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Tensile Properties of Semi-Crystalline Thermoplastics – Performance Comparison under Alternative Testing Standard

ABSTRACT

Due to the wide and ever increasing application of thermoplastic parts in the automotive industry, the measurement and interpretation of their properties must be thoroughly understood before anyone can hope to correctly utilize the results in material selection, product design, and performance analysis while all these can be greatly influenced by the end-use conditions.

Tensile properties of thermoplastics, such as stress and strain at yield, ultimate tensile strength, and Young's modulus, are among the most widely measured and cited mechanical properties for material evaluation, quality control, structure design, modeling, and failure analysis. This paper deals with several major challenges that an engineer may face when attempting to obtain accurate tensile property data for thermoplastics. One such challenge is the trend of automotive industry today to convert from ASTM to ISO procedures for thermoplastics evaluation and product certification.

Our study on the widely used, semi-crystalline polyamides (PA 6) based plastics indicates that, while in most cases the values of tensile properties produced using the two standards are close, difference does exists which might be a result of different specimen geometry, and in some cases different definitions of parameters.

Another challenge is the variation in the material's properties due to the changing environment. In this investigation the tensile properties of PA 6 were studied under two environmental conditions that have the most influence on the structures in use: temperature (from –40°C to 150°C) and relative humidity (dry-as-molded (DAM) and 50% RH). The ultimate tensile strength and Young's modulus have been found to decrease significantly as the temperature or moisture level increases. However, the materials become less sensitive to the environment at elevated temperature or with high moisture content.

Results from this paper should help designers to accurately interpret tensile strength and deformation properties for the semi-crystalline thermoplastics in general and to utilize the material parameters under the end-use conditions for the structural analysis of thermoplastic components.

INTRODUCTION

IMPACT OF ISO AND ASTM TESTING STANDARDS ON TENSILE PROPERTIES OF PLASTICS

In recent years, demands have increased in using polyamide (PA) to replace certain metals and thermosets in the automotive vehicle air induction and power train systems, and lawn / garden and power tools. The combined goal in high performance, weight reduction, and time / cost savings often pushed the structures to perform toward the limit of their properties. The accurate measurement of the material properties, therefore, has become more critical than ever [1-3].

In this regard, global standardization has been playing a more important role in facilitating product manufacturing, marketing, and sales [1-2]. The widely published testing procedures and specifications for plastic materials by the American Society for Testing and Materials (ASTM, Committee D-20 on Plastics) and the International Standard Organization (ISO) have helped product developers, designers, and molders to establish correct and useful baselines. An important development in the standardization area is the fact that the American automotive industry has become one of the first to require ISO test procedures for material and product qualifications [2] when the majority of testing in the North America is still conducted using ASTM standards. The United States Council for Automotive Research (USCAR) recommended the manufacturers of thermoplastic products to fully convert to ISO test procedures by June 1998.

The decision for this conversion will no doubt have a major impact on plastic manufacturers, part molders, designers, and end users when most of the material and product information accumulated for decades and still in use was obtained using ASTM procedures. This paper is part of our effort in assisting this transition [3]. The tensile properties of thermoplastics were analyzed not merely to compare the ASTM and ISO tensile test procedures; they were done also because of the importance of tensile properties in the product design [4-6].

The current investigation has been focused on the short-term (tensile) property evaluation of PA based thermoplastics with the influence of moisture and temperature (from –40 to 150 °C) effects. Material parameters obtained using ISO and ASTM specimens and test procedures were compared for their similarities and differences. Analyses were also made on two important aspects of the tensile property measurements, one was the use of extensometer, and another, the effect of grips and gripping on the accuracy of Young's modulus. The purpose of the investigation was to provide the plastic part designers, product developers, and testing community alike with the guidance in correctly obtaining and interpreting their tensile test results.

MOISTURE EFFECT

One of the insidious disadvantages of certain plastics, such as polyamides, is their tendency to absorb moisture from ambient, then change their properties as a result [4-7]. The moisture may exist during polymerization and washing steps in polymer processing; it can be absorbed from surrounding atmosphere during storage and use. The moisture is known to affect a range of polymer properties, which in turn impact processability, dimensional stability, mechanical, acoustic, electrical, optical, and chemical properties, as well as performance of the products [6-7].

Under dry-as-molded (DAM) conditions, polyamides, or nylon, usually contain 0.1-0.3% water[*]. Under room temperature and 50% relative humidity (RH), type 6 polyamide could eventually absorb 2.75% water. Every 1% moisture increase in nylon may result in 0.2 to 0.3% increase in its dimension [4]. This change in dimension can be accommodated by preconditioning parts prior to service.

