

# Dynamic Mechanical Properties of Extruded Nylon-Wood Composites

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# Global goal

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To characterize the dynamic mechanical properties of extruded nylon-wood composites generated in the AEWC Center at the University of Maine.

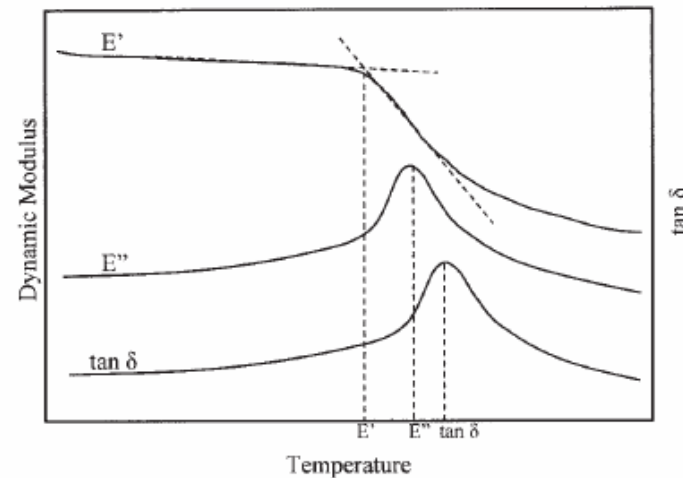
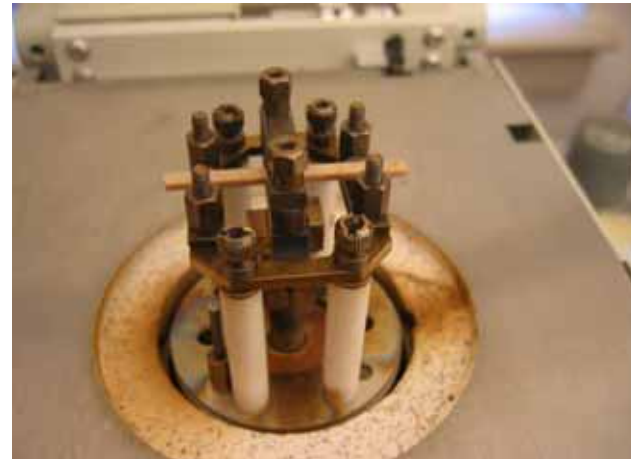
# Specific objectives

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- Temperature dependent storage modulus characterization.
- Glass transition ( $T_g$ ) measurement.
- Physical aging effects.
- Long term performance prediction.
- Comparisons to similar products.

# Glass transition of nylon wood composite— method of measurement

- Instrument: DMTA
- Test mode: three point bending test with temperature sweep.
- Determination: peaks on the curves of  $\tan \delta$ , storage modulus or loss modulus vs. temp.



# Glass transition of nylon wood composite– test parameters

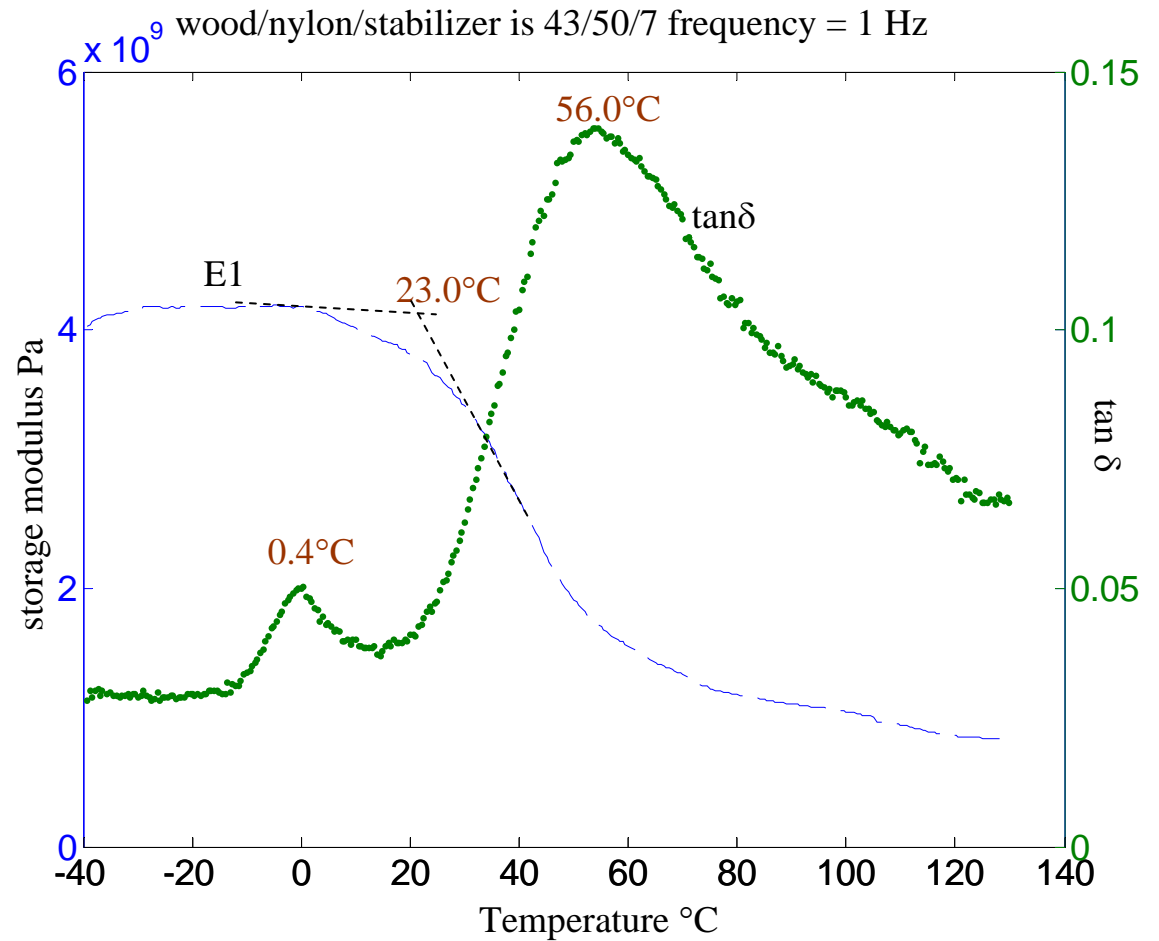
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- Sample preparation:  
Material: wood/nylon  
blend/stabilizer 43 / 50 / 7  
Size: 1.6 to 1.9 × 22 × 3.1 to  
3.3 (mm)  
Conditioning: dried in a  
desiccator 24 hours
- Frequency: 1 Hz
- Temperature: -40 to  
185°C
- Heating rate: 1°C/min
- Strain amplitude: 0.01%.



