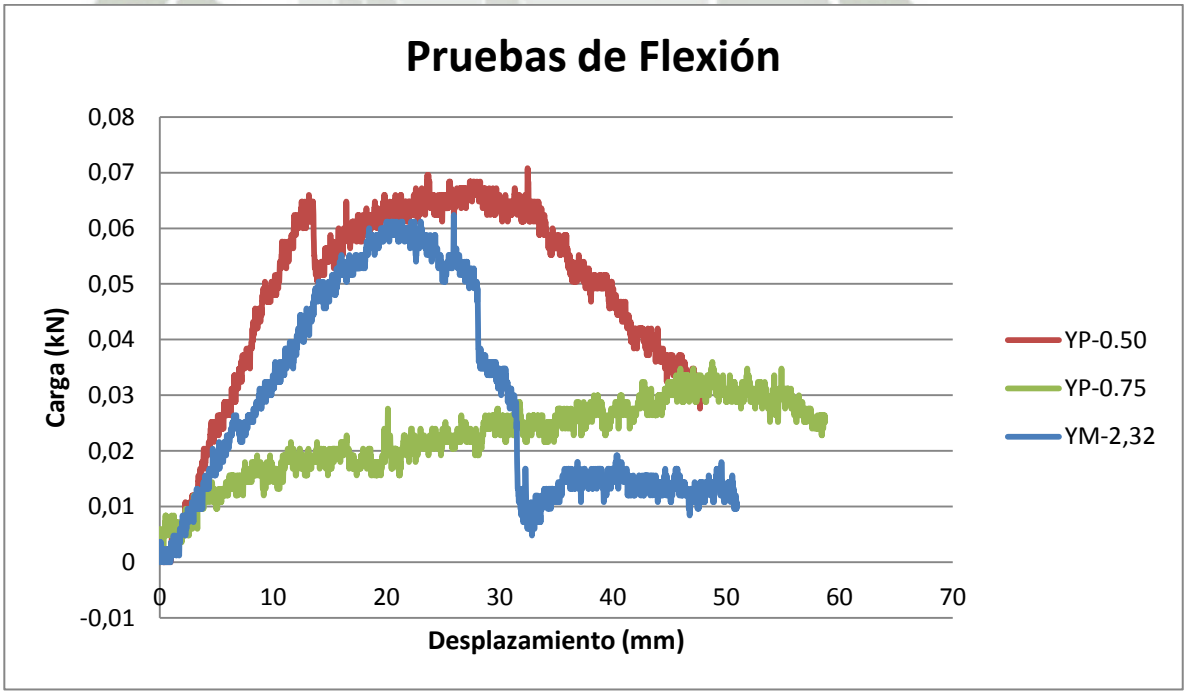
Los siguientes cuadros y gráficas son el resultado del ensayo realizado a cada una de las probetas, los cuales se muestran a continuación:

Cuadro 3.5. Resultados del Ensayo de Flexión

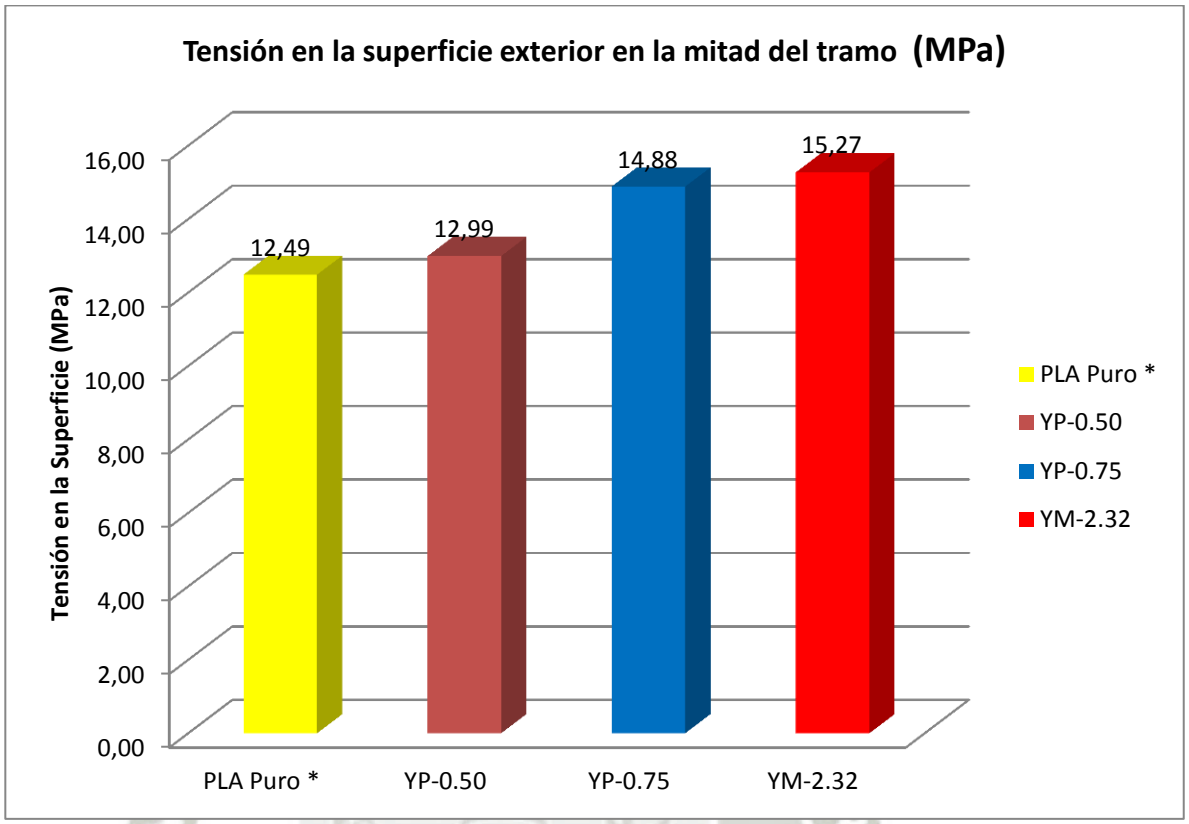
PROBETA	Código	Espesor (mm)	Ancho (mm)	Carga Máxima (N)	Tensión en la superficie exterior en la mitad del tramo (MPa)
1	PLA Puro *	5.00	13.00	54.00	12.49
2	YP-0.50	5.22	15.00	70.80	12.99
3	YP-0.75	3.48	15.00	36.00	14.88
4	YM-2.32	4.52	15.00	62.40	15.27

*Dato obtenido de la tesis de Erick Ponce y Edson Espezua

Gráfica 3.6. Prueba de Flexión



Gráfica 3.7. Tensión en la Superficie



En la gráfica 3.6 se muestran las curvas carga (KN) vs. Desplazamiento (mm) de los materiales compuestos reforzados con fibra de yute, tanto tejido como picado. De acuerdo con lo observado en la Figura 3.6 los materiales compuestos reforzados con fibra de yute YP- 0.50 y YM-2.32 presentan curvas del tipo lineal hasta un desplazamiento aproximado de 12 mm, en el compuesto YP-0.75 la linealidad no es tan notoria.

En la gráfica 3.7 se observa que la tensión superficial aumenta, dicho aumento es progresivo a medida que aumenta la carga, es así que a 11.6 % de yute tejido (YM-2.32) sube de 12.46 MPa (PLA puro) a 15.27 MPa lo que corresponde a un aumento del 22.45 %. En esta gráfica, al igual que en el ensayo de tracción, el material reforzado con fibra de yute tejido de % de carga de 11.6 es aquel que presenta las mejores características a flexión comparado con los demás compuestos ensayados.

3.2.3 ANÁLISIS DEL ENSAYO DE CHARPY

Los siguientes cuadros y gráficas son el resultado del ensayo realizado a cada una de las probetas, los cuales se muestran a continuación:

Cuadro 3.6. Resultados con PLA Puro

ENSAYO CHARPY - PLA PURO		
PROBETA	**Energía (J/m)**	**Angulo (°)**
1	47.3578	134.28
2	52.0653	133.60
3	41.9687	134.44
4	50.7131	133.54
5	52.5708	133.62
PROMEDIO	48.9351	133.89

Cuadro 3.7. Resultados con Probeta YP-0,50

ENSAYO CHARPY (YP-0,50)		
PROBETA	**Energía (J/m)**	**Angulo (°)**
1	50.1542	133.78
2	46.5773	134.23
3	54.4821	133.24
4	50.4733	133.74
5	54.4821	133.24
PROMEDIO	51.2338	133.64

Cuadro 3.8. Resultados con Probeta YP-0,75

ENSAYO CHARPY (YP-0,75)		
PROBETA	**Energía (J/m)**	**Angulo (°)**
1	46.8941	134.17
2	59.2400	133.60
3	54.8847	133.19
4	49.6416	133.84
5	51.5391	133.60
PROMEDIO	52.4399	133.68

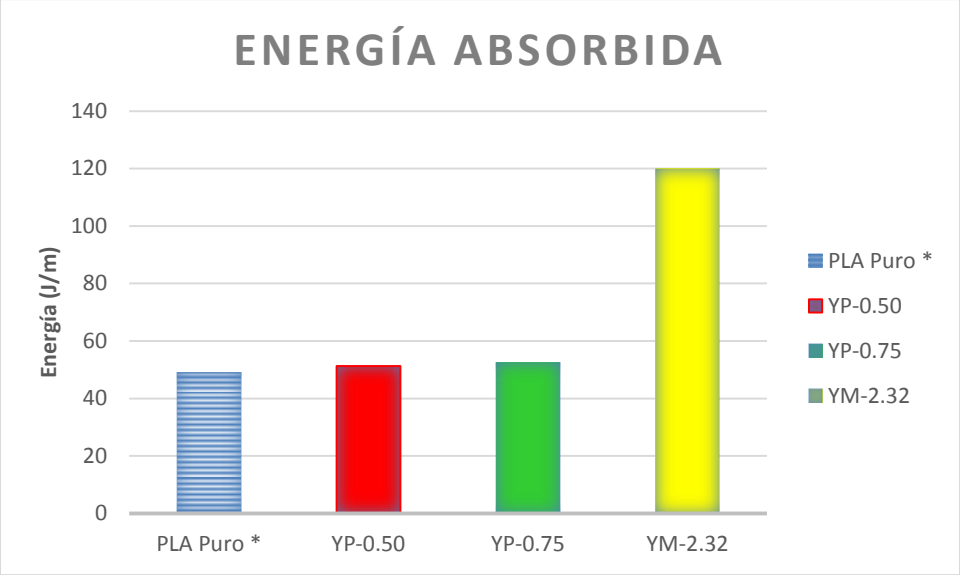
Cuadro 3.9. Resultados con Probeta Yute Tejido YM-2.32

ENSAYO CHARPY CON YUTE TEJIDO		
PROBETA	**Energía (J/m)**	**Angulo (°)**
1	122.6190	130.90
2	135.2148	130.00
3	123.1750	130.86
4	109.5079	131.85
5	109.4906	131.85
PROMEDIO	120.0014	131.09

Cuadro 3.10. Resumen de los resultados obtenidos

PROBETA	Código	Energía (J/m)
1	PLA Puro *	48.9351
2	YP-0.50	51.2338
3	YP-0.75	52.4399
4	YM-2.32	120.0014

Gráfica 3.8. Energía Absorbida por cada Probeta



Comentario: Como podemos observar el compuesto YM-2,32 (yute tejido) absorbe una mayor energía debido a que el compuesto lleva una malla de yute que está distribuida de manera uniforme en toda la probeta por lo cual es más difícil fracturarla. La mezcla YP-0.50 y YP-0.75 también muestra un aumento pero mínimo del valor de la energía absorbida respecto al PLA puro, lo que quizá se atribuya a la mala distribución del yute picado en la matriz de PLA (ver Figura 3.2).

De acuerdo con lo observado en las probetas ensayadas, es notable el efecto de barrera de las fibras frente al mazo impactador. Para el caso de la fibra corta tanto la matriz como las fibras de yute cortadas se fracturaban después de la acción del mazo impactador. Este fenómeno no se producía en las fibras de yute tejidas donde la fractura se daba en la matriz pero las fibras no llegaban a romperse completamente.

3.2.4 ANÁLISIS MECANICO POR DUREZA

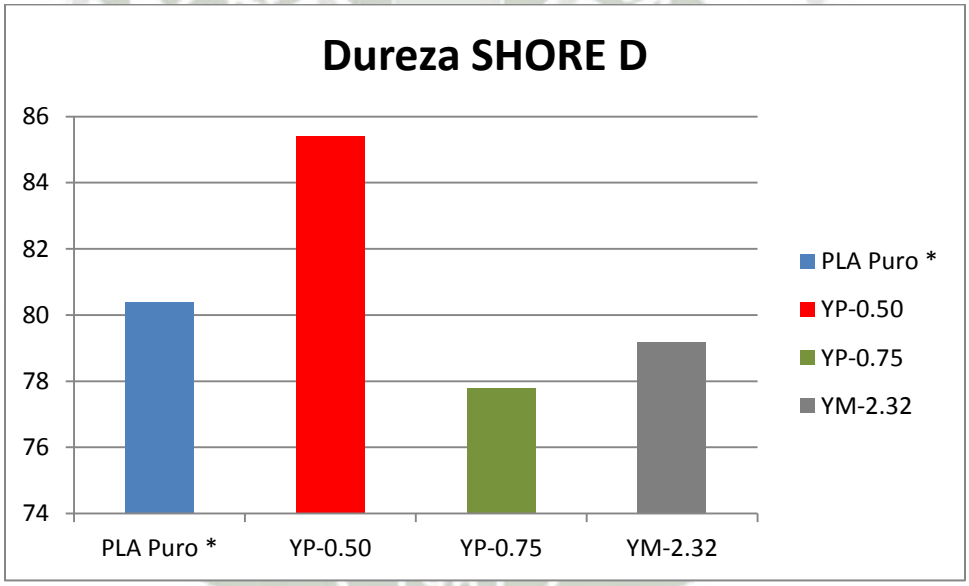
Los siguientes cuadros y gráficas son el resultado del ensayo realizado a cada una de las probetas, los cuales se muestran a continuación:

Cuadro 3.11. Dureza Shore D

PROBETA	Código	D-1	D-2	D-3	D-4	D-5	Dureza SHORE D
1	PLA Puro *	81	79	80	81	81	80.4
2	YP-0.50	84	88	86	85	84	85.4
3	YP-0.75	80	78	76	77	78	77.8
4	YM-2.32	79	80	78	80	79	79.2

*Dato obtenido de la tesis de Erick Ponce y Edson Espezua

Grafica 3.9. Dureza Shore D



Comentario: Podemos observar que la dureza del material compuesto de YP-0.50 es superior a la de los otros compuestos y también a la del mismo PLA puro el valor aumentó en 6.22%; este resultado pudo deberse a que al realizar la mezcla en todos los compuesto el que tuvo mejor dispersión fue el YP-0.50. Por otro lado el compuestos YM-3.32 presenta una ligera disminución del valor de dureza Shore D (1.49%) lo que quizá se deba a la presencia de zonas rugosas en la superficie de la

lámina de este compuesto y por último el compuestos YP-075 disminuye en 3.23 % comparado con el yute puro.

3.2.5 ANÁLISIS DEL INDICE DE FLUIDEZ

Los siguientes cuadros y gráficas son el resultado del ensayo realizado con cada de las probetas, los cuales se muestran a continuación:

Cuadro 3.12. Resultado de Probeta con PLA Puro

PROBETA	PESO (gr)
1	0.45
2	0.43
3	0.40
4	0.43
5	0.43
6	0.40
7	0.46
8	0.47
PROMEDIO	0.43

0.43 gr ---------- 90 seg
X gr ---------- 600 seg

X= (0.43gr x 600seg)/90seg

X= 2.86 gr/10mín

Indice de Fluidez MFR (158°C/7.160Kg)= 2.86 gr/10mín

Cuadro 3.13. Resultado de Probeta YP-0,50

PROBETA	PESO (gr)
1	0.29
2	0.32
3	0.36
4	0.40
5	0.45
6	0.52
7	0.54
PROMEDIO	0.41

0.41 gr ---------- 90 seg
X gr ---------- 600 seg

X= (0.41gr x 600seg)/90seg

X= 2.73 gr/10mín

Índice de Fluidez MFR (158°C/7.160Kg)= 2.73 gr/10mín

Cuadro 3.14. Resultado con Probeta YP-0,75

PROBETA	PESO (gr)
1	0.37
2	0.40
3	0.45
4	0.48
5	0.54
6	0.61
7	0.65
8	0.67
PROMEDIO	0.52

0.52 gr ---------- 90 seg
X gr ---------- 600 seg

X= (0.52gr x 600seg)/90seg

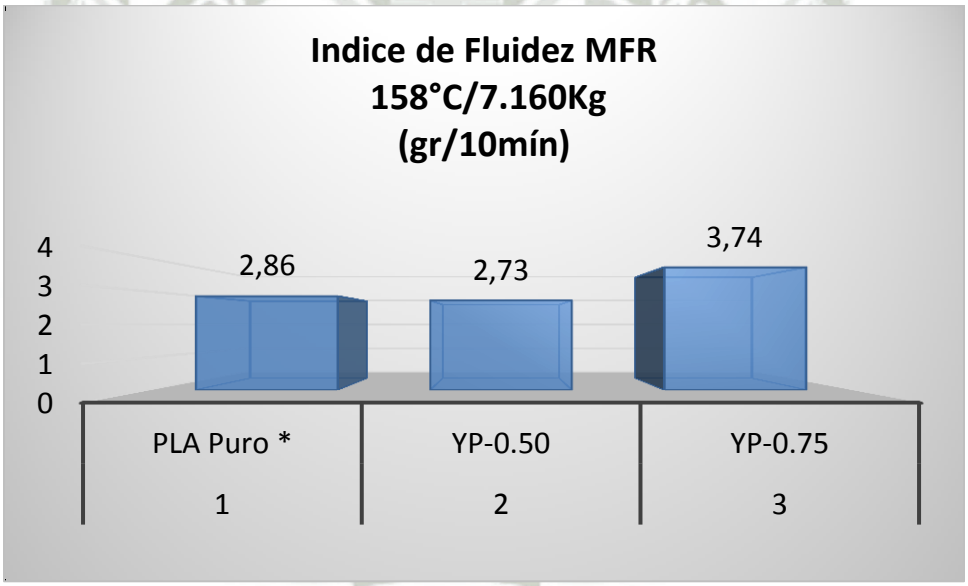
X= 3.74 gr/10mín

Índice de Fluidez MFR (158°C/7.160Kg)= 3.74 gr/10mín

Cuadro 3.15. Resumen de resultados obtenidos

PROBETA	Código	Índice de Fluidez MFR 158°C/7.160Kg (gr/10mín)
1	PLA Puro *	2.86
2	YP-0.50	2.73
3	YP-0.75	3.74

Gráfica 3.10. Índice de Fluidez de los Compuestos



Comentario: Podemos indicar que luego de haber trabajado con un mismo peso, misma temperatura el índice de fluidez del material cambia con forme se le incremento la carga. Lo cual es importante saber, debido a que si esto es llevado a producción dará la idea de los parámetros de trabajo en los equipos de extrusión.

En la gráfica 3.10 se puede observar la diferencia del índice de fluidez en las diferentes muestras. Podemos afirmar que en la mezcla YP-050 se reduce el valor

del índice de fluidez, por otro lado se puede observar también que en el compuesto YP-075 existe un aumento del valor del índice de fluidez.

3.2.6 DENSIDAD

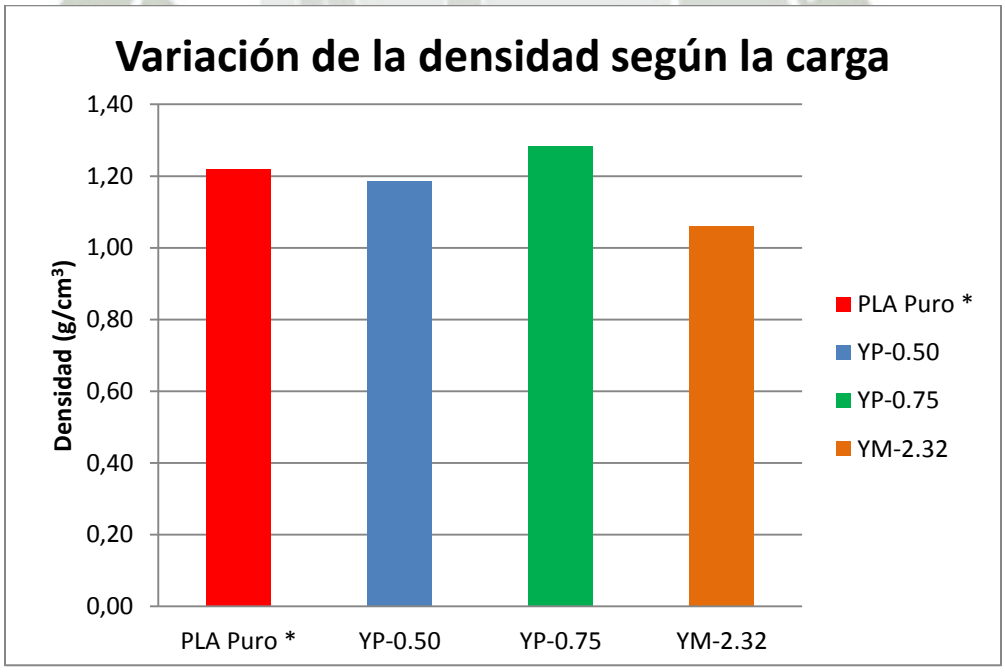
Los siguientes cuadros y gráficas son el resultado del ensayo realizado a cada una de las probetas, los cuales se muestran a continuación:

Cuadro 3.16. Densidad de las muestras

PROBETA	Código	DENSIDAD (g/cm^3)
1	PLA Puro *	1.22
2	YP-0.50	1.18
3	YP-0.75	1.28
4	YM-2.32	1.06

***** Dato obtenido de la tesis de Erick Ponce y Edson Espezúa

Gráfica 3.11. Variación de la Densidad

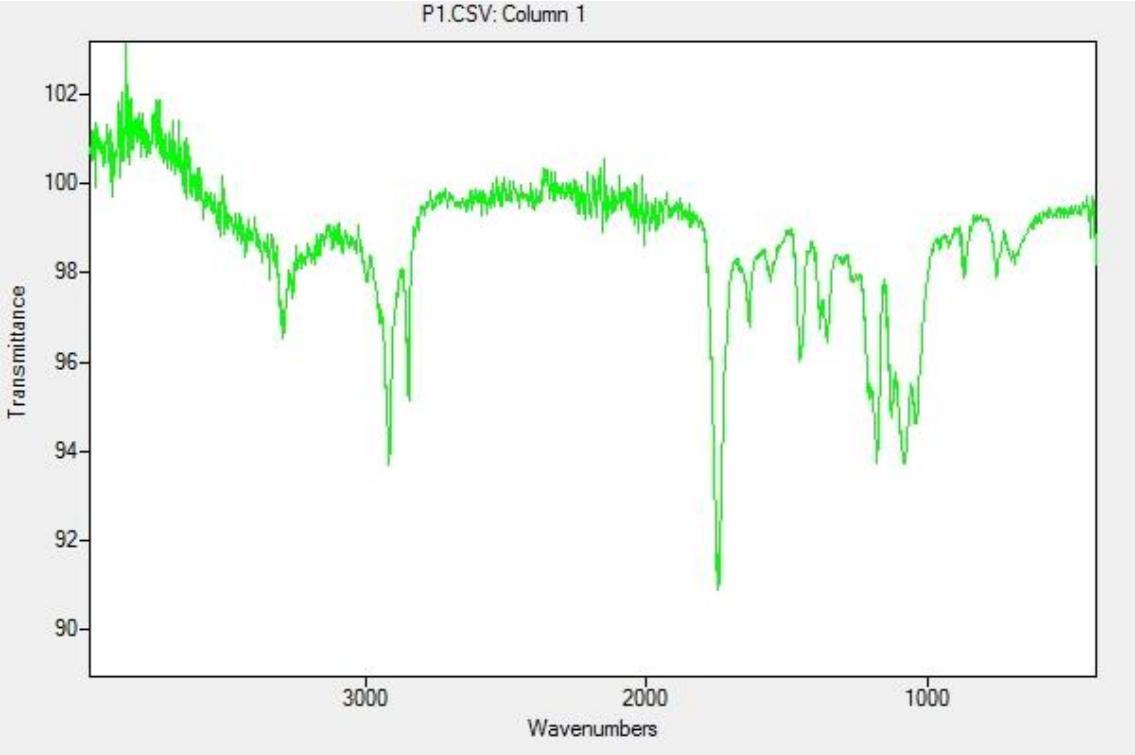


Comentario: Podemos concluir que se da un cambio en la densidad cuando se trabaja con la malla de yute; la disminución de la densidad es de 13.11 % respecto al valor del PLA puro; luego la variación es mínima para las demás mezclas de los demás compuestos. El disminuir la densidad de un compuesto hace que este sea más ligero.

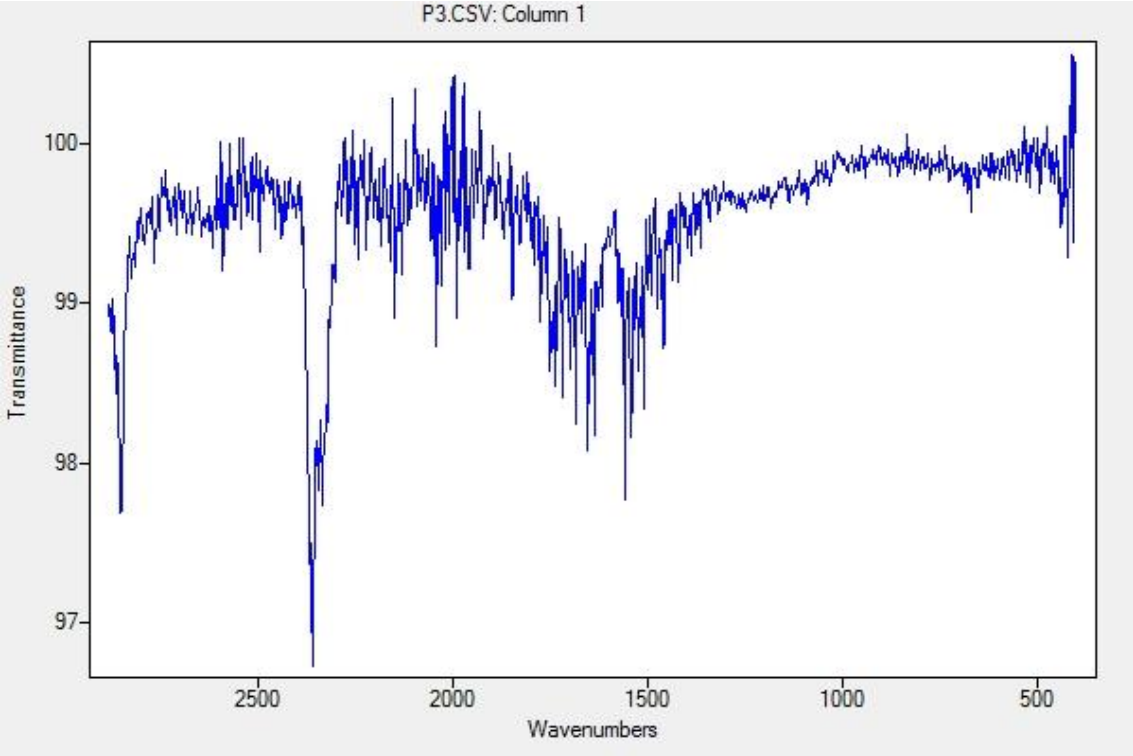
3.2.7 Espectroscopia de Infrarrojo por Transformada de Fourier FTIR

Se basa en la absorción de radiación infrarroja por el material que se analiza. Los análisis fueron realizados en un FTIR Nicolet modelo de Impacto 400D, el método ATR (análisis del sólido).

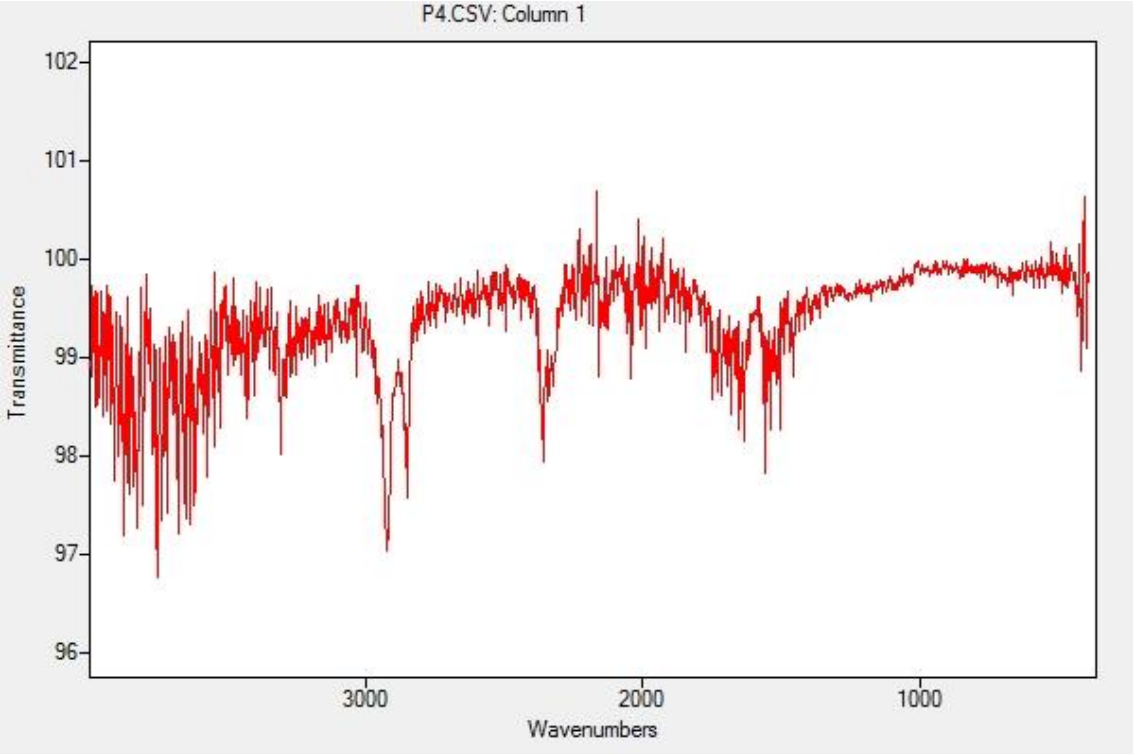
Gráfica 3.12 P1-Probeta PLA Puro



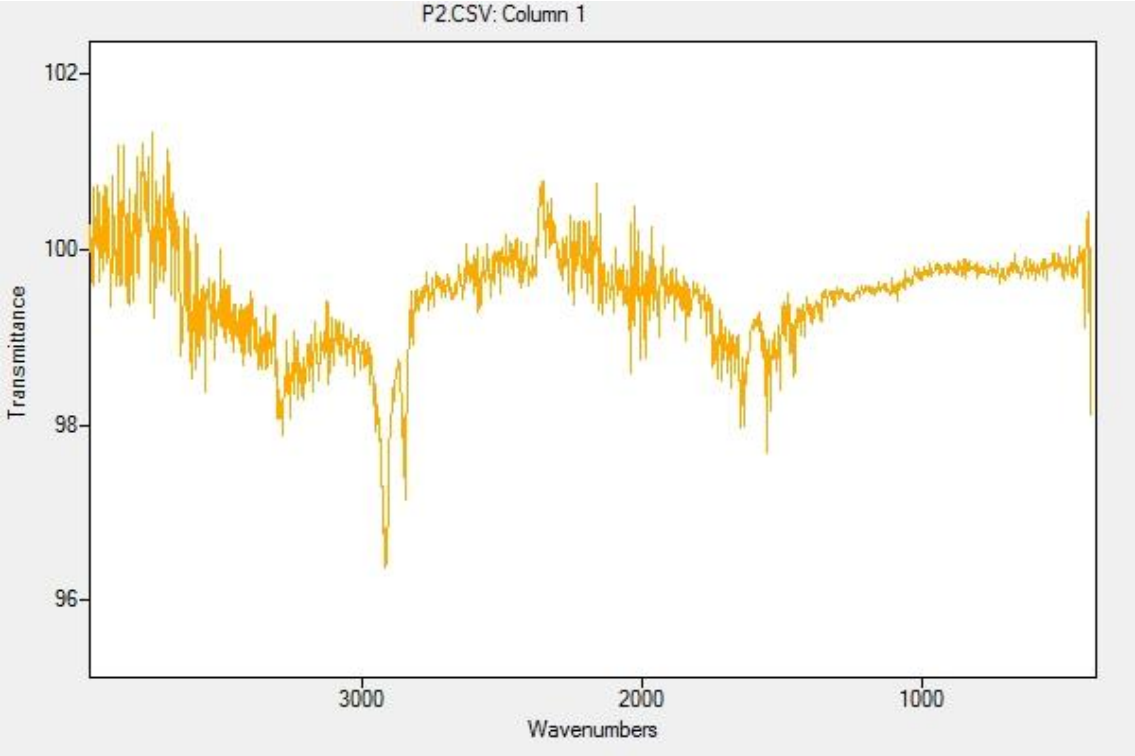
Gráfica 3.13 P2-Probeta YP-0,50



Gráfica 3.14 P3-Probeta YP-0,75



Gráfica 3.15 P4-Probeta YM-2.32



Comentario: Podemos observar que se presentan cambios en las mezclas de PLA en el rango de 1500 a 1100 cm^{-1} (longitud de onda). Lo que evidencia una unión entre el PLA y el yute.