The moisture in nylon behaves as a plasticizer that reduces the entanglement and bonding between molecules, therefore increases their volume and mobility [7]. An increase in moisture lowers a material's strength and stiffness, but increases its total elongation and impact resistance. Practically, the best way to minimize the moisture uptake is to select plastics with low absorption rate or design products in ways to prevent excessive absorption.

The design and analysis (i.e., linear and non-linear FEA) of critically stressed nylon components require input of short and long-term mechanical properties, including effects of moisture, time-temperature, and other typical end-use environmental conditions [8-10]. The properties listed in CAMPUS® (*Computer Aided Material Preselection by Uniform Standards*) include tensile properties of plastics at two material states: dry as molded (DAM) and at 50% RH (relative humidity) at different temperatures [1, 6, 9-10]. CAMPUS®, trade literature, and design manuals normally report properties at 50% RH, which is the environment where most of the materials are conditioned prior to test. Yet reporting RH is not the same as reporting moisture, and the moisture is what directly impacts properties. This issue can be even a greater concern when testing is conducted at elevated temperatures when neither the surrounding RH nor the real moisture level in the material sample is controlled or monitored [1-2]. As the moisture in the sample is being continuously driven out by heat, one may observe significant variations in measured properties [11-13]. This paper is attempted to address some of these moisture/temperature related issues.

MATERIALS

The thermoplastics used in this investigation were heat stabilized, unfilled and glass and/or mineral filled polyamide (PA) 6. Materials were injection molded into ISO multipurpose (ISO 3167) and ASTM Type 1 and Type 2 (ASTM D 638) specimens according to the procedures specified in ISO 294-1, ISO 294-2, ASTM D 3641 and ASTM D 4066. All specimens were sealed (see ASTM D 3892) prior to testing in order to maintain their dry-as-molded (DAM) conditions.

TEST PROCEDURES

TENSILE TESTS (ISO AND ASTM PROCEDURES)

The tensile property tests were conducted using Instron 4505. Most tests were conducted under standard laboratory conditions (temperature = 23 ± 2°C; relative humidity = 50 ± 5%) on dry-as-molded samples. Some samples were also

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[*] ASTM D4066 specified for nylon 6 moisture content wt. % max "as received" equal 0.2% (moisture content measurements by ASTM D 789).

tested at different temperatures (– 40°C and 150°C) using an environmental chamber attached to the Instron. The temperature inside the chamber was controlled at ± 2°C within the set point.

Two crosshead speeds were used for testing: 1 mm/min for Young's modulus, and 5 or 50 mm/min (for reinforced and non-reinforced materials, respectively) for other tensile properties such as stresses and strains at yield and break. The tensile strain was measured from the narrow section of the specimen using a clip-on extensometer (ISO 9513 and ASTM E83) with a gage length of 50.8 mm.

The test control and data acquisition were achieved using Instron Series 9 software. The material parameters for tensile properties, such as tensile strength (σ_M), tensile strain at tensile strength (ε_M), stress at break (σ_B) and strain at break (ε_B), were obtained according to the definitions in ASTM D 638 and ISO-527†. The Young's modulus, *E*, was calculated according to the definition in ISO-527, which gives

$$E = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1} \qquad (1)$$

where ε_1 = 0.0005, ε_2 = 0.0025, and σ_1, σ_2 = stresses at ε_1 and ε_2, respectively.

REMARKS ON SAMPLE CONDITIONING

Many properties of polyamides can be impacted by their moisture content. Values of mechanical, electrical, and thermal properties are reproducible only if the moisture content in the material is under control. Because of this reason, sample conditioning is an important part of property testing and specification.

In this regard, ISO 291 defines the following two standard atmospheres for conditioning:

- "Atmosphere 23": 23/50 (temperature in °C/ relative humidity in %) as recommended for most applications;
- "Atmosphere 27": 27/65, as recommended for tropical countries.

The rate of moisture absorption is very low under "Atmosphere 23". In this environment, it would take more than a year for the moisture in an ISO 3167 multipurpose test specimen (4 mm thick) of PA 66 to reach its equilibrium. To accelerate this process, a higher temperature is required. The following moisture conditioning procedures may be applied for thermoplastics:

- ISO 291 and ASTM D 618: describe standard procedures for plastic specimens conditioning prior to test / evaluation. This standard recommend six conditioning procedures, various by environment (air atmosphere / oven, water), duration, temperature and humidity / water). For moisture sensitive nylons to condition DAM specimens prior to testing is not practical.