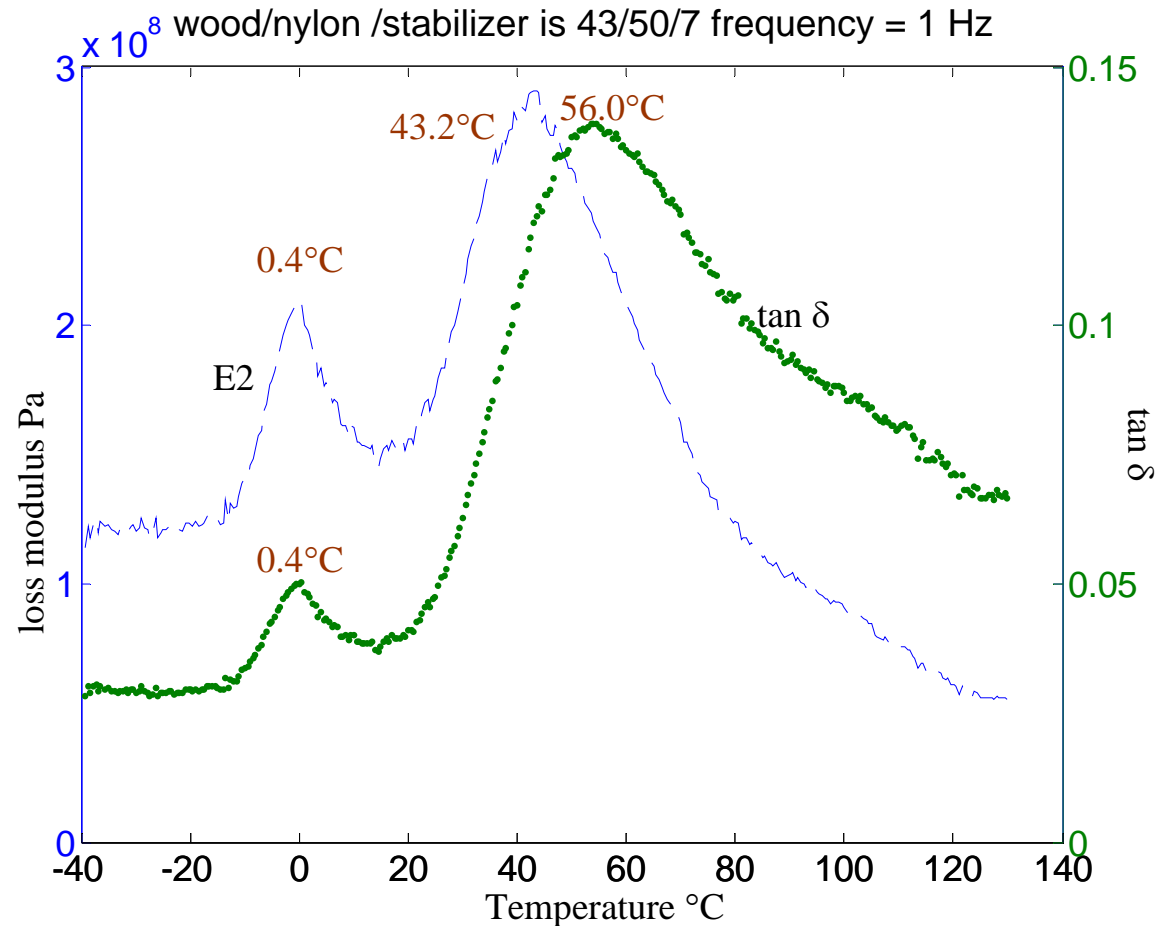
# Glass transition of nylon wood composite— tan $\delta$ and storage modulus

Tan  $\delta$  Tg 56°C  
Tan  $\delta$  0.4°C (phase separation)  
Storage E' Tg 23°C



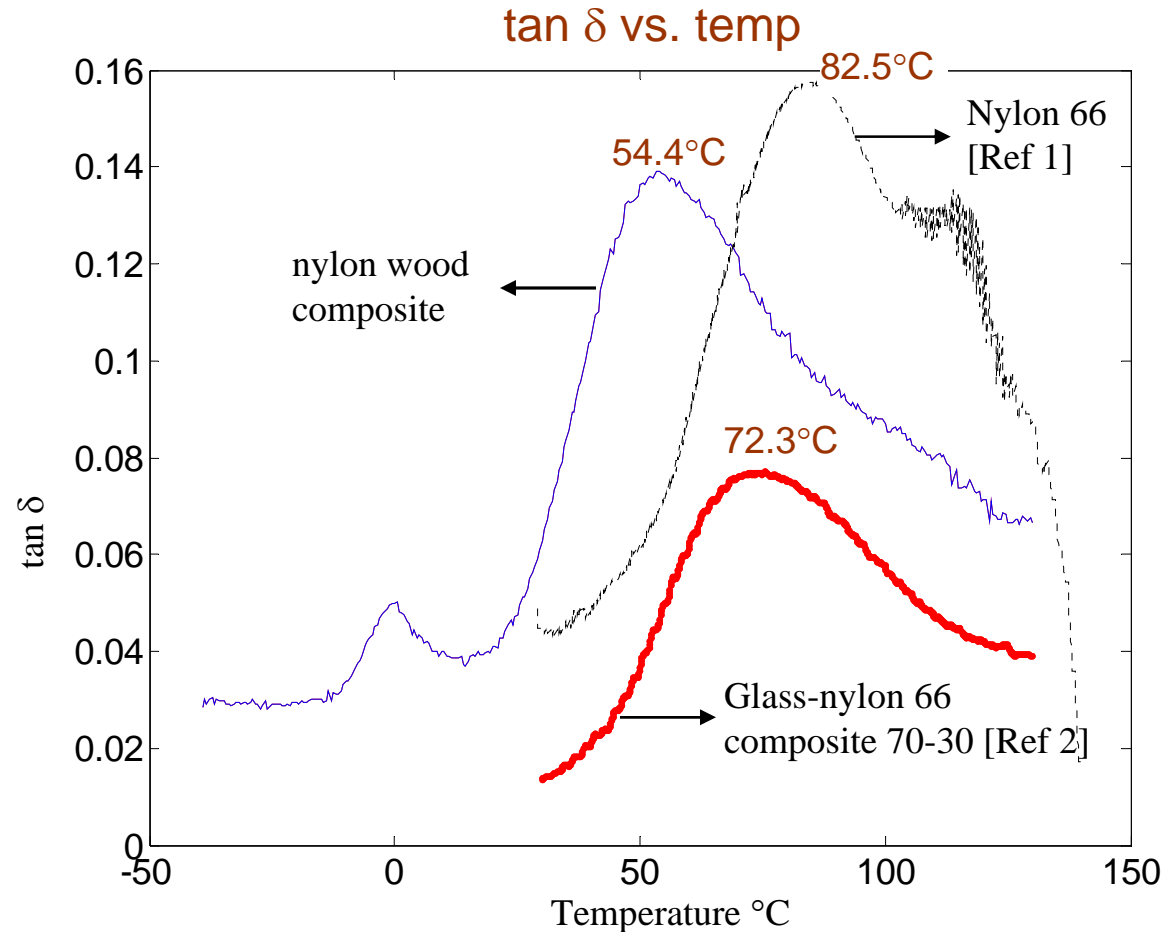
# Glass transition of nylon wood composite– tan $\delta$ and loss modulus

Tan  $\delta$  Tg 56°C  
Tan  $\delta$  0.4°C (phase separation)  
Loss E'' Tg 43.2°C



# TG comparison of nylon wood composite with nylon 66 and glass fiber reinforced

Tg of nylon wood composite shifted to lower temperature  
Caused by additives.