CONCLUSIONES

1. Se ha preparado compuestos en base a PLA con 2.5% y 3.75% yute picado además de 11.6 % de yute tejido, todos fabricados por moldeo por compresión. Luego se ha evaluado las propiedades mecánicas (tracción, flexión, impacto y dureza) y el índice de fluidez de los compuestos obtenidos.
2. Se ha logrado el aumento de la Tensión en la superficie exterior en la mitad del tramo (MPa) en un 22.45 % usando un 11.6 % de tejido de yute en el compuesto con PLA.
3. En los compuestos obtenidos se presenta una disminución de la resistencia a la tracción desde un 26,37 % hasta un 37.96 % al aumentar la carga (g) yute de 2.5 % y 11.6 % respectivamente.
4. El compuesto YM-2.32 absorbe mayor energía (J/m) cuyo valor es 120.0014 y los otros dos compuestos tanto el YP-0.50 y el YP-0.75 absorben menor energía (51.2338 J/m y 52.4399 J/m), siendo uno de los factores más importantes la distribución homogénea del reforzante en la matríz polimeriaca.
5. El empleo de refuerzos de yute en matrices de plásticos biodegradables como el ácido poliláctico (PLA) es interesante desde el punto de vista medioambiental y coste del material. Los plásticos biodegradables todavía presentan un coste elevado en relación a los plásticos de uso común y, en este sentido, la incorporación de refuerzos de yute (con propiedades mecánicas muy inferiores al PLA) no mejora las propiedades mecánicas, pero las mantiene en niveles habituales de muchos de los plásticos de uso común.

RECOMENDACIONES

1. El estudio de la sección de tracción pone en evidencia la falta de interacción entre el polímero base para la matríz de los compuestos (PLA) y las fibras de yute, con lo cual, una línea de trabajo interesante sería emplear algún tratamiento de las fibras para la mejora de la interacción.
2. Dado que la mezcla del PLA y el yute picado no a sido muy uniforme es recomendable emplear compatibilizantes que ayuden a generar una mezcla homogénea entre el PLA y el Yute, lo cual ayudaría aumentar las propiedades del compuesto.
3. Por otro lado, teniendo en cuenta que otras fibras naturales presentan propiedades mecánicas muy superiores a las de los polímeros biodegradables, otra línea de trabajo interesante sería el estudio de compuestos con otras fibras naturales.

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ANEXOS

Designation: D 882 – 02

Standard Test Method for Tensile Properties of Thin Plastic Sheeting[1]

This standard is issued under the fixed designation D 882; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 1013 of Federal Test Method Standard 406.

1. Scope *

1.1 This test method covers the determination of tensile properties of plastics in the form of thin sheeting, including film (less than 1.0 mm (0.04 in.) in thickness).

NOTE 1—Film has been arbitrarily defined as sheeting having nominal thickness not greater than 0.25 mm (0.010 in.).

NOTE 2—Tensile properties of plastics 1.0 mm (0.04 in.) or greater in thickness shall be determined according to Test Method D 638.

1.2 This test method may be used to test all plastics within the thickness range described and the capacity of the machine employed.

1.2.1 *Static Weighing, Constant-Rate-of-Grip Separation Test*—This test method employs a constant rate of separation of the grips holding the ends of the test specimen.

1.3 Specimen extension may be measured in these test methods by grip separation, extension indicators, or displacement of gage marks.

1.4 A procedure for determining the tensile modulus of elasticity is included at one strain rate.

NOTE 3—The modulus determination is generally based on the use of grip separation as a measure of extension; however, the desirability of using extensometers, as described in 5.2, is recognized and provision for the use of such instrumentation is incorporated in the procedure.

1.5 Test data obtained by this test method is relevant and appropriate for use in engineering design.

1.6 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information only.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 4—This test method is similar to ISO 527-3, but is not considered technically equivalent. ISO 527-3 allows for additional specimen configurations, specifies different test speeds, and requires an extensometer or gage marks on the specimen.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[2]

D 638 Test Method for Tensile Properties of Plastics[2]

D 4000 Classification System for Specifying Plastic Materials[3]

D 5947 Test Methods for Physical Dimensions of Solid Plastic Specimens[4]

D 6287 Practice for Cutting Film and Sheeting Test Specimens[4]

E 4 Practices for Force Verification of Testing Machines[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

2.2 *ISO Standard:*

ISO 527-3 Plastics—Determination of Tensile Properties—Part 3: Test Conditions for Films and Sheets[7]

3. Terminology

3.1 *Definitions*—Definitions of terms and symbols relating to tension testing of plastics appear in the Annex to Test Method D 638.

3.1.1 *line grips*—grips having faces designed to concentrate the entire gripping force along a single line perpendicular to the direction of testing stress. This is usually done by combining one standard flat face and an opposing face from which protrudes a half-round.

3.1.2 *tear failure*—a tensile failure characterized by fracture initiating at one edge of the specimen and progressing across the specimen at a rate slow enough to produce an anomalous load-deformation curve.

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 08.02.

[4] *Annual Book of ASTM Standards*, Vol 08.03.

[5] *Annual Book of ASTM Standards*, Vol 03.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

4. Significance and Use

4.1 Tensile properties determined by this test method are of value for the identification and characterization of materials for control and specification purposes. Tensile properties may vary with specimen thickness, method of preparation, speed of testing, type of grips used, and manner of measuring extension. Consequently, where precise comparative results are desired, these factors must be carefully controlled. This test method shall be used for referee purposes, unless otherwise indicated in particular material specifications. For many materials, there may be a specification that requires the use of this test method, but with some procedural modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 in Classification D 4000 lists the ASTM materials standards that currently exist.

4.2 Tensile properties may be utilized to provide data for research and development and engineering design as well as quality control and specification. However, data from such tests cannot be considered significant for applications differing widely from the load-time scale of the test employed.

4.3 The tensile modulus of elasticity is an index of the stiffness of thin plastic sheeting. The reproducibility of test results is good when precise control is maintained over all test conditions. When different materials are being compared for stiffness, specimens of identical dimensions must be employed.

4.4 The tensile energy to break (TEB) is the total energy absorbed per unit volume of the specimen up to the point of rupture. In some texts this property has been referred to as *toughness*. It is used to evaluate materials that may be subjected to heavy abuse or that might stall web transport equipment in the event of a machine malfunction in end-use applications. However, the rate of strain, specimen parameters, and especially flaws may cause large variations in the results. In that sense, caution is advised in utilizing TEB test results for end-use design applications.

4.5 Materials that fail by tearing give anomalous data which cannot be compared with those from normal failure.

5. Apparatus

5.1 *Testing Machine*—A testing machine of the constant rate-of-crosshead-movement type and comprising essentially the following:

5.1.1 *Fixed Member*—A fixed or essentially stationary member carrying one grip.

5.1.2 *Movable Member*—A movable member carrying a second grip.

5.1.3 *Grips*—A set of grips for holding the test specimen between the fixed member and the movable member of the testing machine; grips can be either the fixed or self-aligning type. In either case, the gripping system must minimize both slippage and uneven stress distribution.

5.1.3.1 Fixed grips are rigidly attached to the fixed and movable members of the testing machine. When this type of grip is used, care must be taken to ensure that the test specimen is inserted and clamped so that the long axis of the test specimen coincides with the direction of pull through the center line of the grip assembly.

5.1.3.2 Self-aligning grips are attached to the fixed and movable members of the testing machine in such a manner that they will move freely into alignment as soon as a load is applied so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly. The specimens should be aligned as perfectly as possible with the direction of pull so that no rotary motion that may induce slippage will occur in the grips; there is a limit to the amount of misalignment self-aligning grips will accommodate.

5.1.3.3 The test specimen shall be held in such a way that slippage relative to the grips is prevented insofar as possible. Grips lined with thin rubber, crocus-cloth, or pressure-sensitive tape as well as file-faced or serrated grips have been successfully used for many materials. The choice of grip surface will depend on the material tested, thickness, etc. Line grips padded on the round face with 1.0 mm (40 mil) blotting paper or filter paper have been found superior. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to “neck” into the grips, since pressure is maintained at all times. In cases where samples frequently fail at the edge of the grips, it may be advantageous to increase slightly the radius of curvature of the edges where the grips come in contact with the test area of the specimen.

5.1.4 *Drive Mechanism*—A drive mechanism for imparting to the movable member a uniform, controlled velocity with respect to the stationary member. The velocity shall be regulated as specified in Section 9.

5.1.5 *Load Indicator*—A suitable load-indicating mechanism capable of showing the total tensile load carried by the test specimen held by the grips. This mechanism shall be essentially free of inertial lag at the specified rate of testing (see Note 5). Unless a suitable extensometer is used (see 5.2), the motion of the weighing system shall not exceed 2 % of the specimen extension within the range being measured. The load indicator shall determine the tensile load applied to the specimen with an accuracy of ±1 % of the indicated value, or better. The accuracy of the testing machine shall be verified in accordance with Practices E 4.

5.1.6 *Crosshead Extension Indicator*— A suitable extension-indicating mechanism capable of showing the amount of change in the separation of the grips, that is, crosshead movement. This mechanism shall be essentially free of inertial lag at the specified rate of testing (see Note 5) and shall indicate the crosshead movement with an accuracy of ±1 % of the indicated value, or better.

5.2 *Extensometer (Optional)*—A suitable instrument may, if desired, be used for determining the distance between two designated points on the test specimen as the specimen is stretched. This apparatus, if employed, shall be so designed as to minimize stress on the specimen at the contact points of the specimen and the instrument (see 8.3). It is desirable that this instrument automatically record the distance, or any change in it, as a function of the load on the test specimen or of the elapsed time from the start of the test, or both. If only the latter is obtained, load-time data must also be taken. This instrument must be essentially free of inertial lag at the specified speed of testing (see Note 5).

5.2.1 *Modulus of Elasticity and Low-Extension Measurements*—Extensometers used for modulus of elasticity and low-extension (less than 20 % elongation) measurements shall, at a minimum, be accurate to ±1 % and comply with the requirements set forth in Practice E 83 for a Class C instrument.

5.2.2 *High-Extension Measurements*—Instrumentation and measuring techniques used for high-extension (20 % elongation or greater) measurements shall be accurate to ±10 % of the indicated value, or better.

NOTE 5—A sufficiently high response speed in the indicating and recording system for the load and extension data is essential. The response speed required of the system will depend in part on the material tested (high or low elongation) and the rate of straining.

5.3 *Thickness Gage*—A dead-weight dial micrometer as prescribed in Method C of Test Methods D 5947, or an equivalent measuring device, reading to 0.0025 mm (0.0001 in.) or less.

5.4 *Width-Measuring Devices*—Suitable test scales or other width measuring devices capable of measuring 0.25 mm (0.010 in.) or less.

5.5 *Specimen Cutter*—For the apparatus and techniques for cutting film and sheeting used in this test method, refer to Practice D 6287.

5.5.1 Devices that use razor blades have proven especially suitable for materials having an elongation-at-fracture above 10 to 20 %.

5.5.2 The use of punch press or striking dies are not recommended because poor and inconsistent specimen edges may be produced.

6. Test Specimens

6.1 The test specimens shall consist of strips of uniform width and thickness at least 50 mm (2 in.) longer than the grip separation used.

6.2 The nominal width of the specimens shall be not less than 5.0 mm (0.20 in.) or greater than 25.4 mm (1.0 in.).

6.3 A width-thickness ratio of at least eight shall be used. Narrow specimens magnify effects of edge strains or flaws, or both.

6.4 The utmost care shall be exercised in cutting specimens to prevent nicks and tears which are likely to cause premature failures (Note 6). The edges shall be parallel to within 5 % of the width over the length of the specimen between the grips.

NOTE 6—Microscopical examination of specimens may be used to detect flaws due to sample or specimen preparation.

6.5 Wherever possible, the test specimens shall be selected so that thickness is uniform to within 10 % of the thickness over the length of the specimen between the grips in the case of materials 0.25 mm (0.010 in.) or less in thickness and to within 5 % in the case of materials greater than 0.25 mm (0.010 in.) in thickness but less than 1.00 mm (0.040 in.) in thickness.

NOTE 7—In cases where thickness variations are in excess of those recommended in 6.5, results may not be characteristic of the material under test.

6.6 If the material is suspected of being anisotropic, two sets of test specimens shall be prepared having their long axes respectively parallel with and normal to the suspected direction of anisotropy.

6.7 For tensile modulus of elasticity determinations, a specimen gage length of 250 mm (10 in.) shall be considered as standard. This length is used in order to minimize the effects of grip slippage on test results. When this length is not feasible, test sections as short as 100 mm (4 in.) may be used if it has been shown that results are not appreciably affected. However, the 250-mm gage length shall be used for referee purposes. The speed of testing of shorter specimens must be adjusted in order for the strain rate to be equivalent to that of the standard specimen.

NOTE 8—Two round robin tests[8] have shown that, for materials of less than 0.25-mm (10-mil) thickness, line grips padded on the round side with 1.0-mm (40-mil) blotting paper give the same results with a 100-mm test section as a 250-mm test section produces with flat-face grips.

NOTE 9—Excessive jaw slippage becomes increasingly difficult to overcome in cases where high modulus materials are tested in thicknesses greater than 0.25 mm (0.010 in.).

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 unless otherwise specified by contract or the relevant ASTM material specification. Reference pre-test conditioning, to settle disagreements, shall apply tolerances of ±1°C (1.8°F) and ±2 % relative humidity.

7.2 *Test Conditions*—Conduct the tests at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity unless otherwise specified by contract or the relevant ASTM material specification. Reference testing conditions, to settle disagreements, shall apply tolerances of ±1°C (1.8°F) and ±2 % relative humidity.

8. Number of Test Specimens

8.1 In the case of isotropic materials, at least five specimens shall be tested from each sample.

8.2 In the case of anisotropic materials, at least ten specimens, five normal and five parallel with the principal axis of anisotropy, shall be tested from each sample.

8.3 Specimens that fail at some obvious flaw or that fail outside the gage length shall be discarded and retests made, unless such flaws or conditions constitute a variable whose effect is being studied. However, jaw breaks (failures at the grip contact point) are acceptable if it has been shown that results from such tests are in essential agreement with values obtained from breaks occurring within the gage length.

NOTE 10—In the case of some materials, examination of specimens, prior to and following testing, under crossed optical polarizers (polarizing films) provides a useful means of detecting flaws which may be, or are, responsible for premature failure.

[8] Supporting data are available from ASTM Headquarters. Request RR: D20-1058.

9. Speed of Testing

9.1 The speed of testing is the rate of separation of the two members (or grips) of the testing machine when running idle (under no load). This rate of separation shall be maintained within 5 % of the no-load value when running under full-capacity load.

9.2 The speed of testing shall be calculated from the required initial strain rate as specified in Table 1. The rate of grip separation may be determined for the purpose of these test methods from the initial strain rate as follows:

$$A = BC \quad (1)$$

where:

A = rate of grip separation, mm (or in.)/min,
B = initial distance between grips, mm (or in.), and
C = initial strain rate, mm/mm·min (or in./in.·min).

9.3 The initial strain rate shall be as in Table 1 unless otherwise indicated by the specification for the material being tested.

NOTE 11—Results obtained at different initial strain rates are not comparable; consequently, where direct comparisons between materials in various elongation classes are required, a single initial strain rate should be used. For some materials it may be advisable to select the strain rates on the basis of percent elongation at yield.

9.4 In cases where conflicting material classification, as determined by percent elongation at break values, results in a choice of strain rates, the lower rate shall be used.

9.5 If modulus values are being determined, separate specimens shall be used whenever strain rates and specimen dimensions are not the same as those employed in the test for other tensile properties.

10. Procedure

10.1 Select a load range such that specimen failure occurs within its upper two thirds. A few trial runs may be necessary to select a proper combination of load range and specimen width.

10.2 Measure the cross-sectional area of the specimen at several points along its length. Measure the width to an accuracy of 0.25 mm (0.010 in.) or better. Measure the thickness to an accuracy of 0.0025 mm (0.0001 in.) or better for films less than 0.25 mm (0.010 in.) in thickness and to an accuracy of 1 % or better for films greater than 0.25 mm (0.010 in.) but less than 1.0 mm (0.040 in.) in thickness.

10.3 Set the initial grip separation in accordance with Table 1.

10.4 Set the rate of grip separation to give the desired strain rate, based on the initial distance between the grips, in accordance with Table 1. Zero the calibrated load weighing system, extension indicator(s) and recording system.

NOTE 12—Extensometers may be used for modulus of elasticity determinations with the expectation of obtaining more accurate values than may be obtained using grip separation as the effective gage length. Precautions should be taken to ensure that extensometer slippage and undue stressing of the specimen do not occur. Refer also to 6.7.

10.5 In cases where it is desired to measure a test section other than the total length between the grips, mark the ends of the desired test section with a soft, fine wax crayon or with ink. Do not scratch these marks onto the surface since such scratches may act as stress raisers and cause premature specimen failure. Extensometers may be used if available; in this case, the test section will be defined by the contact points of the extensometer.

NOTE 13—Measurement of a specific test section is necessary with some materials having high elongation. As the specimen elongates, the accompanying reduction in area results in a loosening of material at the inside edge of the grips. This reduction and loosening moves back into the grips as further elongation and reduction in area takes place. In effect, this causes problems similar to grip slippage, that is, exaggerates measured extension.

10.6 Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize slipping of the specimen during test.

10.7 Start the machine and record load versus extension.

10.7.1 When the total length between the grips is used as the test area, record load versus grip separation.

10.7.2 When a specific test area has been marked on the specimen, follow the displacement of the edge boundary lines with respect to each other with dividers or some other suitable device. If a load-extension curve is desired, plot various extensions versus corresponding loads sustained, as measured by the load indicator.

10.7.3 When an extensometer is used, record load versus extension of the test area measured by the extensometer.

10.8 If modulus values are being determined, select a load range and chart rate to produce a load-extension curve of between 30 and 60° to the X axis. For maximum accuracy, use the most sensitive load scale for which this condition can be met. The test may be discontinued when the load-extension curve deviates from linearity.

10.9 In the case of materials being evaluated for secant modulus, the test may be discontinued when the specified extension has been reached.

TABLE 1 Crosshead Speeds and Initial Grip Separation

Percent Elongation at Break	Initial Strain Rate, mm/mm·min (in./in.·min)	Initial Grip Separation		Rate of Grip Separation	
		mm	in.	mm/min	in./min
Modulus of Elasticity Determination					
	0.1	250	10	25	1.0
Determinations other than Elastic Modulus					
Less than 20	0.1	125	5	12.5	0.5
20 to 100	0.5	100	4	50	2.0
Greater than 100	10.0	50	2	500	20.0

10.10 If tensile energy to break is being determined, some provision must be made for integration of the stress-strain curve. This may be either an electronic integration during the test or a subsequent determination from the area of the finished stress-strain curve (see Annex A2).

11. Calculation

11.1 Toe compensation shall be made in accordance with Annex A1 unless it can be shown that the toe region of the curve is not due to the takeup of slack, seating of the specimen, or other artifact, but rather is an authentic material response.

11.2 *Breaking Factor* (nominal) shall be calculated by dividing the maximum load by the original minimum width of the specimen. The result shall be expressed in force per unit of width, usually newtons per metre (or pounds per inch) of width, and reported to three significant figures. The thickness of the film shall always be stated to the nearest 0.0025 mm (0.0001 in.).

Example—Breaking Factor = 1.75 kN/m (10.0 lbf/in.) of width for 0.1300-mm (0.0051-in.) thickness.

NOTE 14—This method of reporting is useful for very thin films (0.13 mm (0.005 in.) and less) for which breaking load may not be proportional to cross-sectional area and whose thickness may be difficult to determine with precision. Furthermore, films which are in effect laminar due to orientation, skin effects, nonuniform crystallinity, etc., have tensile properties disproportionate to cross-sectional area.

11.3 *Tensile Strength* (nominal) shall be calculated by dividing the maximum load by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures.

NOTE 15—When tear failure occurs, so indicate and calculate results based on load and elongation at which tear initiates, as reflected in the load-deformation curve.

11.4 *Tensile Strength at Break* (nominal) shall be calculated in the same way as the tensile strength except that the load at break shall be used in place of the maximum load (Note 15 and Note 16).

NOTE 16—In many cases tensile strength and tensile strength at break are identical.

11.5 *Percent Elongation at Break* shall be calculated by dividing the extension at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation; otherwise the distance between the grips shall be used. The result shall be expressed in percent and reported to two significant figures (Note 15).

11.6 *Yield Strength*, where applicable, shall be calculated by dividing the load at the yield point by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures. Alternatively, for materials that exhibit Hookean behavior in the initial part of the curve, an offset yield strength may be obtained as described in the Appendix of Test Method D 638. In this case the value should be given as "yield strength at —% offset."

11.7 *Percent Elongation at Yield*, where applicable, shall be calculated by dividing the extension at the yield point by the initial gage length of specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation. Before calculating, correct the extension for "toe compensation" as described in Annex A1. The results shall be expressed in percent and reported to two significant figures. When offset yield strength is used, the elongation at the offset yield strength may be calculated.

11.8 *Elastic Modulus* shall be calculated by drawing a tangent to the initial linear portion of the load-extension curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. Before calculating, correct the extension for "toe compensation" as described in Annex A1. For purposes of this determination, the tensile stress shall be calculated by dividing the load by the average original cross section of the test section. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch), and reported to three significant figures.

11.9 *Secant Modulus*, at a designated strain, shall be calculated by dividing the corresponding stress (nominal) by the designated strain. Elastic modulus values are preferable and shall be calculated whenever possible. However, for materials where no proportionality is evident, the secant value shall be calculated. Draw the tangent as directed in A1.3 and Fig. A1.2 of Annex A1, and mark off the designated strain from the yield point where the tangent line goes through zero stress. The stress to be used in the calculation is then determined by dividing the load at the designated strain on the load-extension curve by the original average cross-sectional area of the specimen.

11.10 *Tensile Energy to Break*, where applicable, shall be calculated by integrating the energy per unit volume under the stress-strain curve or by integrating the total energy absorbed and dividing it by the volume of the original gage region of the specimen. As indicated in Annex A2, this may be done directly during the test by an electronic integrator, or subsequently by computation from the area of the plotted curve. The result shall be expressed in energy per unit volume, usually in megajoules per cubic metre (or inch-pounds-force per cubic inch). This value shall be reported to two significant figures.

11.11 For each series of tests, the arithmetic mean of all values obtained shall be calculated to the proper number of significant figures.

11.12 The standard deviation (estimated) shall be calculated as follows and reported to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\bar{X}^2)/(n-1)} \quad (2)$$

where:

s = estimated standard deviation,
X = value of a single observation,
n = number of observations, and
$\bar{X}$ = arithmetic mean of the set of observations.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, and orientation of samples with respect to anisotropy (if any),

12.1.2 Method of preparing test specimens,

12.1.3 Thickness, width, and length of test specimens,

12.1.4 Number of specimens tested,

12.1.5 Strain rate employed,

12.1.6 Grip separation (initial),

12.1.7 Crosshead speed (rate of grip separation),

12.1.8 Gage length (if different from grip separation),

12.1.9 Type of grips used, including facing (if any),

12.1.10 Conditioning procedure (test conditions, temperature, and relative humidity if nonstandard),

12.1.11 Anomalous behavior such as tear failure and failure at a grip,

12.1.12 Average breaking factor and standard deviation,

12.1.13 Average tensile strength (nominal) and standard deviation,

12.1.14 Average tensile strength at break (nominal) and standard deviation,

12.1.15 Average percent elongation at break and standard deviation,

12.1.16 Where applicable, average tensile energy to break and standard deviation,

12.1.17 In the case of materials exhibiting "yield" phenomenon: average yield strength and standard deviation; and average percent elongation at yield and standard deviation,

12.1.18 For materials which do not exhibit a yield point: average —% offset yield strength and standard deviation; and average percent elongation at —% offset yield strength and standard deviation,

12.1.19 Average modulus of elasticity and standard deviation (if secant modulus is used, so indicate and report strain at which calculated), and

12.1.20 When an extensometer is employed, so indicate.

13. Precision and Bias

13.1 Two interlaboratory tests have been run for these tensile properties. The first was run for modulus only, in 1977, in which randomly drawn samples of four thin (~ 0.025 mm (0.001-in.)) materials were tested with five specimens in each laboratory. Elastic (tangent) modulus measurements were made by six laboratories, and secant (1 %) modulus measurements were taken by five laboratories. The relative precision obtained in this interlaboratory study is in Table 2.

13.1.1 In deriving the estimates in Table 2, statistical outliers were not removed, in keeping with Practice E 691.[9]

13.1.2 The within-lab standard deviation of a mean value, $S_{\bar{x}}$, in each case was determined from the standard deviation, $S_{\bar{x}}$, of the five individual specimens as follows: $S_{\bar{x}} = S_x/(5)^{1/2}$. The $S_{\bar{x}}$ values were pooled among laboratories for a given material to obtain the within-lab standard deviation, S_r, of a test result (mean of five specimens). See 13.3-13.3.2 for definitions of terms in the tables.

TABLE 2 Precision Data for Modulus

Material	Thickness, mils	Average, 10^3 psi	S_r, 10^3 psi	S_R, 10^3 psi	I_r, 10^3 psi	I_R, 10^3 psi
Tangent Modulus						
LDPE	1.4	53.9	1.81	8.81	5.12	24.9
HDPE	1.6	191	5.47	16.2	15.5	45.9
PP	1.1	425	10.3	31.5	29.0	89.1
PET	0.9	672	13.8	55.5	39.1	157.1
Secant Modulus						
LDPE	1.4	45.0	2.11	3.43	5.98	9.70
HDPE	1.6	150	3.29	9.58	9.30	27.1
PP	1.1	372	4.66	26.5	13.2	74.9
PET	0.9	640	10.0	27.5	28.4	77.8

13.2 An interlaboratory test was run for all the other tensile properties except modulus in 1981, in which randomly drawn samples of six materials (one of these in three thicknesses) ranging in thickness from 0.019 to 0.178 mm (0.00075 to 0.007 in.) were tested in seven laboratories. A test result was defined as the mean of five specimen determinations. However, each laboratory tested eight specimens, and the $S_{\bar{x}}$ was determined from $S_{\bar{x}} = S_x/(5)^{1/2}$ as above. This was done to improve the quality of the statistics while maintaining their applicability to a five-specimen test result. The materials and their thicknesses are identified in Tables 3-7, each of which contain data for one of the following properties: tensile yield stress, yield elongation, tensile strength, tensile elongation at break, and tensile energy at break (see Note 17).[10]

NOTE 17—Subsequent to filing the research report, examination of the LDPE used in this study between crossed polarizers revealed lengthwise lines representing substantial widthwise variation in molecular orientation that probably was not successfully randomized out of the between-labs component of variance.

NOTE 18—**Caution:** The following explanations of I_r and I_R (13.3-13.3.3) are only intended to present a meaningful way of considering the *Approximate* precision of this test method. The data in Table 2 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.3-13.3.3 would then be valid for such data.

[9] Supporting data are available from ASTM Headquarters. Request RR: D20-1084.

[10] Supporting data are available from ASTM Headquarters. Request RR: D20-1101.

TABLE 3 Precision Data for Yield Stress

Material	Thickness, mils	Average, 10^3 psi	$(S_r)^A$ 10^3 psi	$(S_R)^B$ 10^3 psi	$I(r)^C$ 10^3 psi	$I(R)^D$ 10^3 psi
LDPE	1.0	1.49	0.051	0.13	0.14	0.37
HDPE	1.0	4.33	0.084	0.16	0.24	0.44
PP	0.75	6.40	0.13	0.52	0.37	1.46
PC	4.0	8.59	0.072	0.29	0.20	0.82
CTA	5.3	11.4	0.12	0.50	0.34	1.43
PET	4.0	14.3	0.12	0.23	0.34	0.66
PET	2.5	14.4	0.14	0.54	0.40	1.52
PET	7.0	14.4	0.13	0.36	0.37	1.03

[A] S_r is the within-laboratory standard deviation of the average.
[B] S_R is the between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$.
[D] $I_R = 2.83\ S_R$.

TABLE 4 Precision Data for Yield Elongation

Material	Thickness, mils	Average, %	$(S_r)^A$, %	$(S_R)^B$, %	$I(r)^C$, %	$I(R)^D$, %
PP	0.75	3.5	0.15	0.41	0.42	1.2
PET	2.5	5.2	0.26	0.92	0.74	2.6
PET	4.0	5.3	0.25	0.60	0.71	1.7
PET	7.0	5.4	0.14	1.05	0.40	3.0
CTA	5.3	5.4	0.19	0.99	0.54	2.8
PC	4.0	6.9	0.24	0.98	0.68	2.8
HDPE	1.0	8.8	0.32	1.82	0.91	5.2
LDPE	1.0	10.0	0.55	3.41	1.56	9.6

NOTE 1—See Table 3 for footnote explanation.