- ISO 62 and ASTM D 570: describes standard water absorption procedures prior specimens / parts to test / evaluation. ASTM D 570 recommend seven water absorption procedures (when plastic specimen / part is immersed in to distilled water). Procedures parameters include time (from 0.5 h to 24 h), temperature (23± 1°C, 50± 1 and of 105 to 110°C). High level of temperature (boiling of water) may affect microstructure of nylon (because glass transition temperature of nylon is below boiling point of water). This method is also not practical for plastics containing extractable elements. Some concerns may exist regarding the moisture distribution across the thickness of a specimen or part. The trade literature and manufacturers published data at 20%, 50% and 100% RH was generated using this conditioning method [4,6].

  Table 1 lists the water absorption values for several selected plastics as determined by ASTM D 570 after 24 h immersion at 23°C. Equilibrium value for water absorption will be significantly higher for these plastics, as will water absorption values obtained at elevated temperatures.

  Table 1: Water Absorption Values for Selected Plastics

Material	Water Absorption

† The definitions of these parameters were considered equivalent in these two standards.

PP	< 0.01 %
PC	0.15 %
Nylon 11	0.25 %
Nylon 6	1.3 %
Cellulose Acetate	1.7 %

- ISO 483 and ASTM E 104: list of saturated salt solutions and relative humidity conditioning percent (% RH) to which they correspond at various temperatures. The data at 20%, 50% and 75% RH was generated using this conditioning method [6-7].

- ISO 1110: describes accelerated conditioning at 70°C and 62% relative humidity. The period of time for accelerated conditioning can be calculated using a simple equation. The values of mechanical properties obtained after accelerated conditioning according to this method may differ slightly from those obtained after conditioning in "Atmosphere 23" [6, 9].

MOISTURE ANALYSIS IN NYLON

Moisture analysis is critical for many manufacturing processes. In molding, testing, and end-use of thermoplastics, determining the moisture content in pellets or parts can be critically important. The equilibrium moisture in nylons may vary from 1.0 to 14 % (at 100% RH). Water absorption data for commercial available nylon is presented in Table 2.

Table 2: Influence of Relative Humidity (RH%) on Water Absorption (in %, at 23°C - air) in Non-Reinforced / Non-Filled Nylons

Type of PA	30% RH	50% RH	62% RH	100% RH
PA 46	1.4	3.8	5.0	15
PA 6	1.1	2.75	3.85	9.5
PA 66	1.0	2.5	3.6	8.5

For nylon based plastics the following moisture determination methods are used in practical and laboratories applications [4, 6-7]:

- Karl-Fischer (ASTM D 789):is based on titration with a Karl Fischer Reagent. This test is sensitive to moisture levels of 0.1% to 0.2% water (when 20 g to 30 g of sample are used typically). For higher than 0.2% moisture allow smaller sample size to be used.

- ASTM D 4019: is based on release of water vapor, which is swept by an inert carrier gas into an electrolytic cell. This method allows one to use about 2 ~ 4 g of sample to measure moisture in polyamides at a level of < 0.1%. By ASTM D 4066 a maximum moisture content (in %) in varied nylon based plastics in the range from 0.05 (nylon 46) to 0.3 (nylon 612). For nylon 6 this value is 0.2%.

- ISO 1110, ASTM D 570, and a few other methods are based on weight gain measurements. Specimens of materials whose water-absorption value would be appreciably affected by the temperatures in the neighborhood of 110°C, shall be dried in a oven for 24 h at 50 ± °C, cooled in a desiccator, and immediately weighted to the nearest 0.001 g.

ISO 1110 recommended to determinate period of time needed for accelerate conditioning (at 70±1 °C and (62±1)% RH by simple equation as function of thickness of the specimens. Relation between absorbed moisture (in %) and time of conditioning (in hours) for different thickness of the specimens (3.2; 4.0 and 6.25 mm) is presented in Figure 1.

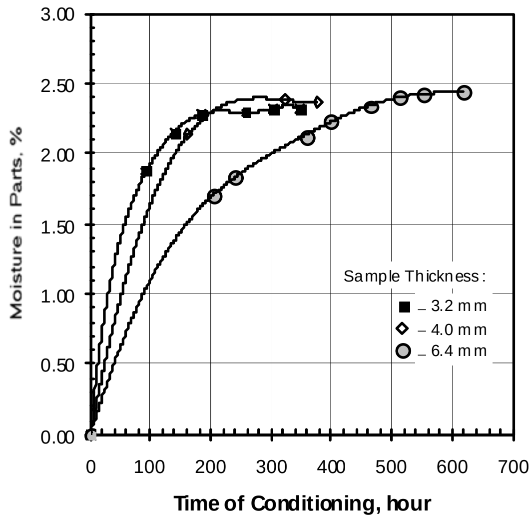

Figure 1: Moisture content vs. time of conditioning for 33 wt% glass fiber reinforced polyamide 6

This moisture-thickness-time data is very helpful for time estimation for a similar moisture content, obtained after conditioning as in the standard Atmosphere 23/50 ("Atmosphere 23").

