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EFFECTS OF MOISTURE CONDITIONING METHODS ON MECHANICAL PROPERTIES OF INJECTION MOLDED NYLON 6

Abstract

The influence of various standard (ASTM, ISO) and experimental moisture conditioning methods on mechanical performance of injection molded nylon 6 is discussed as a result of an in-depth, comprehensive investigation.

The analyzed methods covered a wide range of two basic process parameters for conditioning: temperature (from 23 to 100°C) and relative humidity (from 50% RH to water immersion). The variation of these parameters may result in significantly different moisture absorption rates, equilibrium levels and mechanical properties. The kinetic of mechanical performance and microstructure were evaluated prior to tests and during conditioning in this comprehensive analysis.

The results from this investigation may provide comprehensive, up-to-date information and recommendations concerning accelerated nylon conditioning methods for test specimens and various molded parts, pre-selection of nylon based plastic for design, and prediction and optimization of mechanical performance.

Introduction

Nylons, or polyamides (PA), are high performance semi-crystalline thermoplastics with attractive physical and mechanical properties that provide a wide range of end-use performances important in many industrial applications.

The growth in thermoplastics and composites continued in global market during 2000, at a rate of 4% in North America and ≈ 5-6% worldwide. At the same time, worldwide usage of nylon is growing annually by 8% to 9%. Much of the recent growth in nylon has been found in automotive, where nylon made parts are gradually replacing metals (various steels and light alloys, aluminum and magnesium based), and in some case expensive plastics, including various thermosets.

All nylons are hygroscopic (moisture sensitive), which is an important factor to be considered during material pre-selection, parts design, mechanical performance prediction and optimization. Previously we reported to Antec'2000 [1] our findings on the influence of two very important environmental conditions (absorbed moisture and temperature) for 33 wt.% fiberglass reinforced nylon 6. In general, the moisture content in nylon is a key variable affecting processing (polymerization, compounding, molding, welding, etc.) and end-use performances (mechanical, dimensional, surface appearance, etc.). The absorbed water in polymer behaves like plasticizer, which affects material properties such as strength, stiffness, and ductility. Water also results in deterioration of electrical properties.

At 50% RH and 23°C equilibrium moisture content for various nylons, such as PA 6, PA 66, and PA 46, may vary in wide range from 1.2 wt.% to 3.8 wt. % [2-3]. While many development and research programs, and published reports and articles were focused on the development of engineering databases needed for plastic part design with the influence of end-use conditions, including environmental consideration [2-4], a few studies have been concentrated on:

- Compatibility of the mechanical performance of materials and molded parts conditioned using different standards or methods.
- Efficiency of different conditioning methods in terms of moisture at equilibrium and time to reach equilibrium.
- Development of new conditioning procedures that allow moisture to reach desired equilibrium level in materials within a reasonable time period.

Theoretical Basics in Diffusion and Moisture Absorption in Homogeneous Thermoplastics

The fundamental laws of diffusion in anisotropic solids have been known for a long time [5-7]. With the rapidly increased use of polymer based materials in the early 70', the interest in moisture absorption characteristics increased dramatically.

The amount of moisture absorption during a specific period of time (t) depends on the diffusion coefficients of the individual component in the plastic/composite. For fiber reinforced plastic composites, the diffusion coefficient depends on the following three factors:

- Volume or weight fraction of fibers;
- Diffusion coefficient of the matrix (base resin);
- Temperature.

