



Thermal Analysis

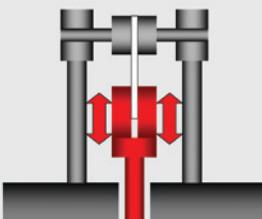


Mechanical Properties of Films and Coatings

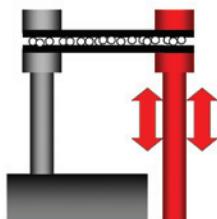
Dynamic Mechanical Analysis (DMA) is a technique used to investigate the stiffness of materials as a function of temperature, humidity, dissolution media or frequency. A mechanical stress is applied to the sample and the resultant strain is measured by the instrument. These parameters are used to evaluate glass transitions, degree of crystallinity and stiffness behavior of the sample.

There are three options when dealing with films and coatings:

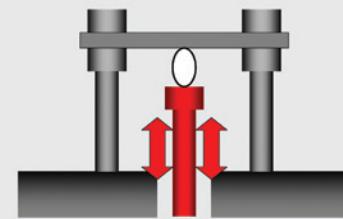
If the coating material can be produced so that it forms a thin film, the film can be mounted in the instrument so that a tension stress is applied.



The coating can be scraped from a substrate as either powder or flakes and suspended in a Material Pocket. A bending stress is then applied.



The coated product (tablet) can be mounted in the instrument and a compression stress applied. The tablet can be immersed in dissolution media to investigate coating effect on tablet integrity.



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A DMA is essentially a relaxation detection instrument. It is many times more sensitive than other techniques (e.g. DSC) at identifying relaxation events, such as a glass transition (T_g).

Data from two coating materials is shown in Figure 1. Each of the coatings had a slightly different pre-treatment prior to analysis. The glass transition temperature is shown as the peak value of the $\tan \delta$ vs temperature and as the drop in the storage modulus. It is shown that the pre-treatment greatly affects the glass transition temperature. For this material, the glass transition temperature was critical for the performance of finished product.

It is possible to investigate the properties of the coating under the influence of a controlled relative humidity environment. Figure 2 shows the effect of moisture on the same coating material. It is clear that the glass transition temperature is lowered by the influence of water. A small beta relaxation is seen in the wet sample indicating that water dramatically affects the mechanical properties of the material.

The development of Material Pockets, which are used to hold a powder or film for use in a DMA, have opened the door for many different materials to be analyzed in the DMA 8000.

Figure 3 shows data from the testing of thin films made by curing a layer of adhesive in the material pocket. This method allows the testing of films that are not self-supporting and also the monitoring of their curing. In this case, two films were made and tested directly in the Material Pocket and reveal a significant difference in the T_g .

Finally, the PerkinElmer Fluid Bath allows us to look at the dissolution of a coating from a capsule. Figure 4 shows a gelcap, a gelatin capsule containing a product, as it is tested in the DMA 8000 and Fluid Bath. In both samples, the modulus decreases with time after immersion reflecting the sample getting less stiff as it dissolves. Eventually, the sample disintegrates so much that the data is meaningless. This is the point where data collection was ceased. The sharp decrease in modulus indicates this point. It is worth noting that the rate of softening and the time taken to destroy the

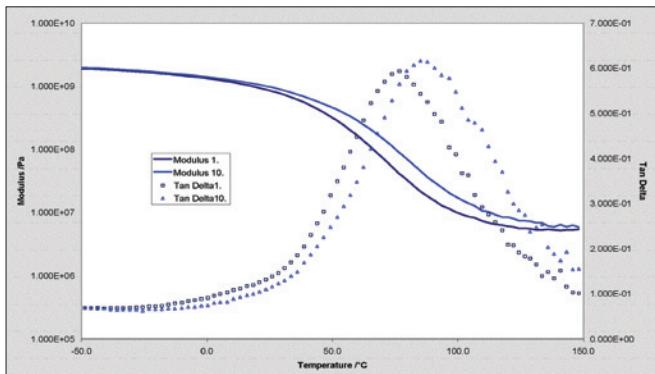


Figure 1. A comparison of pre-treated and untreated coatings run in tension.

sample were both faster at 38 °C than 25 °C. Also, the initial ingress of water into the gelatin to start the dissolution process was much faster at 38 °C, as shown by the short time between immersion and modulus decrease starting.

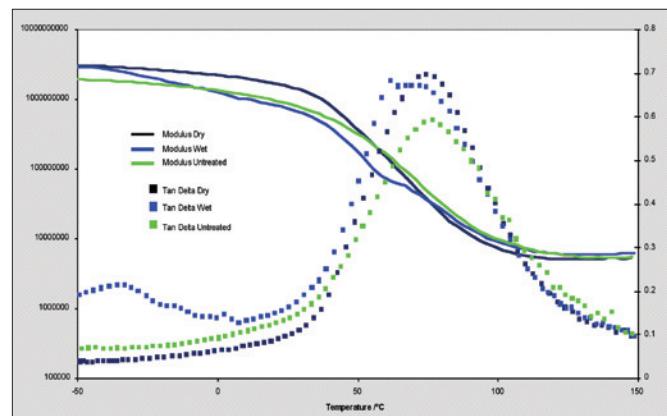


Figure 2. The effect of running the coating from Figure 1 in solution using the Fluid Bath.

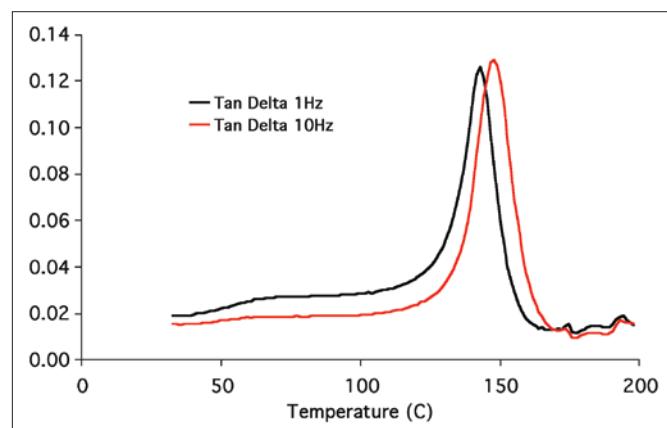


Figure 3. Measurement of thin adhesive films cured in a Material Pocket.

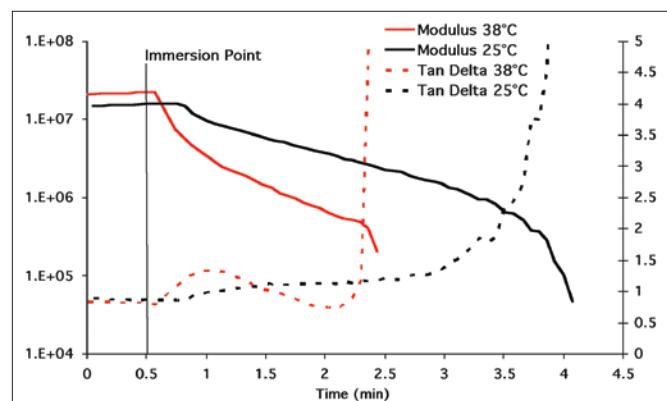


Figure 4. Two gelatin capsules tested in the Fluid Bath.

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