TABLE 5 Precision Data for Tensile Strength

Material	Thickness, mils	Average, 10^3 psi	$(S_r)^A$ 10^3 psi	$(S_R)^B$ 10^3 psi	$I(r)^C$ 10^3 psi	$I(R)^D$ 10^3 psi
LDPE	1.0	3.42	0.14	0.53	0.40	1.5
HDPE	1.0	6.87	0.27	0.81	0.76	2.3
PC	4.0	12.0	0.34	0.93	0.96	2.6
CTA	5.3	14.6	0.20	1.37	0.57	3.9
PP	0.75	28.4	1.57	4.56	4.4	12.9
PET	4.0	28.9	0.65	1.27	1.8	3.6
PET	7.0	30.3	0.83	1.32	2.3	3.7
PET	2.5	30.6	1.22	2.64	3.4	7.5

NOTE 1—See Table 3 for footnote explanation.

TABLE 6 Precision Data for Elongation at Break

Material	Thickness, mils	Average, %	$(S_r)^A$, %	$(S_R)^B$, %	$I(r)^C$, %	$I(R)^D$, %
CTA	5.3	26.4	1.0	4.3	3	12
PP	0.75	57.8	4.4	12.7	12	36
PET	2.5	120	8.0	14.6	23	41
PET	7.0	132	5.8	10.6	16	30
PET	4.0	134	4.4	12.2	12	35
PC	4.0	155	5.4	17.1	15	48
LDPE	1.0	205	24.4	73.3	69	210
HDPE	1.0	570	26.0	91.7	74	260

NOTE 1—See Table 3 for footnote explanation.

13.3 For the purpose of compiling summary statistics, a test result has been defined to be the average of five replicate measurements of a property for a material in a laboratory, as specified in this test method. Summary statistics are given in Table 3. In each table, for the material indicated, *S(r)* is the pooled within-laboratory standard deviation of a test result, *S(R)* is the between-laboratory standard deviation of a test result, where *r* equals 2.83 × *S(r)* (see 13.3.1) and *R* equals 2.83 × *S(R)* (see 13.3.2).

13.3.1 *Repeatability*, I_r (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the I_r value for that material.

13.3.2 *Reproducibility*—In comparing two mean values for the same material obtained by different operators using different equipment on different days, either in the same

TABLE 7 Precision Data for Tensile Energy to Break

Material	Thickness, mils	Average, 10^3 in./lb/in.3	$(S_r)^A 10^3$ in./lb/in.3	$(S_R)^B 10^3$ in./lb/in.3	$I(r)^C 10^3$ in./lb/in.3	$I(R)^D 10^3$ in./lb/in.3
CTA	5.0	3.14	0.14	0.70	0.4	2.0
LDPE	1.0	5.55	0.84	2.47	2.4	7.0
PP	0.75	11.3	1.19	3.11	3.4	8.8
PC	4.0	12.9	0.59	1.55	1.7	4.4
HDPE	1.0	26.0	1.87	5.02	5.3	14.2
PET	2.5	26.1	2.13	4.20	6.0	11.9
PET	4.0	27.1	1.42	2.75	4.0	7.8
PET	7.0	28.4	1.71	2.72	4.8	7.7

NOTE 1—See Table 3 for footnote explanation.

laboratory or in different laboratories, the means should be judged not equivalent if they differ by more than the *R* value for that material.

13.3.3 Any judgment made in accordance with 13.3.1 and 13.3.2 would have an approximate 95 % (0.95) probability of being correct.

13.3.4 For further information, see Practice E 691.

13.4 *Bias*—The systematic error which contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for these test methods.

14. Keywords

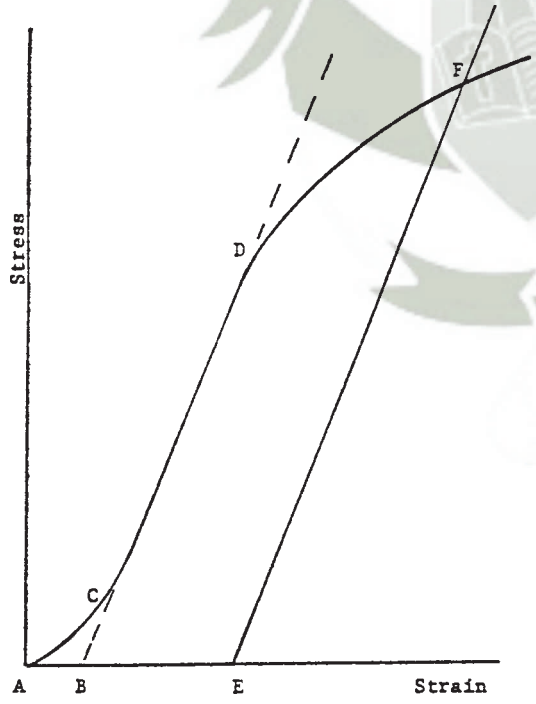
14.1 modulus of elasticity; plastic film; plastic sheeting; tensile properties; tensile strength; toughness; yield stress

ANNEXES

(Mandatory Information)

A1. TOE COMPENSATION

A1.1 In a typical stress-strain curve (Fig. A1.1) there is a toe region, *AC*, which does not represent a property of the material. It is an artifact caused by a takeup of slack, and alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus, strain, and offset yield point, this artifact must be compensated for to give the corrected zero point on the strain or extension axis.

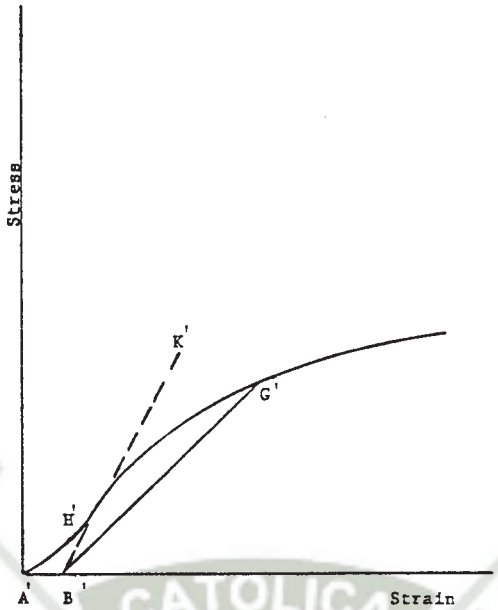


NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.1 Material with Hookean Region

A1.2 In the case of a material exhibiting a region of Hookean (linear) behavior (Fig. A1.1), a continuation of the linear (*CD*) region of the curve is constructed through the zero-stress axis. This intersection (*B*) is the corrected zero-strain point from which all extensions or strains must be measured, including the yield offset (*BE*), if applicable. The elastic modulus can be determined by dividing the stress at any point along the line *CD* (or its extension) by the strain at the same point (measured from point *B*, defined as zero-strain).

A1.3 In the case of a material that does not exhibit any linear region (Fig. A1.2), the same kind of toe correction of the zero-strain point can be made by constructing a tangent to the maximum slope at the inflection point (*H′*). This is extended to intersect the strain axis at point *B′*, the corrected zero-strain point. Using point *B′* as zero strain, the stress at any point (*G′*) on the curve can be divided by the strain at that point to obtain a secant modulus (slope of line *B′ G′*). For those materials with no linear region, any attempt to use the tangent through the inflection point as a basis for determination of an offset yield point may result in unacceptable error.



NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

A2. DETERMINATION OF TENSILE ENERGY TO BREAK

A2.1 Tensile energy to break (TEB) is defined by the area under the stress-strain curve, or

$$\text{TEB} = \int_0^{\epsilon_T} S \, d\epsilon \tag{A2.1}$$

where S is the stress at any strain, ϵ, and ϵ_T is the strain at rupture. The value is in units of energy per unit volume of the specimen's initial gage region. TEB is most conveniently and accurately measured with a tension tester equipped with an integrator. The calculation is then:

$$TEB = (I/K)\frac{\text{(full scale load) (chart speed) (crosshead speed/chart speed)}}{\text{(mean caliper) (specimen width) (gage length)}} \tag{A2.2}$$

where I is the integrator count reading and K is the maximum possible count per unit time for a constant full scale load. This whole calculation is typically done electronically. The results are best expressed in megajoules per cubic metre (or inch-pounds-force per cubic inch).

A2.2 Without an integrator, the area under the recorded stress-strain curve can be measured by planimeter, counting squares, or weighing the cut-out curve. These techniques are time-consuming and likely to be less accurate, since the load scale on some chart paper is not in round-number dimensions. Moreover, if the curve coordinates are in terms of force and extension instead of stress and strain, the calculated energy, corresponding to the measured area, must be divided by the product of gage length, specimen width, and mean caliper:

$$\text{TEB} = \frac{\text{(curve area) (force per unit chart scale) (extension per unit chart travel)}}{\text{(mean caliper) (specimen width) (gage length)}} \tag{A2.3}$$

A2.3 For example, if the area under a force-extension curve is 60 000 mm^2, the load coordinate is 2.0 N/mm of chart scale, the extension coordinate is 0.25 mm of extension per mm of chart travel, and the specimen dimensions are 0.1 mm caliper, 15 mm width and 100 mm gage length, then the calculation for tensile energy to break is:

$$\text{TEB} = \frac{(60\ 000\ \text{mm}^2)\ (2.0\ \text{N/mm})\ (0.25 \times 10^{-3}\ \text{m/mm})}{(0.1 \times 10^{-3}\ \text{m})\ (15 \times 10^{-3}\ \text{m})\ (100 \times 10^{-3}\ \text{m})} \tag{A2.4}$$

$$\text{TEB} = 200\ \text{MJ/m}^3$$

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 882 – 02:
(*1*) Revised 7.1 and 7.2.
D 882 – 01:
(*1*) Section 5.5 rewritten.
(*2*) Note 6 deleted.
(*3*) Added Practice D 6287 to Referenced Documents section.
D 882 – 00:
(*1*) Added 11.1.

D 882 – 97:
(*1*) Note 3 rewritten and moved.
(*2*) ISO equivalency statement changed in Note 4.
(*3*) ISO reference changed in 2.2.
(*4*) Apparatus section (Section 5) rewritten.
(*5*) Table 1 deleted. Table 2 renumbered as Table 1.
(*6*) Sections 10.3 and 10.4 rewritten.
(*7*) Note 13 deleted. New Note 12 added.

Designation: D7264/D7264M – 07

Standard Test Method for Flexural Properties of Polymer Matrix Composite Materials[1]

This standard is issued under the fixed designation D7264/D7264M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method determines the flexural stiffness and strength properties of polymer matrix composites.

1.1.1 *Procedure A*—A three-point loading system utilizing center loading on a simply supported beam.

1.1.2 *Procedure B*—A four-point loading system utilizing two load points equally spaced from their adjacent support points, with a distance between load points of one-half of the support span.

NOTE 1—Unlike Test Method D6272, which allows loading at both one-third and one-half of the support span, in order to standardize geometry and simplify calculations this standard permits loading at only one-half the support span.

1.2 For comparison purposes, tests may be conducted according to either test procedure, provided that the same procedure is used for all tests, since the two procedures generally give slightly different property values.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D2344/D2344M Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates

D3878 Terminology for Composite Materials

D5229/D5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials

D5687/D5687M Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation

D6272 Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending

D6856 Guide for Testing Fabric-Reinforced "Textile" Composite Materials

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E18 Test Methods for Rockwell Hardness of Metallic Materials

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases

E1434 Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases

2.2 *Other Documents:*

ANSI Y14.5-1999 Dimensioning and Tolerancing—Includes Inch and Metric[3]

ANSI B46.1-1995 Surface Texture (Surface Roughness, Waviness and Lay)[3]

3. Terminology

3.1 *Definitions*—Terminology D3878 defines the terms relating to high-modulus fibers and their composites. Terminology E6 defines terms relating to mechanical testing. Terminology E456 and Practice E177 define terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other documents.

[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

Current edition approved April 1, 2007. Published April 2007. Originally approved in 2006. Last previous edition approved in 2006 as D7264/D7264M – 06. DOI: 10.1520/D7264_D7264M-07.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *flexural strength*, *n*—the maximum stress at the outer surface of a flexure test specimen corresponding to the peak applied force prior to flexural failure.

3.2.2 *flexural modulus*, *n*—the ratio of stress range to corresponding strain range for a test specimen loaded in flexure.

3.3 *Symbols*:

b = specimen width
CV = sample coefficient of variation, in percent
E_f^{chord} = flexural chord modulus of elasticity
E_f^{secant} = flexural secant modulus of elasticity
h = specimen thickness
L = support span
m = slope of the secant of the load-deflection curve
n = number of specimens
P = applied force
s_{n-1} = sample standard deviation
x_i = measured or derived property
$\bar{x}$ = sample mean
δ = mid-span deflection of the specimen
ε = strain at the outer surface at mid-span of the specimen
σ = stress at the outer surface at mid-span of the specimen

4. Summary of Test Method

4.1 A bar of rectangular cross section, supported as a beam, is deflected at a constant rate as follows:

4.1.1 *Procedure A*—The bar rests on two supports and is loaded by means of a loading nose midway between the supports (see Fig. 1).

4.1.2 *Procedure B*—The bar rests on two supports and is loaded at two points (by means of two loading noses), each an equal distance from the adjacent support point. The distance between the loading noses (that is, the load span) is one-half of the support span (see Fig. 2).

4.2 Force applied to the specimen and resulting specimen deflection at the center of span are measured and recorded until the failure occurs on either one of the outer surfaces, or the deformation reaches some pre-determined value.

4.3 The major difference between four-point and three-point loading configurations is the location of maximum bending moment and maximum flexural stress. With the four-point configuration the bending moment is constant between the central force application members. Consequently, the maximum flexural stress is uniform between the central force application members. In the three-point configuration, the maximum flexural stress is located directly under the center

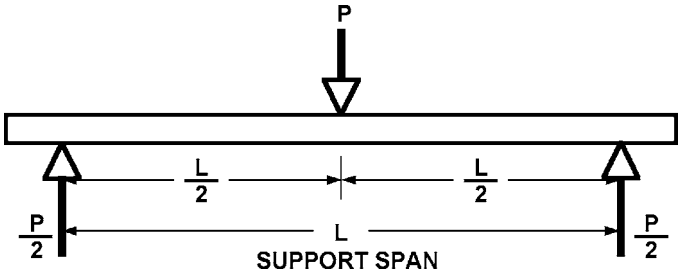


FIG. 1 Procedure A—Loading Diagram

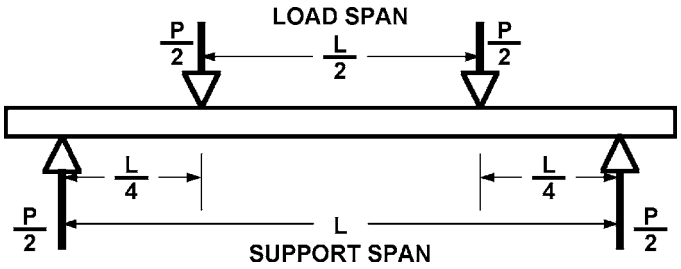


FIG. 2 Procedure B—Loading Diagram

force application member. Another difference between the three-point and four-point configurations is the presence of resultant vertical shear force in the three-point configuration everywhere in the beam except right under the mid-point force application member whereas in the four-point configuration, the area between the central force application members has no resultant vertical shear force. The distance between the outer support members is the same as in the equivalent three-point configuration.

4.4 The test geometry is chosen to limit out-of-plane shear deformations and avoid the type of short beam failure modes that are interrogated in Test Method D2344/D2344M.

5. Significance and Use

5.1 This test method determines the flexural properties (including strength, stiffness, and load/deflection behavior) of polymer matrix composite materials under the conditions defined. Procedure A is used for three-point loading and Procedure B is used for four-point loading. This test method was developed for optimum use with continuous-fiber-reinforced polymer matrix composites and differs in several respects from other flexure methods, including the use of a standard span-to-thickness ratio of 32:1 versus the 16:1 ratio used by Test Methods D790 (a plastics-focused method covering three-point flexure) and D6272 (a plastics-focused method covering four-point flexure).

5.2 This test method is intended to interrogate long-beam strength in contrast to the short-beam strength evaluated by Test Method D2344/D2344M.

5.3 Flexural properties determined by these procedures can be used for quality control and specification purposes, and may find design applications.

5.4 These procedures can be useful in the evaluation of multiple environmental conditions to determine which are design drivers and may require further testing.

5.5 These procedures may also be used to determine flexural properties of structures.

6. Interferences

6.1 Flexural properties may vary depending on which surface of the specimen is in compression, as no laminate is perfectly symmetric (even when full symmetry is intended); such differences will shift the neutral axis and will be further affected by even modest asymmetry in the laminate. Flexural properties may also vary with specimen thickness, conditioning and/or testing environments, and rate of straining. When evaluating several datasets these parameters should be equivalent for all data in the comparison.

6.2 For multidirectional laminates with a small or moderate number of laminae, flexural modulus and flexural strength may be affected by the ply-stacking sequence and will not necessarily correlate with extensional modulus, which is not stacking-sequence dependent.

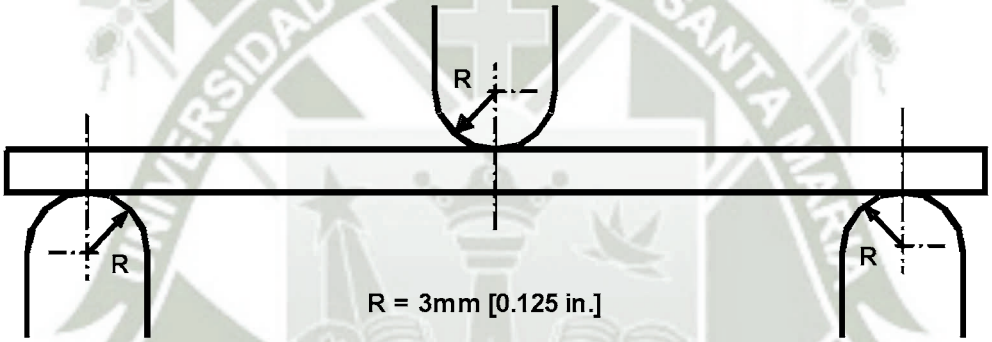
6.3 The calculation of the flexural properties in Section 13 of this standard is based on beam theory, while the specimens in general may be described as plates. The differences may in some cases be significant, particularly for laminates containing a large number of plies in the ±45° direction. The deviations from beam theory decrease with decreasing width.

6.4 Loading noses may be fixed, rotatable or rolling. Typically, for testing composites, fixed or rotatable loading noses are used. The type of loading nose can affect results, since non-rolling paired supports on either the tension or compression side of the specimen introduce slight longitudinal forces and resisting moments on the beam, which superpose with the intended loading. The type of supports used is to be reported as described in Section 14. The loading noses should also uniformly contact the specimen across its width. Lack of uniform contact can affect flexural properties by initiating damage by crushing and by non-uniformly loading the beam. Formulas used in this standard assume a uniform line loading at the specimen supports across the entire specimen width; deviations from this type of loading is beyond the scope of this standard.

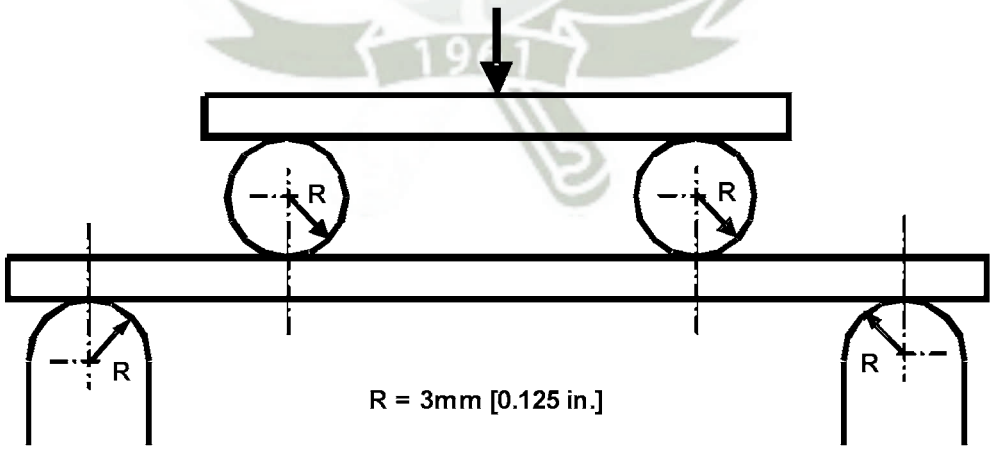
7. Apparatus

7.1 *Testing Machine*—Properly calibrated, which can be operated at a constant rate of crosshead motion, and in which the error in the force application system shall not exceed ±1 % of the full scale. The force indicating mechanism shall be essentially free of inertia lag at the crosshead rate used. Inertia lag shall not exceed 1 % of the measured force. The accuracy of the testing machine shall be verified in accordance with Practices E4.

7.2 *Loading Noses and Supports*—The loading noses and supports shall have cylindrical contact surfaces of radius 3.00 mm [0.125 in.] as shown in Fig. 3, with a hardness of 60 to 62 HRC, as specified in Test Methods E18, and shall have finely



Three-Point Loading Configuration with Fixed Supports and Loading Nose



Four-Point Loading Configuration with Fixed Supports and Rolling Loading Noses

FIG. 3 Example Loading Nose and Supports for Procedures A (top) and B (bottom)

ground surfaces free of indentation and burrs with all sharp edges relieved. Loading noses and supports may be arranged in a fixed, rotatable or rolling arrangement. Typically, with composites, rotatable or fixed arrangements are used.

7.3 *Micrometers*—For width and thickness measurements the micrometers shall use a 4 to 7 mm [0.16 to 0.28 in.] nominal diameter ball-interface on an irregular surface such as the bag side of a laminate, and a flat anvil interface on machined edges or very smooth tooled surfaces. A micrometer or caliper with flat anvil faces shall be used to measure the length of the specimen. The accuracy of the instrument(s) shall be suitable for reading to within 1 % or better of the specimen dimensions. For typical section geometries, an instrument with an accuracy of ±0.02 mm [±0.001 in.] is desirable for thickness and width measurement, while an instrument with an accuracy of ±0.1 mm [±0.004 in.] is adequate for length measurement.

7.4 *Deflection Measurement*—Specimen deflection at the common center of the loading span shall be measured by a properly calibrated device having an accuracy of ±1 % or better of the expected maximum displacement. The device shall automatically and continuously record the deflection during the test.

7.5 *Conditioning Chamber*—When conditioning materials at non-laboratory environments, a temperature/vapor-level controlled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to within ±1°C [±2°F] and the required vapor level to within ±3 % relative humidity, as outlined in Test Method D5229/D5229M. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

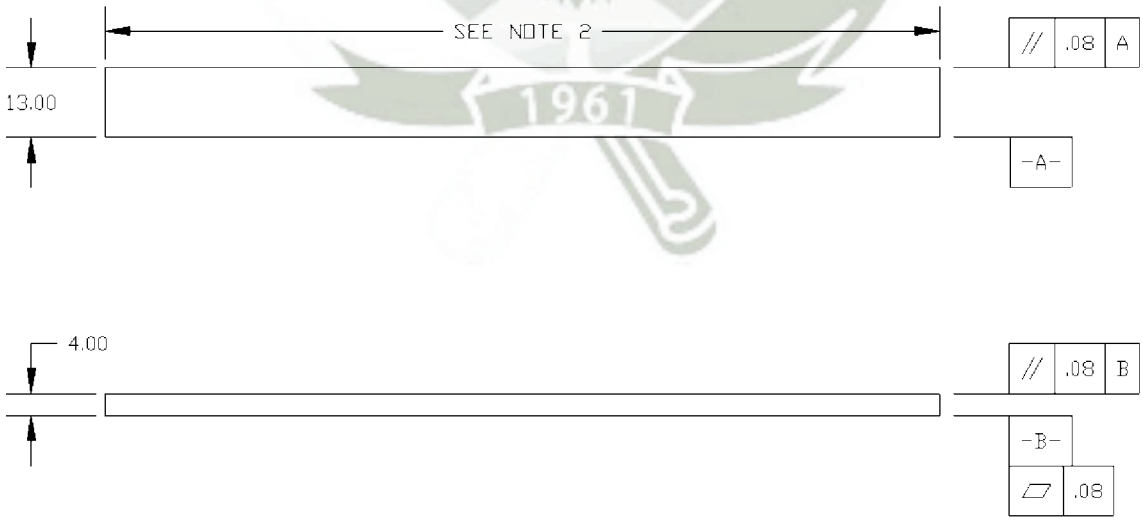
7.6 *Environmental Test Chamber*—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the test specimen at the required temperature within ±3°C [±5°F] and the required vapor level to within ±5 % relative humidity.

8. Test Specimens

8.1 *Specimen Preparation*—Guide D5687/D5687M provides recommended specimen preparation practices and should be followed when practical.

8.2 *Specimen Size* is chosen such that the flexural properties are determined accurately from the tests. For flexural strength, the standard support span-to-thickness ratio is chosen such that failure occurs at the outer surface of the specimens, due only to the bending moment (see Notes 2 and 3). The standard span-to-thickness ratio is 32:1, the standard specimen thickness is 4 mm [0.16 in.], and the standard specimen width is 13 mm [0.5 in.] with the specimen length being about 20 % longer than the support span. See Figs. 4 and 5 for a drawing of the standard test specimen in SI and inch-pound units, respectively. For fabric-reinforced textile composite materials, the width of the specimen shall be at least two unit cells, as defined in Guide D6856. If the standard specimen thickness cannot be obtained in a given material system, an alternate specimen thickness shall be used while maintaining the support span-to-thickness ratio [32:1] and specimen width. Optional support span-to-thickness ratios of 16:1, 20:1, 40:1, and 60:1 may also be used provided it is so noted in the report. Also, the data obtained from a test using one support span-to-thickness ratio may not be compared with the data from another test using a different support span-to-thickness ratio.

8.2.1 Shear deformations can significantly reduce the apparent modulus of highly orthotropic laminates when they are tested at low support span-to-thickness ratios. For this reason, a high support span-to-thickness ratio is recommended for flexural modulus determinations. In some cases, separate sets of specimens may have to be used for modulus and strength determination.



NOTE 1—Drawing interpretation per ANSI Y14.5-1999 and ANSI B46.1-1995.
NOTE 2—See 8.2 and 11.3 of this test standard for the required values of span and overall length.

FIG. 4 Standard Flexural Test Specimen Drawing (SI)

NOTE 1—Drawing interpretation per ANSI Y14.5-1999 and ANSI B46.1-1995.
NOTE 2—See 8.2 and 11.3 of this test standard for the required values of span and overall length.

FIG. 5 Standard Flexural Test Specimen Drawing (Inch-Pound)

NOTE 2—A support span-to-thickness ratio of less than 32:1 may be acceptable for obtaining the desired flexural failure mode when the ratio of the lower of the compressive and tensile strength to out-of-plane shear strength is less than 8, but the support span-to-thickness ratio must be increased for composite laminates having relatively low out-of-plane shear strength and relatively high in-plane tensile or compressive strength parallel to the support span.

NOTE 3—While laminate stacking sequence is not limited by this test method, significant deviations from a lay-up of nominal balance and symmetry may induce unusual test behaviors and a shift in the neutral axis.

9. Number of Test Specimens

9.1 Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. For statistically significant data the procedures outlined in Practice E122 should be consulted. Report the method of sampling.

10. Conditioning

10.1 The recommended pre-test specimen condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D5229/D5229M; however, if the test requester does not explicitly specify a pre-test conditioning environment, conditioning is not required and the test specimens may be tested as prepared.

NOTE 4—The term *moisture*, as used in Test Method D5229/D5229M, includes not only the vapor of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

10.2 The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with the data.

10.3 If there is no explicit conditioning process, the conditioning process shall be reported as "unconditioned" and the moisture content as "unknown."

11. Procedure

11.1 Condition the specimens as required. Store the specimens in the conditioned environment until test time.

11.2 Following final specimen machining and any conditioning but before testing, measure and record the specimen width and thickness at the specimen mid–section, and the specimen length, to the specified accuracy.

11.3 Measure the span accurately to the nearest 0.1 mm [0.004 in.] for spans less than 63 mm [2.5 in.] and the nearest 0.3 mm [0.012 in.] for spans greater than or equal to 63 mm [2.5 in.]. Use the measured span for all calculations. See Annex A1 for information on the determination of and setting of the span.

11.4 *Speed of Testing*—Set the speed of testing at a rate of crosshead movement of 1.0 mm/min [0.05 in./min] for a specimen with standard dimensions. For specimens with dimensions that vary greatly from the standard dimensions, a crosshead rate that will give a similar rate of straining at the outer surface can be obtained via the method outlined in Test Methods D790 for Procedure A and Test Method D6272 for Procedure B.

11.5 Align the loading nose(s) and supports so that the axes of the cylindrical surfaces are parallel. For Procedure A, the loading nose shall be midway between the supports. For Procedure B, the load span shall be one-half of the support span and symmetrically placed between the supports. The parallelism may be checked by means of plates with parallel grooves into which the loading nose(s) and supports will fit when properly aligned. Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading noses and supports. See Annex A1 for setting and measuring span.

11.6 Apply the force to the specimen at the specified crosshead rate. Measure and record force-deflection data at a

rate such that a minimum of 50 data points comprise the force deflection curve. (A higher sampling rate may be required to properly capture any nonlinearities or progressive failure of the specimen.) Measure deflection by a transducer under the specimen in contact with it at the center of the support span, the transducer being mounted stationary relative to the specimen supports. Do not use the measurement of the motion of the loading nose relative to the supports as this will not take into account the rotation of the specimen about the load and support noses, nor account for the compliance in the loading nose or crosshead.

11.7 *Failure Modes*—To obtain valid flexural strength, it is necessary that the specimen failure occurs on either one of its outer surfaces, without a preceding interlaminar shear failure or a crushing failure under a support or loading nose. Failure on the tension surface may be a crack while that on the compression surface may be local buckling. Buckling may be manifested as fiber micro-buckling or ply-level buckling. Ply-level buckling may result in, or be preceded by delamination of the outer ply.

11.7.1 *Failure Identification Codes*—Record the mode, area, and location of failure for each specimen. Choose a standard failure identification code based on the three-part code shown in Fig. 6. A multimode failure can be described by including each of the appropriate failure-mode codes between the parentheses of the M failure mode.

12. Validation

12.1 Values for properties at failure shall not be calculated for any specimen that breaks at some obvious, fortuitous flaw, unless such flaws constitute a variable being studied. Specimens that fail in an unacceptable failure mode shall not be included in the flexural property calculations. Retests shall be made for any specimen for which values are not calculated. If a significant fraction (>50 %) of the specimens fail in an unacceptable failure mode then the span-to-thickness ratio (for excessive shear failures) or the loading nose diameter (crushing under the loading nose) should be reexamined.

