MEASURING THE THERMAL EXPANSION OF ULTRA-THIN SAMPLES

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A new instrument can accurately measure the thermal expansion of even extremely thin glass and ceramic materials.

Measuring the thermomechanical behavior of ultra-thin materials has always been a challenge due to the low mechanical strength of very thin sheets. Even when reducing the force applied by the push rod of a standard electronic dilatometer to a minimum, it is nearly impossible to get a reliable measurement on sheets with a thickness below 100 microns because they bend too easily.

Recently, a new instrument* has been introduced specifically for measuring the thermal expansion of ultra-thin materials. The instrument can carry out thermomechanical measurements without making any contact with the sample, providing extremely accurate results. Additionally, the test procedure is designed to be easy and hassle-free, with no need to run calibration curves.

Test Method

The new instrument uses a double-beam optical dilatometer based on a new concept. Two microscopes with a very long focal length use blue light to illuminate both ends of a specimen placed inside the dilatometer's furnace (see Figure 1). Since the specimen is very thin and unable to stand alone, the sample is placed horizontally on a special sample holder (see Figure 2).

The blue light used to illuminate the specimen has a wavelength of 478 nm, which enables an optical resolution of 0.5 micron. For example, on a specimen with a total length of 50 mm, the resolution of the measurement is of one part in 100,000. This is of the same order of magnitude as standard electronic dilatometers. Two digital cameras capture images of the ends of the specimen, and the instrument counts the number of illuminated pixels on the charge-

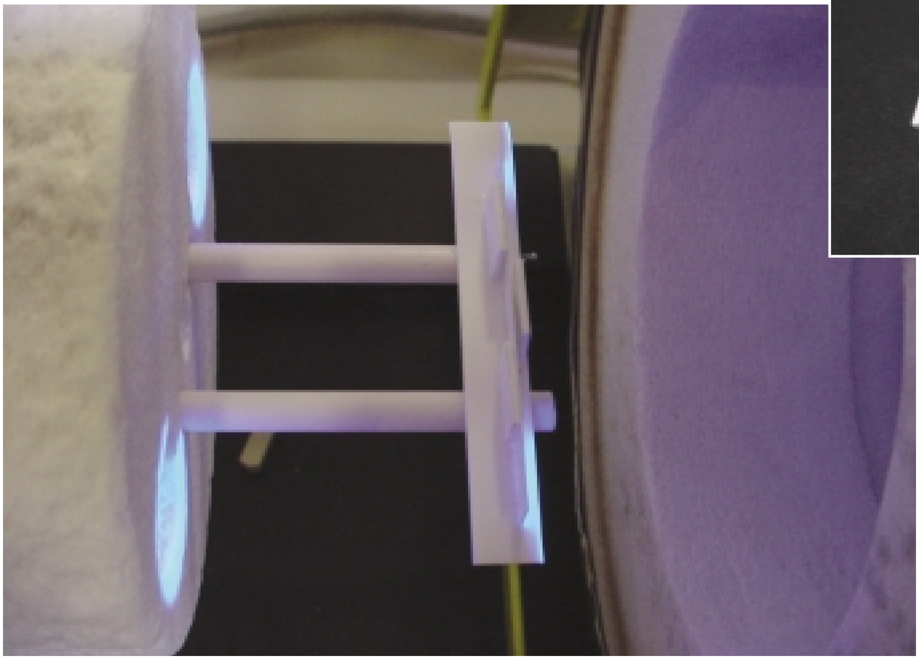

Figure 1. The instrument illuminates the sample on both ends with a blue light.

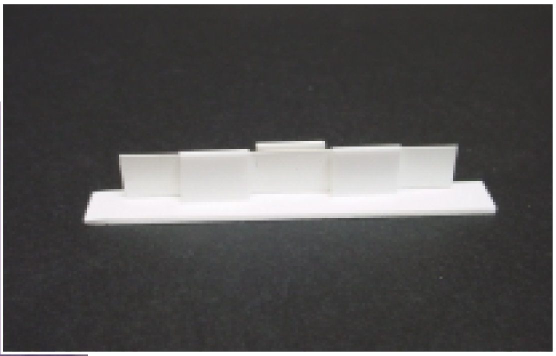

Figure 2. A sample holder is used for thin specimens.

*The MISURA LT, developed and supplied by Expert System Solutions, Modena, Italy, U.S. Patent No. 6,476,922 B2.

coupled device (CCD) of the cameras during the heat treatment.

One potential problem with this measuring method is that the sample is free to expand in both directions. In some cases, a negligible amount of friction between one side of the sample and the sample holder might cause the actual expansion to occur only on the other side of the sample. As a result, the image of the edge of the sample that is expanding can go out of the field of view of one camera due to the extremely high degree of magnification. However, this problem can easily be corrected by moving the optical path of the camera with a linear motor to bring the edge of the specimen back into the camera's field of view. The displacement is registered electronically, and the final data plot is completely seamless.

Measuring both ends of the specimen at the same time without making contact provides an absolute measurement. Nothing interferes with the measurement itself, so once the magnification factor of the optical paths is established there is no need for time-consuming calibration curves. However, operators who wish to verify the instrument's accuracy can easily check its calibration using a certified standard reference material. Figure 3 shows a comparison between the certified values of the SRM 738 from the National Institute of Standards and Technology (NIST) and the actual measurement carried out at 5°C/min using the new double-beam optical dilatometer.