RESULTS AND DISCUSSIONS

TENSILE PROPERTIES BY ISO AND ASTM STANDARDS AT DRY AS MOLDED (DAM) CONDITIONS

In Figures 2 to 4, properties obtained using ISO specimens were plotted against those obtained using ASTM D 638 (Type 1) specimens. The solid line, Y = X, indicates on the graph where the two sets of property values are equal to each other.

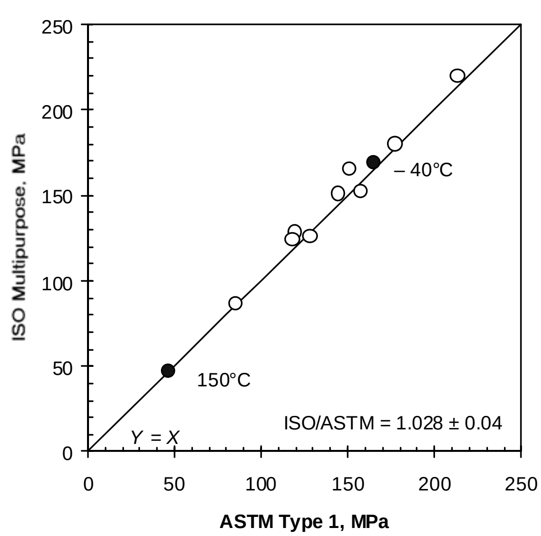

Figure 2: Tensile Strength of Materials, σ_M. T = 23°C unless otherwise indicated

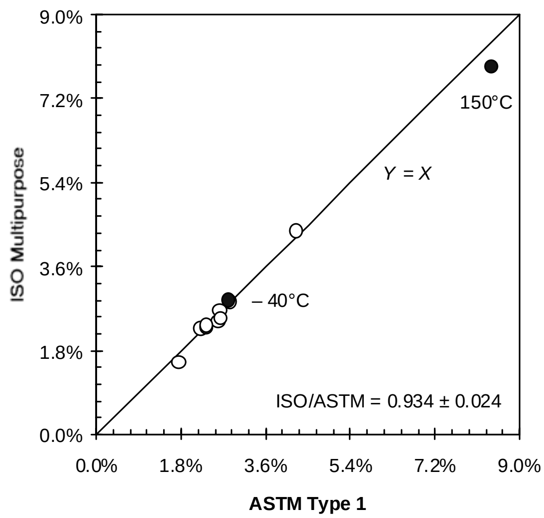

Figure 3: Tensile Strain at Tensile Strength, ε_M. T = 23°C unless otherwise indicated

For the tensile strength (Figure 2) and strain at tensile strength (Figure 3), the closeness of the data points to this line suggests that the properties obtained using the two standards are very close [3, 5, 9]. Statistically, the two sets of property values were compared using the least square method that generated a ratio between the ISO and ASTM data, as seen on each figure. The results indicate that, among the materials in the investigation, the ultimate stresses (σ_M and σ_B) obtained from ISO specimens are on average 2 ~ 3% higher than those from ASTM specimens, and 8% or more can be received from the modulus when test is done on ISO specimens. On the other hand, the opposite trend was found in tensile strains where the numbers for ε_M and ε_B are 5 ~ 6% lower in ISO 3167 specimens.

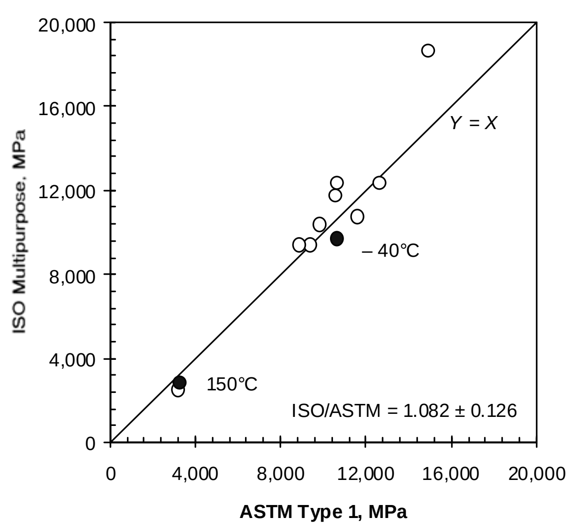

Figure 4: Young's Modulus, E. T = 23°C unless otherwise indicated

Despite the small difference in the nominal cross-sectional areas (10 mm × 4 mm = 40 mm2 for ISO, 12.7 mm × 3.18 mm = 40.4 mm2 for ASTM Type 1), the different linear dimensions of the two specimens (e.g., the ASTM specimen is wider but thinner than the ISO specimen) might have had an impact on the injection molding process and the distribution of the reinforcement, especially the orientation and distribution of fiber-glass. If so, this may be enough to cause a difference in the measured properties. The fact that the deviation in the modulus tends to increase with the amount of glass fibers (Figure 4) further suggests such a possibility.