13. Calculation

NOTE 5—In determination of the calculated value of some of the properties listed in this section it is necessary to determine if the toe compensation (see Annex A2) adjustment must be made. This toe compensation correction shall be made only when it has been shown that the toe region of the curve is due to take up of the slack, alignment, or seating of the specimen and is not an authentic material response.

13.1 *Maximum Flexural Stress, Procedure A*—When a beam of homogenous, elastic material is tested in flexure as a beam simply supported at two points and loaded at the midpoint, the maximum stress at the outer surface occurs at mid-span. The stress may be calculated for any point on the load-deflection curve by the following equation (Note 6):

$$\sigma = \frac{3PL}{2bh^2} \quad (1)$$

where:

σ = stress at the outer surface at mid-span, MPa [psi],
P = applied force, N [lbf],
L = support span, mm [in.],
b = width of beam, mm [in.], and
h = thickness of beam, mm [in.].

NOTE 6—Eq 1 applies strictly to materials for which the stress is linearly proportional to strain up to the point of rupture and for which the strains are small. Since this is not always the case, a slight error will be introduced in the use of this equation. The equation will however, be valid for comparison data and specification values up to the maximum fiber strain of 2 % for specimens tested by the procedure herein described. It should be noted that the maximum ply stress may not occur at the outer surface of a multidirectional laminate.[4] Laminated beam theory must be applied to determine the maximum tensile stress at failure. Thus, Eq 1 yields an apparent strength based on homogeneous beam theory. This apparent strength is highly dependent on the ply-stacking sequence for multidirectional laminates.

13.2 *Maximum Flexural Stress, Procedure B*—When a beam of homogeneous, elastic material is tested in flexure as a beam simply supported at two outer points and loaded at two central points separated by a distance equal to ½ the support span and at equal distance from the adjacent support point, the maximum stress at the outer surface occurs between the two central loading points that define the load span (Fig. 2). The stress may be calculated for any point on the load-deflection curve by the following equation (Note 7):

$$\sigma = \frac{3PL}{4bh^2} \quad (2)$$

where:

σ = stress at the outer surface in the load span region, MPa [psi],
P = applied force, N [lbf],
L = support span, mm [in.],
b = width of beam, mm [in.], and

[4] For the theoretical details, see Whitney, J. M., Browning, C. E., and Mair, A., "Analysis of the Flexure Test for Laminated Composite Materials," *Composite Materials: Testing and Design (Third Conference), ASTM STP 546* , 1974, pp. 30-45.

First Character

Failure Mode	Code
Tension	T
Compression	C
Buckling	B
interlaminar Shear	S
Multi-mode	M(xyz)
Other	O

Second Character

Failure Area	Code
At loading nose	A
Between loading noses	B
at Support nose	S
between Load and support nose	L
Unknown	U

Third Character

Failure Location	Code
Top	T
Bottom	B
Left	L
Right	R
Middle	M
Various	V
Unknown	U

FIG. 6 Flexure Test Specimen Three-Part Failure Identification Code

h = thickness of beam, mm [in.].

NOTE 7—The limitations defined for Eq 1 in Note 6 apply also to Eq 2.

13.3 *Flexural Strength*—The flexural strength is equal to the maximum stress at the outer surface corresponding to the peak applied force prior to failure. (for multidirectional laminates, see Note 6). It is calculated in accordance with Eq 1 and 2 by letting P equal the peak applied force.

13.4 *Flexural Stress at a Given Strain*—The maximum flexural stress at any given strain may be calculated in accordance with Eq 1 and 2 by letting P equal the applied force read from the force-deflection curve at the deflection corresponding to the desired strain (for multidirectional laminates, see Note 6). Equations for calculating strains from the measured deflection are given in 13.5 and 13.6.

13.5 *Maximum Strain, Procedure A*—The maximum strain at the outer surface also occurs at mid-span, and it may be calculated as follows:

$$\varepsilon = \frac{6\delta h}{L^2} \tag{3}$$

where:
ε = maximum strain at the outer surface, mm/mm [in./in.],
δ = mid-span deflection, mm [in.],
L = support span, mm [in.], and
h = thickness of beam, mm [in.].

13.6 *Maximum Strain, Procedure B*—The maximum strain at the outer surface also occurs at mid-span, and it may be calculated as follows:

$$\varepsilon = \frac{4.36\delta h}{L^2} \tag{4}$$

where:
δ = mid-span deflection, mm [in.],
ε = maximum strain at the outer surface, mm/mm [in./in.],
L = support span, mm [in.], and
h = thickness of beam, mm [in.].

13.7 *Flexural Modulus of Elasticity*:

13.7.1 *Flexural Chord Modulus of Elasticity*—The flexural chord modulus of elasticity is the ratio of stress range and corresponding strain range. For calculation of flexural chord modulus, the recommended strain range is 0.002 with a start point of 0.001 and an end point 0.003. If the data is not available at the exact strain range end points (as often occurs with digital data), use the closest available data point. Calculate the flexural chord modulus of elasticity from the stress-strain data using Eq 5 (for multidirectional or highly orthotropic composites, see Note 8).

$$E_f^{chord} = \frac{\Delta\sigma}{\Delta\varepsilon} \tag{5}$$

where:
E_f^{chord} = flexural chord modulus of elasticity, MPa [psi],
$\Delta\sigma$ = difference in flexural stress between the two selected strain points, MPa [psi], and
$\Delta\varepsilon$ = difference between the two selected strain points (nominally 0.002).

13.7.1.1 Report the chord modulus of elasticity in MPa [psi] for the strain range 0.001 to 0.003. If a different strain range is used in the calculations, also report the strain range used.

NOTE 8—Shear deformation can seriously reduce the apparent flexural modulus of highly orthotropic laminates when they are tested at low span-to-thickness ratios.[5] For this reason, a high span-to-thickness ratio is recommended for flexural modulus determinations. In some cases, separate sets of specimens may have to be used for modulus and strength determination.

13.7.2 *Flexural Secant Modulus of Elasticity*—The flexural secant modulus of elasticity is the ratio of stress to corresponding strain at any given point on the stress-strain curve. The flexural secant modulus is same as the flexural chord modulus in which the initial strain point is zero. It shall be expressed in MPa [psi]. It is calculated as follows (for multidirectional or highly orthotropic composites, see Note 8):

13.7.2.1 *For Procedure A*:

$$E_f^{secant} = \frac{L^3 m}{4bh^3} \tag{6}$$

where:
E_f^{secant} = flexural secant modulus of elasticity, MPa [psi],
L = support span, mm [in.],
b = width of beam, mm [in.],
h = thickness of beam, mm [in.] and
m = slope of the secant of the force-deflection curve.

13.7.2.2 *For Procedure B*:

$$E_f^{secant} = \frac{0.17L^3 m}{bh^3} \tag{7}$$

where E_f^{secant}, m, L, b, and h are the same as for Eq 6.

13.7.3 Chord modulus of elasticity shall be reported although other definitions of moduli may also be used. However, when other definitions of moduli are used, it should be clearly indicated in the report.

13.8 *Statistics*—For each series of tests calculate the average value, standard deviation, and coefficient of variation for each property determined:

$$\bar{x} = \frac{1}{n}\left(\sum_{i=1}^{n} x_i\right) \tag{8}$$

$$s_{n-1} = \sqrt{\frac{\left(\sum_{i=1}^{n} x_i^2 - n\bar{x}^2\right)}{n-1}}$$

$$CV = 100 \cdot \frac{s_{n-1}}{\bar{x}}$$

where:
$\bar{x}$ = average value or sample mean,
x_i = value of single measured or derived property,
n = number of specimens,
s_{n-1} = estimated standard deviation,
CV = coefficient of variation in percentage.

[5] For discussion of these effects, see Zweben C., Smith, W. S., and Wardle, M. W., "Test Methods for Fiber Tensile Strength, Composite Flexural Modulus, and Properties of Fabric-Reinforced Laminates," *Composite Materials: Testing and Design (Fifth Conference), ASTM STP 674*, 1979, pp. 228-262.

14. Report

14.1 The information reported for this test method includes material identification and mechanical testing data. These data shall be reported in accordance with Guides E1309 and E1434. At a minimum, the following should be reported:

14.1.1 The revision level or date of issue of the test method used.

14.1.2 The date(s) and location(s) of the testing.

14.1.3 The name(s) of the test operator(s).

14.1.4 The test Procedure used (A or B).

14.1.5 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.

14.1.6 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch number, source (if not from the manufacturer), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, prepreg matrix content, and prepreg volatiles content.

14.1.7 Description of the fabrication steps used to prepare the laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used.

14.1.8 Ply orientation stacking sequence of the laminate.

14.1.9 If requested, report density, reinforcement volume fraction, and void content test methods, specimen sampling method and geometries, test parameters, and test data.

14.1.10 Average ply thickness of the material.

14.1.11 Results of any nondestructive evaluation tests.

14.1.12 Method of preparing the test specimens, including specimen labeling scheme and method, specimen geometry, sampling method, and specimen cutting method.

14.1.13 Calibration dates and methods for all measurement and test equipment.

14.1.14 Type of test machine, grips, jaws, alignment data, and data acquisition sampling rate and equipment type.

14.1.15 Dimensions of each specimen to at least three significant figures, including specimen width, thickness, and overall length.

14.1.16 Conditioning parameters and results, and the procedure used if other than that specified in this test method.

14.1.17 Relative humidity and temperature of the testing laboratory.

14.1.18 Environment of the test machine environmental chamber (if used) and soak time at environment.

14.1.19 Number of specimens tested.

14.1.20 Load-span length, support-span length, and support span-to-thickness ratio.

14.1.21 Loading and support nose type and dimensions.

14.1.22 Speed of testing.

14.1.23 Transducer placement on the specimen, transducer type, and calibration data for each transducer used.

14.1.24 Force-deflection curves for each specimen. Note method and offset value if toe compensation was applied to force-deflection curve.

14.1.25 Tabulated data of flexural stress versus strain for each specimen.

14.1.26 Individual flexural strengths and average value, standard deviation, and coefficient of variation (in percent) for the population. Note if the failure load was less than the maximum load prior to failure.

14.1.27 Individual strains at failure and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.28 Strain range used for the flexural chord modulus of elasticity determination.

14.1.29 Individual values of flexural chord modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.30 If an alternate definition of flexural modulus of elasticity is used in addition to chord modulus, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

14.1.31 Individual values of the alternate (see above) flexural modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.32 Individual maximum flexural stresses, and the average, standard deviation, and coefficient of variation (in percent) values for the population. Note any test in which the failure load was less than the maximum load before failure.

14.1.33 For flexural modulus only tests: maximum load applied, strain at maximum applied load, and calculated flexural modulus of elasticity (E_f).

14.1.34 Individual maximum flexural strains and the average, standard deviation, and coefficient of variation (in percent) values for the population. Note any test that was truncated to 2 % strain.

14.1.35 Failure mode and location of failure for each specimen.

15. Precision and Bias

15.1 *Precision*—The data required for the development of precision is not currently available for this test method.

15.2 *Bias*—Bias cannot be determined for this test method as no acceptable reference standard exists.

16. Keywords

16.1 fiber-reinforced composites; flexural properties; stiffness; strength

ANNEXES

(Mandatory Information)

A1. MEASURING AND SETTING SPAN

A1.1 For flexural fixtures that have adjustable spans, it is important that the span between the supports is maintained constant or the actual measured span is used in the calculation of flexural stress, flexural modulus and strain, and the loading noses are positioned and aligned properly with respect to the supports. Some simple steps as follows can improve the repeatability of results when using adjustable span fixtures.

A1.2 *Measurement of Span*:

A1.2.1 This technique is needed to ensure that the correct span, not an estimated span, is used in calculation of results.

A1.2.2 Scribe a permanent line or mark at the exact center of the support where the specimen makes complete contact. The type of mark depends on whether the supports are fixed or rotatable (see Figs. A1.1 and A1.2).

A1.2.3 Using a vernier caliper with pointed tips that is readable to at least 0.1 mm [0.004 in.], measure the distance between the supports, and use this measurement of span in the calculations.

A1.3 *Setting the Span and Alignment of Loading Nose(s)*—To ensure a constant day-to-day setup of the span and ensure the alignment and proper positioning of the loading nose(s), simple jigs should be manufactured for each of the standard setups used. An example of a jig found to be useful is shown in Fig. A1.3.

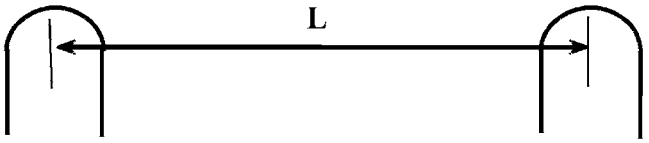


FIG. A1.1 Markings on Fixed Specimen Supports

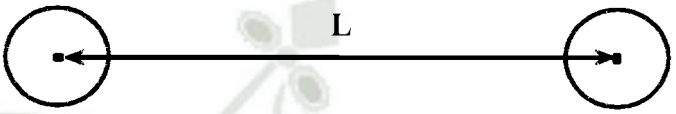


FIG. A1.2 Markings on Rotatable Specimen Supports

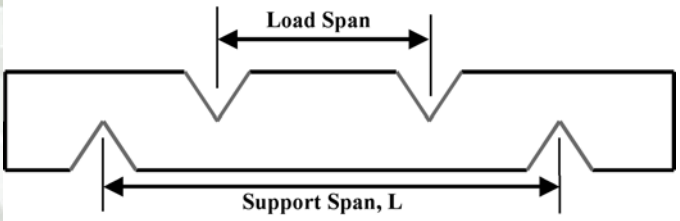


FIG. A1.3 Fixture Used to Align Loading Noses and Supports

A2. TOE COMPENSATION

A2.1 In a typical force-deflection curve (see Fig. A2.1) there is a toe region, AC, which does not represent a property of the material. It is an artifact caused by a take-up of slack and alignment, or seating of the specimen. In order to obtain correct values of such parameters as flexural modulus, and deflection at failure, this artifact must be compensated for to give the corrected zero point on the deflection, or extension axis.

A2.2 In the case of a material exhibiting a region of Hookean (linear) behavior (see Fig. A2.1), a continuation of the linear (CD) region is constructed through the zero axis. This intersection (B) is the corrected zero deflection point from which all deflections must be measured. The slope can be determined by dividing the change in force between any two points along the line CD (or its extension) by the change in deflection at the same two points (measured from Point B, defined as zero-deflection).

A2.3 In the case of a material that does not exhibit any linear region (see Fig. A2.2), the same kind of toe correction of zero-deflection point can be made by constructing a tangent to the maximum slope at the inflection Point H'. This is extended to intersect the deflection axis at Point B', the corrected zero-deflection point. Using Point B' as zero deflection, the force at any point (G') on the curve can be divided by the deflection at that point to obtain a flexural chord modulus (slope of Line B'G').

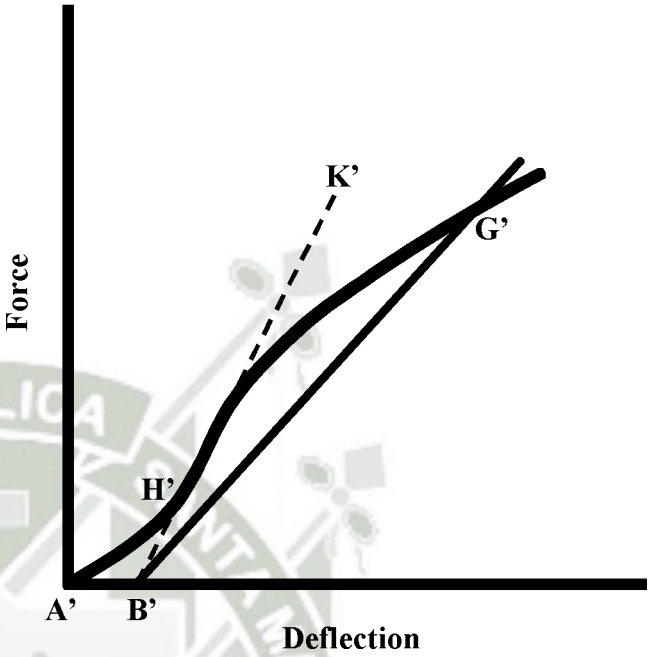


FIG. A2.2 Material without a Hookean Region

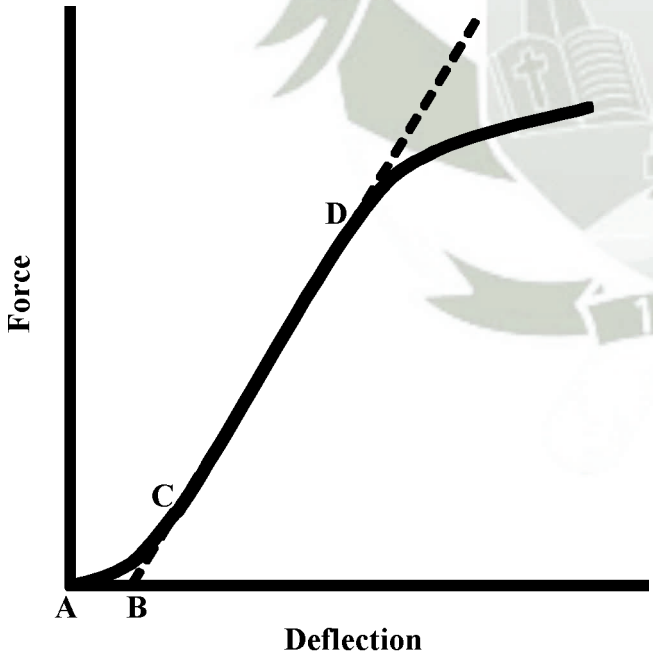


FIG. A2.1 Material with a Hookean Region

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Designation: D 2240 – 97$^{\epsilon 1}$

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Rubber Property—Durometer Hardness[1]

This standard is issued under the fixed designation D 2240; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 NOTE—A footnote was removed from Note 5 in February 1999.

1. Scope

1.1 This test method covers seven types of durometers A, B, C, D, DO, O and OO, and the procedure for determining indentation hardness of substances classified as rubber, cellular materials, elastomeric materials, thermoplastic elastomers and some hard plastics.

1.2 This test method is not applicable to the testing of fabrics.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[2]

D 785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials[2]

D 1349 Practice for Rubber—Standard Temperatures For Testing[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

3. Summary of Test Method

3.1 This test method permits hardness measurements based on either initial indentation or indentation after a specified period of time, or both.

NOTE 1—Durometers with maximum reading pointers used to determine initial hardness values may yield lower hardness when the maximum pointer is used.

4. Significance and Use

4.1 This test method is based on the penetration of a specific type of indentor when forced into the material under specified conditions. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and viscoelastic behavior of the material. The shape of the indentor and the applied force influence the results obtained so there may be no simple relationship between the results obtained with one type of durometer and those obtained with another type of durometer or other instruments for measuring hardness. This test method is an empirical test intended primarily for control purposes. No simple relationship is known to exist between indentation hardness determined by this test method and any fundamental property of the material tested. For specification purposes it is recommended that Test Method D 785 be used for hard material.

NOTE 2—Durometer scale comparison chart only. This is not and cannot be used as a conversion reference.

Type A	10 20 30 40 50 60 70 80 90 100
Type B	10 20 30 40 50 60 70 80 90 100
Type C	10 20 30 40 50 60 70 80 90 100
Type D	10 20 30 40 50 60 60 70 80 90 100
Type DO	10 20 30 40 50 60 70 80 90 100
Type O	10 20 30 40 50 60 70 80 90 100
Type OO	10 20 30 40 50 60 70 80 90 100

5. Apparatus

5.1 Hardness measuring apparatus or durometer consisting of the following components:

5.1.1 *Presser Foot*, with a hole having a diameter as specified in Fig. 1(*a*), (*b*), or (*c*) with its center at least 6 mm (0.25 in.) from any edge of the foot.

5.1.2 *Indentor*, formed from hardened steel rod and shaped in accordance with Fig. 1(*a*), (*b*), or (*c*) with full extension adjustable between 2.46 to 2.54 mm (0.97 to 0.100 in.).

5.1.3 *Indentor Extension Indicating Device* (analog or electronic), having a scale reading from 0 to 100 with equal divisions throughout the range. The scale reading is an inverse function of the indentor extension. The device shall have a pointer that moves on the scale at a rate of one hardness point for each 0.025 mm (0.001 in.) of indentor movement.

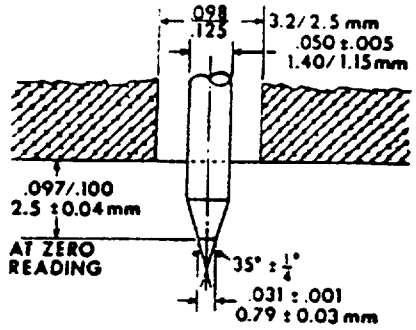
NOTE 3—Type A Shore Durometers serial numbers 1 through 16 300 and 16 351 through 16 900 and Type A-2 Shore Durometers numbers 1 through 8077 do not meet the requirement of 2.46 to 2.54 mm (0.097 to 0.100 in.) extension of the indentor at zero reading. These durometers will give readings which are low by amounts ranging from 3 units at 30

[1] This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

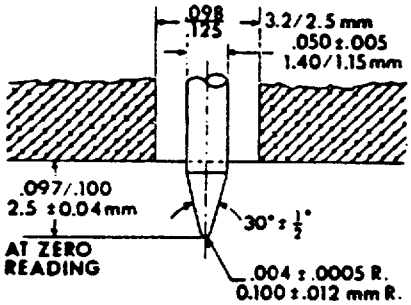
Current edition approved Feb. 10, 1997. Published March 1997. Originally published as D 2240 – 64 T. Last previous edition D 2240 – 95.

[2] *Annual Book of ASTM Standards*, Vol 08.01.

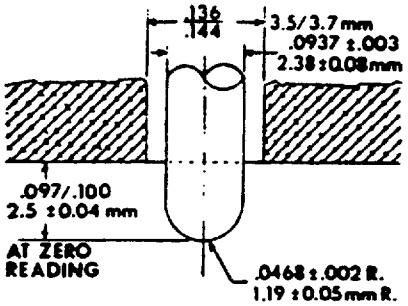
[3] *Annual Book of ASTM Standards*, Vol 09.01.



(a) Indentor for Type A and C Durometers



(b) Indentor for Type B and D Durometers



(c) Indentor for Type DO, O, and OO Durometers

NOTE 1—Spring Force Combinations:

Force, N = $0.550 + 0.075\ H_x$

where H_x = hardness reading on Type A, B and O durometers.

Force, N = $0.4445\ H_y$

where H_y = hardness reading on Type C, D and DO durometers.

Force, N = $0.203 + 0.00908\ H_{oo}$

where H_{oo} = hardness reading on Type OO durometers.

FIG. 1 Durometer, Indentor and Spring Force Combinations

hardness to 1 unit at 90 hardness.

5.1.4 *Timing Device* (optional), capable of being set to a desired elapsed time, signalling the operator or holding the hardness reading when the desired elapsed time has been reached. The timer should be automatically activated when the presser foot is in firm contact with the specimen being tested.

5.1.5 *Calibrated Spring*, for applying force to the indentor in accordance with Fig. 1.

6. Test Specimen

6.1 The test specimen shall be at least 6 mm (0.25 in.) in thickness unless it is known that results equivalent to the 6 mm values are obtained with a thinner specimen (see Note 4). A specimen may be composed of plied pieces to obtain the necessary thickness, but determinations made on such specimens may not agree with those made on solid specimens because the surface faces between plies may not be in complete contact. The lateral dimensions of the specimen shall be sufficient to permit measurements at least 12 mm (0.5 in.) from any edge unless it is known that identical results are obtained when measurements are made at lesser distance from an edge. The surfaces of the specimen shall be flat and parallel over a sufficient area to permit the presser foot to contact the specimen over an area having a radius of at least 6 mm (0.25 in.) from the indentor point. A suitable hardness determination cannot be made on a rounded, uneven, or rough surface.

NOTE 4—The minimum requirement for the thickness of the specimen is dependent on the extent of penetration of the indentor into the specimen; that is, thinner specimens may be used for materials having hardness values at the upper end of the scale. The minimum distance from the edge at which measurements may be made likewise decreases as the hardness increases. For materials having hardness values above 50 Type D durometer, the thickness of the specimen should be at least 3 mm (0.12 in.) and measurements should not be made closer than 6 mm (0.25 in.) to any edge.

7. Calibration

7.1 The durometer spring shall be calibrated by supporting the durometer in a vertical position and applying a measurable force to the indentor tip (see Fig. 2). The device used to apply the force may be a dead weight or electronic load cell device capable of measuring applied force at 50 % of the calibration tolerance. Care should be taken to ensure that the force is applied vertically to the indentor tip, as side loads will cause errors in calibration. Spring calibration shall be verified on all durometer at scale readings of 20, 30, 40, 50, 60, 70, 80 and 90. The measured force (9.8 × mass in kilograms) shall be equivalent to the force calculated by the equation in Fig. 1. The measured force for Type A, B and O durometers shall be within ±0.08 N. For Type C, D and DO durometers it shall be within ±0.44 N, and for Type OO durometers it shall be within ±0.025 N.

NOTE 5—Instruments specifically designed for the calibration of durometers may be used.

7.2 Indentor extension and shape must be in accordance with 5.1.2. With the durometer placed firmly on a flat surface the indicator should read a number equal to the indentor extension measured in inches × 1000, within ±0.5 durometer points.

NOTE 6—When performing the procedure in 7.2 on Type B and D durometers care should be used not to damage the indentor tip.

7.3 Test blocks (rubber or spring type) provided for checking durometer operation are not to be relied upon as calibration standards. The calibration procedures outlined in 7.1 and 7.2 are the only valid calibration methods.

8. Conditioning

8.1 Tests shall be made at 23 ± 2°C (73.4± 3.6°F). For materials whose hardness depends on relative humidity, the specimen shall be conditioned in accordance with Procedure A of Practice D 618 and tested under the same conditions.

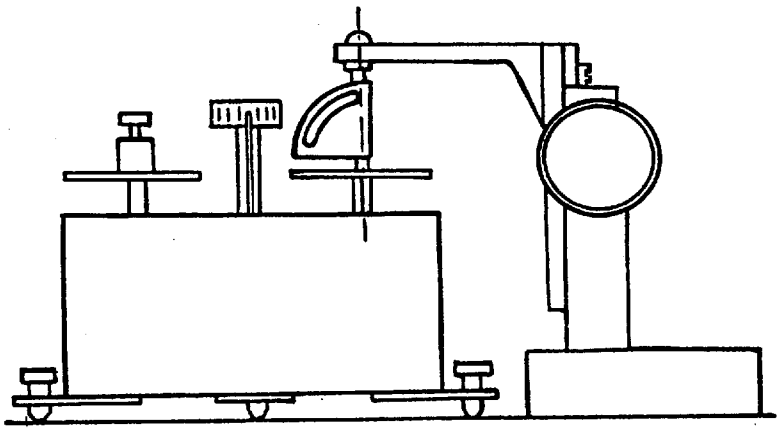


FIG. 2 Apparatus for Calibration of Durometer Spring

NOTE 7—No conclusive evaluation has been made on durometers at temperatures other than 23 ± 2°C (73.4 ± 3.6°F). Conditioning at temperatures other than the above may show changes in calibration. Durometer use at temperatures other than the above should be decided locally (see Practice D 1349).

9. Procedure

9.1 Place the specimen on a hard, horizontal surface. Hold the durometer in a vertical position with the point of the indentor at least 12 mm (0.5 in.) from any edge of the specimen, unless it is known that identical results are obtained when measurements are made with the indentor at a lesser distance. Apply the presser foot to the specimen as rapidly as possible, without shock, keeping the foot parallel to the surface of the specimen. Apply just sufficient pressure to obtain firm contact between presser foot and specimen.

NOTE 8—Better repeatability may be obtained by using a mass centered on the axis of the indentor. Recommended masses are 1 kg for Type A, B and O durometers, 5 kg for Type C, D and DO durometers, and 400 g for Type OO durometers. Durometer stands using the masses above as a constant load and a controlled descent speed, without shock, produce maximum repeatability.

9.2 For any material covered in 1.1, after the presser foot is in firm contact with the specimen, the scale reading is to be taken within 1 s or after any period of time agreed upon between supplier and user unless the durometer has a maximum indicator, in which case the maximum reading is taken. The hardness reading may progressively decrease with time delay.

9.3 Make one measurement at each of three or five different points distributed over the specimen at least 6 mm (0.25 in.) apart using the median of these measurements for the hardness value.

NOTE 9—The type of durometer should be selected with the knowledge that readings below 10 or above 90 are not considered reliable by the manufacturer. It is suggested that readings in these ranges not be recorded.

10. Report

10.1 Report the following information:

10.1.1 Hardness value obtained,

10.1.2 Complete identification of the material tested,

10.1.3 Vulcanization date,

10.1.4 Description of specimen, including thickness and number of pieces plied, if less than 6 mm (0.25 in.),

10.1.5 Temperature of test if other than 23°C,

10.1.6 Relative humidity when hardness of material is dependent on humidity,

10.1.7 Type and serial number of durometer,

10.1.8 Indentation hardness time interval at which reading was taken, and

10.1.9 Date of test.

NOTE 10—Readings may be reported in the form: A/45/15 where A is the type of durometer, 45 the reading, and 15 the time in seconds that the pressure foot is in firm contact with the specimen. Similarly, D/60/1 indicates a reading of 60 on the Type D durometer obtained either within 1 s or from a maximum indicator.

11. Precision and Bias [4]

11.1 These precision and bias statements have been prepared in accordance with Practice D 4483. Refer to this Practice for terminology and other testing and statistical concepts.

11.2 The Type 1 precision for both Type A and D methods was determined from an interlaboratory program with three materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for both A and D testing programs. All materials were supplied from a single source.

11.3 A test result for hardness (both A and D) was the median of five individual hardness readings on each day in each laboratory.

11.4 Table 1 shows the precision results for Type A method. Table 2 gives the precision results for Type D method.

11.5 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described above. The precision parameters should not be used for acceptance or rejection testing, or both, of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

[4] Supporting data are available from ASTM Headquarters. Request RR:D11-1029.

TABLE 1 Type 1 Precision—Type A Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr [A]	r [B]	(r) [C]	SR [D]	R [E]	(R) [F]
1	51.4	0.646	1.83	3.56	1.56	4.41	8.59
2	65.3	0.878	2.48	3.81	2.14	6.06	9.27
3	68.0	0.433	1.23	1.80	2.28	6.45	9.49
Pooled	61.6	0.677	1.92	3.11	2.018	5.72	9.28

[A] Sr = repeatability standard deviation, measurement units.
[B] r = repeatability = 2.83 × Sr, measurement units.
[C] (r) = repeatability, relative, (that is, in percent).
[D] SR = reproducibility standard deviation, measurement units.
[E] R = reproducibility = 2.83 × SR, measurement units.
[F] (R) = reproducibility, relative, (that is, in percent).

