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## Time-Temperature Superposition Using DMA Creep Data

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### ABSTRACT

The principle of time-temperature superposition (TTS) extends the range of frequencies or times of viscoelastic properties beyond that measurable. It provides a unique way of estimating material viscoelastic properties over time. This principle is successfully used to determine the long-term properties of a magnetic tape/film held under a stress. Multiple creep experiments show excellent repeatability of 2.3 %.

### INTRODUCTION

The concept of time-temperature superposition (TTS) comes from the observation that the time-scales of the motions of constituent molecules of a polymer are affected by temperature. More specifically, the motions or relaxations occur at shorter times at high temperatures ( $T$ ). Furthermore, by assuming that the whole relaxation spectrum of a polymer can be affected by increasing temperature, it is possible to look at the long-time properties of materials by simply changing the temperature.

From an experimental point of view, data from oscillation frequency sweeps, and creep and stress-relaxation experiments performed at various isothermal temperatures can be superposed to a reference temperature. These tests may be performed on solid polymer samples using a DMA or melts using a rheometer.

A good application of TTS is the changing mechanical properties of films/fibers wound on a spool at high speeds and then stored. During production, tension is maintained while winding the film/fiber. Under this tension, the materials stretch with storage time. Creep is a perfect analogue of this process as a stress is applied to sample and the deformation observed over time. It is shown that with creep TTS, the resultant information is extended to months, even years.

### EXPERIMENTAL

The test sample is a film cut from a roll of magnetic tape, similar to that of audio or videotape. Such film is wound at high speeds onto the spool and stored, awaiting shipment. The width and thickness were 8.00 mm and 8  $\mu\text{m}$ , respectively.

The tension-film clamp of the TA Instruments Q800 Dynamic Mechanical Analyzer is used in all experiments. The loading of the sample requires some care to assure that there was no twists or folds. Improper loading becomes evident when the sample appears to be “breathing” when the MEASURE button is pressed and the

oscillations begin. When the sample is correctly loaded, the tensile oscillations do “pulse” when viewed from the side. The MEASURE step also determines the exact length of the sample. The temperature range was 30 to 165 °C, in 5 °C steps. The 10 MPa creep stress is applied for 10 min at each temperature. All these parameters are entered using the Creep TTS template available in the TA Instruments Thermal Advantage™ software.

## RESULTS AND DISCUSSION

All the log-log creep curves are overlaid in Figure 1 with compliance on the Y-axis and time on the X-axis. The increase in compliance with increasing temperature is clearly seen. This is expected since at higher temperatures, the relaxation occurs in a shorter time, enabling the sample to deform under the stress producing higher compliance.

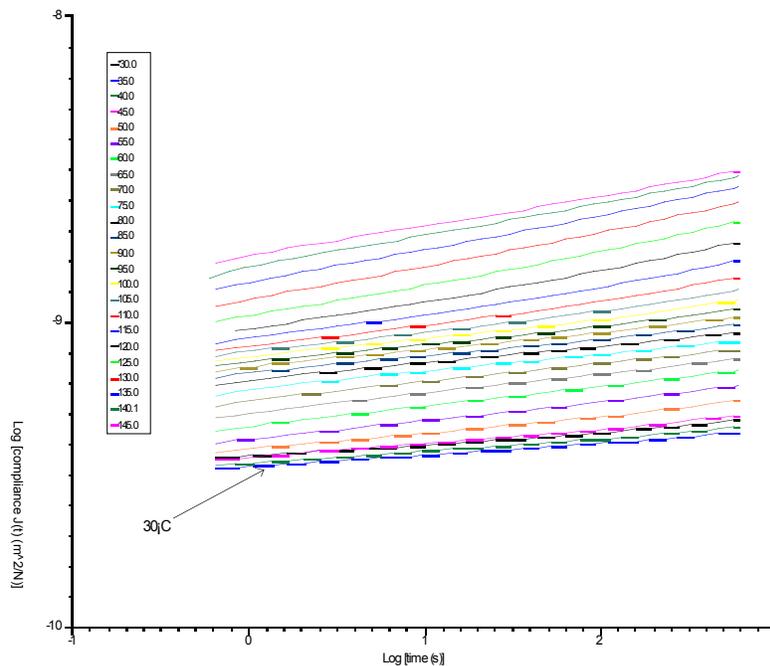


Figure 1- Creep Curves for TTS in the Thermal Advantage TTS Software

If the selected reference temperature is taken at 30 °C and the shifting performed automatically by the software, then the mastercurve shown in Figure 2 is obtained. The data spans a time range of approximately 0.1s to  $10^{12}$  s (ca. 300 centuries).

Such an experiment shows that the sample is stretched with time. It is possible to characterize several such samples with known behavior and generate a data library. The mastercurve of a new product may then be compared with those in the library to estimate behavior over time.

To test the repeatability of creep measurements, replicate experiments are performed at 30 °C as shown in Figure 4. A comparison of compliance values at 8 min, produces a mean value of  $364 \mu\text{m}^2/\text{N}$  with a relative standard deviation of  $\pm 2.3$  %. Since TTS is performed on log scales, the repeatability on this presentation is even better as shown in Figure 4.