EFFECTS OF MOISTURE ON TENSILE PROPERTIES

Whichever standard a user may choose for testing, the properties of materials cannot be appropriately presented without knowing the moisture content associated with the properties. In polyamides, water exists only in the amorphous phase, yet its present could influence crystallization and crystalline phases [6-7]. In nylon 6, two crystalline types, α and γ, are known to exist. With water absorption, the γ phase is transformed to the more stable α phase. Water also behaves as a plasticizer that decreases glass transition temperature (T_g) and lowers tensile strength and Young's modulus. At room temperature (23°C), T_g for DAM nylon 6 is above the ambient [6-7]. After conditioning at 50% RH, T_g has been reduced to below ambient. Moisture and temperature have similar effects on the tensile properties. In this regard, the moisture and temperature have similar effects on the properties. The decrease in the tensile stress at the yield and increase of strains (at the yield) as function of moisture content is shown in Figure 5-moisture.

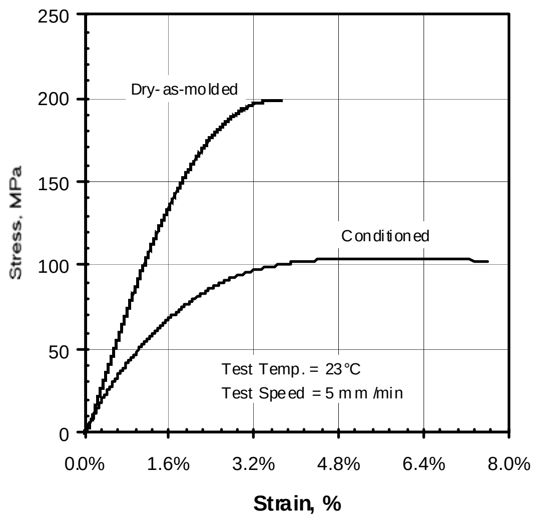

Figure 5: Tensile stress-strain curves at 23°C for 33 wt% glass fiber reinforced polyamide 6, dry-as-molded (DAM) and conditioned

At minus temperature (-40°C) and room temperature (23°C) the stress-strain curves are very similar (Figure 6). At elevated temperature (120°C) the decrease both in the tensile stress and strains at the yield and as function of moisture content is shown in Figure 7.

Changes in moisture content (from 0.2% to 1.2%) affect significantly the decrease of tensile strength and Young's modulus and increase of tensile strain at tensile strength (Figures 8).
Moisture content increase from 1.2% to 1.75% is not so highly affect to tensile properties of fiber-glass reinforced nylon 6 in comprizing with the range 0.2 – 1.2 % (Figure 9).

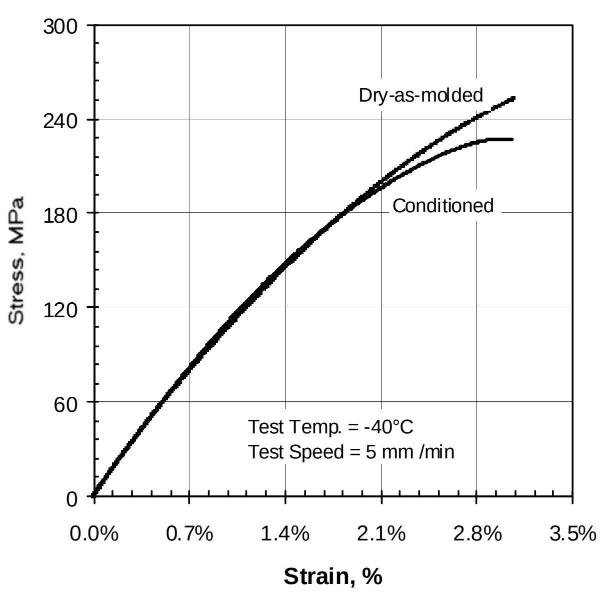

Figure 6: Tensile stress-strain curves at -40°C for 33 for 33 wt% glass fiber reinforced polyamide 6, dry-as-molded and