TABLE 2 Type 1 Precision—Type D Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr [A]	r [B]	(r) [C]	SR [D]	R [E]	(R) [F]
1	42.6	0.316	0.894	2.10	2.82	7.98	18.7
2	54.5	0.791	2.24	4.11	3.54	10.0	18.4
3	82.3	1.01	2.86	3.47	3.54	10.0	12.2
Pooled	59.8	0.762	2.16	3.61	3.32	9.40	15.7

[A] Sr = repeatability standard deviation, measurement units.
[B] r = repeatability = 2.83 × Sr, measurement units.
[C] (r) = repeatability, relative, (that is, in percent).
[D] SR = reproducibility standard deviation, measurement units.
[E] R = reproducibility = 2.83 × SR, measurement units.
[F] (R) = reproducibility, relative, (that is, in percent).

11.6 *Precision*—The precision of this test method may be expressed in the format of the following statements which use as appropriate value r, R, (r) or (R), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of r or R associated with a mean level in Table 1 and Table 2 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

11.6.1 *Repeatability*—The repeatability, r, of this test method has been established as the appropriate value tabulated in Table 1 and Table 2. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated r (for any given level) must be considered as derived from different or nonidentical sample populations.

11.6.2 *Reproducibility*—The reproducibility, R, of this test method has been established as the appropriate value tabulated in Table 1 and Table 2. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated R (for any given level) must be considered to have come from different or nonidentical sample populations.

11.6.3 Repeatability and reproducibility expressed as a percentage of the mean level, (r) and (R), have equivalent application statements as above for r and R. For the (r) and (R) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by this test method. Bias, therefore, cannot be determined.

12. Keywords

12.1 durometer hardness

Designation: D 1238 – 04

Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer[1]

This standard is issued under the fixed designation D 1238; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers measurement of the rate of extrusion of molten resins through a die of a specified length and diameter under prescribed conditions of temperature, load, and piston position in the barrel as the timed measurement is being made.

1.2 Procedure A is a manual cutoff operation based on time used for materials having flow rates that fall generally between 0.15 and 50 g/10 min. Procedure B is an automatically timed flow rate measurement used for materials having flows from 0.50 to 900 g/10 min. By both procedures, the piston travel is generally the same during the timed measurement; the piston foot is about 46 and 20.6 mm above the die. Comparable flow rates have been obtained by these procedures in interlaboratory round-robin measurements of several materials described in 13.1. Provision is made for calculation of melt volume-flow rate as well as melt mass-flow rate.

NOTE 1—Round-robin testing indicates this test method may be suitable at flow rates up to 1500 g/10 min if the timing clock resolves the elapsed time to the nearest 0.01 s.

NOTE 2—This test method and ISO 1133-1991 are technically equivalent.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 5.7, 10.2.12, and 14.1.2.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 618 Practice for Conditioning Plastics for Testing

D 883 Terminology Relating to Plastics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ANSI Standard:*

B46.1 on Surface Texture[3]

2.3 *ISO Standard:*

ISO 1133-1991 Determination of the Melt-Mass Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics[3]

3. Terminology

3.1 *General:*

3.1.1 For definition of some of the technical terms used in this test method refer to Terminology D 883.

4. Significance and Use

4.1 This test method is particularly useful for quality control tests on thermoplastics.

NOTE 3—Polymers having flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.

4.2 This test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process and, in this case, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.

4.3 The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior.

4.4 The flow rate of a material may be measured under any of the conditions listed for it in 8.2. Additional characterization of a material can be obtained if more than one condition is used. In case two conditions are employed, a Flow Rate Ratio (FRR) may be obtained by dividing the flow rate at one condition by the flow rate at the other condition.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.08).

Current edition approved March 1, 2004. Published April 2004. Originally approved in 1965. Last previous edition approved in 2001 as D 1238 - 01ε1.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

5. Apparatus

5.1 *Plastometer*:

5.1.1 The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and 2, are described in 5.2-5.8. All dimensional measurements shall be made when the article being measured is at 23 ± 5°C.

5.1.2 Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results among laboratories. It is important, therefore, for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results.

5.2 *Cylinder*—The steel cylinder shall be 50.8 mm in diameter, 162 mm in length with a smooth, straight hole 9.5504 ± 0.0076 mm in diameter, displaced 4.8 mm from the cylinder axis. Wells for a thermal sensor (thermoregulator, thermistor, etc.) and thermometer shall be provided as shown in Fig. 1. A 3.2-mm plate shall be attached to the bottom of the cylinder to retain the die. A hole in this plate, centered under the die and countersunk from below, allows free passage of the extrudate. The cylinder may be supported by at least two 6.4-mm high-strength screws at the top (radially positioned at right angles to the applied load) or by at least two 10-mm diameter rods screwed into the side of the cylinder for attaching to a vertical support. The essential dimensions of a satisfactory cylinder of this type are shown in Fig. 1 (Note 4). The cylinder bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

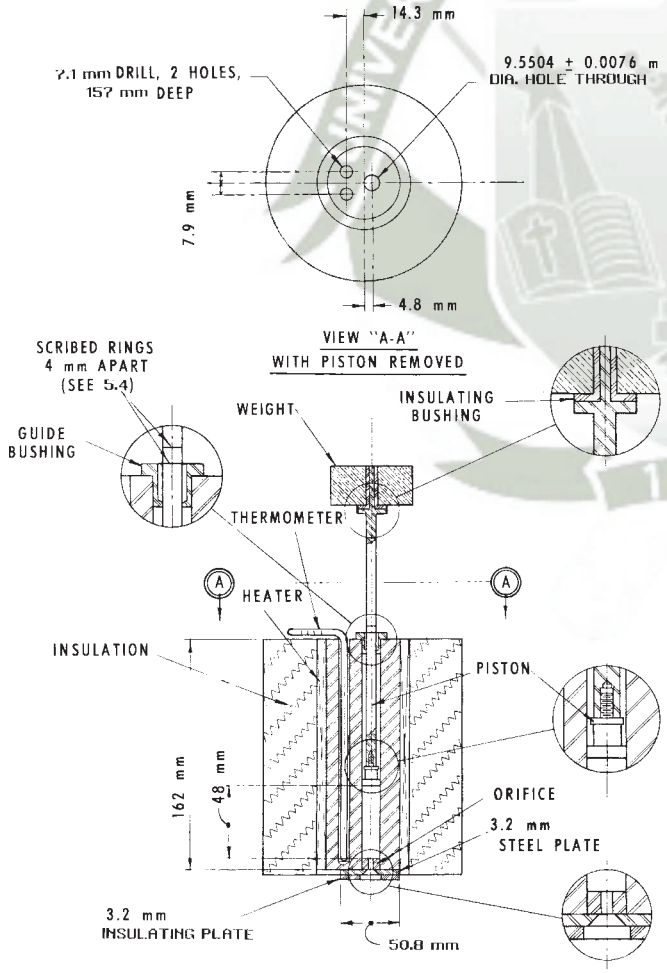


FIG. 1 General Arrangement of Extrusion Plastometer

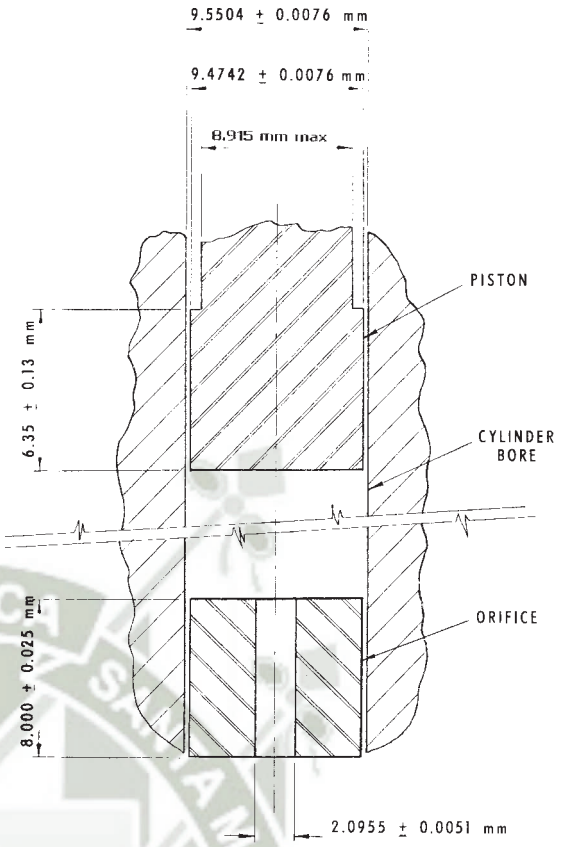


FIG. 2 Details of Extrusion Plastometer

NOTE 4—Cylinders made of SAE 52100 or other equivalent steel heat-hardened to 60–65 Rockwell Hardness Scale C give good service when used at temperatures below 200°C. Cylinder liners of cobalt-chromium-tungsten alloy are also satisfactory to 300°C.

5.3 *Die*—The outside of the steel die shall be such diameter that it will fall freely to the bottom of the 9.5504 ± 0.0076 mm diameter hole in the cylinder (Note 5). The die shall have a smooth straight bore 2.0955 ± 0.0051 mm in diameter and shall be 8.000 ± 0.025 mm in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The die bore shall be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

NOTE 5—Recommended die material is tungsten carbide. Also satisfactory are steel, synthetic sapphire, and cobalt-chromium-tungsten alloy.

5.4 *Piston*:

5.4.1 The piston shall be made of steel with an insulating bushing at the top as a barrier to heat transfer from the piston to the weight. The land of the piston shall be 9.4742 ± 0.0076 mm in diameter and 6.35 ± 0.13 mm in length. The piston

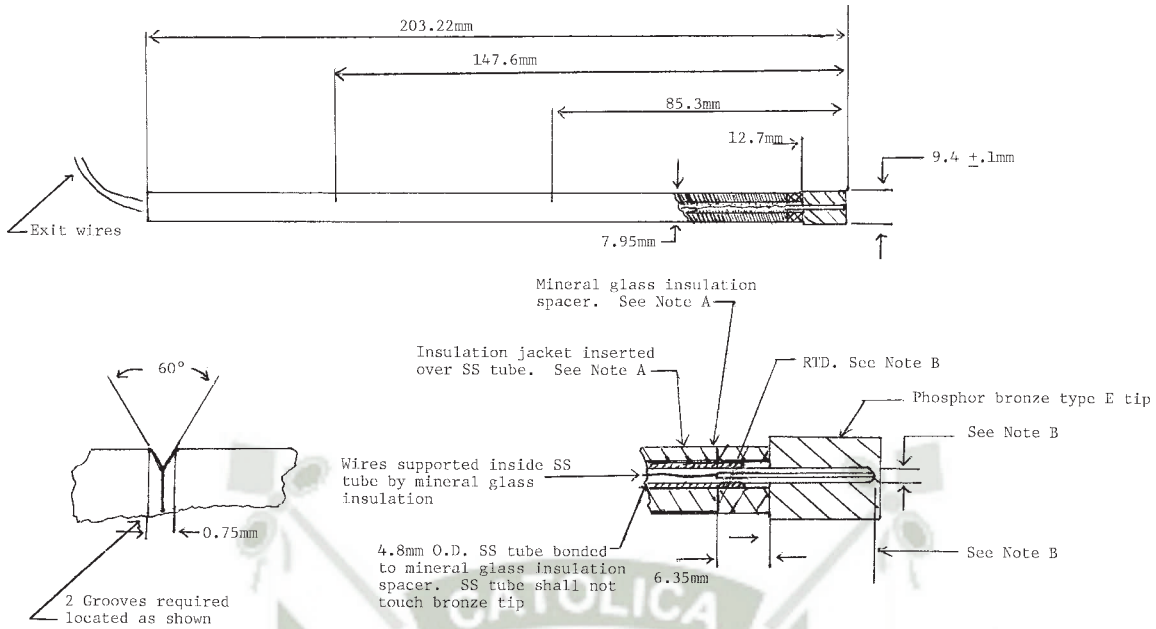


FIG. 3 Example of a Temperature Calibration Device

Note A—Mineral glass insulation or equivalent spacer shall be bonded to tip and SS tube. Bond material shall be low conductivity type, 400°C minimum rating. Insulation jacket material shall be low conductivity type (400°C minimum rating preferred, see Note 5).

Note B—The RTD shall be inserted into bronze tip and bonded using high conductivity, 400°C rated material. Tip of RTD element shall touch the bronze tip. Minimum insertion depth of 11.2 mm clearance between RTD and tip wall shall be minimized.

design may incorporate means for land replacement, for example, having threads and flats immediately above the land. Above the land, the piston shall be no larger than 8.915 mm in diameter (Note 6). The finish of the piston foot shall be 12 rms in accordance with ANSI B46.1. If wear or corrosion is a problem, the piston should be of stainless steel and equipped with a detachable foot for ease of replacement.

NOTE 6—To improve standardization it is preferable that the piston be guided with a loose-fitting metal sleeve at the top of the cylinder.

NOTE 7—Pistons of SAE 52100 steel with the bottom 25 mm, including the foot, hardened to a Rockwell hardness, C scale, of 55 to 59 have been found to give good service when used at temperatures below 200°C.

5.4.2 The piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder or other suitable reference point, the bottom of the piston is 48 mm above the top of the die (see Fig. 1).

5.4.3 The combined weight of piston and load shall be within a tolerance of ±0.5 % of the selected load.

5.5 *Heater*:

5.5.1 The equipment must have a heater capable of heating the apparatus so that the temperature at 10 mm above the die can be maintained within ±0.2°C of the desired temperature during the test. The temperature of the barrel, from 10 mm to 75 mm above the top of the die, must be maintained within ±1 % of the set temperature (°C).

NOTE 8—At temperatures higher than 200°C this degree of temperature control may be more difficult to obtain.

5.5.2 Calibrate the temperature-indicating device by means of a light-gage probe-type thermocouple or a platinum-resistance temperature sensor having a short sensing length.[4] The thermocouple should be encased in a metallic sheath having a diameter of approximately 1.6 mm with its hot junction grounded to the end of the sheath. Insert the temperature sensor into the melt from the top of the cylinder so that it is 10 ± 1 mm above the upper face of the die. The temperature sensors shall be used with a potentiometer having a sensitivity of at least 0.005 mV, or a temperature readout having a sensitivity of at least 0.1°C. Calibration should also be verified at 75 mm above the upper face of the die. An alternate technique for calibration is to use a sheathed thermocouple or platinum-resistance temperature sensor with tip diameter of 9.4 ± 0.1 mm for insertion in the bore without material present. An example of this is shown in Fig. 3. Calibration of the temperature-indicating device shall be verified at each run temperature.

NOTE 9—The response of the temperature sensing device may be affected by immersion level. Take care to ensure adequate insulation of the device sensor and stabilization of the barrel temperature.

5.5.3 Heat shall be supplied by electric band heater(s) covering the entire length of the cylinder. The heater(s) may be single- or multi-element, depending upon the manufacturer's control means. The heater(s) plus control system must be capable of maintaining the temperature within the required ±0.2°C of the set point. The temperature sensor and readout equipment must be calibrated to a traceable national standard

[4] Round-robin data showing flow rate and corresponding temperature profile of the melt obtained using probe-type thermocouples and platinum resistance temperature sensors can be obtained from ASTM Headquarters. Request RR:D20-1094.

(that is, NIST) at least once per year. The cylinder with the heater(s) shall be lagged with 38 mm of foamed-glass insulation. An insulating plate 3.2 mm in thickness shall be attached to the bottom of the cylinder to minimize heat loss at this point.

5.6 *Temperature Controller*—The type of controller and sensor must be capable of meeting the required control tolerance specified in 5.5.1.

5.7 *Thermometer*— Thermometers having a range of 4°C graduated in 0.2°C divisions may be used to indicate temperature. The temperature at this point may not necessarily be the temperature of the material 10 mm above the die. The thermometer may be used to monitor indirectly the temperature of the material 10 mm above the die and may be calibrated by reference to a thermocouple or platinum resistance temperature sensor inserted in the material 10 mm above the die. See 5.5.2 for a description of a method for measuring temperature. **Warning—**Caution should be observed with the use of a mercury-filled thermometer. Mercury vaporization occurs if the thermometer is broken. Mercury thermometers are not to be used at or above the boiling point of mercury, which is 357°C.

NOTE 10—**Warning:**

5.8 *Level*—Provision shall be made for vertical alignment of the bore of the extrusion plastometer. This is necessary to minimize subtractive loads resulting from rubbing or friction between the piston tip and sidewall. Means of alignment are discussed in Appendix X1.

5.9 *Accessory Equipment*—Necessary accessories include equipment for charging samples to the cylinder, a funnel, a die plug, a tool for cutting off the extruded sample, a timer or stop watch, cleaning equipment, go/no-go gages, a balance accurate to ±0.001 g, and, when required, a weight or weight-piston support.

NOTE 11—Satisfactory operation of the apparatus for polyethylenes can be ascertained by making measurements on NIST Standard Reference Materials (SRMs) certified for melt flow rate. The four SRMs certified under condition 190/2.16 are SRM 1473 with a flow rate of 1.29 g/min, SRM 1474 with a flow rate of 5.03 g/10 min, SRM 1496 with a flow rate of 0.26 g/10 min, and SRM 1497 with a flow rate of 0.19 g/10 min. SRM 1475a is certified under condition 190/3.25 with a flow rate of 2.20 g/10 min.[5]

6. Test Specimen

6.1 The test specimen may be in any form that can be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs. It may be desirable to preform or pelletize a powder.

7. Conditioning

7.1 Many thermoplastic materials do not require conditioning prior to testing. Materials which contain volatile components, are chemically reactive, or have other special characteristics most probably require appropriate conditioning procedures. Moisture not only affects reproducibility of flow rate measurement but, in some types of materials, degradation is accelerated by moisture at the high temperatures used in testing. Check the applicable material specification for any conditioning requirements before using this test. See Practice D 618 for appropriate conditioning practices.

8. Procedural Conditions

8.1 Standard conditions of test are given in Table 1. Test conditions shall be shown as: Condition _ _ _ / _ _ _, where the temperature in degrees Celsius is shown first, followed by the weight in kilograms. For example: Condition 190/2.16.

8.2 The following conditions have been found satisfactory for the material listed:

Material	Condition	
Acetals (copolymer and homopolymer)	190/2.16	190/1.05
Acrylics	230/1.2	230/3.8
Acrylonitrile-butadiene-styrene	200/5.0	230/3.8
	220/10	
Acrylonitrile/butadiene/styrene/polycarbonate	230/3.8	250/1.2
blends	265/3.8	265/5.0
Cellulose esters	190/0.325	190/2.16
	190/21.60	210/2.16
Ethylene-chlorotrifluoroethylene copolymer	271.5/2.16	
Ethylene-tetrafluoroethylene copolymer	297/5.0	
Nylon	275/0.325	235/1.0
	235/2.16	235/5.0
	275/5.0	
Perfluoro(ethylene-propylene) copolymer	372/2.16	
Perfluoroalkoxyalkane	372/5.0	
Polycaprolactone	125/2.16	80/2.16
Polychlorotrifluorethylene	265/12.5	
Polyether sulfone (PES)	380/2.16	360/10
	343/2.16	
Polyethylene	125/0.325	125/2.16
	250/1.2	
	190/0.325	190/2.16
	190/21.60	190/10
	310/12.5	
Polycarbonate	300/1.2	
Polymonochlorotrifluoroethylene	265/21.6	
	265/31.6	
Polypropylene	230/2.16	
Polyphenyl sulfone (PPSU)	365/5.0	380/2.16
Polystyrene	200/5.0	230/1.2
	230/3.8	190/5.0
Polysulfone (PSU)	343/2.16	360/10
Polyterephthalate	250/2.16	210/2.16
	285/2.16	
Poly(vinyl acetal)	150/21.6	
Poly(vinylidene fluoride)	230/21.6	
	230/5.0	
Poly(phenylene sulfide)	315/5.0	
Styrene acrylonitrile	220/10	230/10
	230/3.8	
Styrenic Thermoplastic Elastomer	190/2.16	200/5.0
Thermoplastic Elastomer-Ether-Ester	190/2.16	220/2.16
	230/2.16	240/2.16
		250/2.16
Thermoplastic elastomers (TEO)	230/2.16	
Vinylidene fluoride copolymers	230/21.6	
	230/5.0	
	for T_m= 100° use 120/5.0 or 21.6	

NOTE 12—Some materials may require special materials of construction or handling for performing this test. Please refer to the material specification for appropriate recommendations.

8.3 If more than one condition is used and the material is polyethylene, the determination of Flow Rate Ratio (FRR) has been found to be useful. The FRR is a dimensionless number derived by dividing the flow rate at Condition 190/10 by the flow rate at Condition 190/2.16.

NOTE 13—When determining such a ratio of flow rates for a material at

[5] These standard polyethylenes are available from the National Institute of Standards and Technology, Office of Standard Reference Materials, Washington, DC 20234.

TABLE 1 Standard Test Conditions, Temperature, and Load

Condition	Temperature, °C	Total Load Including Piston, kg	Approximate Pressure	
Standard Designation			kPa	psi
80/2.16	80	2.16		
125/0.325	125	0.325	44.8	6.5
125/2.16	125	2.16	298.2	43.25
150/2.16	150	2.16	298.2	43.25
190/0.325	190	0.325	44.8	6.5
190/2.16	190	2.16	298.2	43.25
190/21.60	190	21.60	2982.2	432.5
200/5.0	200	5.0	689.5	100.0
230/1.2	230	1.2	165.4	24.0
230/3.8	230	3.8	524.0	76.0
265/12.5	265	12.5	1723.7	250.0
275/0.325	275	0.325	44.8	6.5
230/2.16	230	2.16	298.2	43.25
190/1.05	190	1.05	144.7	21.0
190/10.0	190	10.0	1379.0	200.0
300/1.2	300	1.2	165.4	24.0
190/5.0	190	5.0	689.5	100.0
235/1.0	235	1.0	138.2	20.05
235/2.16	235	2.16	298.2	43.25
235/5.0	235	5.0	689.5	100.0
250/2.16	250	2.16	298.2	43.25
310/12.5	310	12.5	1723.7	250.0
210/2.16	210	2.16	298.2	43.25
285/2.16	285	2.16	298.2	43.25
315/5.0	315	5.0	689.5	100.0
372/2.16	372	2.16	298.2	43.25
372/5.0	372	5.0	689.5	100
297/5.0	297	5.0	689.5	100
230/21.6	230	21.6	2982.2	432.5
230/5.0	230	5.0	689.5	100
265/21.6	265	21.6	2982.2	432.5
265/31.6	265	31.6	4361.2	632.5
271.5/2.16	271.5	2.16	298.2	43.25
220/10	220	10.0	1379.0	200.0
250/1.2	250	1.2	165.4	24.0
265/3.8	265	3.8	524.0	76.0
265/5	265	5.0	689.5	100.0

the same temperature under different loads, it has been found that precision is maximized when one operator uses one Procedure (A or B), the same plastometer, and the same die for both measurements (the die need not be removed from the plastometer between the two determinations).

9. Procedure A—Manual Operation

9.1 Select conditions of temperature and load from Table 1 in accordance with material specifications such that flow rates will fall between 0.15 to 50 g/10 min.

9.2 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1.)

9.3 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11.

NOTE 14—The degree of cleanliness can significantly influence the flow rate results, therefore a thorough method of cleaning must be established. It has been found that three swabs of the barrel is satisfactory for most materials and that the die, barrel, and piston are more easily cleaned while hot.

9.4 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at 23 ± 5°C) is within the tolerances given in 5.3.

NOTE 15—Cleaning and usage can result in a die diameter that is out of specifications. Data has shown that erroneous results will be obtained if the die diameter is not within the appropriate tolerances.

9.5 Verify that the temperature is stable and within ±0.2°C of the appropriate test temperature as specified in 5.5.1.

9.6 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

NOTE 16—The reduction in heating time when the unit is being used repetitiously is only allowed when runs of the same or similar material are being measured over a continuous time frame. If the piston and die are removed and cleaned, they should be considered “cold” and the full 15 minutes heating stabilization time required.

9.7 Remove the piston and place it on an insulated surface. Charge the cylinder within 1 min with a weighed portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add the appropriate weight.

NOTE 17—Experience has shown that for the best reproducibility the piston should operate within the same part of the cylinder for each measurement. The piston is scribed so the starting point for each extrusion is roughly the same. Some excess of material over the minimum required for the actual flow measurement portion of the test is provided by the charging weights shown in Table 2. This is necessary to achieve a void-free extrudate and flow equilibrium before start of rate measurements.

NOTE 18—It is frequently helpful to take interim cuts of the extrudate at uniform time intervals during the specified extrusion time. Weights of

TABLE 2 Standard Test Conditions, Sample Mass,[A] and Testing Time[B]

Flow Range, g/10 min	Suggested Mass of Sample in Cylinder, g	Time Interval, min	Factor for Obtaining Flow Rate in g/10 min
0.15 to 1.0	2.5 to 3.0	6.00	1.67
>1.0 to 3.5	3.0 to 5.0	3.00	3.33
>3.5 to 10	4.0 to 8.0	1.00	10.00
>10 to 25	4.0 to 8.0	0.50	20.00
>25	4.0 to 8.0	0.25	40.00

[A] This is a suggested mass for materials with melt densities of about 0.7 g/cm^3. Correspondingly, greater quantities are suggested for materials of greater melt densities. Density of the molten resin (without filler) may be obtained using the procedure described by Terry, B. W., and Yang, K., "A New Method for Determining Melt Density as a Function of Pressure and Temperature," *SPE Journal*, SPEJA, Vol. 20, No. 6, June 1964, p. 540 or the procedure described by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 23, 1979, p. 1051. It may also be obtained from the weight of an extruded known volume of resin at the desired temperature. For example, 25.4 mm (1 in.) of piston movement extrudes 1.804 cm^3 of resin. An estimate of the density of the material can be calculated from the following equation:

$$\text{resin density at test temperature} = M/1.804$$

where:
M = mass of extruded resin.

[B] See 9.14.

these individual cuts give an indication of the presence of bubbles which may be masked due to their size or to opacity of the sample. This technique is particularly helpful in the case of highly pigmented materials. Forcing out some of the resin manually during the preheat period often eliminates bubbles in the test extrudate.

9.8 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position 7.0 ± 0.5 min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min.

NOTE 19—It has been found that purging within 60 s of the start time will result in higher variability in the data.

NOTE 20—There may be cases where the 7.0 ± 0.5 min is too much or not enough preheat time. For those materials, provisions must be in the material documents. It is necessary to refer to the appropriate material document before beginning any test.

NOTE 21—Additional care may be necessary to prevent thermal degradation in the extrusion plastometer. This is sometimes done by the addition of an appropriate antioxidant. For highly unstable materials, it may be necessary to use alternative techniques as an indication of flow characteristics.

9.9 For materials with flow rates greater than 10 g/10 min, a weight (and if needed, a piston) support must be used after the initial purge. The support shall be removed at such a time as to allow the test to begin within 7 ± 0.5 min of the completion of the charge. The piston/weight support should be of such a length that the lower scribe mark of the supported piston/weight will be 25 mm above the top of the guide bushing or other suitable reference mark.

NOTE 22—It has been found that the effect of choosing plugging, weight support, or both, is significant to the flow rate results. The choice of piston support was made to cover all conditions and flow rates 10 to 50 g/10 min.

9.10 For all tests, start collecting a timed extrudate when requirements for the piston position are met, provided this is within 7.0 ± 0.5 min from the end of charging; otherwise, discard the charge and repeat the test with readjusted piston position after the initial purge, or change weights. Requirements are that the top scribed mark on the piston be visible above the cylinder or index and that the lower scribe mark be in the cylinder or below the index. As the lower scribed mark approaches the top of the cylinder or index, reset the timer to zero, then simultaneously start the timer and make the initial cut-off when the position requirements are met. Make the final cut-off exactly when the time interval given in Table 2 is reached. Collect the timed extrudate. If the extrudate contains visible bubbles, discard the complete charge and begin the test again.

NOTE 23—The charge weight should only be increased if no material is being purged and there is still not enough material to complete the test.

9.11 Discharge the remainder of the specimen and push the die out through the top of the cylinder. Swab out the cylinder with cloth patches after the manner of cleaning a pistol barrel. The die may be cleaned by dissolving the residue in a solvent. A better method is pyrolytic decomposition of the residue in a nitrogen atmosphere. Place the die in a tubular combustion furnace or other device for heating to 550 ± 10°C and clean with a small nitrogen purge through the die. This method is preferable to flame or solvent cleaning, being faster than solvent cleaning and less detrimental to the die than an open flame. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim die cleaning may be unnecessary. In such cases, however, the effect of cleaning upon flow rate determination must be shown to be negligible if this step is avoided.

9.12 Once the extrudate is cool, weigh to the nearest 1 mg.

9.13 Multiply the weight of the extrudate by the appropriate factor shown in Table 2 to obtain the flow rate in grams per 10 min.

NOTE 24—Frequently, errors in test technique, apparatus geometry, or test conditions, which defy all but the most careful scrutiny exist, causing discrepancy in flow rate determinations. The existence of such errors is readily determined by periodically measuring a reference sample of known flow rate. The flow rate value and range to be tolerated can be determined using a statistically correct test program composed of multiple determinations with various instruments. Standard samples of polyethylene, linear or branched, are available from the National Institute of Standards and Technology.

9.14 In case a specimen has a flow rate at the borderline of the ranges in Table 2 and slightly different values are obtained at different time intervals, the referee value shall be obtained at the longer time interval.

10. Procedure B—Automatically Timed Flow Rate Measurement

10.1 *Apparatus*:

10.1.1 Extrusion plastometer and auxiliary equipment are detailed in Section 4 and below.

10.1.2 A timing device shall electrically, optically, or mechanically time piston movement within the specified travel range. The requirements of the system are as follows:

10.1.2.1 Sense and indicate the piston travel time within ±0.01 s (see Note 1).

10.1.2.2 Measure piston travel within ±0.4 % of the nominal preset value (see 10.1.2.4 and 10.1.2.5) for use in the flow rate calculations.

10.1.2.3 Any effects on the applied load must be included in the allowable tolerance given in 5.4.3.

10.1.2.4 It should be preset or be settable for measuring piston travel of 6.35 ± 0.25 mm for flow rates up to 10 g/10 min.

10.1.2.5 It should be preset or be adjustable for measuring piston travel of 25.4 ± 0.25 mm for flow rates greater than 10 g/10 min.

10.1.2.6 To ensure high interlaboratory reproducibility, it is important that the timing device operates within a fixed portion of the cylinder. This is defined as the portion of the cylinder between 46 ± 2 mm and 20.6 ± 2 mm above the top of the die.

10.1.2.7 Check die, cylinder, and position dimensions for conformance to 5.2-5.4 and Figs. 1 and 2.

10.2 *Procedure*:

10.2.1 Refer to Table 1 for selection of conditions of temperature and load in accordance with the material specification.

10.2.2 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at 23 ± 5°C) is within the tolerances given in 5.3 (see Note 15).

10.2.3 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction (see Appendix X1).

10.2.4 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11 and Note 14.

10.2.5 Check the die bore diameter with appropriately sized no-go/go gages before beginning the test. Make frequent checks to determine whether the die diameter is within the tolerances given in 5.3 (see Note 15).

10.2.6 Verify that the temperature is stable and within ±0.2°C of the appropriate test temperature as specified in 5.5.1.