1. Clark, R.L., Influence of the Interphase on the Mechanical Properties of Nylon66 Composites, dissertation, Virginia Polytechnic Institute and State University, 1996

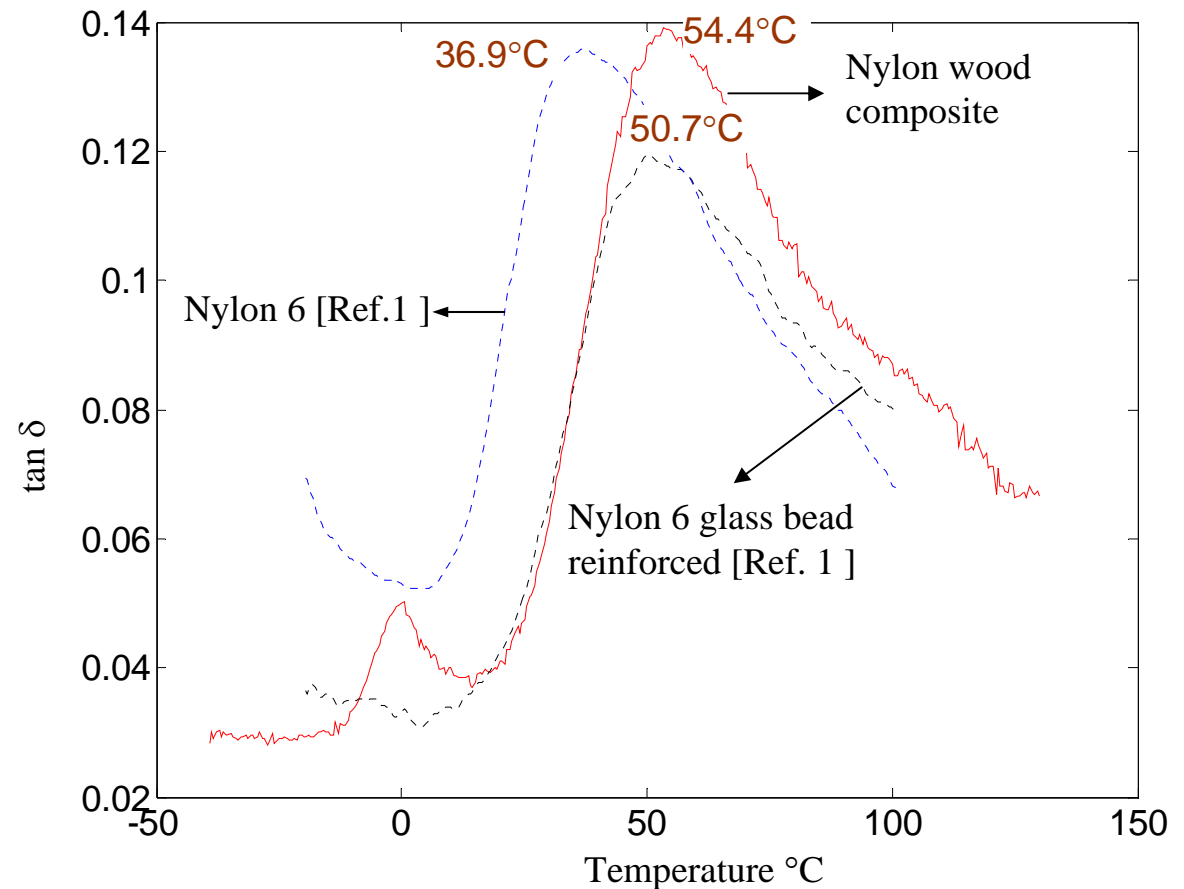
2. Feng, W.; Ait-Kadi, A.; Brisson, J.; Riedl, B. Polymer Composites 2003, 24, 512