The inhomogeneities in the material, such as voids, micro-porosity, and damages related to thermos-mechanical loading history in material, may affect the diffusion coefficient. The current investigation was focused on moisture absorption in unfilled nylon 6, a material considered homogeneous. The driving force for moisture diffusion is the gradient in the moisture concentration. For homogeneous materials, the moisture diffusion follows Flick's law. It was observed [5-6] that moisture absorption in fiber reinforced plastics correlated very well with Flick's laws. The moisture flux is given by Fick's first law:

$$J_i = -D_{ij} \frac{\partial C}{\partial x_j} \qquad (1)$$

where J_i is the moisture flux, D_{ij} the mass diffusion tensor, C the moisture weight fraction content and x_i the spatial coordinate. Some details in C calculation for various composites are discussed in [4-7]. From eq.(1) it is possible to derive the change of moisture content with time (t) and Flick's second law of diffusion is obtained by:

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x_i}\{D_{ij} \frac{\partial C}{\partial x_j}\} \qquad (2)$$

For a unidirectional fiber reinforced composite the diffusion coefficient is anisotropic and depends on fiber orientation (longitudinal - L and transverse T). These coefficients may be expressed in terms of the fiber and matrix diffusion coefficients by the rule of mixture (D_L), using an analogy with thermal conductivity [5]:

$$D_L = \nu_f D_f + D_m(1-\nu_f) \qquad (3)$$

$$D_T = D_m f(\nu_f, D_f, D_m) \qquad (4)$$

where the subscripts f and m stand for fiber and matrix, respectively. For glass or carbon fiber reinforcement, D_f is negligible compared to D_m, and equation (3) and (4) can be simplified to:

$$D_L = D_m(1-\nu_f) \qquad (5)$$

$$D_T = [1 - 2(\nu_f/\pi\}^{0.5}] \qquad (6)$$

Experimental

Prediction of equilibrium moisture level in various thermoplastics (unfilled and fiber-reinforced) by equations (1) – (6) requires experimental data on kinetic of moisture absorption, which enables one to verify the accuracy used in test & evaluation methods and theoretical assumptions. Accurately developed data for the matrix (base polymer) is the key data for a comprehensive analysis of this dynamic process.

Materials and Specimens

The material used in this investigation was heat-stabilized, unfilled nylon 6 (Capron 8202 HS)[1]. The material was injection molded into 4-mm thick ISO multi-purpose tensile bars (ISO-3167). Molded specimens were properly sealed in special bags prior to conditioning in order to preserve their dry-as-molded state while the moisture content remains at ~ 0.2%.

Moisture Measurement

The moisture content of the all specimens was calculated from the weight gains using a Mettler balance (Figure 1). A Karl-Fischer moisture analyzer was also used to measure water content in a selected group of specimens. The comparison of the two methods is shown in Figure 2. Samples were then sealed tightly in moisture proof bags until tests or analyses were performed.

Mechanical Tests

The tensile and flexural properties of nylon 6 in this study were obtained on ISO multipurpose specimens using ISO and ASTM standards. Tests were conducted using an Instron 4505 universal testing system.

Advantages and Limitation of the Conditioning Procedures

The following two basic standard and experimental moisture conditioning methods (Table 1) were utilized in various studies [1—5]:

- Immersion in water (at room temperature and boiling water temperature - 100°C);
- Standing in air (temperatures - from 23°C to 70°C, and relative humidity – from 50%RH to 100%RH).

The basic procedures were based on ISO-1110 (when the temperature and relative humidity are maintained at 70°C and 62%, respectively. Although ISO-1110 can greatly accelerate the moisture absorption in nylon compared to the standard method "Atmosphere 23" ("AT-23"), it can also cause "over conditioning [1]" (more moisture than what can be obtained under "AT-23"). For this reason, the conditioning was terminated after the moisture in the material reached the equilibrium level under "AT-23".

The properties and performance of nylon 6 are greatly influenced by the level of moisture, which makes it important to analyze properties at various moisture levels. The effect of moisture can be a major factor in products that were exposed to different weather

[1] Capron® - is a registered trademark for BASF Corporation nylon/polyamide plastic products.

conditions due to changing in seasons or geographic locations.

Discussions

In general, the effect of moisture can only be properly analyzed after it has reached equilibrium and is uniformly distributed in the material. Practically, however, conditioning materials to the equilibrium can be difficult due to the following:

- The rate of moisture uptake in nylon, under the conditions suggested in the standard practice as in ASTM method, is very low. The moisture may take more than a year to reach equilibrium therefore it is impractical for material testing and qualification.
- The equilibrium moisture is strongly dependent on temperature and relative humidity. As a result, materials conditioned using one method cannot be directly compared with those conditioned using a different method.
- Although greatly desired and often practiced, the rapid moisture uptake as that seen in water immerse, may result in additional, undesirable structural changes in nylon.