10.2.7 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

10.2.8 Adjust the travel arm to 6.35 ± 0.25 mm for measuring materials with expected flow rates of up to 10 g/10 min or 25.40 ± 0.25 mm for measuring materials with expected flow rates of 10 g/10 min or higher.

TABLE 3 Factors for Calculation of Flow Rate

Material (Unpigmented)	Temperature, °C	Piston Travel, *L*, cm [in.]	Factor for Calculation of Flow Rate, *F*[A]
Polyethylene	190	2.54 [1]	826
Polyethylene	190	0.635 [0.25]	207
Polypropylene	230	2.54 [1]	799
Polypropylene	230	0.635 [0.25]	200

[A] Factors calculated using melt-density values of 0.7636 g/cm^3 for polyethylene and 0.7386 g/cm^3 for polypropylene, as expressed in article by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 23, 1979, P. 1051. The base densities at 23°C for which the melt densities are reported were 0.917 g/dm^3 for annealed low-density polyethylene and polypropylene homopolymer.

NOTE 25—It has been found that for some materials the melt flow rates obtained on a material will be different depending on which timer length is chosen; therefore, it is important to adhere to the protocol in 10.2.8 to compare interlaboratory results.

10.2.9 Remove the piston and place it on an insulated surface. Charge the cylinder within 60 s with a weighted portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add weight.

10.2.10 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position 7.0 ± 0.5 min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min (see Note 19).

10.2.11 Weight and piston support, if needed, must be used after the initial purge. The support will be removed at such a time as to allow the timer to activate within 7.0 ± 0.5 min after completion of the charge. If the timer is not activated within 7 ± 0.5 min after the completion of the charge, the test must be repeated with readjusted piston position after the initial purge, or change weights. The piston/weight support should be of such a length that the lower scribe mark of the supported piston/weight will be at least 25 mm above the top of the cylinder. Only use piston support if there is excessive material flow (see Notes 22 and 23).

10.2.12 For materials greater than 50 g/10 min a die plug must be used in addition to the piston/weight support. The die plug is inserted before charge and is removed prior to removing the piston/weight support. The initial charge should be adjusted to reduce excess flow. If the timer arm is not activated within 7 ± 0.5 min after the completion of the charge the test must be repeated with readjusted piston position, or change weights (see Notes 22 and 23). **Warning—**Rapid expulsion of material when die plug is removed may be hazardous.

NOTE 26—**Warning:**

10.2.13 If the timed extrudate contains visible bubbles, repeat the test (see Note 24).

10.2.14 Record the time to the nearest 0.01 s for the piston to complete the calibrated distance of travel.

10.2.15 Discharge any remaining resin and clean the die, piston, and cylinder as detailed in 9.11.

11. Procedure C— Automatically Timed Flow Rate Measurement for High Flow Rate Polyolefins Using Half-Height, Half Diameter Die

11.1 *Apparatus*:

11.1.1 Extrusion plastometer and auxiliary equipment are detailed in Sections 5 and 10.

11.1.2 For polyolefins with a MFR of 75 or greater using the standard die (See 5.3), an alternate die can be used to reduce the flow rate of these materials and improve the reproducibility of results. The alternate die dimensions shall be: Height 3.985 ± 0.025 mm; bore diameter 1.048 ± 0.005 mm. No spacer shall be used with this die. Bore and finish requirements are the same as 5.3.

11.1.3 For calibration of the temperature indicating device, 5.5.2 shall be used with the variation that temperatures are

measured at 14 ± 1 mm and at a nominal 79 mm above the upper surface of the die.

11.1.4 If a thermometer as described in 5.7 is used to indicate temperature, it can be used to monitor indirectly the temperature of the material 14 mm above the upper surface of the die and may be calibrated via 11.1.3.

11.2 *Procedure*:

11.2.1 Use procedure described in 10.2 with the exception that the die diameter and tolerances are given in 11.1.2.

12. Calculation (Procedures B and C)

12.1 Calculate the flow rate in grams per 10 min or volume rate in cm^3 per 10 min as follows (see Note 24):

$$\text{Flow rate} = (426 \times L \times d)/t$$

or

$$\text{Volume rate} = 426 \times L/t$$

where:

L = length of calibrated piston travel, cm,
d = density of resin at test temperature, g/cm^3(see reference under Table 2),
t = time of piston travel for length L, s, and
426 = mean of areas of piston and cylinder × 600.

NOTE 27—Factors that may be substituted in the following equation are given for some materials in Table 3.

$$\text{Flow rate, g/10 min} = F/t$$

where:

F = factor from Table 3, and
t = time of piston travel for length L, s.

12.2 Agreement between Procedures A and B may be optimized if an average melt density for a particular type of material is determined with the actual equipment used and that value is substituted into the equation given in 12.1.

13. Report

13.1 Report the following information:

13.1.1 Statement indicating the nature and physical form of the material charged to the cylinder.

13.1.2 Temperature and load at which the test is run shall be reported. The results and test conditions can be referred to as FR-condition, where the standard designation for the condition from Table 1 is shown (for example: FR-190/2.16).

NOTE 28—It has become customary to refer to the flow rate of polyethylene as "melt index" when obtained under Condition 190/2.16. However, for all other materials the use of melt index or any term other than "flow rate" is discouraged, regardless of the condition used.

13.1.3 Flow rate reported as the rate of extrusion in grams per 10 min or volume rate in cm^3 per 10 min.

13.1.4 Procedure used (A, B, or C).

13.1.5 Any unusual behavior of the test specimen such as discoloration, sticking, extrudate surface irregularity or roughness, etc.

13.1.6 Details of conditioning, if any.

14. Precision and Bias (Procedures A, B, and C)

14.1 *Precision*:

14.1.1 Tables 4 and 5 are based on a round robin[6] conducted in 1986 and 1987, involving polypropylene, polyethylene, polystyrene, polycarbonate and acrylic materials. Tables 6 and 7 are based on a round robin[7] completed in 1997 involving low and high density polyethylene, polypropylene, polystyrene, polycarbonate, PMMA, and acetal. The number of participating laboratories is shown for each material. Data for Tables 4 and 5 were generated through each lab testing two specimens for each material on three different days, while data for Tables 6 and 7 were generated through each lab testing two specimens for each material on two different days. The analysis in Practice E 691 is based on a test result being the average of two specimens.

14.1.2 Table 8 is based on a round robin[8] conducted in 1980 using Procedure B. Four polypropylene samples having flow

[6] Supporting data are available from ASTM Headquarters. Request RR: D20-1164.
[7] Supporting data are available from ASTM Headquarters.
[8] Supporting data are available from ASTM Headquarters. Request RR: D20-1124.

TABLE 4 Precision, Procedure A (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	I_r[C]	I_R[D]	Number of Laboratories
Polyethylene	190/2.16	0.27	0.008	0.022	0.023	0.063	9
Polyethylene	190/2.16	0.40	0.012	0.038	0.035	0.108	9
Polyethylene	190/2.16	2.04	0.026	0.079	0.073	0.224	9
Polyethylene	190/2.16	44.1	0.919	1.232	2.560	3.486	7
Polypropylene	230/2.16	2.23	0.106	0.226	0.299	0.639	9
Polypropylene	230/2.16	7.09	0.222	0.471	0.627	1.331	9
Polypropylene	230/2.16	32.8	0.581	1.051	1.644	2.974	9
Polystyrene	200/5	1.67	0.024	0.122	0.068	0.344	6
Polystyrene	200/5	8.82	0.190	0.667	0.538	1.886	6
Polystyrene	200/5	13.3	0.305	0.925	0.864	2.617	6
Polycarbonate	300/1.2	2.41	0.076	0.115	0.215	0.326	4
Polycarbonate	300/1.2	10.5	0.429	0.647	1.215	1.830	4
Polycarbonate	300/1.2	16.2	0.155	1.109	0.438	3.140	4
Acrylic	230/3.8	2.59	0.051	0.051	0.145	0.145	3

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$, and
[D] $I_R = 2.83\ S_R$.

TABLE 5 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	I_r[C]	I_R[D]	Number of Laboratories
Polyethylene	190/2.16	0.27	0.009	0.014	0.026	0.039	8
Polyethylene	190/2.16	0.40	0.016	0.027	0.045	0.076	8
Polyethylene	190/2.16	2.04	0.040	0.094	0.112	0.266	9
Polyethylene	190/2.16	43.7	0.997	1.924	2.819	5.443	8
Polypropylene	230/2.16	2.25	0.052	0.214	0.1466	0.604	8
Polypropylene	230/2.16	7.16	0.143	0.589	0.4051	1.666	8
Polypropylene	230/2.16	32.6	0.693	0.945	1.959	2.672	8
Polystyrene	200/5	1.65	0.037	0.166	0.106	0.470	4
Polystyrene	200/5	8.39	0.144	0.423	0.406	1.197	4
Polystyrene	200/5	13.0	0.108	0.387	0.306	1.097	4

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$, and
[D] $I_R = 2.83\ S_R$.

TABLE 6 Precision, Procedure A (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	r[C]	R[D]	Number of Labs
PMMA	230/3.8	1.51	0.013	0.086	0.037	0.242	7
LDPE	190/2.16	1.74	0.027	0.052	0.075	0.144	11
Polystyrene	200/5.0	1.86	0.040	0.085	0.112	0.238	9
HDPE	190/2.16	5.35	0.049	0.103	0.137	0.288	11
Polypropylene	230/2.16	10.94	0.088	0.473	0.247	1.324	10
Polycarbonate	300/1.2	13.59	0.109	0.233	0.305	0.653	4[E]
Acetal	190/2.16	25.30	0.235	0.571	0.658	1.599	7

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $r = 2.83\ S_r$.
[D] $R = 2.83\ S_R$.
[E] Insufficient laboratories to meet Practice E 691.

TABLE 7 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	r[C]	R[D]	Number of Labs
PMMA	230/3.8	1.54	0.026	0.102	0.072	0.286	6
LDPE	190/2.16	1.76	0.015	0.053	0.042	0.149	11
Polystyrene	200/5.0	1.89	0.042	0.102	0.117	0.285	7
HDPE	190/2.16	5.41	0.041	0.113	0.114	0.316	10
Polypropylene	230/2.16	10.96	0.357	0.491	0.999	1.376	10
Polycarbonate	300/1.2	13.79	0.104	0.477	0.292	1.335	3[E]
Acetal	190/2.16	25.64	0.182	0.822	0.508	2.302	6

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $r = 2.83\ S_r$.
[D] $R = 2.83\ S_R$.
[E] Insufficient laboratories to meet Practice E 691.

TABLE 8 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	I_r[C]	I_R[D]
Polypropylene	230/2.16	245	13.2	16.6	37.4	46.9
Polypropylene	230/2.16	482	31.8	40.0	89.9	113
Polypropylene	230/2.16	837	20.9	58.6	59.1	166
Polypropylene	230/2.16	1603	129	243	365	688

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$, and
[D] $I_R = 2.83\ S_R$.

rates from 250 to 1500 were tested in nine laboratories. **Warning—**The following explanations of I_r and I_R (14.1.4-14.1.6) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 4-8 should not be vigorously applied to acceptance or rejection of material since those data are specific to the round robin and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials. The principles of 14.1.4-14.1.7 would then be valid for such data.

14.1.3 Table 9 is based on a round robin conducted in 1999 on Procedure C. Data for seven of the eight participating

TABLE 9 Precision Data for High Melt Flow Polyolefins Procedure C (Values in g/10 min)

Materials[A]	Average	S_r	S_R	r	R
PE-A (35)	4.67	0.068	0.119	0.191	0.334
PE-B (185)	24.30	0.688	1.168	1.928	3.270
PE-C (2350)	315.7	10.81	19.89	30.27	55.69
PE-D (122)	16.20	0.188	0.348	0.526	0.975

[A]Numbers in parentheses are approximate melt flow rate values of materials using standard die (5.3).

laboratories were included in the statistics for this table. Four polyethylene materials were tested with melt flow rates using the standard die ranging from approximately 35 to 2350 g/10 min using the half-height, half-diameter die.

14.1.4 *Concept of I_r and I_R*—Relevant if S_r and S_R have been calculated from a large enough body of data, and if test results are averages obtained from testing two specimens.

14.1.5 *Repeatability, I_r*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the I_r value for that material.

14.1.6 *Reproducibility, I_R*— In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R value for that material.

14.1.7 Any judgment in accordance with 14.1.4 and 14.1.6 would have an approximate 95 % (0.95) probability of being correct.

14.2 *Bias*—There are no recognized standards by which to estimate bias of this test method.

15. Keywords

15.1 melt flow rate; melt index; volume flow rate

APPENDIXES

(Nonmandatory Information)

X1. EXTRUSION PLASTOMETER BORE ALIGNMENT

X1.1 A fixture consisting of a circular level mounted on a shaft having two bearing points 9.47 + 0.00 − 0.0076 mm in diameter that can be inserted into the bore has been found suitable. A circular level that can be rigidly mounted on the piston rod for insertion into the bore may also be satisfactory. A circular level having a sensitivity of 20 min/2.5 mm has been found satisfactory. Other alignment techniques that give comparable alignment sensitivity would be considered satisfactory.

X2. TROUBLESHOOTING GUIDE

INTRODUCTION

Appendix X2 is offered in an effort to help a laboratory improve melt flow rate testing and to get to the root cause of problems which may be caused by equipment, environment, or testing technique. This guide is not meant to be an all-inclusive trouble-shooting check list, but merely tries to help users to evaluate testing to some degree.

X2.1 Basic Programs

X2.1.1 The following are basic programs in which all laboratories should participate:

X2.1.1.1 *Standard Reference Materials*—If available, SRMs can usually be obtained from NIST. These SRMs provide accurate information on the melt flow rate of these materials. However, these SRMs are expensive and are available for a very limited number of materials.

X2.1.1.2 *Internal Controls*—An internal control for each type of material should be set up. This involves setting aside enough material to last a long time (at least one year). These materials will have to be tested many times to establish statistical parameters. Each time an internal standard is run, the results should be plotted on a SQC chart so that any problems or trends can be detected quickly. A SQC chart should be set up for each extrusion plastometer in the laboratory. A replacement standard should be introduced before the old standard runs out and should be compared to the old standard to ensure that any shifts seen in the SQC chart are due to the material and not to the equipment.

X2.1.1.3 *Proficiency Tests*—Participation in proficiency test programs is important for demonstrating how the laboratory's results compare with other laboratories. Over a period of time, laboratory bias can be demonstrated, if bias exists.

X2.1.1.4 *ASTM Round Robins (Interlaboratory Tests)*—These programs are similar to proficiency test programs but are limited to only a few laboratories and a few materials. However, these programs do provide information on how well the laboratory performs the test.

X2.1.1.5 *Calibration, Verification, and Maintenance*—The extrusion plastometer should be calibrated. Proper maintenance of the instrument will help to ensure proper calibration.

(*1*) As a first step to obtaining reproducible results, operators should be well trained. Using internal standards can demonstrate that repeatable results can be provided. The operators should also understand the test and know what can affect the results.

(*2*) Before starting the test, the following should be verified:

(*a*) The protocol is understood by the operator.

(*b*) The barrel, piston, and die orifice have been properly cleaned.

(*c*) The extrusion plastometer, including the piston and die, are at equilibrium at the proper temperature.

(*d*) The extrusion plastometer is level.

(*e*) A standard has been run and the results fall within established parameters.

(*3*) When a problem arises, the following questions should be asked:

(*a*) Did anything unusual happen?

(*b*) Does the extrudate contain air bubbles?

(*c*) Were the proper weights applied?

(*d*) Is the unit at the proper temperature?

(*e*) Was the piston stored in the barrel?

(*f*) Is the die damaged, that is, chipped?

(*g*) Is the die bore worn, that is, the diameter is larger than the maximum specified?

(*h*) Was the proper amount of material used?

(*i*) Has the balance been properly calibrated?

(*j*) Was the proper purge time used?

(*k*) Was the plug pulled at the proper time?

(*l*) Was the correct warm-up time used?

(*m*) Was the barrel cleaned properly?

(*n*) Is the piston rod straight?

(*o*) Is the piston tip diameter OK?

X2.2 Understanding How Melt Flow Rate is Affected

X2.2.1 *Levelness of the Instrument*—The piston must be free to move in a vertical position. If the instrument is not level, the piston can be slowed by friction as it touches the side of the barrel. This will not only introduce an error into the results but may also scratch the barrel. The piston must move in an exact vertical plane, indicated by a small bubble level that should be placed on the top of the barrel or on the top of the piston when placed in the barrel. The level should be checked on a regular schedule.

X2.2.2 *Die Orifice Diameter*—An undersized die orifice (which can result from a buildup of residue) will cause low results. Conversely, an oversized orifice (which can result from wear) will cause high results. It is important that the die orifice be cleaned after each test and that the die orifice diameter be verified frequently using a calibrated go/no-go gage. Remember, the calibrated pin gage can also wear and the diameter should be verified regularly.

X2.2.3 *Die Cleanliness*—The die should be completely cleaned after each test. Any residue left in the orifice will eventually char and be very difficult, if not impossible, to clean. Buildup of material will reduce the diameter of the die and change the surface smoothness, resulting in erroneous results.

X2.2.4 *Temperature in the Barrel*—Melt flow rates are very dependent on temperature. The temperature within the barrel is the only important temperature. Temperature indicators must be calibrated to the temperature within the barrel. High flow rates will result from high temperatures and low flow rates from low temperatures. Defective heaters may be difficult to detect and can cause variable results.

X2.2.5 *Preheat Time*—Proper preheat time is required to allow the material in the barrel to fully melt and to come to temperature equilibrium throughout the barrel. If material is not fully melted, die plugging and low melt flow rates can result. If not at temperature equilibrium, the melt flow rate will change as the test is conducted.

X2.2.5.1 It is important that the die and piston be at temperature equilibrium with the rest of the system before any sample is introduced into the barrel. If the piston and die are not at temperature, the large mass of metal involved must be heated to temperature during testing and the energy required may not allow the polymer to reach thermal equilibrium. This can result in erroneous data.

X2.2.6 *Barrel Condition*—The barrel should be properly cleaned after each test. Failure to do so can result in contamination of the next sample, and buildup and degradation of material in the barrel, resulting in a decrease in the diameter of the barrel. This can cause friction with the piston tip resulting in low melt flow rates.

X2.2.6.1 Frequently overlooked is the condition of the barrel itself. In addition to being clean, the barrel wall must be smooth and of the proper diameter. The inside diameter of the barrel is as important as the diameter of the die orifice. The barrel diameter should be measured regularly, and changed if so indicated. The melt flow rate is a function of the fourth power of the barrel diameter.

X2.2.7 *Piston Parameters*—The diameter of the piston foot should be checked. If it is worn, material can flow back past the tip. This would result in erroneous data.

X2.2.7.1 The piston tip, or foot, is sometimes screwed into the end of the piston and can be changed easily. However, because it is easily unscrewed, it can work itself loose. The piston tip must be checked frequently and kept tightly screwed into the piston.

X2.2.7.2 Care must be taken not to bend a piston. Even a slight, almost non-detectable curvature in the piston can result in the force not being applied directly to the vertical position, resulting in excessive pressure on the wall of the barrel. This will cause low results and can also scratch the barrel wall.

X2.2.7.3 The piston has two reference marks. The lower scribe mark on the piston must be at the reference start position (top of the guide ring or barrel) at 7.0 ± 0.5 min, as stated in 9.8 and 10.2.10. Starting each test at a different position can give variable results.

X2.2.8 *Sample Mass*—Small variations in sample mass can cause significant variability in melt flow rates. Any balance used to weigh the sample should be calibrated and verified on a regular schedule.

X2.2.9 *Moisture in Sample*—Samples should be dried before testing. Some materials may be affected more than others by moisture. However, the presence of moisture during the test, in general, will affect the melt flow rates of most materials.

X2.2.10 *Sample Purge Time*—Purging material from the barrel before the actual test starts serves two primary functions: (*1*) to expel entrapped air or volatiles before applying the full test load, and (*2*) to move the lower scribe mark on the piston to the reference start position. For flow rate consistency, it is important that the extrudate be free of voids and that the test always starts with the piston in the same position in the barrel. Whether in the extrudate for Procedure A or in the barrel for Procedure B, voids will affect test results.

X2.2.11 *Load Weight*—The actual weight (load) applied to the material during the test will affect the test results. Higher weights will produce higher results. The load weight, which includes the piston weight, should be verified at regular intervals.

X2.2.12 *Extrudate Cut Technique*—It is important that good extrudate cutting technique be developed. A sharp, clean tool should always be used for this operation. The timing of the cut is critical since a shorter than target cut time will produce low results and a longer than target cut time will give high results. The timing intervals should be verified with certified timers. When cut, the extrudate end should not be ragged or stringy. These variables, if not controlled, can cause poor reproducibility of test results.

X2.2.13 *Purging*—Purging the barrel between runs or when changing materials is sometimes necessary. Purging with the same material which will be tested after the purge is best. However, if for some reason another purge material is used, run the test material through the barrel before the actual test run in order to ensure that the purge material is no longer in the barrel.

X2.2.13.1 Purging does not replace cleaning. After purging, the equipment must be cleaned properly to avoid the effects of contamination, resin buildup, and so forth, as discussed previously.

X2.2.14 *Melt Density*—It is important that the correct melt density be used, that is, 0.7636 g/cm^3 for PE at 190°C and 0.7386 g/cm^3 for PP at 230°C. These values may be different for copolymers or when additives are incorporated into the resin. Small errors in these values can affect the end results. If not known, the melt density can be determined as described at the end of Footnote A of Table 2.

X2.2.15 *Piston (Flag) Travel Distance*—The setting of the proper piston travel distance (6.35 ± 0.25 mm for MFRs up to 10 g/10 min and 25.40 ± 0.25 mm for MFRs greater than 10 g/10 min) is important in Procedure B. The reproducibility of Procedure B is better if these parameters are strictly adhered to. A calibrated distance verification device will be required to maintain the proper piston travel.

X2.2.16 *Calculation Factors*—When trouble shooting, always check the calculations and the factors used.

X2.2.17 *Power (Electrical) Fluctuation*—Constant power (voltage) is important to maintain the temperature desired. Periodic changes in voltage will cause changes in the temperature of the unit, creating test values that fluctuate because of inconsistent sample temperatures. Though this is a rare situation, this fluctuation has been found to cause erratic test results.

REFERENCES

(**1**) "Polyethylene Insulation and Sheathing for Electrical Cables," *Government Department Electrical Specification No. 27*, Great Britain, 1950.

(**2**) Tordella, J. P., and Jolly, R. E., "Melt Flow of Polyethylene," *Modern Plastics*, Vol 31, No. 2, 1953, p. 146.

(**3**) Dexter, F. D., "Plasticity Grading of Fluorothenes," *Modern Plastics*, Vol 30, No. 8, 1953, p. 125.

(**4**) Harban, A. A., and McClamery, R. M., "Limitations on Measuring Melt Flow Rates of Polyethylene and Ethylene Copolymers by Extrusion Plastometer," *Materials Research and Standards*, Vol 3, No. 11, 1963, p. 906.

(**5**) Rudin, A., and Schreiber, H. P., "Factors in Melt Indexing of Polyolefins," *SPE Journal*, Vol 20, No. 6, 1964, p. 533.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 1238 – 04:

(*1*) Editorially added sulfone plastic melt flow rate conditions as found in Specification D 6394 for Sulfone Plastics (SP).

(*2*) Additional melt flow rate conditions for polyether sulfone and polyphenylsulfone placed in 8.2.

D 1238 – 01:

(*1*) Procedure C for high flow polyolefins including precision data for the procedure, as well as Table 9 in Section 14 was added.

D 1238 – 00:

(*1*) Added Appendix X2, Troubleshooting Guide.

D 1238-99:

(*1*) Revised title to include the word "melt."

(*2*) Added conditions for polycaprolactone to 8.2 and Table 1.

(*3*) In Note 21, replaced "melt indexer" with "extrusion plastometer."

(*4*) In Note 25, replaced incorrect reference to 10.2.9 with 10.2.8.

(*5*) Editorially corrected equation in Footnote A of Table 2.

D 1238-98:

(*1*) Revision to Section 14.

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Designation: D 6110 – 04

Standard Test Method for Determining the Charpy Impact Resistance of Notched Specimens of Plastics[1]

This standard is issued under the fixed designation D 6110; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method is used to determine the resistance of plastics to breakage by flexural shock as indicated by the energy extracted from standardized (see Note 1) pendulum-type hammers, mounted in standardized machines, in breaking standard specimens with one pendulum swing. This test method requires specimens to be made with a milled notch (see Note 2). The notch produces a stress concentration which promotes a brittle, rather than a ductile, fracture. The results of this test method are reported in terms of energy absorbed per unit of specimen width (see Note 3).

NOTE 1—The machines with pendulum-type hammers have been standardized in that they must comply with certain requirements including a fixed height of hammer fall, which results in a substantially fixed velocity of the hammer at the moment of impact. Hammers of different initial energies (produced by varying their effective weights), however, are recommended for use with specimens of different impact resistance. Moreover, manufacturers of the equipment are permitted to use different lengths and constructions of pendulums with possible differences in pendulum rigidities resulting (see Section 5). Be aware that other differences in machine design do exist.

NOTE 2—The specimens are standardized in that they have a fixed length and fixed depth, however, the width of the specimens is permitted to vary between limits. One design of milled notch is allowed. The notch in the specimen serves to concentrate the stress, minimize plastic deformation, and direct the fracture to the part of the specimen behind the notch. Scatter in energy-to-break is thus reduced. Because of differences in the elastic and viscoelastic properties of plastics, however, response to a given notch varies among materials.

NOTE 3—Caution must be exercised in interpreting the results of this test method. The following testing parameters have been shown to affect test results significantly: method of specimen fabrication, including but not limited to processing technology, molding conditions, mold design, and thermal treatment; method of notching; speed of notching tool; design of notching apparatus; quality of the notch; time between notching and test; test specimen thickness; test specimen width under notch; and environmental conditioning.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 4—This standard resembles ISO 179 in title only. The content is significantly different.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 618 Practice for Conditioning Plastics for Testing

D 647 Practice for Design of Molds for Test Specimens of Plastic Molding Materials[3]

D 883 Terminology Relating to Plastics

D 4000 Classification System for Specifying Plastic Materials

D 4066 Classification System for Nylon Injection and Extrusion Materials

D 5942 Test Method for Determination of Charpy Impact Strength

D 5947 Test Methods for Physical Dimensions of Solid Plastics Specimens

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods

3. Terminology

3.1 *Definitions*—For definitions related to plastics, see Terminology D 883.

4. Summary of Test Method

4.1 A notched specimen is supported as a horizontal simple beam and is broken by a single swing of the pendulum with the impact line midway between the supports and directly opposite the notch.

5. Significance and Use

5.1 Before proceeding with this test method, refer to the material specification for the material being tested. Any test specimen preparation, conditioning, dimensions and testing parameters required by the materials specification shall take

[1] This test method are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *Discontinued 1994; Replaced by D 1896, D 3419, D 3641, D 4703, D 5227.*

***A Summary of Changes section appears at the end of this standard.**

precedence over those required by this test method. Table 1 of Classification D 4000 lists the ASTM materials standards that currently exist. If there is no material specification, then the requirements of this test method apply.

5.2 The excess energy pendulum impact test indicates the energy to break standard test specimens of specified size under stipulated conditions of specimen mounting, notching (stress concentration), and pendulum velocity at impact.

5.3 For this test method, the energy lost by the pendulum during the breakage of the specimen is the sum of the energies required to initiate fracture of the specimen; to propagate the fracture across the specimen; to throw the free ends of the broken specimen (toss energy); to bend the specimen; to produce vibration in the pendulum arm; to produce vibration or horizontal movement of the machine frame or base; to overcome friction in the pendulum bearing and in the excess energy indicating mechanism, and to overcome windage (pendulum air drag); to indent or deform, plastically, the specimen at the line of impact; and to overcome the friction caused by the rubbing of the striking nose over the face of the bent specimen.

NOTE 5—The toss energy, or the energy used to throw the free ends of the broken specimen, is suspected to represent a very large fraction of the total energy absorbed when testing relatively dense and brittle materials. No procedure has been established for estimating the toss energy for the Charpy method.

5.4 For tough, ductile, fiber-filled, or cloth-laminated materials, the fracture propagation energy is usually large compared to the fracture initiation energy. When testing these materials, energy losses due to fracture propagation, vibration, friction between the striking nose and the specimen has the potential to become quite significant, even when the specimen is accurately machined and positioned, and the machine is in good condition with adequate capacity (see Note 6). Significant energy losses due to bending and indentation when testing soft materials have also been observed.

NOTE 6—Although the frame and the base of the machine should be sufficiently rigid and massive to handle the energies of tough specimens without motion or excessive vibration, the pendulum arm cannot be made very massive because the greater part of its mass must be concentrated near its center of percussion at its striking nose. Locating the striking nose precisely at the center of percussion reduces the vibration of the pendulum arm when used with brittle specimens. Some losses due to pendulum arm vibration (the amount varying with the design of the pendulum) will occur with tough specimens even when the striking nose is properly positioned.

5.5 In a well-designed machine of sufficient rigidity and mass the losses due to vibration and friction in the pendulum bearing and in the excess energy indicating mechanism should be very small. Vibrational losses are observed when wide specimens of tough materials are tested in machines of insufficient mass, or in machines that are not securely fastened to a heavy base.

5.6 Since this test method permits a variation in the width of the specimens and since the width dictates, for many materials, whether a brittle, low-energy break (as evidenced by little or no drawing down or necking and by a relatively low energy absorption) or a ductile, high-energy break (as evidenced by considerable drawing or necking down in the region behind the notch and by a relatively high energy absorption) will occur, it is necessary that the width be stated in the specification covering that material and that the width be stated along with the impact value.

5.7 This test method requires that the specimen break completely. Results obtained when testing materials with a pendulum that does not have sufficient energy to complete the breaking of the extreme fibers and toss the broken pieces shall be considered a departure from standard and should not be reported as a standard result. Impact values cannot be directly compared for any two materials that experience different types of failure.

5.8 The value of this impact test method lies mainly in the areas of quality control and materials specification. If two groups of specimens of supposedly the same material show significantly different energy absorptions, critical widths, or critical temperatures, it is permitted to assume that they were made of different materials or were exposed to different processing or conditioning environments. The fact that a material shows twice the energy absorption of another under these conditions of test does not indicate that this same relationship will exist under another set of test conditions.

6. Apparatus

6.1 *Pendulum Impact Machine*—The machine shall consist of a massive base on which are mounted a pair of supports for holding the specimen and to which is connected, through a rigid frame and bearings, one of a number of pendulum-type hammers having an initial energy suitable for use with the particular specimen to be tested (or one basic pendulum designed to accept add-on weights), plus a pendulum holding and releasing mechanism and a mechanism for indicating the excess energy remaining in the pendulum after breaking specimen. The specimen anvil, pendulum, and frame shall be sufficiently rigid to maintain correct alignment of the striking edge and specimen, both at the moment of impact and during the propagation of the fracture, and to minimize energy losses due to vibration. The base shall be sufficiently massive so that the impact will not cause it to move. The machine shall be designed, constructed, and maintained so that energy losses due to pendulum air drag (windage), friction in the pendulum bearings, and friction and inertia in the excess energy indicating mechanism are held to a minimum.