# TG comparison of nylon wood composite with nylon 6 and glass bead reinforced

**T<sub>g</sub>:** of both nylon wood composite and nylon6-glass bead reinforced composite are higher than nylon 6.

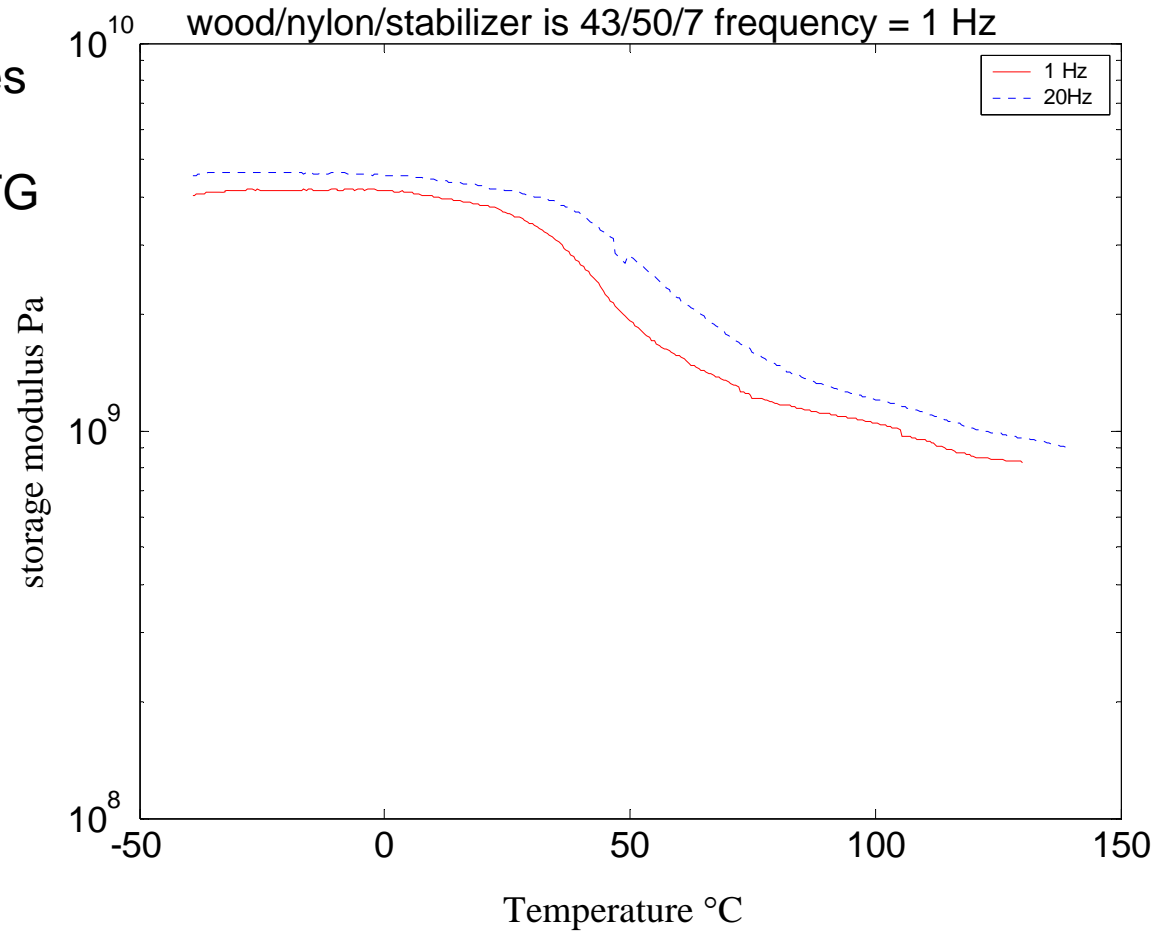
**Reason:**

- 1) Fillers raise TG
- 2) Additives lower TG



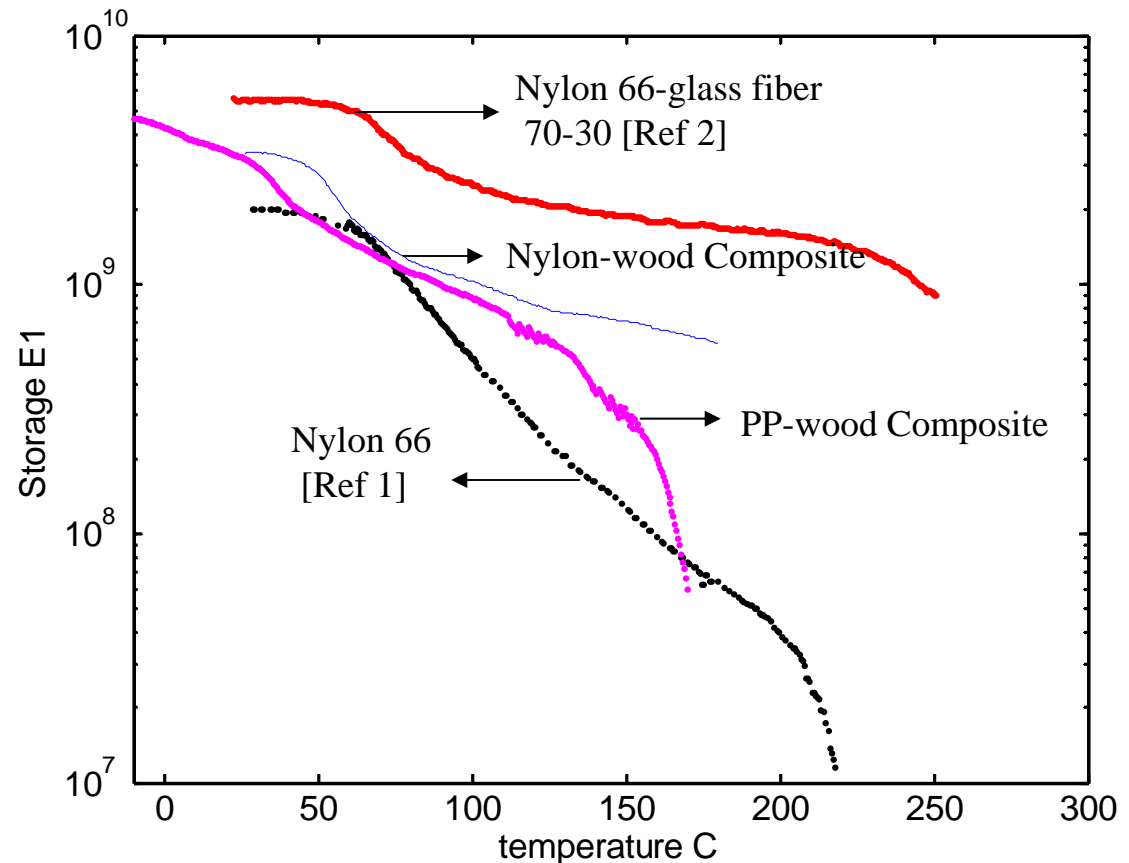
# Storage modulus of nylon wood composite

- 1) Storage modulus decreases with temperatures.
- 2) A significant drop around TG



# Storage modulus of nylon wood composite with nylon 66 and glass fiber reinforced

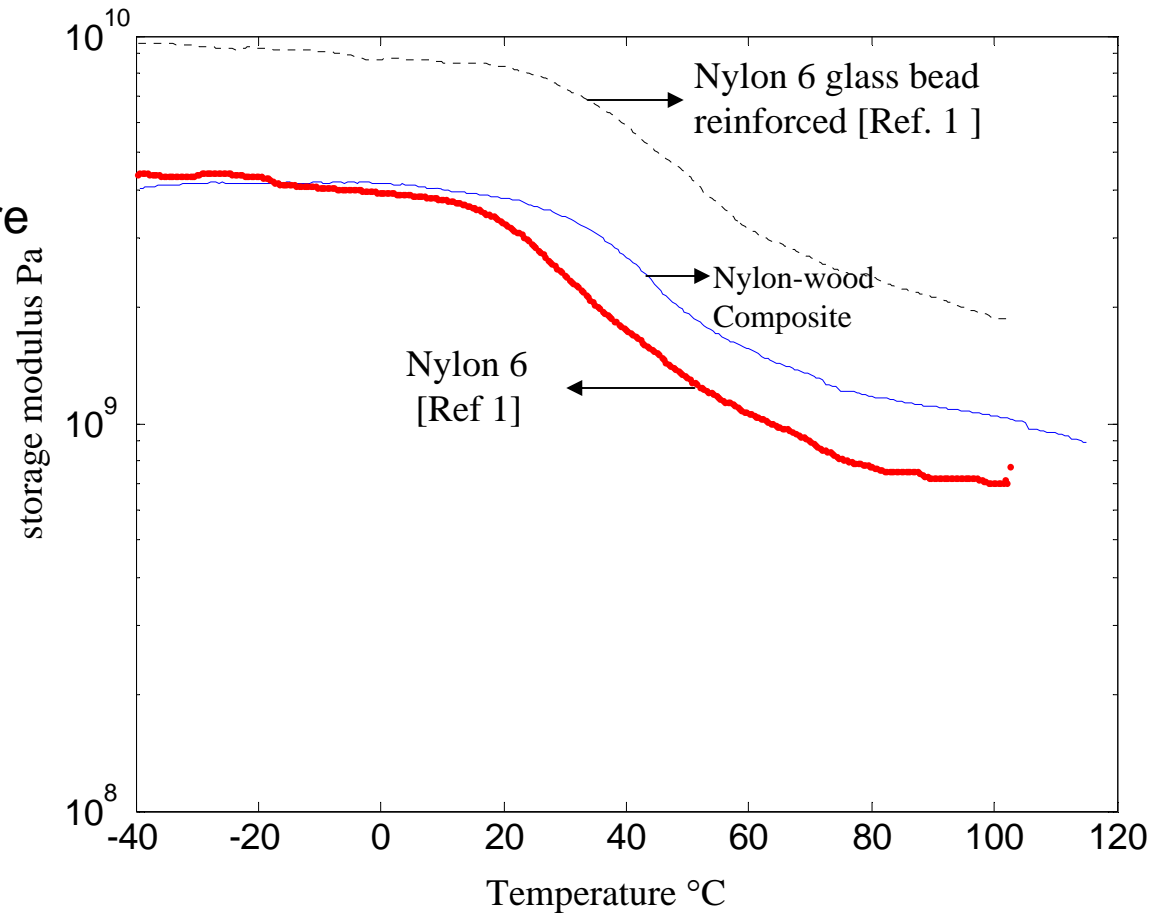
- 1) All curves have a distinct drop around TG.
- 2) Both glass fiber and wood flour improved the temperature stability of storage modulus of nylon 66.
- 3) The effects of glass fiber is more distinct than wood flour.



