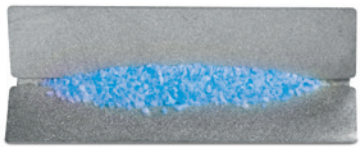
APPLICATION NOTE

Thermal Analysis



Use of Material Pockets for Mechanical Analysis of Powders

Dynamic Mechanical Analysis is a technique used to investigate the stiffness of materials as a function of temperature, humidity, frequency or other variable. A mechanical stress is applied to the sample and the resultant strain is measured by using a PerkinElmer® DMA 8000 instrument. Due to the nature of the experiment, the sample required is normally at least a few millimeters in size as well as self-supporting. A Material Pocket is available to overcome this restriction and enables the study of powders, coating, flakes, liquids or semi-solids. The Material Pocket is run in Single Cantilever Bending geometry in the DMA 8000.

The Material Pocket is a stainless steel envelope that holds the sample so it can be mounted in a DMA 8000 instrument. As stainless steel does not have any relaxations or phase transitions over the temperature range of the instrument, this is an ideal sample mounting material.

The preparation of the sample as illustrated below in Figure 1, is very straightforward.

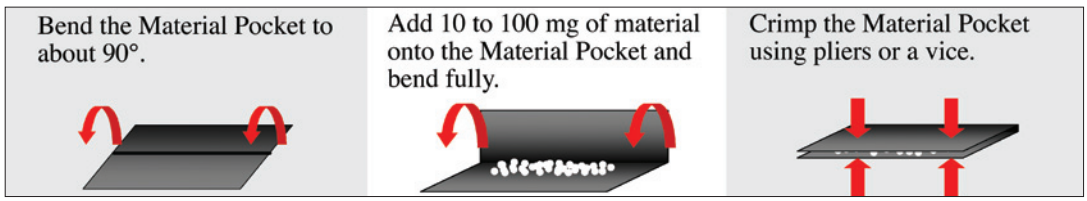


Figure 1. Sample preparation using the Material Pocket.

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Comparison of bar sample to Material Pocket sample

Figure 2 shows tan δ data from two DMA experiments with Polystyrene. The original sample was the same but the red line shows an experiment run with a bar sample run in single cantilever bending and the black line shows an experiment run with grated PS in a Material Pocket. Both were run at 1 Hz. It is clear that the glass transition, shown as a peak in the tan δ data, is the same for both experiments. The peak value is less for the Material Pocket but this is a reflection of the lower sample mass. This experiment demonstrates that it is possible to use the Material Pocket to obtain relaxation data on a sample and that the stainless steel of the pocket does not affect the observed Tg.

Quantization of amorphicity of a powder using a Material Pocket

Tan δ data for two samples of lactose is graphed in Figure 3. The red line is for 100% amorphous and the black line for 100% crystalline. Amorphous lactose is more hygroscopic than crystalline lactose, hence, there is a peak in the graph at around 100 °C corresponding to latent water being driven off. A glass transition is observed in the amorphous sample, then an event corresponding to recrystallization of the amorphous material. Also under this event, is a loss of hydration water which shows in the crystalline sample as well. As the temperature is increased further, the sample begins to melt.

This technique can also be quantitative. Figure 4 shows relaxation strength (1-peak value of the Tg transition) plotted against amorphicity of lactose. A sample of unknown amorphicity can be analyzed by the DMA 8000 using the Material Pocket and, depending on the relaxation strength, the amorphicity can be calculated.

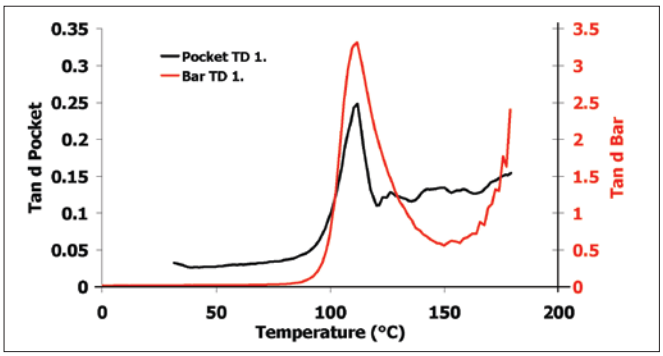

Figure 2. Tan δ from experiments with polystyrene.