6.1.1 *Pendulum*—The simple pendulum shall consist of a single or multi-membered arm with a bearing on one end and a head, containing the striking nose, on the other. Although a large proportion of the mass of the pendulum should be concentrated in the head, the arm must be sufficiently rigid to maintain the proper clearances and geometric relationships between the machine parts and the specimen and to minimize vibrational energy losses, which are always included in the measured impact value. A machine with a simple pendulum design is illustrated in Fig. 1. Instruments with a compound-pendulum design also have been found to be acceptable for use. A compound-pendulum design is illustrated in Fig. 2.

6.1.1.1 The machine shall be provided with a basic pendulum capable of delivering an energy of 2.7 ± 0.14 J [2.0 ± 0.10 ft-lbf]. This pendulum shall be used for specimens that extract less than 85 % of this energy when breaking a specimen. Heavier pendulums or additional weights designed to

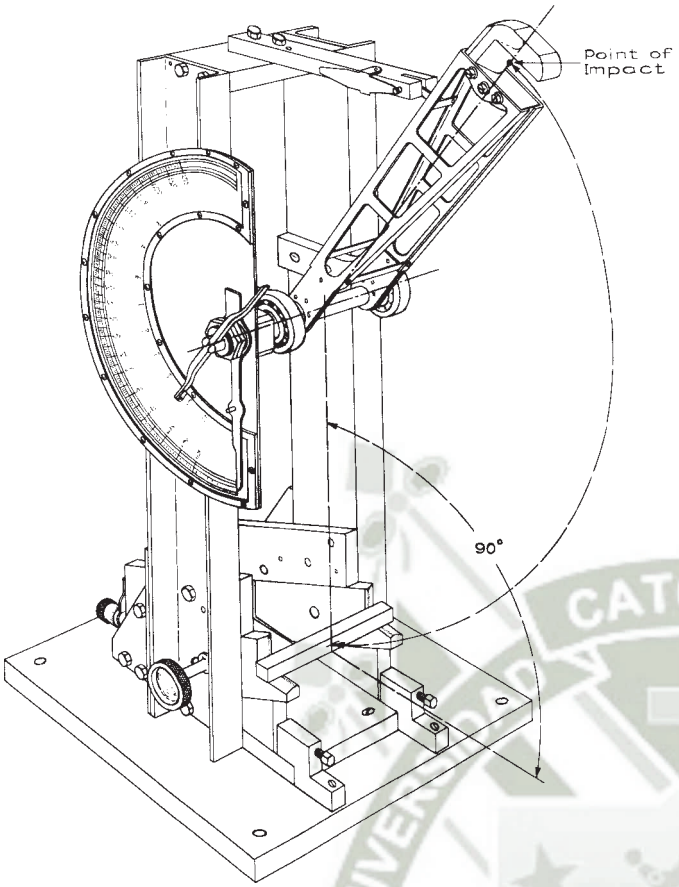


FIG. 1 Simple Beam (Charpy-Type) Impact Machine

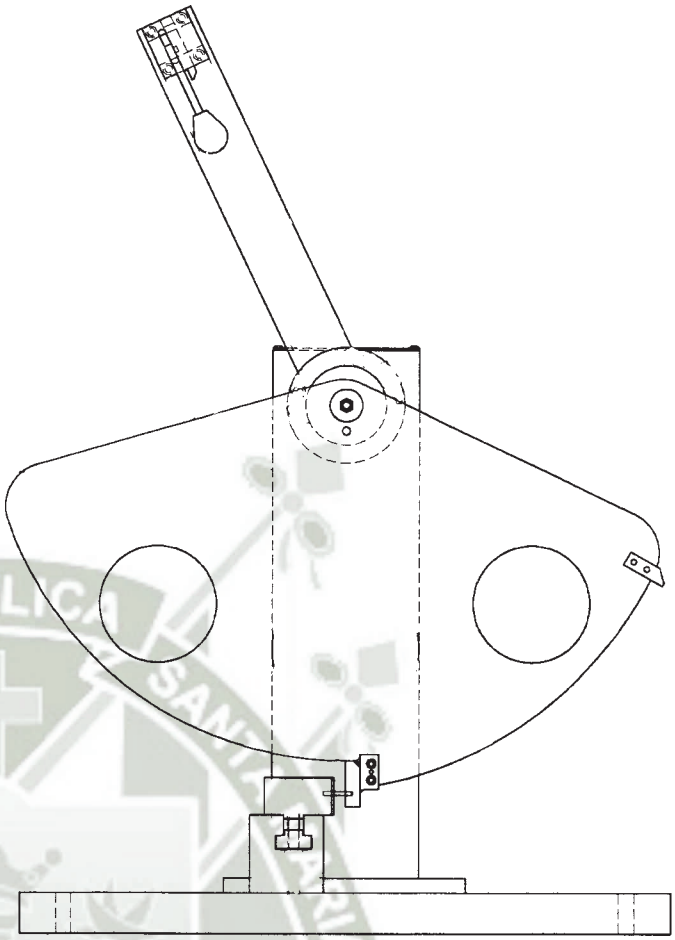


FIG. 2 Example of Compound–Pendulum–Type Machine

attach to the basic pendulum shall be provided for specimens that require more energy to break. A series of pendulums such that each has twice the energy of the next lighter one has been found convenient.

6.1.1.2 The effective length of the pendulum shall be between 0.325 and 0.406 m [12.8 an 16.0 in.] so that the required elevation of the striking nose is obtained by raising the pendulum to an angle between 60 and 30° above the horizontal.

6.1.2 *Striking Edge*—The striking edge (nose) of the pendulum shall be made of hardened steel, tapered to have an included angle of 45 ± 2° and shall be rounded to a radius of 3.17 ± 0.12 mm [0.125 ± 0.005 in.]. The pendulum shall be aligned in such a way that when it is in its free hanging position, the center of percussion of the pendulum shall lie within ±2.54 mm [0.10 in.] of the middle of the line of contact made by the striking nose upon the face of a standard specimen of square cross section. The distance from the axis of support to the center of percussion is determined experimentally from the period of motion of small amplitude oscillations of the pendulum by means of the following equation:

$$L = (g/4\pi^2)\, p^2 \quad (1)$$

where:

L = distance from the axis of support to the center of percussion, m,

g = local gravitational acceleration (known to an accuracy of one part in one thousand), m/s^2

π = 3.1416 ($4\pi^2$= 39.48), and

p = period, in s, of a single complete swing (to and fro) determined from at least 20 consecutive and uninterrupted swings. The angle of swing shall be less than 5° each side of center.

6.1.3 *Pendulum Holding and Releasing Mechanism*—The mechanism shall be designed, constructed, and operated so that it will release the pendulum without imparting acceleration or vibration to the pendulum. The position of the pendulum holding and releasing mechanism shall be such that the vertical height of fall of the striking nose shall be 610 ± 2 mm [24.0 ± 0.005 in.]. This will produce a velocity of the striking nose at the moment of impact of approximately 3.46 m [11.4 ft]/s as determined by the following equation:

$$v = \sqrt{2gh} \quad (2)$$

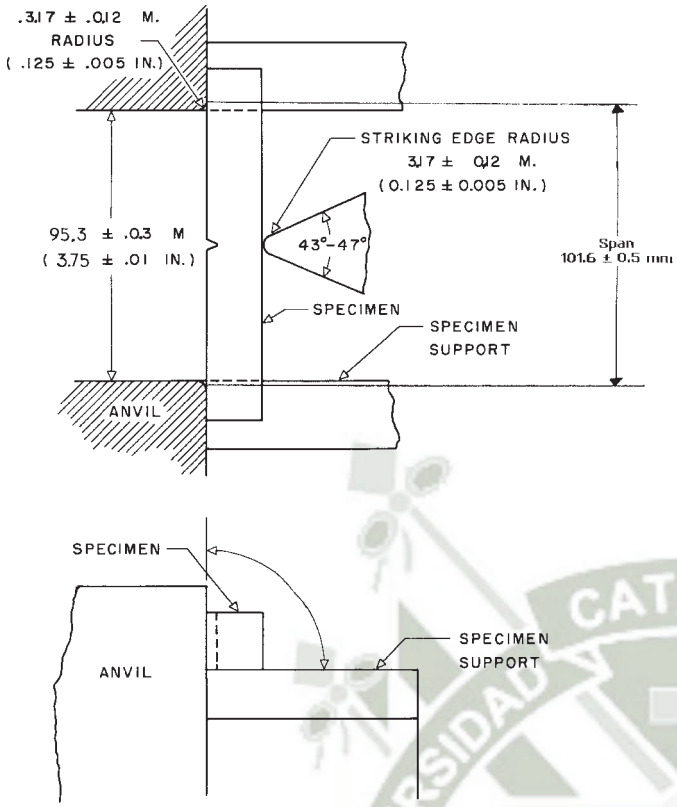


FIG. 3 Relationship of Anvil, Specimen, and Striking Edge to Each Other for Charpy Test Method

where:

v = velocity of the striking nose at the moment of impact,
g = local gravitational acceleration, and
h = vertical height of fall of the striking nose.

This assumes no windage or friction.

6.1.4 *Specimen Supports*—The test specimen shall be supported against two rigid anvils in such a position that its center of gravity and the center of the notch shall lie on tangent to the arc of travel of the center of percussion of the pendulum drawn at the position of impact. The edges of the anvils shall be rounded to a radius of 3.17 ± 0.12 mm [0.125 ± 0.005 in.] and the anvils' lines of contact (span) with the specimen shall be 101.6 ± 0.5 mm [4.0 ± 0.02 in.] apart (see Fig. 3). A jig for positioning the specimen on the supports should be supplied by the machine manufacturer.

NOTE 7—Some machines currently in use employ a 108.0–mm span. Data obtained under these conditions are valid.[4]

6.1.5 *Excess Energy Indicator*—Means shall be provided for determining energy remaining in the pendulum after breaking a specimen. One acceptable method is the use of a pointer and dial mechanism which indicate the height of rise of the pendulum beyond the point of impact in terms of energy removed from that specific pendulum. More modern instruments use an electronic digital display or computer to measure the energy loss and indicate the breaking energy of the specimen. The indicated remaining energy must be corrected for pendulum bearing friction, pointer friction, pointer inertia, and pendulum windage. The equipment manufacturer should provide graphs or tables to aid in the calculation of the correction friction and windage. Instructions for making these corrections are found in Annex A1 and Annex A2. Many digital indicating systems automatically correct for windage and friction. The equipment manufacturer should be consulted for information on how this is performed.

6.1.6 The calibration procedure in Appendix X2 should be used to establish the accuracy of the equipment. A check of the calibration of an impact machine is difficult to make under dynamic conditions. The basic parameters normally are checked under static conditions. If the machine passes the static tests, then it is assumed to be accurate. Appendix X2, however, also describes a dynamic test for checking certain features of the machine and specimen. For some machine designs, it might be necessary to change the recommended method of obtaining the required calibration measurements. Additional instructions for adjusting a particular machine should be supplied by the manufacturer. Other methods of performing the required checks are acceptable provided that they are proven to result in an equivalent accuracy.

6.2 *Specimen Notching Machine*—Notching shall be done on a milling machine, engine lathe, or other suitable machine tool. A carbide-tipped or industrial diamond-tipped notching cutter is recommended. Both cutter speed and feed rate shall be controllable. Provision for cooling the specimen is recommended. Water and compressed air are suitable coolants for many plastics.

6.2.1 The profile of the cutting tooth or teeth shall be such as to produce a notch in the test specimen of the contour and depth specified in Fig. 4 and in the manner specified in Section 8.

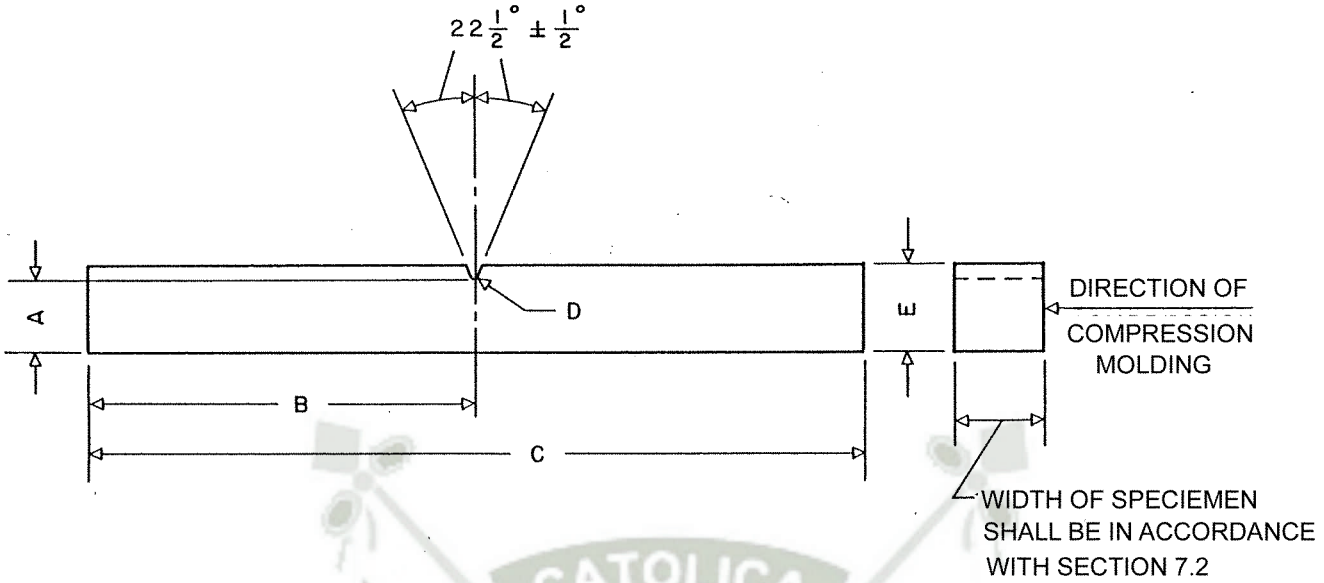
6.2.2 A single-tooth cutter shall be used for notching the specimen, unless it is demonstrated that notches of an equivalent quality are produced with a multi-tooth cutter. Single-tooth cutters are preferred because of the ease of grinding the cutter to the specimen contour and because of the smoother cut on the specimen. The cutting edge shall be ground and honed carefully to ensure sharpness and freedom from nicks and burrs. Tools with no rake and a work relief angle of 15 to 20° have been found satisfactory.

6.3 *Micrometers*—Apparatus for measurement of the width of the specimen shall comply with the requirements of Test Methods D 5947. Apparatus for the measurement of the depth of plastic material remaining in the specimen under the notch shall comply with requirements of Test Methods D 5947, provided however that the one anvil or presser foot shall be a tapered blade conforming to the dimensions given in Fig. 5. The opposing anvil or presser foot shall be flat and conforming to Test Methods D 5947.

7. Test Specimen

7.1 The test specimen shall conform to the dimensions and geometry of Fig. 4, except as modified in accordance with 7.2-7.5. To ensure the correct contour and conditions of the specified notch, all specimens shall be notched in accordance with Section 8.

[4] Supporting data is available from ASTM Headquarters. Request Research Report RR: D20–1033.



A	10.16 ± 0.05	0.400 ± 0.002
B	63.5 max	2.50 max
	61.0 min	2.40 min
C	127.0 max	5.00 min
	124.5 min	4.90 min
D	0.25R ± 0.05	0.010R ± 0.002
E	12.70 ± 0.15	0.500 ± 0.006

FIG. 4 Dimensions of Simple Beam, Charpy Type, Impact Test Specimen

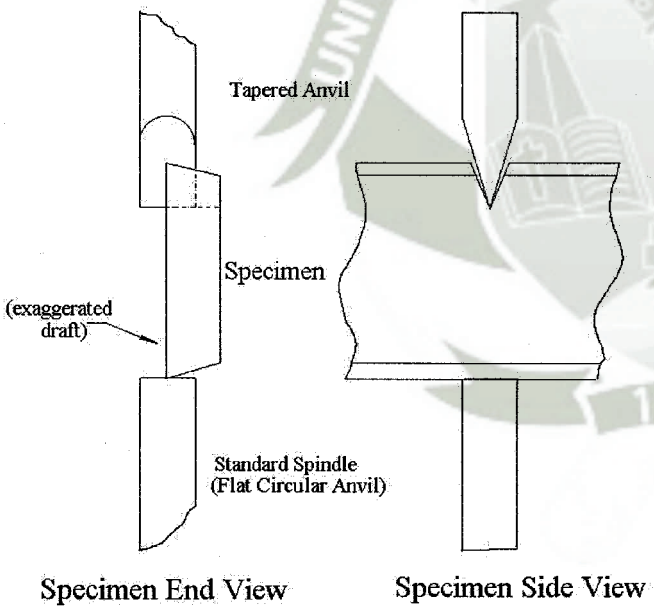


FIG. 5 Notch Depth Measurement on Test Specimens

7.2 Molded specimens shall have a width between 3.00 and 12.7 mm [0.118 and 0.500 in.]. Use the specimen width as specified in the material specification or as agreed upon between the supplier and the customer.

7.2.1 The type of mold and molding machine used and the flow behavior in the mold cavity will influence the strength obtained. It is possible that results from a specimen taken from one end of a molded bar will give different results than a specimen taken from the other end. Cooperating laboratories should therefore agree on standard molds conforming to Practice D 647, and upon a standard molding procedure for the material under investigation.

7.2.2 A critical investigation of the mechanics of impact testing has shown that tests made upon specimens under 6.35 mm [0.250 in.] in width absorb more energy due to crushing, bending, and twisting than do wider specimens. Specimens 6.35 mm [0.250 in.] or over in width are therefore recommended. The responsibility for determining the minimum specimen width shall be the investigator's, with due reference to the specification for that material.

7.2.3 The impact resistance of a plastic material will be different if the notch is perpendicular to, rather than parallel to, the direction of molding.

7.3 For sheet materials, the specimens shall be cut from the sheet in both the lengthwise and crosswise directions unless otherwise specified. The width of the specimen shall be the thickness of the sheet if the sheet thickness is between 3.00 and 12.7 mm [0.118 and 0.500 in.]. Sheet material thicker than 12.7 mm [0.500 in.] shall be machined down to 12.7 mm [0.500 in.]. It is acceptable to test specimens with a 12.7–mm [0.500–in.] square cross section either edgewise or flatwise as cut from the sheet. When specimens are tested flatwise, the notch shall be made on the machined surface if the specimen is machined on one face only. When the specimen is cut from a thick sheet, notation shall be made of the portion of the thickness of the sheet from which the specimen was cut, for example, center, top, or bottom surface.

7.3.1 The impact resistance of a plastic material will be different if the notch is perpendicular to, rather than parallel to, the grain of an anisotropic bar cut from a sheet. Specimens cut from sheets that are suspected of being anisotropic shall be prepared and tested both lengthwise and crosswise to the direction of the anisotropy.

7.4 The practice of cementing, bolting, clamping, or otherwise combining specimens of substandard width to form a composite test specimen is not recommended and should be avoided since test results will be seriously affected by interface effects or effects of solvents and cements on energy absorption of composite test specimens, or both. If Charpy test data on such thin materials are required, however, and if possible sources of error are recognized and acceptable, the following technique of preparing composites ought to be utilized. The test specimens shall be a composite of individual thin specimens totaling 6.35 to 12.7 mm [0.125 to 0.500 in.] in width. Individual members of the composite shall be aligned accurately with each other and clamped, bolted, or cemented together. Care must be taken to select a solvent or adhesive that will not affect the impact resistance of the material under test. If solvents or solvent–containing adhesives are employed, a conditioning procedure shall be established to ensure complete removal of the solvent prior to test. The composite specimens shall be machined to proper dimensions and then notched. In all such cases, the use of composite specimens shall be noted in the report of test results.

7.5 Each specimen shall be free of twist and shall be bounded by mutually perpendicular pairs of plane, paralleled surfaces and free from scratches, pits, and sink marks. The specimens shall be checked for conformity with these requirements by visual observation against straight edges, squares or flat plates, and by measuring with micrometer calipers. Any specimen showing observable or measurable departure from one or more of these requirements shall be rejected or machined to the proper size and shape before testing. A specimen that has a slight twist to its notched face of 0.05 mm [0.002 in.] at the point of contact with the pendulum striking edge will be likely to have a characteristic fracture surface with considerable greater fracture area than for a normal break. In this case, the energy to break and toss the broken section will be considerably larger (20 to 30 %) than for a normal break.

8. Notching Test Specimens

NOTE 8—When testing a material for the first time, it is necessary to study the effect of all variations in the notching conditions, including cutter dimensions, notch depth, cutter speed, and feed rate. To establish that the notching parameters are suitable, it is advisable to notch several specimens of the material and inspect both the tool entrance and tool exit side of each notched specimen, in accordance with Appendix X1. Adjust the notching machine as required. The specimens used to determine notching conditions shall not be used to make determinations of impact resistance.

8.1 *Notch Dimensions*—The included angle of the notch shall be 45 ± 1° with a radius of curvature at the apex of 0.25 ± 0.05 mm [0.010 ± 0.002 in.]. The plane bisecting the notch angle shall be perpendicular to the face of the test specimen within 2°.

8.1.1 The notch is a critical factor of this test. It is extremely important, therefore, that dimensions of the notch in the specimen are verified. There is evidence that the contour of notches cut in materials of widely differing physical properties by the same cutter will differ. It is sometimes necessary to alter the cutter dimensions in order to produce the required notch contour for certain materials.

8.1.2 Both the notch and the cutter used to make the notch shall be inspected, at a minimum, after every 500 notches. The specimen used to verify the notch shall be the same material that is being prepared for testing. Follow the procedure in Appendix X1 when inspecting and verifying the notch in the specimen. If the angle or radius of the notch does not meet the requirements of 8.1, the cutter should be replaced.

NOTE 9—The contour of the notch made using multi-tooth cutters is checked by measuring the contour of the notch on a strip of soft metal that is inserted between two specimens during the notching process.

NOTE 10—When the same material is being tested on a repetitive basis, and it is demonstrated that the notch in the specimen takes the contour of the tip of the cutter and that the notch meets the contour requirements when checked in accordance with Appendix X1, then it is acceptable to check the contour of the tip of the cutter instead of the notch in the specimen.

8.2 *Notch Depth*—The depth of the plastic material remaining in the specimen under the notch shall be 10.16 ± 0.05 mm [0.400 ± 0.002 in.]. This dimension shall be measured with apparatus in accordance with 6.3. The tapered blade will be fitted to the notch. The specimen will be approximately vertical between the anvils. Position the edge of the non-cavity (wider edge) surface centered on the micrometer's flat circular anvil.

8.3 *Cutter Speed and Feed Rate*—The cutter speed and feed speed should be selected based on the material being tested. The quality of the notch will be adversely affected by thermal deformations and stresses induced during the cutting operation if proper conditions are not selected.[5] The notching parameters used shall not alter the physical state of the material, such as by raising the temperature of a thermoplastic above its glass transition temperature.

8.3.1 In general, high cutter speeds, slow feed rates, and lack of coolant induce more thermal damage than a slow cutter speed, fast feed speed, and the use of a coolant. Too high a feed speed/cutter speed ratio, however, has been shown to cause impacting and cracking of the specimen. The range of cutter speed/feed ratios possible to produce acceptable notches has been shown to be extended by the use of a suitable coolant.

8.3.1.1 For some thermoplastics, suitable notches have been produced using cutter speeds from 54 to 150 m/min and a feed rate of 89 to 160 mm/min without a water coolant. Satisfactory notches also have been produced using the same cutter speeds at feed speeds of from 36 to 160 mm/min with water coolant.

8.3.1.2 Embedded thermocouples have been used to determine the temperature rise in the material near the apex of the notch during machining. Thermal stresses induced during the notching operation have been observed in transparent materials by viewing the specimen at low magnification between crossed

[5] Supporting data is available from ASTM Headquarters. Request Research Report RR: D20–1066.

polars in monochromatic light. The specimens used to determine temperature rise shall not be used to make determinations of impact resistance.

8.3.2 The feed rate and the cutter speed shall remain constant throughout the notching operation.

8.4 It is acceptable to notch specimens individually or in a group. In either case, however, an unnotched backup or dummy bar shall be placed behind the last specimen in the sample holder to prevent distortion and chipping by the cutter as it exits from the last test specimen.

8.5 All specimens having one dimension less than 12.7 mm [0.500 in.] shall have the notch cut on the shorter side. Compression molded specimens shall be notched on the side parallel to the direction of application of molding pressure. The impact resistance of a plastic material will be different if the notch is perpendicular to rather than parallel to the direction of molding, as with or across the grain of an anisotropic bar cut from a plate.

9. Conditioning

9.1 Check the materials specification for the material that is being tested. If there are no conditioning requirements stated by the materials specification, the test specimens shall be conditioned at 23 ± 2°C [73 ± 3.6°F] and 50 ± 5 % relative humidity for not less than 40 h after notching and prior to testing in accordance with Procedure A of Practice D 618. unless documented (between supplier and customer) that shorter conditioning time is sufficient for a given material to reach equilibrium of impact resistance.

9.2 For hygroscopic materials, such as nylons, the material specifications (for example, Classification System D 4066) call for testing dry–as–molded specimens. Such requirements take precedence over the above routine preconditioning to 50 % relative humidity. These specimens shall be sealed in water vapor-impermeable containers as soon as molded. When notching these specimens, minimize the exposure time during notching and return the specimens to a dry container after notching to allow for full cooling of the specimens prior to testing.

9.3 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C [73 ± 3.6°F] and 50 ± 5 % relative humidity, unless otherwise specified. In cases of disagreement, the tolerances shall be ±1°C and ±2 % relative humidity.

10. Procedure

10.1 *Specimen Preparation*:

10.1.1 Prepare the test specimens in accordance with the procedures in Section 7. At least five and preferably ten or more individual determinations of impact resistance shall be made to determine the average impact resistance for a particular sample. The specimens shall be of nominal width only.

10.1.2 Notch the specimens in accordance with the procedure in Section 8.

10.1.3 Condition the specimens in accordance with the materials specification for the material that is being tested. If there are no conditioning requirements detailed in the materials specification, follow the conditioning requirements in Section 9.

10.2 *Machine Preparation*:

10.2.1 Estimate the breaking energy for the sample and select a pendulum of suitable energy. Select the lightest standard pendulum that is expected to break all specimens in the group with an energy loss of not more than 85 % of its capacity (see 6.1). If the breaking energy cannot be estimated, the correct pendulum can be determined by performing trial runs. Caution should be used to avoid damaging the pendulum by selecting a pendulum that is too light for a particular sample.

NOTE 11—Ideally, an impact test would be conducted at a constant test velocity. In a pendulum-type test, however, the velocity decreases as the fracture progresses. For specimens that have an impact energy approaching the capacity of the pendulum, there is insufficient energy to complete the break and toss. By avoiding the higher 15 % scale energy readings, the velocity of the pendulum will not be reduced below 1.33 m/s. On the other hand, the use of a pendulum that is too heavy would reduce the sensitivity of the reading.

10.2.2 After installing the selected pendulum on the machine, check the machine for conformity with the requirements of Section 6 before starting the tests.

10.2.3 When using a machine equipped with a pointer and dial mechanism or an electronic indicator that does not automatically correct for windage and friction, determine the windage and friction correction factors for the machine before testing specimens. Windage and friction correction factors shall be determined on a daily basis and shall be calculated each time weights are added to the pendulum or the pendulum is changed. Refer to Annex A1 for information on constructing windage and friction correction charts or refer to Annex A2 for a procedure to calculate the windage and friction correction. If excessive friction is indicated (see X2.12 and X2.13) the machine shall be adjusted before testing specimens. Follow the machine manufacturer's instructions to correct for excessive windage and friction.

NOTE 12—The actual correction factors for windage and friction will be smaller than these factors in an actual test because the energy absorbed by the specimen prevents the pendulum from making a full swing. The indicated breaking energy of the specimen, therefore, must be included in the calculation of the machine correction.

10.2.4 Some machines equipped with an electronic digital display or computer automatically compensate for windage and friction.

10.3 *Specimen Testing*:

10.3.1 Check all of the specimens in the sample group for conformity with the requirements of Sections 7 and 8 and 10.1.

10.3.2 Measure and record the width of each specimen after notching to the nearest 0.025 mm [0.001 in]. Measure the width in one location adjacent to the notch centered about the anticipated fracture plane.

10.3.3 Measure and record the depth of material remaining in the specimen under the notch of each specimen to the nearest 0.025 mm [0.001 in]. The tapered blade will be fitted to the notch. The specimen will be approximately vertical between the anvils. Position the edge of the non-cavity (wider edge) surface so that it is centered on the micrometer's flat circular anvil. See Fig. 5.

10.3.4 Position a test specimen horizontally on the supports and against the anvils so that it will be impacted on the face

opposite the notch (see Fig. 3). The notch should be centered between the anvils. A centering jig is useful for this purpose.

10.3.5 Raise and secure the pendulum in the release mechanism. Zero the excess energy indicating mechanism.

10.3.6 Release the pendulum, allowing the striking edge of the pendulum to impact the specimen. Note the indicated breaking energy.

10.3.7 Calculate the net breaking energy (see 11.1). If the net breaking energy is greater than 85 % of the pendulum's nominal energy, the wrong pendulum was used. Discard the result. Select and install a pendulum with a greater available energy or add additional weight to the pendulum, determine the windage and friction correction factor, and repeat the test on a new specimen.

10.3.8 If the proper pendulum was used, test the remaining specimens as described in 10.3.1-10.3.6. Results from specimens that do not break should be discarded. A specimen that does not break completely into two or more pieces is not considered to be broken.

10.3.9 After all of the specimens for the sample have been tested, calculate the impact resistance, in joules per metre, for each individual specimen (see 11.2).

10.3.10 Calculate the average impact resistance for the group of specimens (see 11.3). Values obtained from specimens that did not break completely shall not be included in the average.

10.3.11 Calculate the standard deviation for the group of specimens (see 11.4).

11. Calculation

11.1 *Net Breaking Energy*—Subtract the windage and friction loss energy from the indicated breaking energy.

11.2 *Impact Resistance*—Divide the net breaking energy by the measured width of each individual specimen.

11.3 Calculate the average impact resistance for a group of specimens by adding the individual impact resistance values for the group and dividing the sum by the total number of specimens in the group.

11.4 Calculate the standard deviation as follows and report it to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\,\bar{X}^2/(n-1)} \quad (3)$$

where:

s = estimated standard deviation,
X = value of single observation,
n = number of observations, and
$\bar{X}$ = arithmetic mean of the set of observations.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type source, manufacturer's code number, and previous history.

12.1.2 A statement of how the specimens were prepared, the testing conditions used, the number of hours the specimens were conditioned after notching, and for sheet materials, the direction of testing with respect to anisotropy, if any.

12.1.3 The capacity of the pendulum, J.

12.1.4 The span.

12.1.5 The width and depth under the notch of each specimen tested.

12.1.6 The total number of specimens tested per sample of material (that is five, ten, or more).

12.1.7 The average impact resistance, J/m. Impact resistance is not to be reported for other than complete breaks. Reporting results in kJ/m^2 is optional (see Appendix X4).