1. Clark, R.L., Influence of the Interphase on the Mechanical Properties of nylon66 Composites, dissertation, Virginia Polytechnic Institute and State University, 1996
2. Feng, W.; Ait-Kadi, A.; Brisson, J.; Riedl, B. Polymer Composites 2003, 24, 512

# Storage modulus of nylon wood composite with nylon 6 and glass bead reinforced

- 1) All curves have a distinct drop around TG.
- 2) Both glass beads and wood flour reinforced composite are more temp. stable than that of nylon 6.
- 3) The effects of glass bead is more distinct than wood flour.



# Aging effects

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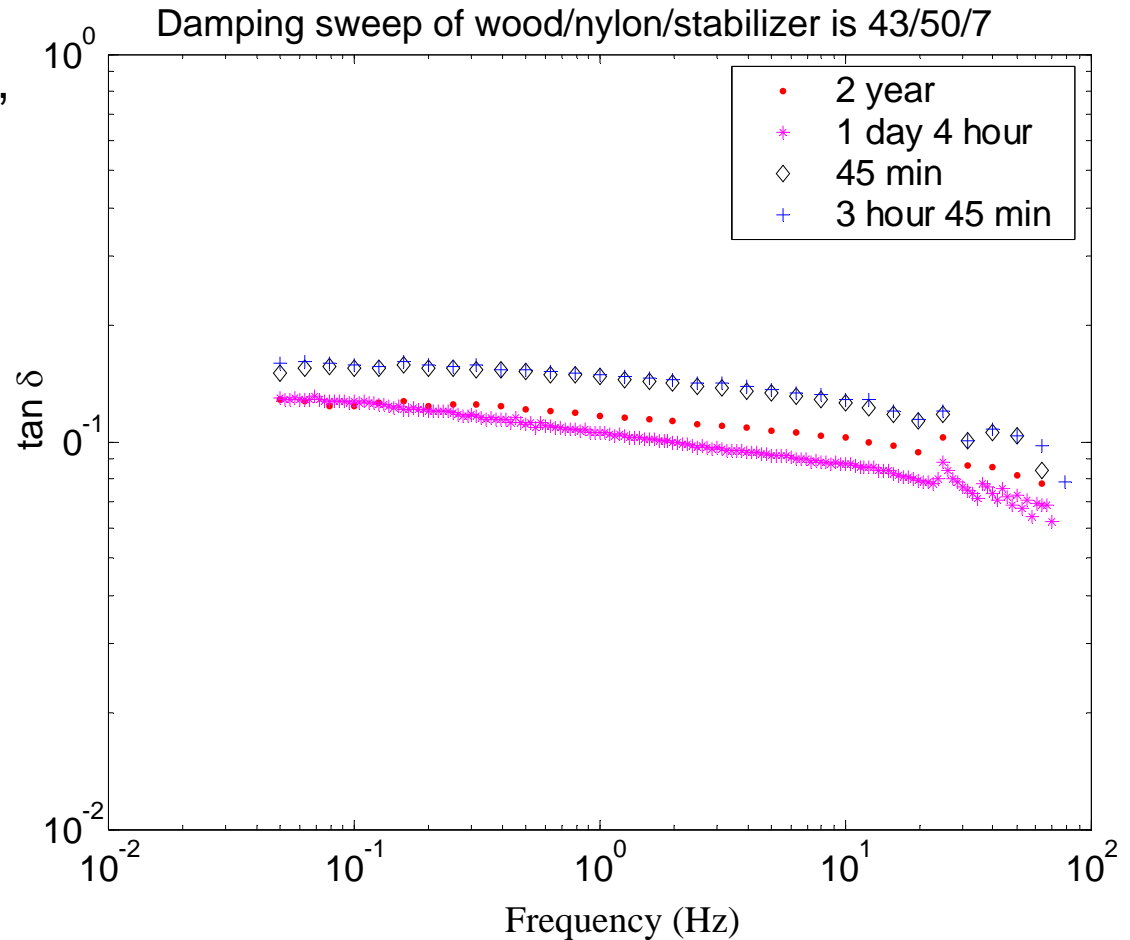
- Aging phenomenon:
  - more brittle
  - damping decrease
- Evaluation method
  - Changes of dynamic mechanical properties
  - Changes of creep and stress-relaxation

# Aging effects evaluation

- Damping changes ( $\tan\delta$ ) of composite are used to evaluate the aging effects.
- Frequency sweep at room temperature was used to conduct the tests.
- Samples were heated in oven at 90°C about 2 hours and quenched to room temperature.
- Samples were aged at room temperature lasting in different time.

# Aging effects of Nylon wood composite – damping changes as a function of aging time

- 1) Generally these curves distribute in a narrow band, which indicate that aging effects are not pronounced among these curves.
- 2) Curves at aging time =45min and 3 hours 45min are higher than that of aging time=2 year and 1 day 4 hour, which indicate that slight aging effects caused the damping decrease.



# Creep curves and long term prediction

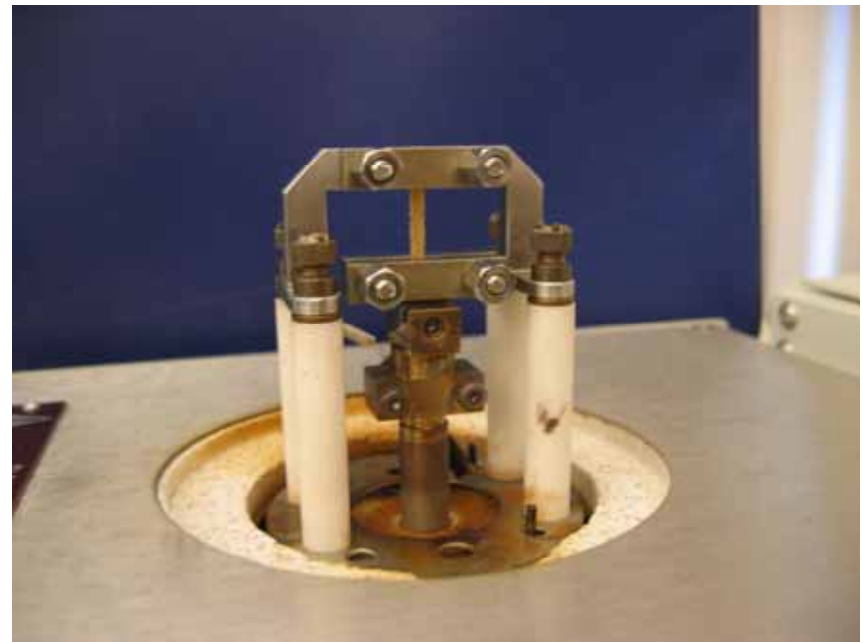
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- Temperature dependent creep behavior:  
creep curves measured at various temp.
- Long term mechanical behavior prediction:  
Time-Temperature Superposition Principle  
(TTSP)