conditioned

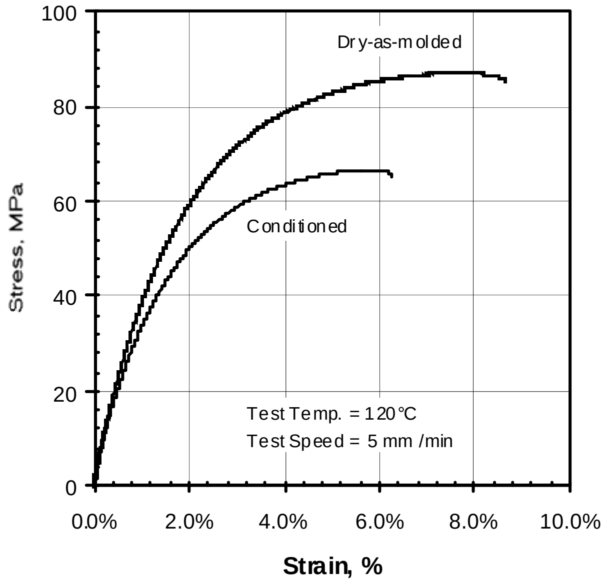

Figure 7: Tensile stress-strain curves at 120°C for 33 wt% glass fiber reinforced polyamide 6, dry-as-molded and conditioned

At this range of moisture content changes the reduction of the tensile strength is 5-6% approximately. Tensile strains will increase by 15-20%.

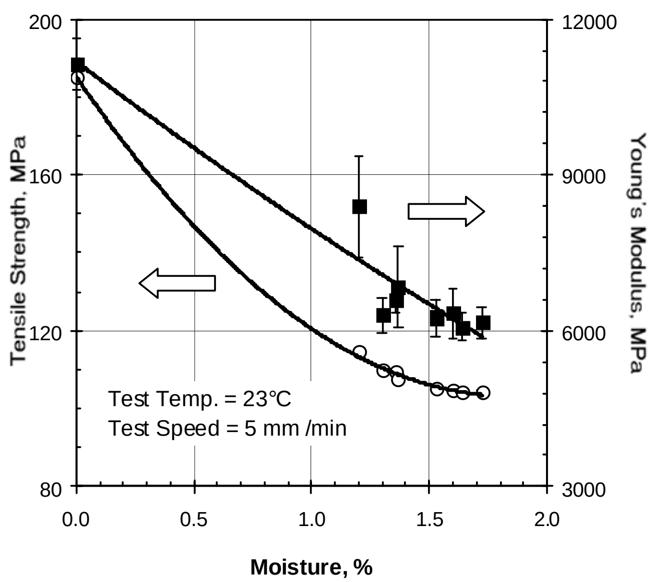

Figure 8: Tensile strength and Young's modulus vs. moisture for 33 wt% glass fiber reinforced polyamide 6

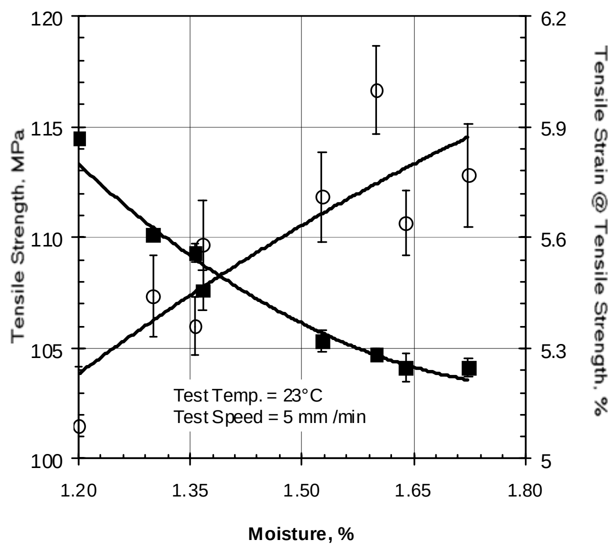

Figure 9: Change of tensile strength and tensile strain at tensile strength vs. moisture content for 33 wt% glass fiber reinforced polyamide 6

Figure 10 show changes in tensile strength of fiber-glass reinforced nylon at wide range of moisture (up to 2.7%) and temperature (-40, 23; 80; 100; 120and 150 °C) effects. At –40°C tensile strength was changed non-significantly (decreases by 10% approximately). Very significant changes were observed at room (23°C) temperature: tensile strength decreases by 45% approximately. At elevated temperatures (from 80 to 150°C) tensile strength decrease by 20% approximately.

