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## Measurement of the Coefficient of Hygroscopic Expansion (CHE) Using Controlled Humidity Dynamic Mechanical Analysis

### ABSTRACT

This paper discusses the direct measurement of the coefficient of hygroscopic expansion of a Nylon 6 sample using the TA Instruments Q800 Dynamic Mechanical Analyzer equipped with the DMA-RH Accessory to control relative humidity.

### INTRODUCTION

Hygroscopy is defined as the ability of a substance to attract water molecules from the surrounding environment through either absorption or adsorption. Materials exhibit differing degrees of hygroscopicity, which can dramatically affect the mechanical viability of the material, particularly when used in a composite structure. The effect of moisture sorption on the mechanical characteristics of a material can be quantified by the Coefficient of Hygroscopic Expansion, or CHE (also referred to as Coefficient of Hygroscopic Swelling or CHS). This term is analogous to the more commonly determined Coefficient of Thermal Expansion (CTE), and is the constant which relates the dimensional change of a material to a change in the surrounding relative humidity.

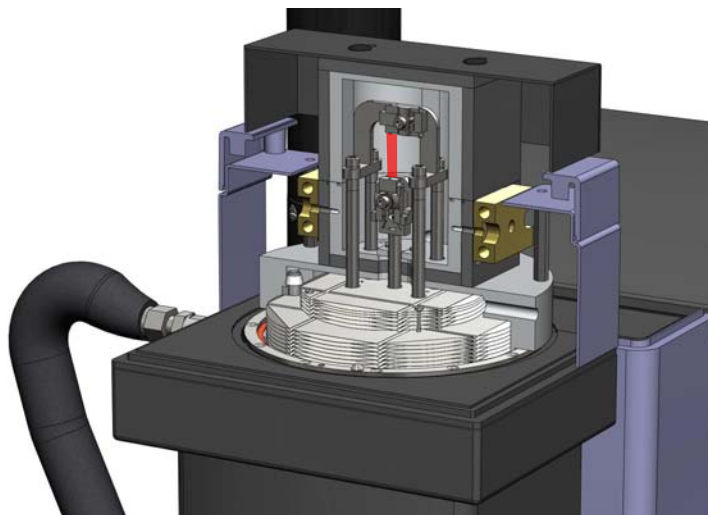
If materials of dramatically different CHE are combined in a composite structure, the resultant expansion differences can cause stress and/or deformation to occur. A common example where this effect can be seen is in a paperback book cover. In a humid environment, the unlaminated side of the cover will absorb more moisture than the laminated side, and the resultant increase in area will cause a stress that curls the cover toward the laminated side. This is similar to how simple bimetal strips are used as torsion springs in thermometers. It is thus important to understand the CHE of materials, to minimize potential detrimental sorptive effects.

The TA Instruments DMA-RH Accessory allows the mechanical properties of a sample to be analyzed under controlled conditions of both relative humidity and temperature. It is designed for use with the Q800 Dynamic Mechanical Analyzer. The DMA-RH accessory is an integrated unit and contains the following components:



Figure 1: The TA Instruments Q800 Dynamic Mechanical Analyzer and DMA-RH Accessory

1. The sample chamber mounts to the DMA in place of the standard furnace and encloses the sample. Peltier elements in the chamber precisely control the temperature to within  $\pm 0.1^\circ\text{C}$ . The sample chamber accommodates standard DMA clamps including tension, cantilever, and 3-point bending, and can be easily removed for rapid conversion back to the standard DMA furnace.
2. The DMA-RH Accessory contains the humidifier and electronics which continuously monitor and control temperature and humidity of the sample chamber. The DMA Q800 and the DMA-RH Accessory are fully software-integrated.
3. A heated vapor transfer line is maintained above the dew point temperature of the humidified gas in order to avoid condensation and provide accurate results.



**Figure 2: Sample Chamber of the DMA-RH Accessory**

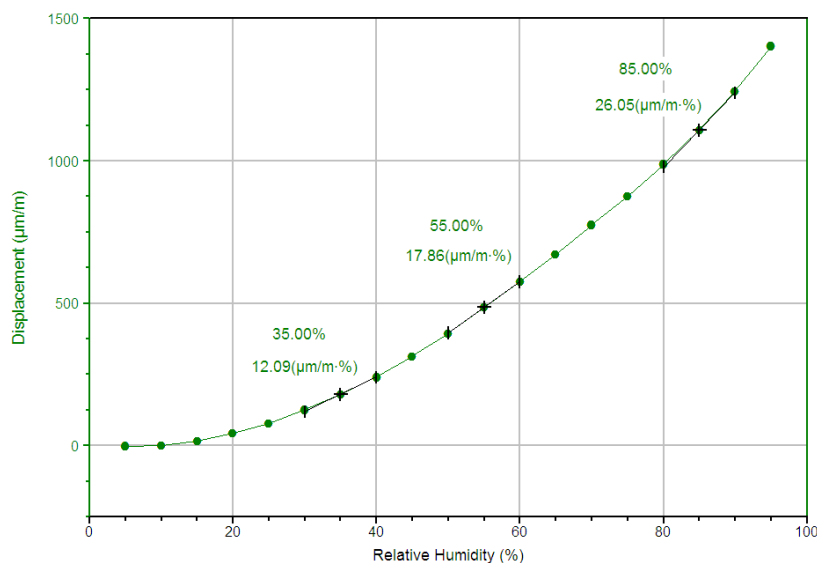
The DMA-RH accessory allows for the control of temperature over the range  $5\text{-}120^\circ\text{C}$ , and humidity over the range  $5\text{-}95\%$  RH. As such, it is well-suited to materials in which mechanical properties are of interest this temperature and humidity range.