# Creep tests of nylon wood composite

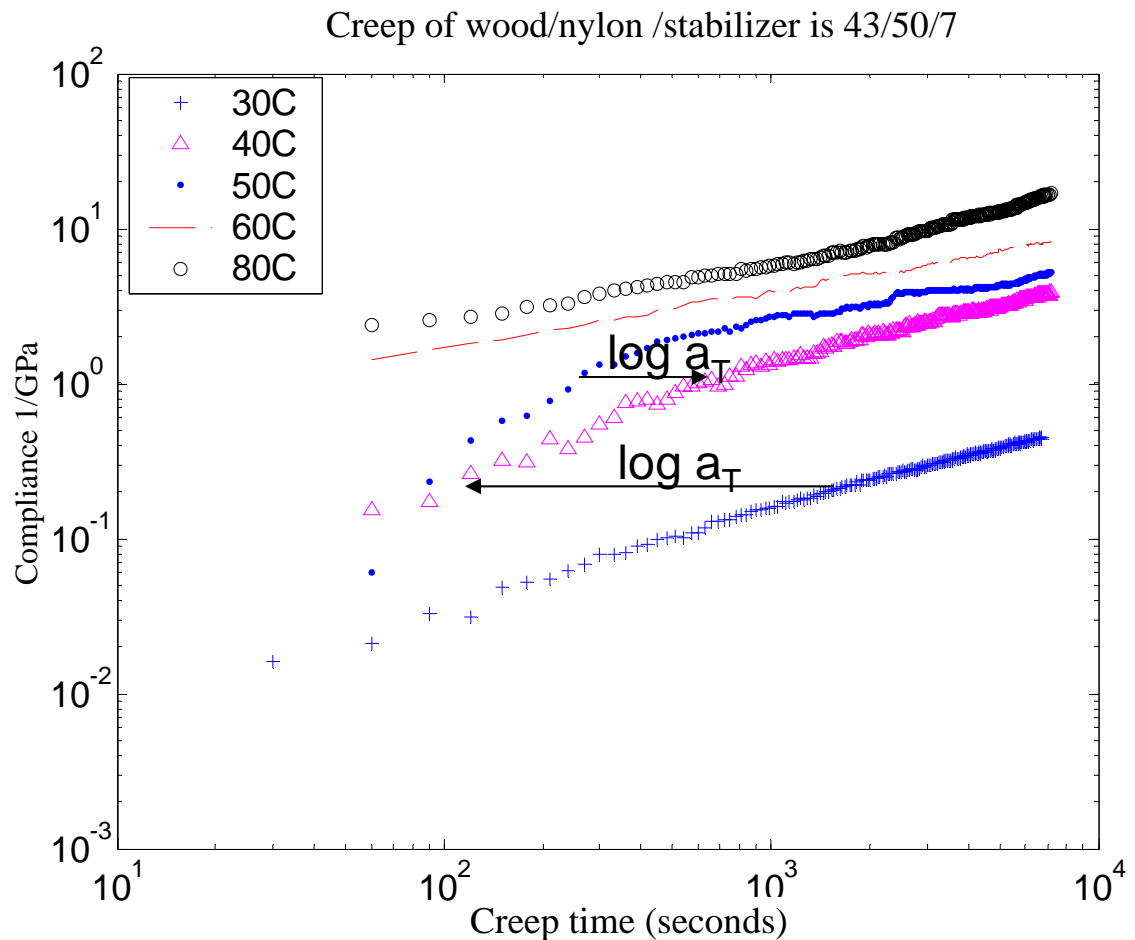
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- Instrument: DMTA
- Mode: creep test with constant temperature
- Sample:
  - size: 10 mm × (0.3 to 0.4) mm × 1.5 to 2.5
  - Conditioning: dried in a desiccator 24 hours
- Temperature: ranging from 30°C to 80°C



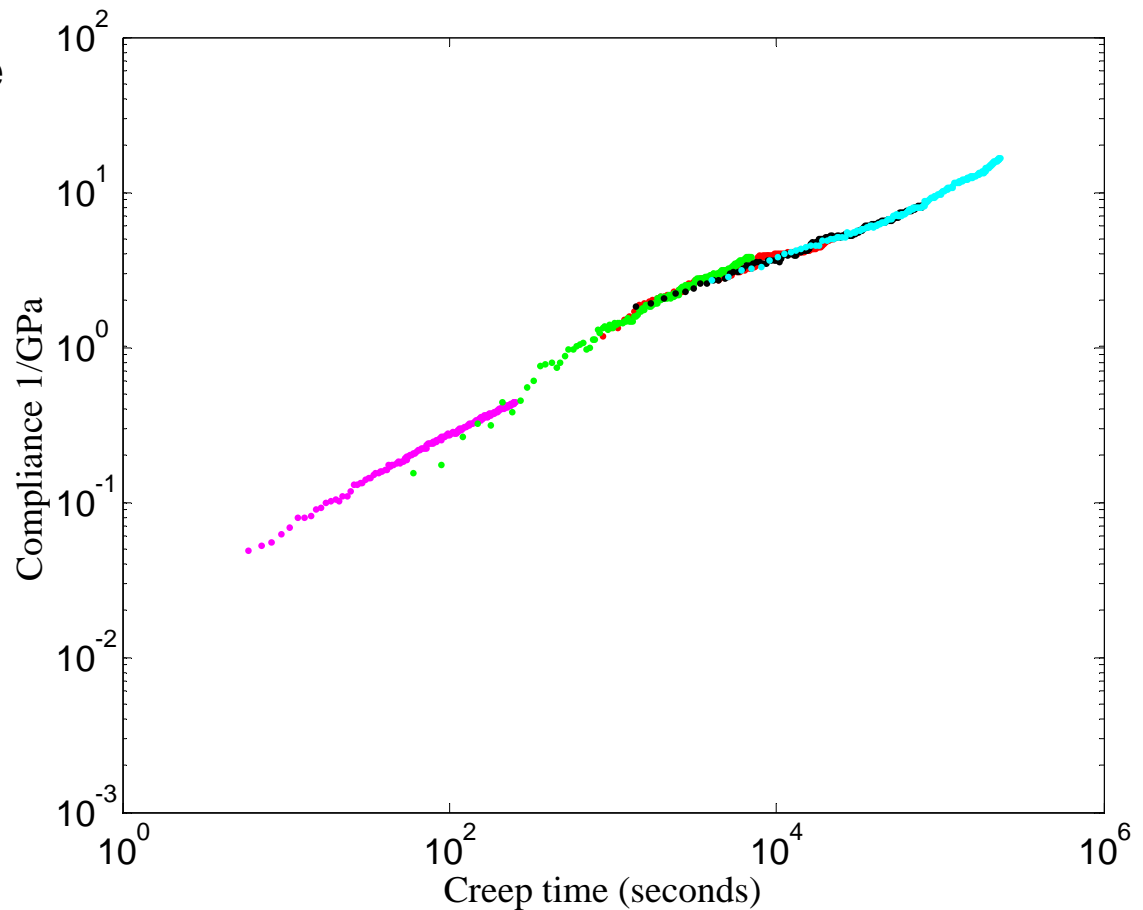
# Creep curves at various temperatures and shift factor