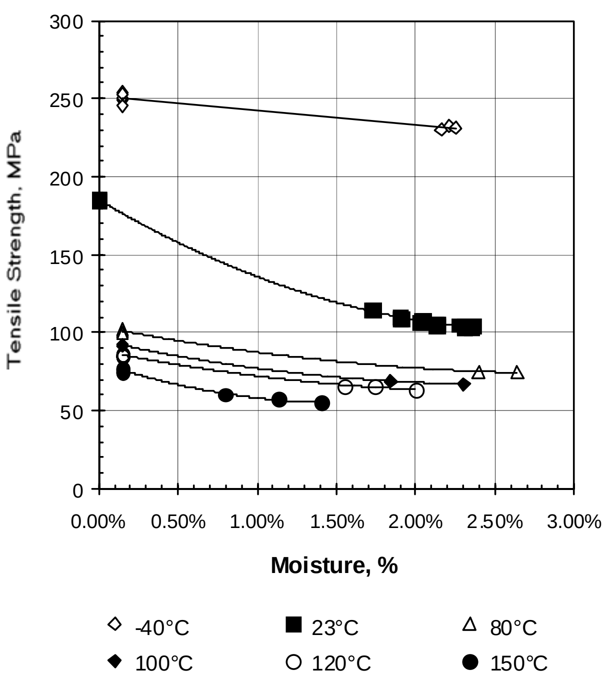

Figure 10: Tensile strength vs. moisture at different testing temperature for 33 wt% glass fiber reinforced polyamide 6

EFFECT OF GRIPS AND GRIPPING ON MODULUS MEASUREMENT

One of the observations from the tensile test was that although the sample standard deviation for stress (e.g., σ_M and σ_B) is normally very small, the same deviation is greater for strain, and greater still for Young's modulus. Using the coefficient of variation (CV) to characterize the data scattering, where CV = (sample standard deviation) ÷ (sample mean), it was found that CV is 0.2 ~ 1.5% for stress, 2 ~ 5% for strain, and 2 ~ 10% for modulus.

In order to understand the increase in CV from stress to strain, and from strain to modulus, a closer examination was made on the stress-strain relationship between $\varepsilon = 0$ and 0.3% which encloses the region where the modulus was calculated. It was found that in many cases the initial portion of the stress-strain curve around $\varepsilon = 0$ was rather complicated (Figure 11).

The variability in the modulus could increase significantly if the initial part of the stress-strain curve extended into $\varepsilon >$ 0.05%. This situation was found to be worse in some samples than in others. To find out why this was the case, the specimen elongation and the applied force were compared from one sample point to the next, as shown in Figure 11.

It was noticed that, at the beginning of the tensile test, the applied force does not always increase as the position of the crosshead changes. Instead the force remains unchanged or even decreases following an initial increase. A moment later it increases again and this time the change is more rapid. Corresponding to the force, the elongation measured by the extensometer also exhibits a strange pattern in the same region.

An explanation for this phenomenon can be given knowing that the force has been transferred to the specimen through a pair of wedge action, or self-tightening, grips. The decrease in force following an initial increase can be considered to be a result of the grips biting into the material (Figure 12). The indentation by the serrated grip faces may have caused certain plastic flow on the surface of the specimen, and it apparently has been sensed by the extensometer as suggested by the elongation behavior seen in Figure 16. The combination of the surface indentation and the surface plastic flow appears to be what gave the erroneous stress-strain behavior that in turn caused large variations in strain and modulus.

To verify this hypothesis, tensile tests were conducted on a few samples using a pair of side-action grips in which the on-going surface indentation is not an issue due to the lack of self-tightening.

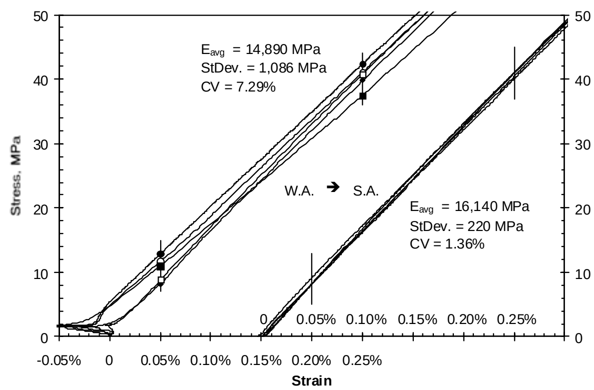

Figure 11: Effect of Grips on the Tensile Behavior of Thermoplastics (PA 6, 50% G.F.). The variation in modulus measurement associated with the wedge-action grips (W.A.) is seen to be reduced significantly with the use of side-action grips (S.A.)