## **RESULTS & DISCUSSION**

In this study, the CHE of Nylon 6 at  $25^\circ\text{C}$  is determined. Nylon 6 shows significant adsorption of and is plasticized by water; as a result the mechanical properties of this polymer will be dependent on the imposed relative humidity. A thin strip of Nylon 6 ( $13\text{mm} \times 6.4\text{mm} \times 0.12\text{mm}$ ) was cut from a sheet and mounted in a film tension clamp on the Q800 DMA. The following thermal/RH method was employed. (Explanations of specific method steps are given.)

- 1) Force = 0 N (removes all residual force)
- 2) Relative humidity 0.00 % (purges sample chamber with dry nitrogen)
- 3) Equilibrate at  $25.00^\circ\text{C}$
- 4) Isothermal for 120.00 min (allows for desorption of residual moisture)
- 5) Measure Length (normalized displacement after contraction to due desorption)
- 6) Data storage Off (disregards transient data)
- 7) Increment humidity 5.00 % (sets RH control)
- 8) Isothermal for 44.00 min (allows time for sample to equilibrate under set RH)
- 9) Data storage On
- 10) Isothermal for 1.00 min (collects equilibrium data)
- 11) Data storage Off
- 12) Repeat segment 7 for 18 times (programs RH steps up to 95%)

The resulting data (disregarding the initial drying step) is shown in Figure 3.



**Figure 3: Coefficient of Hygroscopic Expansion Data for Nylon 6 at 25°C**

The data in Figure 3 show the effect of imposed relative humidity on the Nylon 6 sample. As the relative humidity is increased the sample expands. TA Instruments Universal Analysis software allows the displacement data to be normalized to the initial sample length, as is typical in CTE experiments. The resulting slope of the line is equivalent to the CHE for the material. The data is nonlinear over the entire relative humidity range, but exhibits narrow regions of linearity sufficient to calculate a representative slope. Note that Nylon 6 demonstrates an increasing CHE as relative humidity is increased. The calculated value at 35% RH is 12  $\mu\text{m}/\text{m}\cdot\%$ , which more than doubles to 26  $\mu\text{m}/\text{m}\cdot\%$  at 85% RH. This is not unexpected, as Nylon 6 is known to show significant adsorption and is heavily plasticized by water.

## CONCLUSIONS

The data presented illustrates the determination of the Coefficient of Hygroscopic Expansion of Nylon 6. This experiment requires an instrument with excellent sensitivity for small dimensional changes, as well as precise relative humidity control. The TA Instruments Q800 DMA and DMA-RH Accessory provide the ideal platform for the study of relative humidity effects on the mechanical properties of polymeric materials, and the accurate and straightforward determination of CHE.

## **TA Instruments**

### **United States**

109 Lukens Drive, New Castle, DE 19720 • Phone: 1-302-427-4000 • E-mail: [info@tainstruments.com](mailto:info@tainstruments.com)

### **Canada**

Phone: 1-905-309-5387 • E-mail: [shunt@tainstruments.com](mailto:shunt@tainstruments.com).

### **Mexico**

Phone: 52-55-5200-1860 • E-mail: [mdufinguez@tainstruments.com](mailto:mdufinguez@tainstruments.com)

### **Spain**

Phone: 34-93-600-9300 • E-mail: [spain@tainstruments.com](mailto:spain@tainstruments.com)

### **United Kingdom**

Phone: 44-1-293-658-900 • E-mail: [uk@tainstruments.com](mailto:uk@tainstruments.com)

### **Belgium/Luxembourg**

Phone: 32-2-706-0080 • E-mail: [belgium@tainstruments.com](mailto:belgium@tainstruments.com)

### **Netherlands**

Phone: 31-76-508-7270 • E-mail: [netherlands@tainstruments.com](mailto:netherlands@tainstruments.com)

### **Germany**

Phone: 49-6196-400-7060 • E-mail: [germany@tainstruments.com](mailto:germany@tainstruments.com)

### **France**

Phone: 33-1-304-89460 • E-mail: [france@tainstruments.com](mailto:france@tainstruments.com)

### **Italy**

Phone: 39-02-2742-11 • E-mail: [italia@tainstruments.com](mailto:italia@tainstruments.com)

### **Sweden/Norway**

Phone: 46-8-555-11-521 • E-mail: [sweden@tainstruments.com](mailto:sweden@tainstruments.com)

### **Japan**

Phone: 813-5479-8418 • E-mail: [j-marketing@tainstruments.com](mailto:j-marketing@tainstruments.com)

### **Australia**

Phone: 613-9553-0813 • E-mail: [sshamis@tainstruments.com](mailto:sshamis@tainstruments.com)

### **India**

Phone: 91-80-2839-8963 • E-mail: [india@tainstrument.com](mailto:india@tainstrument.com)

### **China**

Phone: 8610-8586-8899 • E-mail: [info@tainstruments.com.cn](mailto:info@tainstruments.com.cn)

### **Taiwan**

Phone: 886-2-2563-8880 • E-mail: [skuo@tainstruments.com](mailto:skuo@tainstruments.com)

### **Korea**

Phone: 82.2.3415.1500 • E-mail: [dhchee@tainstruments.com](mailto:dhchee@tainstruments.com)

To contact your local TA Instruments representative visit our website at [www.tainstruments.com](http://www.tainstruments.com)

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