- 1) Compliance increase along with creep time.
- 2) Compliance increase along with temp. increase.
- 3) Shift factor: horizontal distance from an individual curve to a reference curve along creep time axis in logarithm scale. It is a value to tell the relaxation time difference.



# Master curve of nylon wood composite from TTSP

Master curve covering a large creep time is used to predict long term performance.



# Arrhenius model

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- Barrier model: two positions (energy minima) separated by a barrier of height  $E_h$  (activation energy)

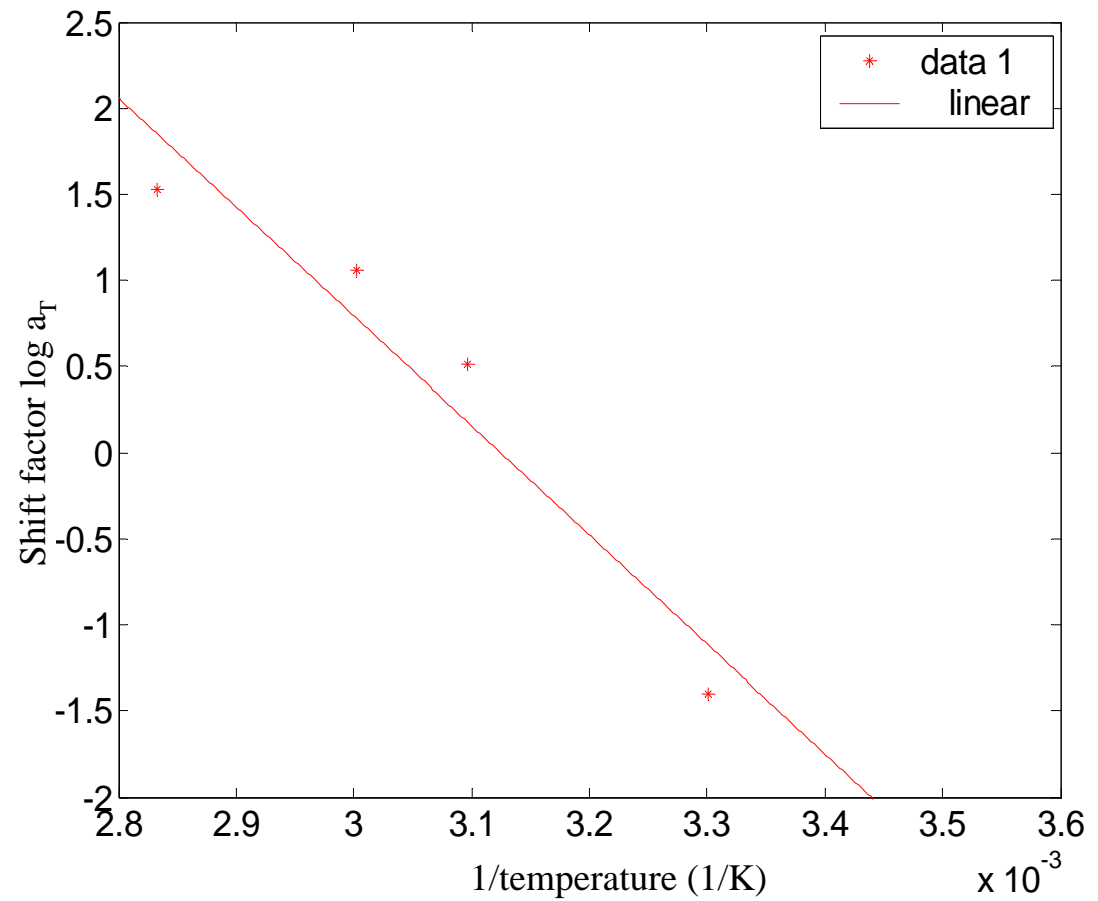
$$\log a_T = \frac{E_h}{R} \left[ \frac{1}{T_2} - \frac{1}{T_1} \right]$$

$\log a_T$  is the shift factor,  $R$  is the gas constant ( $R=8.30434$  J) and  $E_h$  is the activation energy,  $T_1$  and  $T_2$  are temperature at two different position.

# Linear relationship of shift factor and $(1/T_2 - 1/T_1)$

$$\log a_T = \frac{E_h}{R} \left[ \frac{1}{T_2} - \frac{1}{T_1} \right]$$

According to Arrhenius Equation, if temperature dependent behavior follow Arrhenius format, the relationship of shift factor  $\log a_T$  and  $(1/T_2 - 1/T_1)$  is linear. The result is very close to Arrhenius behavior.