Problems may also arise when materials are conditioned to the same moisture level but using different methods. Often in this case the properties of materials (e.g. tensile modulus) are found different (Figure 3), presumably due to the uneven moisture distribution across the thickness of the test specimens or structures. Figures 4-6 show the similar effect of conditioning on flexural properties from analysis of four conditioning methods.

It is highly desirable to develop an accelerated conditioning method that engineers could use to achieve needed moisture levels in nylon at much shorter period of time without significant deviation in properties comparing to “AT-23”. Several Temp/RH combinations used in this investigation have been found to result in very close property values at each moisture absorption level. This has made it possible in practice to choose conditions that take shorter time to reach a given moisture level, therefore accelerate the conditioning process.

Table 1 shows the change in T_g as the result of moisture absorption. With moisture increase from 0.15% (this level is typical for “dry-as-molded conditions”) to 1.36% glass-transition temperature T_g decreases from 47°C to 8°C. By these reasons conditioning temperature at 55°C will be of a less concern for nylon 6. Additional examination of the changes in crystallinity (type, size and distribution) under different conditionings methods, should further enrich this study by relating property changes to changes in microstructure.

Summary and Conclusions

Nylons are high performance semi-crystalline thermoplastics with a number of attractive physical and mechanical properties, utilized in many industrial applications. At the same time, all nylons are hygroscopic (moisture sensitive), and one needs to take into account this very important factor in material pre-selection stage, plastic parts design, mechanical performance prediction and optimization.

The properties and performance of nylon 6 are greatly influenced by the level of moisture, therefore it’s important to analyze properties at various moisture levels. The effect of moisture can be a big factor in products that were exposed to different weather conditions due to change in seasons or geographic locations.

The following two basic standard and experimental moisture conditioning methods were investigated in this study:

- Immersion in water (at room temperature and boiling water temperature - 100°C);
- Standing in air (temperatures - from 23°C to 70°C, and relative humidity – from 50%RH to 100%RH).

It is very important to explore the possibility of acceleration in conditioning that engineers could use to achieve desired moisture level in nylon at much shorter time, yet without causing significant deviation in properties comparing to “AT-23”. Several temperatures (55°C and 70°C) and RH (62%, 50%) combinations are shown to result in very close property values at given moistures. This has made it possible in practice to choose conditions that take shorter time to reach a given moisture level, therefore accelerate the conditioning process.

The results from this investigation will provide comprehensive, uniform, and up-to-date information and recommendations for moderate accelerated methods of conditioning of the test specimens and various molded parts, pre-selection of nylon based plastic for design, mechanical performance prediction and optimization.

Acknowledgment

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Table 1. Influence of Moisture Content (in %) on Glass Transition Temperature (in °C)

% H_2O	T_g, °C	Temp/RH, °C/%
0.15	47	23/50
0.258	37	23/50
0.74	25	23/50
1.36	8	23/50

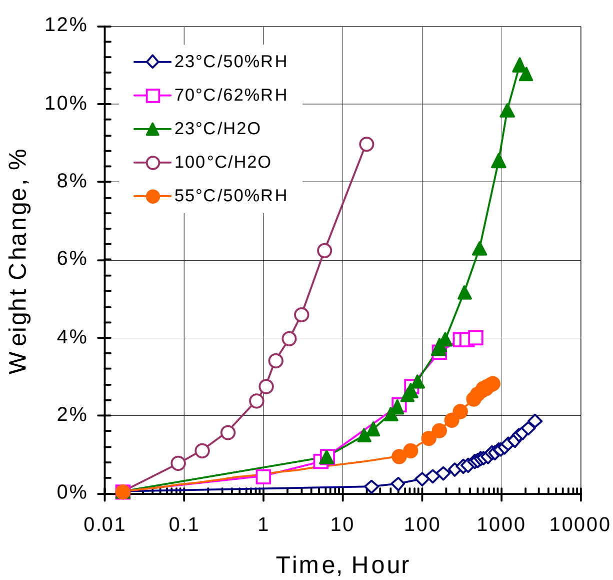

Figure 1. Moisture Gain (in %) vs. Time (in hours)