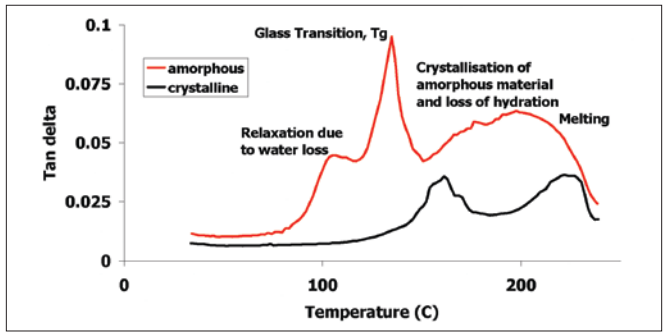

Figure 3. Tan δ from experiments with lactose.

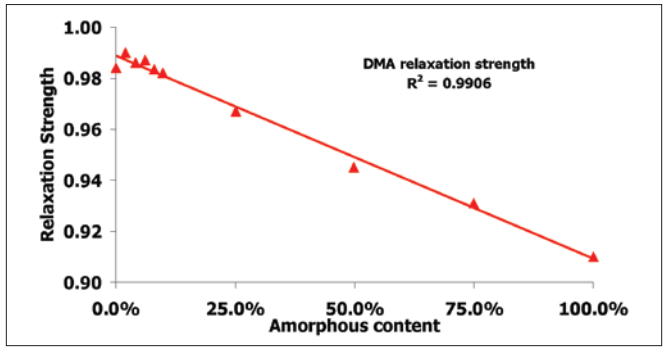

Figure 4. Relaxation strength plotted against amorphicity of lactose.

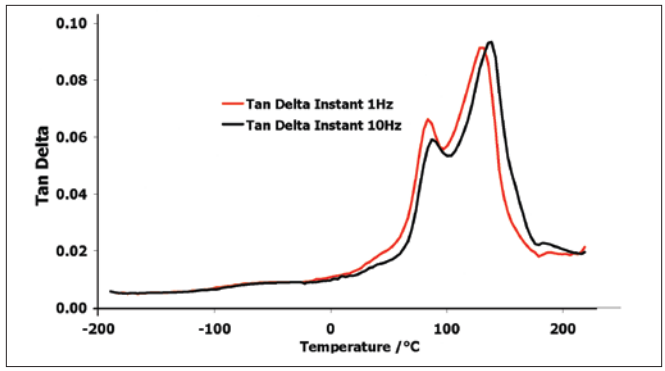

Figure 5. Tan δ from experiment with instant coffee.

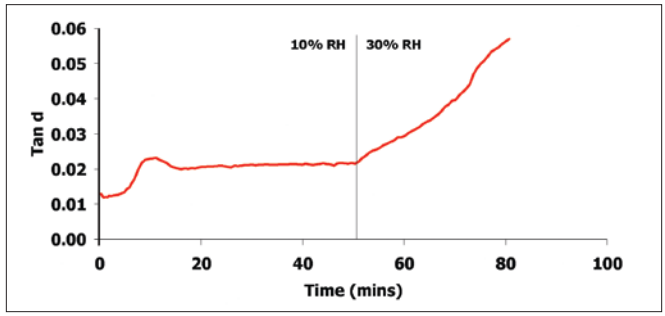

Figure 6. Humidity dependence of tan δ for sucrose at 40 ˚C.

Investigation of complex formulations using the Material Pocket

Figure 5 shows tan δ information from a sample of instant coffee run in a Material Pocket by DMA. The large dual peak around 100 °C is a glass transition as there is a phase shift between the two frequencies. There is also a smaller pre-transition at about -100 °C. This experiment illustrates that DMA, with the Material Pocket, can be used to investigate complex samples. The Tg of individual components can be separated in order to non-invasively investigate those components without masking effects of either the formulation or the Material Pocket itself.

Use of humidity control with Material Pockets

Because the Material Pockets are not sealed, the sample inside can be exposed to relative humidity. Figure 6 shows the tan δ response for a sample of sucrose run at 40 °C. After a short temperature equilibration at 10% RH, the relative humidity was increased to 30%. As the temperature of the experiment was close to the Tg for sucrose, the small increase in humidity drives the sample towards the Tg indicated by the increasing tan δ.

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