# KWW creep equation

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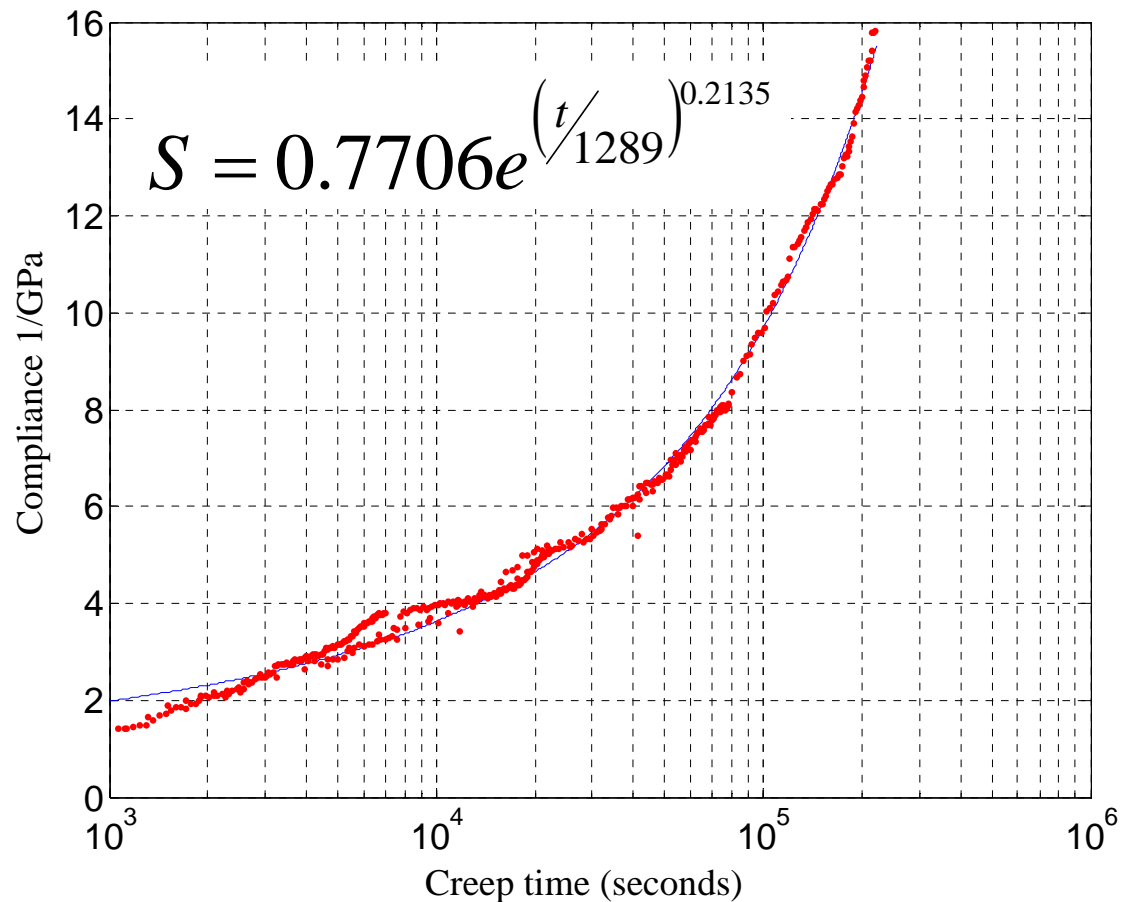
$$S(t) = S_0 e^{\left(\frac{t}{\tau}\right)^\beta}$$

In this equation,  $S_0$  is the initial compliance,  $\beta$  is a shape parameter,  $\tau$  is the relaxation time spectrum, and  $t$  is creep time

$\tau$  is the parameter that is related to the aging time and temperature effects.

# Curve fit of KWW equation

Curve fit result of KWW equation describes the time and temperature dependent properties of the material. It is used to conduct further viscoelastic behavior analysis of a component.



# Shift factor at target temperature

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In KWW equation,  $\tau(T)$  is obtained from curve fit and modified by shift factor.

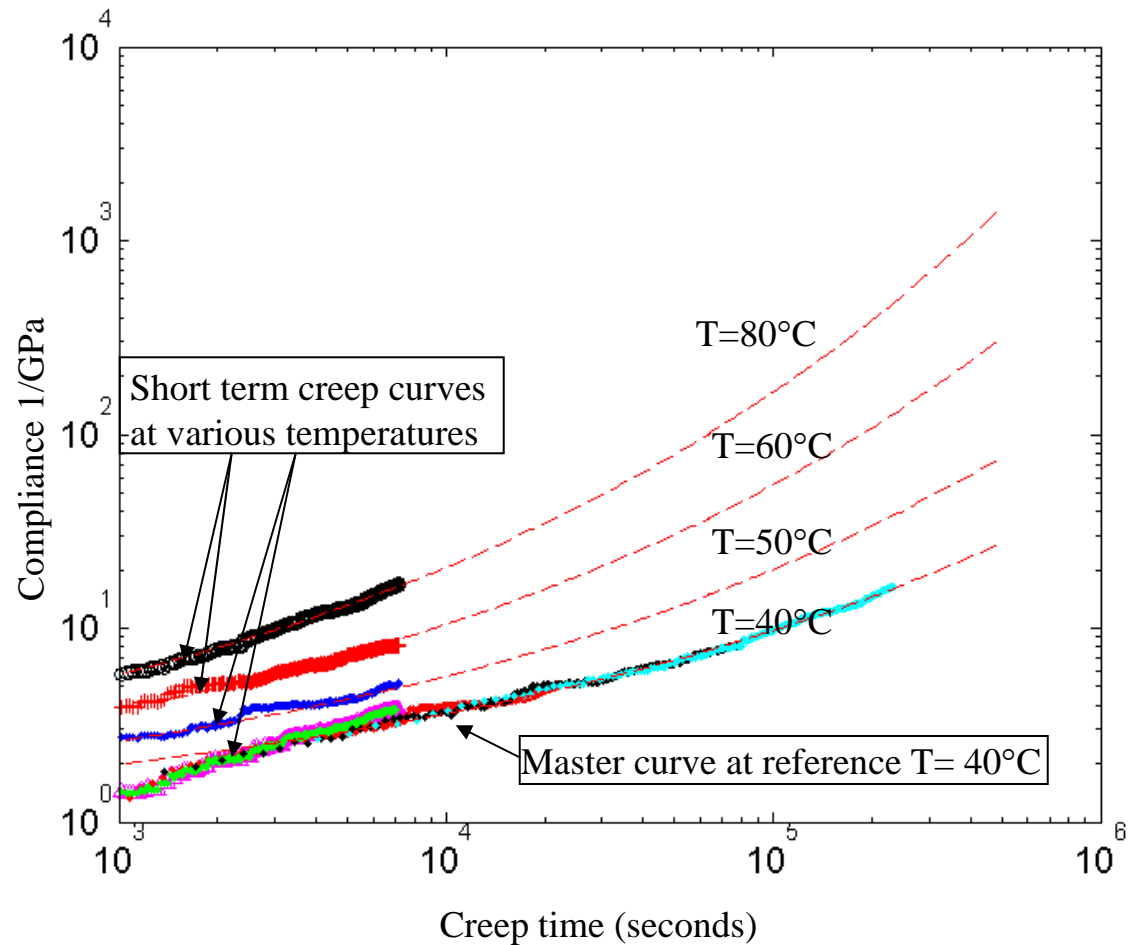
$$S(t) = S_0 e^{\left(\frac{t}{\tau}\right)^\beta}$$

In the equation of shift factor at target temperature,  $\tau(T_{ref})$  is the relaxation time at the reference temperature,  $a_T$  is the shift factor at a target temperature relative to the reference temperature.

$$\tau(T) = \tau(T_{ref}) / a_T$$

# Long term creep curves at various temperatures

- 1) Long term creep curves at various temperatures are obtained by curve fit result equation, shift factor at target temperature.
- 2) Long term creep curves are well matched each individual short term creep curve at the beginning, which partly shows the reliability of our method.



# Conclusion

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- $T_g$  of the nylon 6,66 -wood composite is between the  $T_g$  of nylon 6 and nylon 66.
- The storage modulus behavior as a function of temperature for the nylon-wood composite compares favorably with nylon-glass.
- Temperature-dependent properties of nylon improved by wood filler compared to unfilled nylon 66.
- Aging effects are reduced by wood filler.
- Curve fit of KWW equation provides a foundation equation for further viscoelastic behavior analysis.
- Long term creep curves at various temperatures obtained by means of the KWW equation and temperature shift factors.