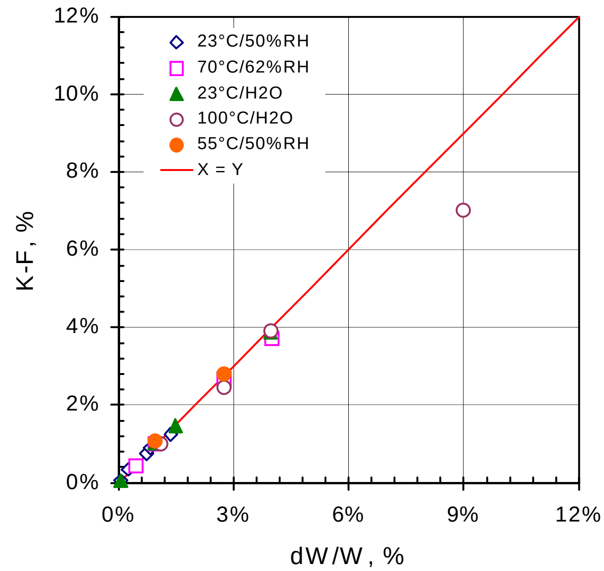

Figure 2. Efficiency of Moisture Measurement Methods: Weight change vs. Karl-Fischer

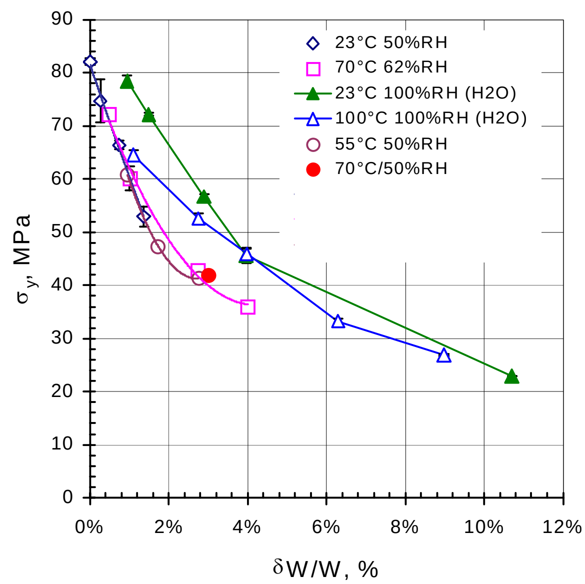

Figure 3. Tensile Stress at Yield vs. Moisture.

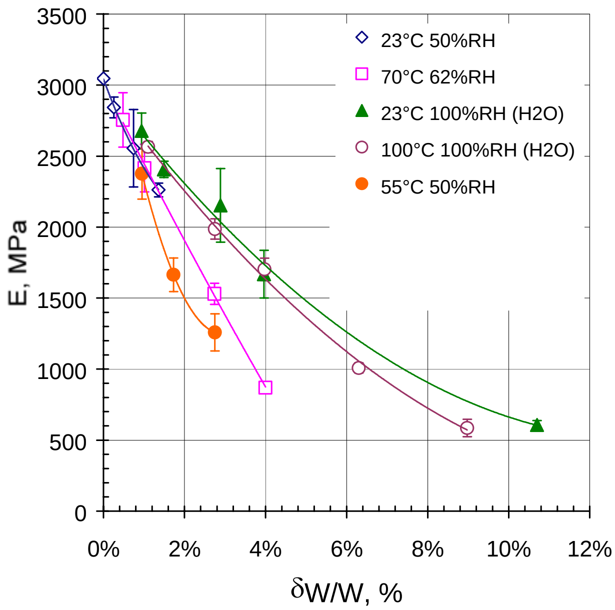

Figure 4. Tensile Modulus vs. Moisture.