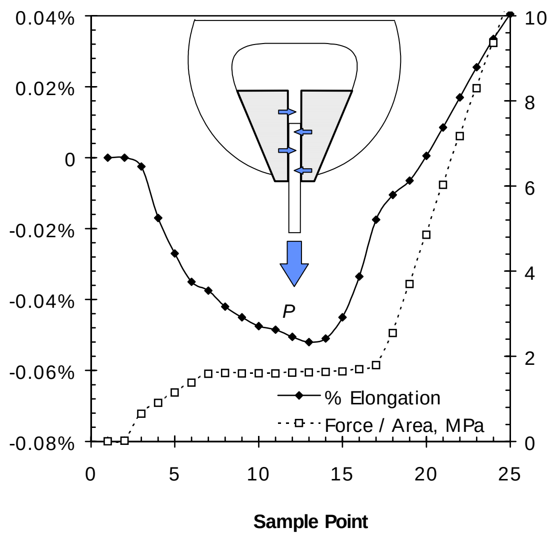

Figure 12: The Self-Tightening of the Wedge-Action Grips Was Considered to be Responsible for the Large Variability in Strain and Modulus Measurement (Figure 5)

Figure 13 shows the stress-strain in the same region as Figure 12. Sure enough, the force and elongation behavior that caused large errors is no longer there. The significantly reduced variability is obvious in Figure 12 where the stress-strain curves with wedge-action and side-action grips are compared.

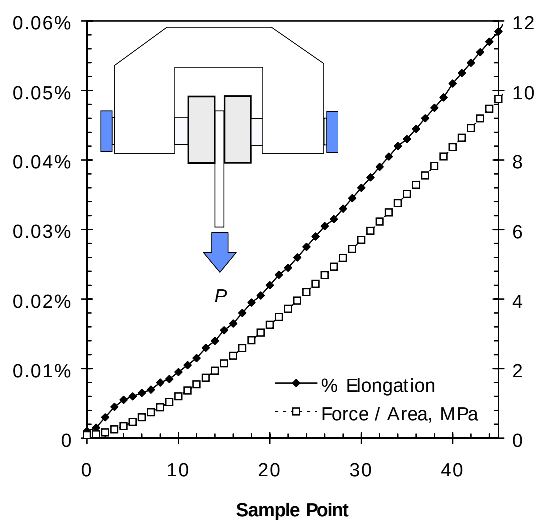

Figure 13: “Well-Behaved” Stress and Strain Curves with the Use of Side-Action Grips

CONCLUSIONS

- Tensile strength and deformation parameters of PA 6 obtained by ISO and ASTM methods are generally compatible; both can be used for the design of injection molded, non-reinforced and glass fiber reinforced parts and the material pre-selection.
- The ISO tensile stress and Young’s modulus values are found to be slightly higher than those of ASTM for reinforced and non-reinforced semi-crystalline PA 6, and amorphous PP [2].
- The value of Young’s modulus can be significantly affected by the method of tensile strain calculation, which can be obtained with or without an extensometer.
- Use of wedge-action grips may cause large variability in strain and modulus calculation, and this variability can be reduced significantly by using a pair of side-action grips.
- Moisture and temperature have similar effects on the tensile properties. With moisture growth tensile strength

decreases.
- Affect of moisture content on tensile strength is more effective at room temperature conditions, and is less significant at minus at elevated temperatures.

ACKNOWLEDGMENTS

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KEYWORDS

Nylon; polyamide; plastic; stress-strain; Young's modulus; thermoplastics; tensile; moisture; absorption; water; relative humidity; conditioning; temperature, fiber-glass; reinforcement.

ABBREVIATIONS

BK – carbon black; GF – fiber-glass reinforcement; IM – impact modified; MF – mineral filled; PA – polyamide; RH – relative humidity; ASTM – American Society of Testing and Materials; ISO – International Organization for Standardization.

TERM

Conditioning – the whole series of operations intended to bring a sample / plastic part into a state of equilibrium with regard to temperature and humidity.

Conditioning atmosphere – the atmosphere in which a sample / plastic part is kept before being subjected to test.

Test atmosphere – the atmosphere to which a sample / plastic part is exposed throughout the test.

Moisture absorption – the pickup of water vapor from air by a material, in reference to vapor withdrawn from the air only, as distinguished from water absorption which is the gain in weight due to the absorption of water by immersion.

The water absorption may be expressed in the following ways:
- as the mass of water absorbed;
- as the mass of water absorbed per unit of surface area;

- as a percentage by mass of water absorbed with the respect to the mass of the test specimen.

Moisture content – the amount of moisture in a material determined under prescribed conditions and expressed as a percent of the mass of the moist specimen, that is, the mass of the dry substance plus the moisture.

Moisture equilibrium – the condition reached by a sample when it no longer takes up moisture from, or gives up moisture to, surrounded environment.

Moisture regain – the moisture in material determined under prescribed conditions and expressed as a percent of the weight of the moisture-free specimen. Moisture regain may result from either sorption or de-sorption, and differs from the moisture content only in the basics used for calculation.

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