12.1.8 The standard deviation of the values of the impact resistance of the specimens in 10.3.11.

13. Precision and Bias

13.1 Table 1 is based on a round robin[6] conducted in 1987 in accordance with Practice E 691, involving five materials tested by nine laboratories. For each material, all samples were prepared at one source, but the individual specimens were notched and conditioned at the laboratories which tested them. Each laboratory tested an average of nine specimens for each material. **Warning—**The explanations of *r* and *R* (13.2-13.2.3) are intended only to present a meaningful way of considering the approximate precision of this test method. The data presented in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their materials and laboratory, or between specific laboratories. The principles of 13.2-13.2.3 would then be valid for such data.

13.2 *Concept of r and R in Table 1* —If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing nine specimens for each test result, then:

13.2.1 *Repeatability—r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the

[6] Supporting data is available from ASTM Headquarters. Request Research Report RR: D20–1041 and 1134.

TABLE 1 Precision for Charpy Test

Values in ft·lbf/in. of Width

Material	Average	S_r^A	S_R^B	r^C	R^D	Number of Laboratories
Phenolic	0.55	0.029	0.050	0.08	0.14	7
Reinforced nylon	1.98	0.065	0.143	0.18	0.40	7
Polycarbonate	2.85	0.083	0.422	0.23	1.19	8
Polypropylene	4.06	0.151	0.422	0.42	1.19	9
ABS	10.3	0.115	0.629	0.32	1.78	9

[A] S_r = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test result from all of the participating laboratories:

$$S_r = [\,[(S_1)^2 + (S_2)^2 \ldots + (Sn)^2]\,/n]^{1/2}$$

[B] S_R = between-laboratories reproducibility, expressed as standard deviation:

$$S_R = [\,S_r^2 + S_L^2\,]^{1/2}$$

where S_L = standard deviation of laboratory means.
[C] r = within-laboratory critical interval between two test results = 2.8 × S_r.
[D] R = between laboratories critical interval between two test results = 2.8 × S_R.

same day in the same laboratory. Two tests results shall be judged not equivalent if they differ by more than the *r* value for that material.

13.2.2 *Reproducibility—R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories, not necessarily on the same day. Two test results shall be judged not equivalent if they differ by more than the *R* value for that material.

13.2.3 Any judgement in accordance with 13.2.1 or 13.2.2 would have an approximate 95 % (0.95) probability of being correct.

13.3 There are no recognized standards by which to estimate bias of this test method.

14. Keywords

14.1 Charpy impact; impact resistance; notch sensitivity; notched specimen

ANNEXES

(Mandatory Information)

A1. INSTRUCTIONS FOR THE CONSTRUCTION OF A WINDAGE AND FRICTION CORRECTION CHART

A1.1 The construction and use of the chart herein described is based upon the assumption that the friction and windage losses are proportional to the angle through which these loss torques are applied to the pendulum. Fig. A1.1 shows the assumed energy loss versus the angle of the pendulum position during the pendulum swing. The correction chart to be described is principally the left half of Fig. A1.1. The windage and friction correction charts should be available from commercial testing machine manufacturers. The energy losses designated as A or B are described in 10.3.

A1.2 Start the construction of the correction chart (Fig. A1.2) by laying off to some convenient linear scale on the abscissa of a graph the angle of pendulum position for the portion of the swing beyond the free hanging position. For convenience, place the free hanging reference point on the right end of the abscissa with the angular displacement increasing linearly to the left. The abscissa is referred to as Scale C. Although angular displacement is the quantity to be represented linearly on the abscissa, this displacement is more conveniently expressed in terms of indicated energy read from the machine dial. This yields a nonlinear Scale C with indicated pendulum energy increasing to the right.

A1.3 On the right hand ordinate lay off a linear Scale B starting with zero at the bottom and stopping at the maximum expected pendulum friction and windage value at the top.

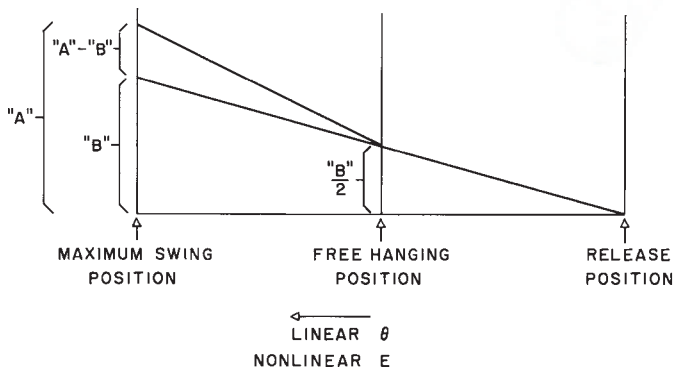


FIG. A1.1 Method of Construction of a Windage and Friction Correction Chart

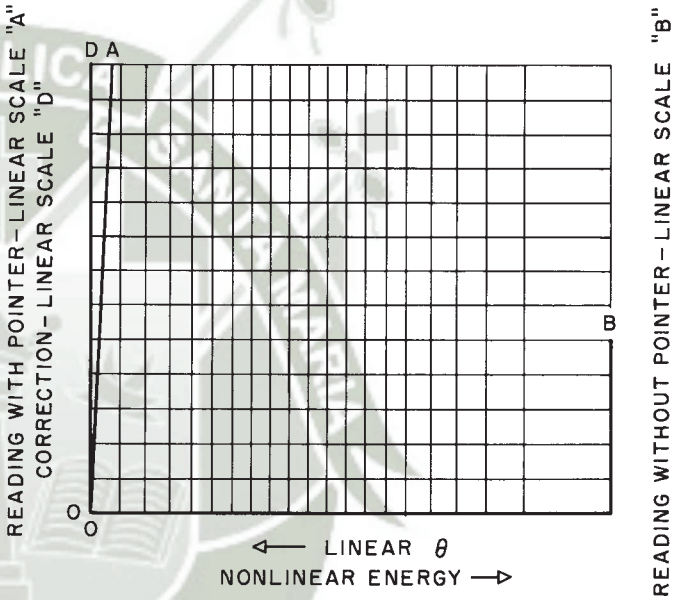


FIG. A1.2 Sample Windage and Friction Correction Chart

A1.4 On the left ordinate construct a linear Scale D ranging from zero at the bottom to 1.2 times the maximum ordinate value appearing on Scale B, but make the scale twice the scale used in the construction of Scale B.

A1.5 Adjoining Scale D draw a curve OA which is the focus of points whose coordinates have equal values of energy correction on Scale D and indicated energy on Scale C. This curve is referred to as Scale A and utilizes the same divisions and numbering system as the adjoining Scale D.

A1.6 *Instructions for Using Chart*:

A1.6.1 Locate and mark on Scale A the reading A obtained from the free swing of the pendulum with the pointer prepositioned in the free hanging or maximum indicated energy position on the dial.

A1.6.2 Locate and mark on Scale B the reading B obtained after several free swings with the pointer pushed up close to zero indicated energy position of the dial by the pendulum in accordance with instructions in 10.3.

A1.6.3 Connect the two points thus obtained by a straight line.

A1.6.4 From the indicated impact energy on Scale C project up to the constructed line and across to the left to obtain the correction for windage and friction from Scale D.

A1.6.5 Subtract this correction from the indicated impact reading to obtain the energy delivered to the specimen.

A2. PROCEDURE FOR THE CALCULATION OF WINDAGE AND FRICTION CORRECTION

A2.1 The procedure for the calculation of the windage and friction correction in this annex is based on the equations developed by derivation in Appendix X3. This procedure is acceptable as a substitute for the graphical procedure described in Annex A1 and is applicable to small electronic calculator and computer analysis.

A2.2 Calculate L, the distance from the axis of support to the center of percussion as indicated in 6.3. It is assumed here that the center of percussion is approximately the same as the center of strike.

A2.3 Measure the maximum height, h_M, of the center of percussion (center of strike) of the pendulum at the start of the test as indicated in X2.16.

A2.4 Measure and record the energy correction, E_A, for windage of the pendulum plus friction in the dial, as determined with the first swing of the pendulum with no specimen in the testing device. This correction must be read on the energy scale, E_M, appropriate for the pendulum used.

A2.5 Without resetting the position of the indicator obtained in A2.4, measure the energy correction, E_B, for pendulum windage after two additional releases of the pendulum with no specimen in the testing device.

A2.6 Calculate β_{max} as follows:

$$\beta_{max} = \cos^{-1}\{1 - [(h_M/L)(1 - E_A/E_M)]\} \quad \text{(A2.1)}$$

where:

E_A = energy correction for windage of pendulum plus friction in dial, J [ft·lbf],
E_M = full-scale reading for pendulum used, J [ft·lbf],
L = distance from fulcrum to center of strike of pendulum, m [ft],
h_M = maximum height of center of strike of pendulum at start of test, m [ft], and
β_{max} = maximum angle pendulum will travel with one swing of the pendulum.

A2.7 Measure specimen breaking energy, E_S, J [ft·lbf].

A2.8 Calculate β for specimen measurement E_s as:

$$\beta = \cos^{-1}\{1 - [(h_M/L)(1 - E_S/E_M)]\} \quad \text{(A2.2)}$$

where:

β = angle pendulum travels for a given specimen, and
E_S = dial reading breaking energy for a specimen, J [ft·lbf].

A2.9 Calculate total correction energy, E_{TC} as:

$$E_{TC} = (E_A - (E_B/2))(\beta/\beta_{max}) + (E_B/2) \quad \text{(A2.3)}$$

where:

E_{TC} = total correction energy for the breaking energy, E_s, of a specimen, J [ft·lbf], and
E_B = energy correction for windage of the pendulum, J [ft·lbf].

A2.10 Calculate the impact resistance using the following formula:

$$I_s = (E_s - E_{TC})/t \quad \text{(A2.4)}$$

where:

I_s = impact resistance of specimen, J/m [ft·lbf/in.] of width, and
t = width of specimen or width of notch, m [in.]

APPENDIXES

(Nonmandatory Information)

X1. PROCEDURE FOR THE INSPECTION AND VERIFICATION OF NOTCH

X1.1 The purpose of this procedure is to describe the microscopic method to be used for determining the radius and angle of the notch. These measurements could also be made using a comparator if available.

NOTE X1.1—The notch shall have a radius of 0.25 ± 0.05 mm [0.010 ± 0.002 in.] and an angle of 45 ± 1°.

X1.2 *Apparatus*:

X1.2.1 *Optical Device*, with minimum magnification of 60×, Filar glass scale and camera attachment.

X1.2.2 *Transparent Template*, that will be developed in this procedure.

X1.2.3 *Ruler*.

X1.2.4 *Compass*.

X1.2.5 *Plastic Drafting Set Squares (Triangles)*, 45–45–90°.

X1.3 A transparent template must be developed for each magnification and for each microscope used. It is preferable that each laboratory standardize on one microscope and one magnification. It is not necessary for each laboratory to use the same magnification because each microscope and camera combination have somewhat different blowup ratios.

X1.3.1 Set the magnification of the optical device at a suitable magnification with a minimum magnification of 60×.

X1.3.2 Place the Filar glass slide on the microscope platform. Focus the microscope so the most distinct of the Filar scale is visible.

X1.3.3 Take a photograph of the Filar scale (see Fig. X1.1).

X1.3.4 Create a template similar to that shown in Fig. X1.2.

X1.3.4.1 Find the approximate center of the piece of paper.

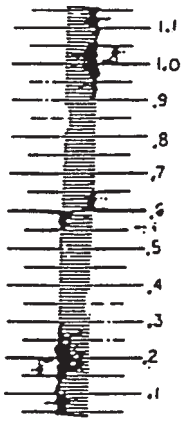
X1.3.4.2 Draw a set of perpendicular coordinates through the center point.

X1.3.4.3 Draw a family of concentric circles that are spaced in accordance with the dimensions of the Filar scale. This task is accomplished by first setting a mechanical compass at a distance of 0.1 mm [0.004 in.] as referenced by the magnified photograph of the Filar eyepiece. Subsequent circles shall be spaced 0.02 mm apart [0.001 in.], as rings, with the outer ring being 0.4 mm [0.016 in.] from the center.

X1.3.5 Photocopy the paper with the concentric circles to make a transparent template of the concentric circles.

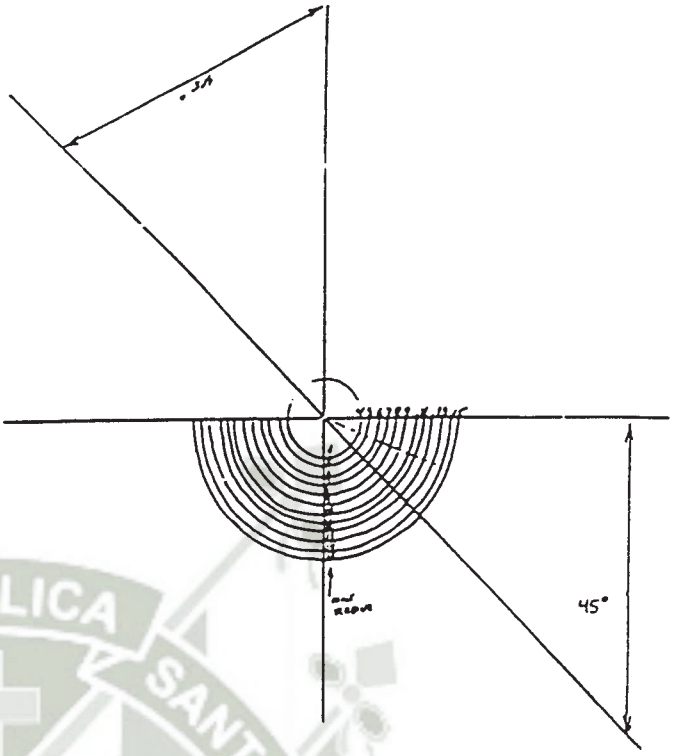
X1.3.6 Construct Fig. X1.3 by taking a second piece of paper, finding its approximate center, and marking this point. Draw one line through this center point. Label this line zero degree (0°). Draw a second line perpendicular to the first line through this center point. Label this line 90°. From the center draw a line that is 44° relative to the 0°. Label the line 44°. Draw another line at 46°. Label the line 46°.

X1.4 Place a microscope glass slide on the microscope platform. Place the notched specimen on top of the slide. Focus



NOTE 1—100× reference.
NOTE 2—0.1 mm major scale; 0.01 mm minor scale.

FIG. X1.1 Filar Scale



NOTE 1—Magnification = 100×.

FIG. X1.2 Example of Transparent Template for Determining Radius of Notch

the microscope. Move the specimen around using the platform adjusting knobs until the specimen's notch is centered and near the bottom of the viewing area. Take a picture of the notch.

X1.4.1 *Determination of Notching Radius (*Fig. X1.4*)*:

X1.4.1.1 Place the picture on a sheet of paper. Position the picture so that bottom of the notch in the picture faces downwards and is about 64 mm [2.5 in.] from the bottom of the paper. Tape the picture down to the paper.

X1.4.1.2 Draw two lines along the sides of the notch projecting down to a point where they intersect below the notch Point I (see Fig. X1.4B).

X1.4.1.3 Open the compass to about 51 mm [2 in.]. Using Point I as a reference, draw two arcs intersecting both sides of the notch (see Fig. X1.4C). These intersections are called 1a and 1b.

X1.4.1.4 Close the compass to about 38 mm [1.5 in.]. Using Point 1a as the reference point, draw an arc (2a) above the notch, draw a second arc (2b) that intersects with arc 2a at Point J. Draw a line between I and J. This establishes the centerline of the notch (see Fig. X1.4D)

X1.4.1.5 Place the transparent template on top of the picture and align the center of the concentric circles with the drawn centerline of the notch (see Fig. X1.4E).

X1.4.1.6 Slide the template down the centerline of the notch until one concentric circle touches both sides of the notch. Record the radius of the notch and compare it against the limits of 0.2 to 0.3 mm [0.008 to 0.012 in.].

X1.4.1.7 Examine the notch to ensure that there are no flat spots along the measured radius.

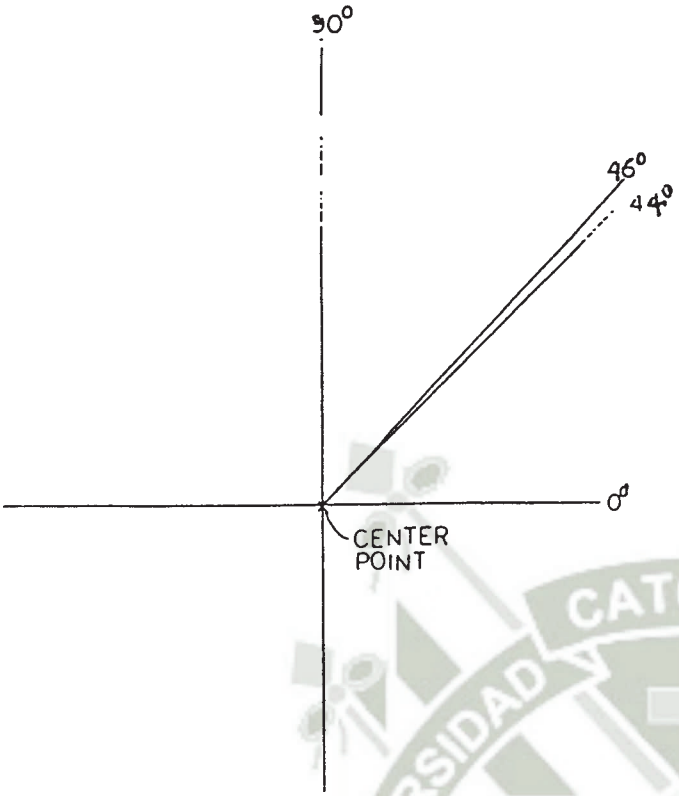


FIG. X1.3 Example of Transparent Template for Determining Angle of Notch

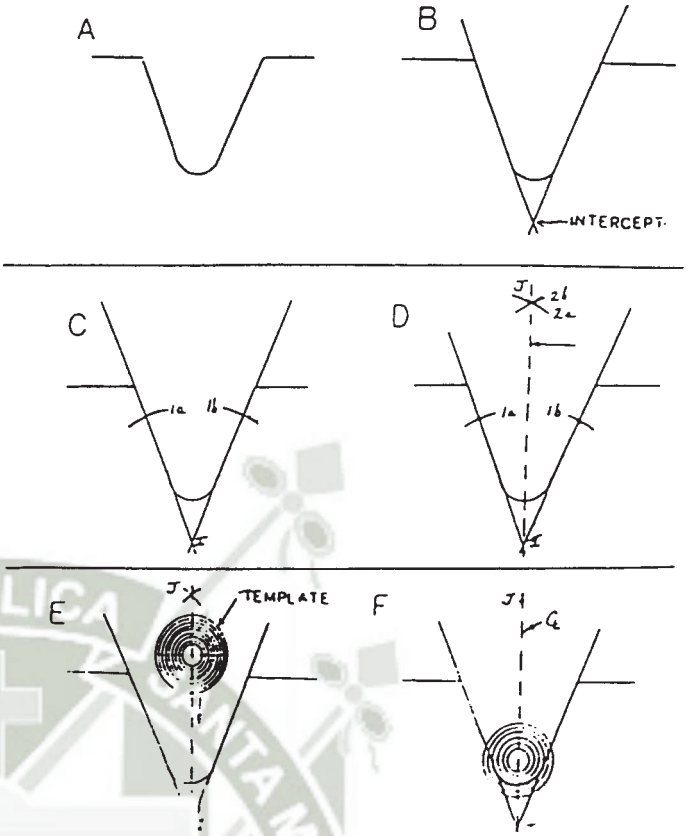


FIG. X1.4 Determining Radius

X1.4.2 *Determination of Notch Angle*—Place transparent template for determining notch angle (Fig. X1.3) on top of the photograph attached to the sheet of paper. Rotate the picture so that the notch tip is pointed towards you. Position the center point of the template on top of the Point I established in 0° axis of the template with the right side straight portion of the notch. Check the left side straight portion of the notch to ensure that this portion falls between the 44° and 46° lines. If not, replace the blade.

X1.5 A picture of a notch shall be taken at least every 500 notches or if a control sample gives a value outside its 3-sigma limits for that test.

X1.6 If the notch in the control specimen is not within the requirements, a picture of the notching blade should be taken and analyzed by the same procedure used for the specimen notch. If the notching blade does not meet ASTM requirements or shows damage, it should be replaced with a new blade which has been checked for proper dimensions.

X1.7 If a cutter has the correct dimensions, but does not cut the correct notch in the specimen, it will be necessary to evaluate other conditions (cutter and feed speeds) to obtain the correct notch dimension for that material.

X2. CALIBRATION OF PENDULUM-TYPE HAMMER IMPACT MACHINES FOR USE WITH PLASTIC SPECIMENS

X2.1 This calibration procedure applies specifically to the Charpy impact machine.

X2.2 Locate the impact machine on a sturdy base. It shall not walk on the base and the base shall not vibrate appreciably. Loss of energy from vibrations will give high readings. It is recommended that the impact tester be bolted to a base having a mass of at least 23 kg if it is used at capacities higher than 2.7 J [2 ft·lbf].

X2.3 Check the level of the machine in both directions on the plane of the base with spirit levels mounted in the base, by a machinist's level if a satisfactory reference surface is available, or with a plumb bob. The machine should be made level to within $\tan^{-1}$ 0.001 in the plane of swing and to within $\tan^{-1}$ 0.002 in the place perpendicular to the swing.

X2.4 Contact the machine manufacturer for a procedure to ensure the striker radius is in tolerance (3.17 ± 0.12 mm) (see 6.3).

X2.5 Check the transverse location of the center of the pendulum striking edge that shall be within 0.40 mm [0.016 in.] of the center of the anvil. Readjust the shaft bearings or relocate the anvil or straighten the pendulum shaft as necessary to attain the proper relationship between the two centers.

X2.6 Check the pendulum arm for straightness within 1.2

mm [0.05 in.] with a straightedge or by sighting down the shaft. This arm is sometimes bent by allowing the pendulum to slam against the catch when high–capacity weights are on the pendulum.

X2.7 Center a notched 12.7–mm square metal bar having opposite sides parallel within 0.025 mm and 125 mm long on the Charpy anvils. Place a thin oil film on the striking edge of the pendulum with an oiled cloth and let the striking edge rest gently against the bar. A thin line of oil should be transferred across the entire width of the bar, thereby verifying that the striking edge is in contact across the entire specimen width.

X2.8 When the pendulum is hanging free in its lowest position, the energy reading must be within 0.2 % of full scale.

X2.9 Swing the pendulum to a horizontal position, and support it by the striking edge in this position with a vertical bar. Allow the other end of this bar to rest at the center of a load pan on a balanced scale. Subtract the weight of the bar from the total weight to find the effective weight of the pendulum. The effective pendulum weight should be within 0.4 % of the required weight for that pendulum capacity. If weight must be added or removed, take care to balance the added or removed weight without affecting the center of percussion relative to the striking edge. It is not advisable to add weight to the opposite side of the bearing axis from the striking edge to decrease the effective weight of the pendulum since the distributed mass has the potential to result in large energy losses from vibration of the pendulum.

X2.10 Calculate the effective length of the pendulum arm or the distance to the center of percussion from the axis of rotation by the procedure in Note 5. The effective length must be within the tolerance stated in 6.3.

X2.11 Determine the vertical distance of fall of the pendulum striking edge from its latched height to its lowest point. This distance should be 610 ± 2 mm. This measurement is made with a half-width specimen positioned on the anvils. Place a thin oil film on the specimen and bring the striking edge against it. The upper end of the oil line on the striking edge is the center of strike. Measure the change in vertical height of the center of strike from the latched to the free hang position (the lowest point). This vertical fall distance is adjusted by varying the position of the pendulum latch.

X2.12 If a pointer and dial mechanism is used to indicate the energy, the pointer friction should be adjusted so that the pointer will just maintain its position anywhere on the scale. The striking pin of the pointer should be securely fastened to the pointer. Friction washers with glazed surfaces should be replaced with new washers. Friction washers should be on either side of the pointer collar. The last friction washer installed should be backed by a heavy metal washer. Pressure on this metal washer is produced by a thin bent spring washer and locknuts. If the spring washer is placed next to the fiber friction washer, the pointer will tend to vibrate during impact.

X2.13 The free-swing reading of the pendulum (without specimen) from the latched height should be less than 2.5 % of pendulum capacity on the first swing. If the reading is higher than this, the friction in the indicating mechanism is excessive or the bearings are dirty. To clean the bearings, dip them in grease solvent and spin dry in an air jet. Clean the bearings until they spin freely or replace them. Oil very lightly with instrument oil before replacing. A reproducible method of starting the pendulum from the proper height must be devised.

X2.14 The shaft about which the pendulum rotates shall have no detectable radial play, less than 0.05 mm [0.002 in.]. An end play of 0.25 mm [0.010 in.] is permissible when a 9.8–N [2.2–lbf] axial force is applied in alternate directions.

X2.15 The machine should not be used to indicate more than 85 % of the energy capacity of the pendulum. Extra weight added to the pendulum will increase available energy of the machine. This weight must be added so as to maintain the center of percussion within the tolerance stated in 6.3. Correct effective weight for any range is calculated as follows:

$$W = E_p / h \quad \text{(X2.1)}$$

where:

W = the effective pendulum weight, N [lbf] (see X2.13),
E_p = potential or available energy of the machine, J [ft × lbf], and
h = the vertical distance of fall of the pendulum striking edge, m [ft] (see X2.11).

Each 4.5 N [1 lbf] of added effective weight increases the capacity of the machine by 2.7 J [2 ft × lbf].

NOTE X2.1—If the pendulum is designed for use with add-on weight, it is recommended that they be obtained through the equipment manufacturer.

X3. DERIVATION OF PENDULUM IMPACT CORRECTION EQUATIONS

X3.1 From right triangle distances in Fig. X3.1:

$$L - h = L \cos \beta \quad \text{(X3.1)}$$

X3.2 The potential energy gain of pendulum, E_p, is:

$$E_p = hW_p g \quad \text{(X3.2)}$$

X3.3 Combining Eq X3.1 and Eq X3.2 gives the following:

$$L - E_p/W_p g = L \cos \beta \quad \text{(X3.3)}$$

X3.4 The maximum energy of the pendulum is the potential energy at the start of the test, E_M, or

$$E_M = h_M W_p g \quad \text{(X3.4)}$$

X3.5 The potential energy gained by the pendulum, E_p, is related to the absorption of energy of a specimen, E_s, by the following equation:

$$E_M - E_s = E_p \quad \text{(X3.5)}$$

X3.6 Combining Eq X3.3-X3.5 gives the following:

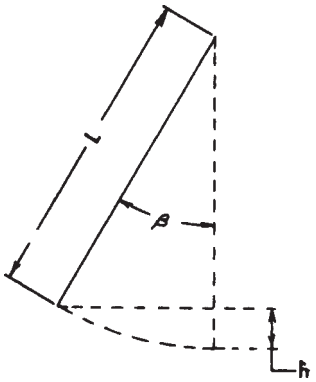


FIG. X3.1 Swing of Pendulum from Its Rest Position

$$(E_M - E_S)/E_M = L/h_M(1 - \cos\beta) \quad \text{(X3.6)}$$

X3.7 Solving Eq X3.6 for β gives the following:

$$\beta = \cos^{-1}\{1 - [(h_M/L)(1 - E_S/E_M)]\} \quad \text{(X3.7)}$$

X3.8 From Fig. X3.2, the total energy correction, E_{TC}, is given as:

$$E_{TC} = m\beta + b \quad \text{(X3.8)}$$

X3.9 At the zero point of the pendulum the potential energy is:

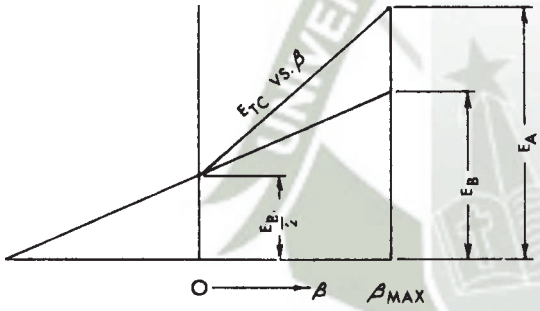


FIG. X3.2 Total Energy Correction for Pendulum Windage and Dial Friction as a Function of Pendulum Position

$$E_B/2 = m(0) + b$$

or

$$b = E_B/2 \quad \text{(X3.9)}$$

X3.10 The energy correction, E_A, on the first swing of the pendulum occurs at the maximum pendulum angle, β_{max}. Substituting in Eq X3.8 gives the following:

$$E_A = m\beta_{max} + (E_B/2) \quad \text{(X3.10)}$$

X3.11 Combining Eq X3.8 and Eq X3.11 gives the following:

$$E_{TC} = (E_A - (E_B/2))(\beta/\beta_{max}) + (E_B/2) \quad \text{(X3.11)}$$

X3.12 *Nomenclature*:

b = intercept of total correction energy straight line,
E_A = energy correction, including both pendulum windage plus dial friction, J,
E_B = energy correction for pendulum windage only, J,
E_M = maximum energy of the pendulum (at the start of test), J,
E_p = potential energy gain of pendulum from the pendulum rest position, J,
E_S = uncorrected breaking energy of specimen, J,
E_{TC} = total energy correction for a given breaking energy, E_S, J,
g = acceleration of gravity, m/s^2,
h = distance center of gravity of pendulum rises vertically from the rest position of the pendulum, m,
h_m = maximum height of the center of gravity of the pendulum, m,
m = slope of total correction energy straight line,
L = distance from fulcrum to center of gravity of pendulum, m,
W_p = weight of pendulum, as determined in X2.13, kg, and
β = angle of pendulum position from the pendulum rest position.

X4. UNIT CONVERSIONS

X4.1 Joules per metre cannot be converted directly into kilojoules per square metre.

NOTE X4.1—The optional units of kJ/m^2 [ft·lbf/in.^{2}] also may be required; therefore, the cross-sectional area under the notch must be reported.

X4.2 The following examples are approximations:

1ft·lbf/39.37 in. = 1.356 J/m

1ft·lbf/in. = (39.37)(1.356) J/m
1ft·lbf/in. = 53.4 J/m
1ft·lbf/in. = 0.0534 kJ/m

1ft·lbf/1550 in.^{2} = 1.356 J/m^2
1ft·lbf/in.^{2} = (1550)(1.356) J/m^2
1ft·lbf/in.^{2} = 2101 J/m^2
1ft·lbf/in.^{2} = 2.1 kJ/m^2

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that impact the use of this test method. This section also includes descriptions of the changes or reasons for the changes, or both.

D6110-04:

(*1*) Add reference to D5947 in paragraph 2.1.
(*2*) Changed requirements for micrometer in paragraph 6.3.
(*3*) Removed requirement to mill draft angle from specimens.
(*4*) Changed notch depth dimensions to 10.16 ± 0.05 mm in order to agree with Figure 4; and clarified procedure to measure notch depth in paragraph 8.2.
(*5*) Added paragraphs 10.3.2 and 10.3.3 on clarifying notch depth measuring technique. Renumbered subsequent sections.
(*6*) Added Figure 6.
(*7*) Permissive language removed.
(*8*) Imperial units added for reference.