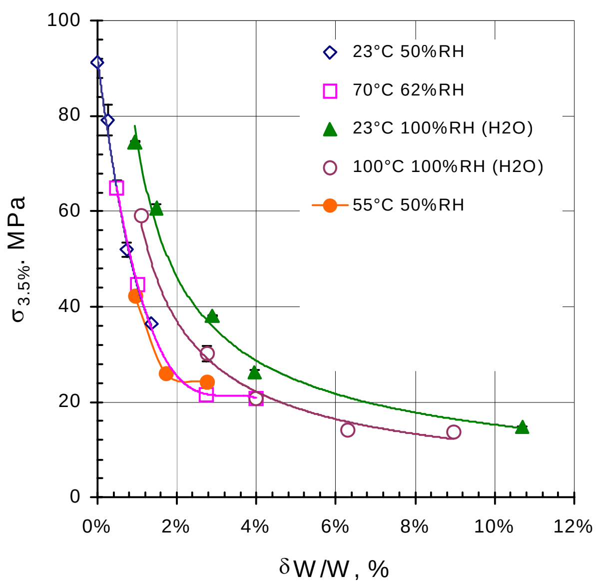

Figure 5. Flexural Stress @ 3.5% Flexural Strain vs. Mositure.

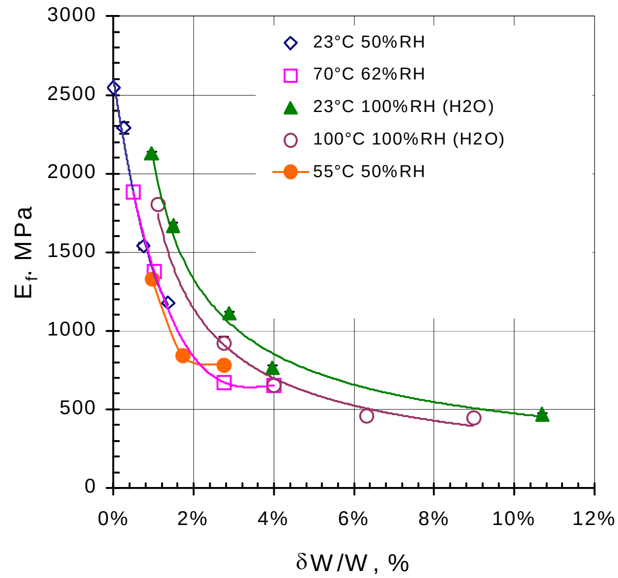

Figure 6. Flexural Modulus vs. Moisture.

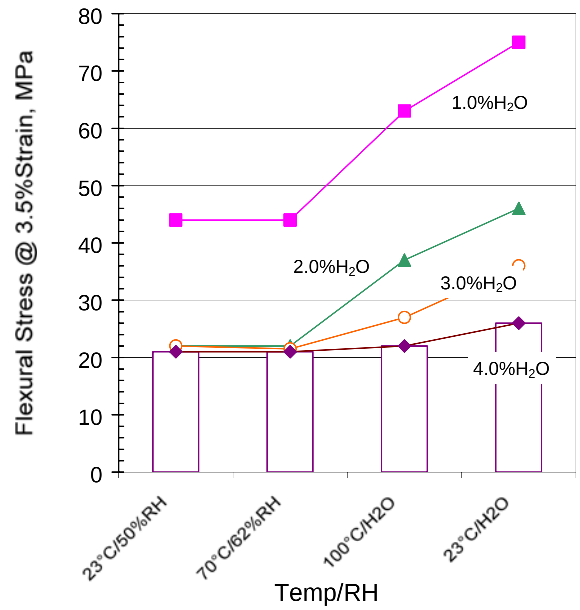

Figure 7. Effects of Conditioning Methods on Flexural Stress.

Keywords

Nylon 6, moisture, conditioning, relative humidity, strength, mechanical performance.

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