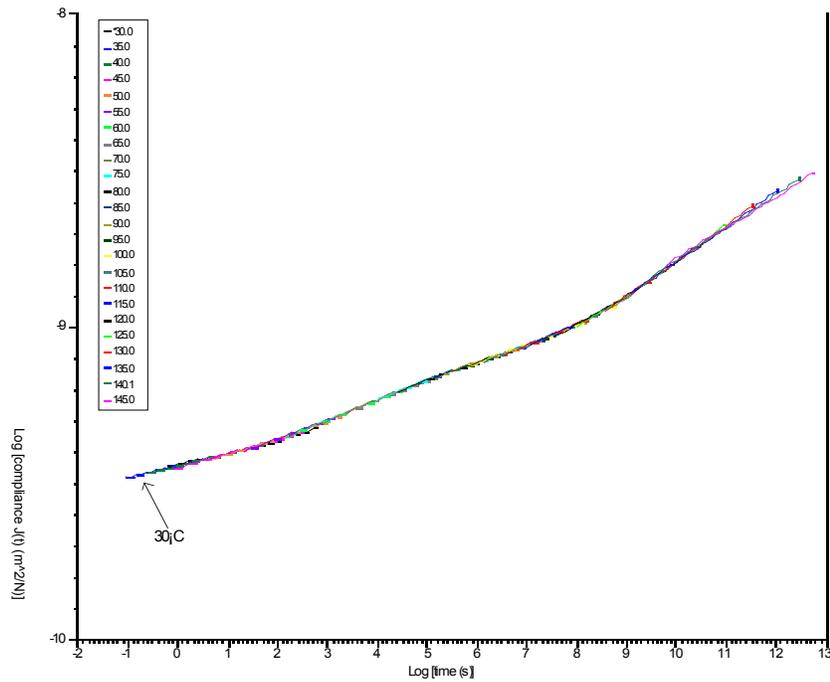


Figure 2 - TTS Mastercurve at 30 °C.

## SUMMARY

TTS is a very useful tool to look at long-term mechanical properties of materials. It is shown that there is stretching of wound magnetic tapes with time due to the stress during winding. The DMA is a superior analytical tool, with excellent repeatability, to predict the long-term mechanical properties of materials when used in conjunction with TTS principles. The TA Instruments Advantage software allows easy shifting of raw data to generate a mastercurve (just 5 easy clicks of the mouse from starting a session to a complete mastercurve!).

## REFERENCES

1. John D. Ferry, *Viscoelastic Properties of Polymers*, 3<sup>rd</sup> Ed., John Wiley & Sons, 1980, Chapter 11.

## KEYWORDS

creep/stress relaxation, dynamic mechanical analysis, films/fibers, relaxations

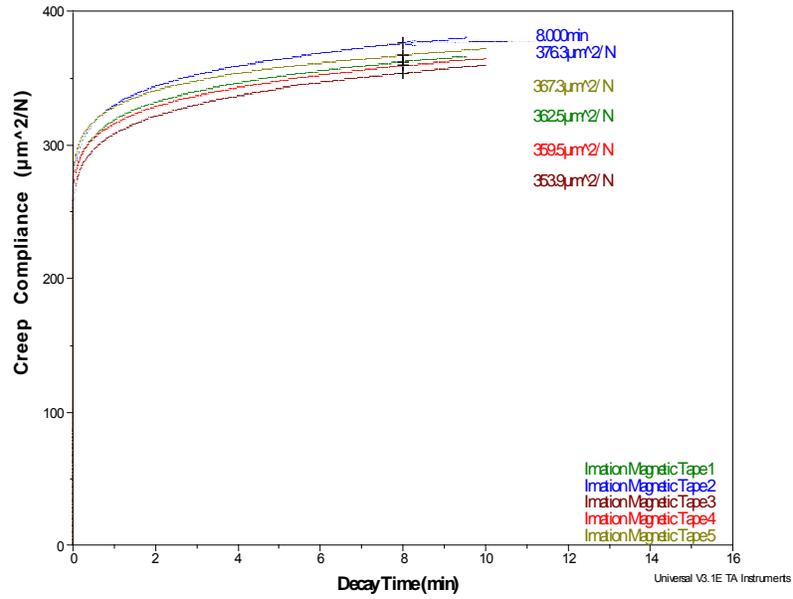


Figure 3 - Creep at 30°C

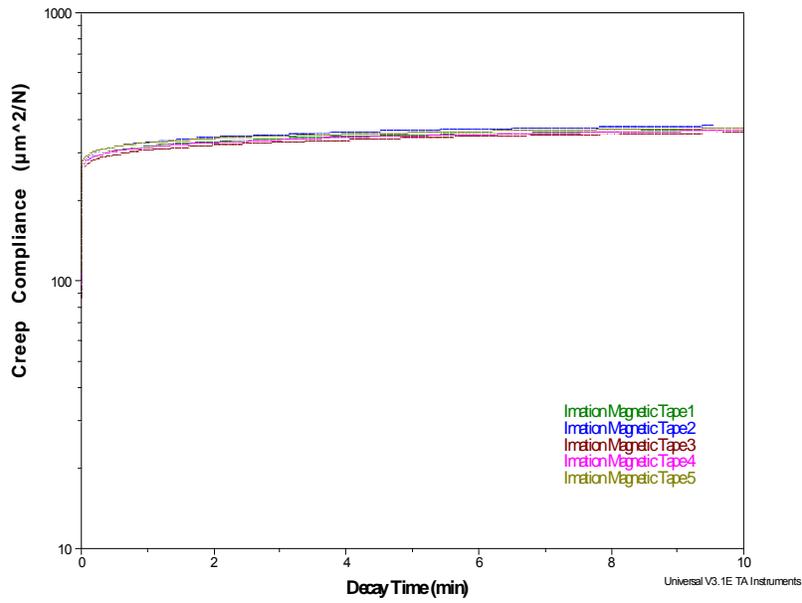


Figure 4 - Creep Compliance Repeatability at 30°C.