Because there is no need for multiple calibration curves and the sample is measured without making contact, the heating rate can be changed during the test to more closely replicate actual heat treatment curves.

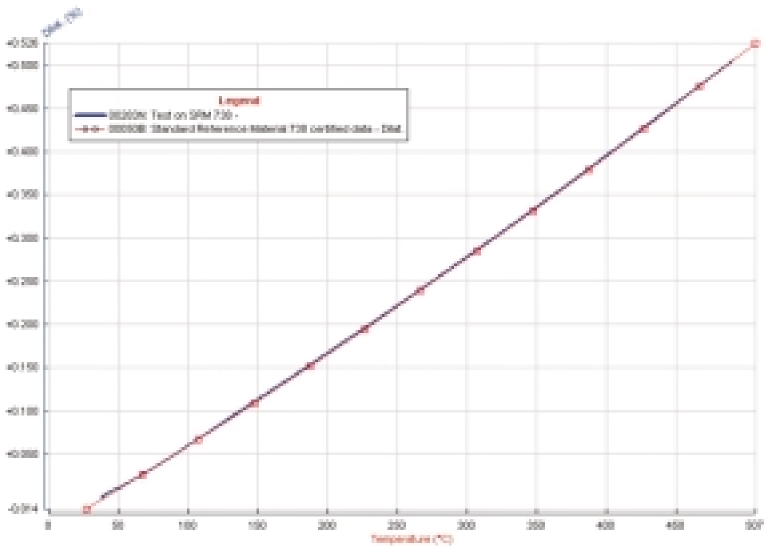

Figure 3. Comparison of the certified SRM 738 expansion (red line) and the actual measurement (blue line).

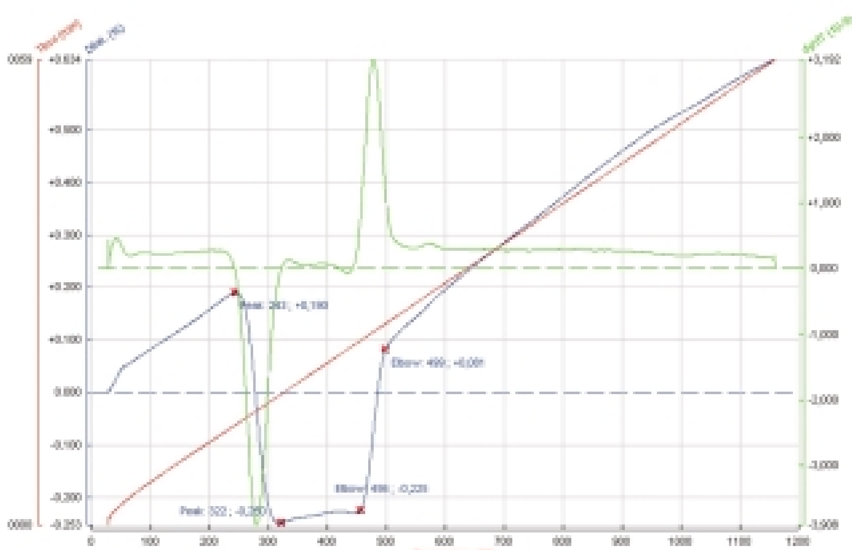

Figure 4. The thermomechanical behavior of the 100-micron-thick green ceramic tape at a heating rate of 20°C/min.

Thermal Expansion of Thin Sheets

To test the new instrument, researchers used it to measure the thermal expansion of several different samples:

- A 10-micron-thick foil of rolled aluminum
- A 50-micron-thick sheet of rolled brass
- A 100-micron-thick green ceramic tape, cast with a doctor blade

None of the samples were able to stand alone under their own weight, so all were placed inside the special sample holder to keep them in place during the measurement.

For the 10-micron thick aluminum foil, the heating rate was 20°C/min. As expected, the average thermal expansion coefficient in the first part of the curve was 24 x 10^{-6}/°C. At around 275°C, the material underwent a volume reduction, which represented a reorganization of an internal structure stressed by the cold rolling. The curve then proceeded smoothly to the melting of the metal, which occured at 650°C.

For the rolled brass sample, the average thermal expansion was 21 x 10^{-6}/°C, and the sample kept expanding up to 600°C. At this temperature, the sample started relaxing the stresses caused by the cold rolling, and it showed an obvious reduction in volume and incoherent melting. After the test, the surface of the sample was indeed showing phase separations.

The graph shown in Figure 4 was recorded for the 100-micron-thick green ceramic tape. The material is electronic-grade aluminum oxide, which is used for electronic substrates. The first curve was recorded with a heating rate of 20°C per minute and shows the actual behavior of the material during sintering up to 1150°C. The curve shows a quite complex behavior, highlighting a shrinkage phase that starts at 243°C and ends at 322°C.

The optical dilatometer can also be used to measure the thermal expansion of glass samples.

This corresponds to the burnout of the binder (in this case polyvinyl acetate [PVA]). The expansion process continues and shows a sharp change between 456 and 499°C. After that, the material keeps expanding with constant thermal expansion coefficient up to 1150°C, where the actual sintering starts.

The second curve, shown in Figure 5 (p. 20), was recorded with a complex heating cycle to allow more time for the burnout of the binder. In this case, the heat treatment included a low heating rate of up to 240°C and a pause of 10 minutes at 240°C. After the burnout, the heating rate was increased to 20°C/min up to 440°C, which was followed by a second 10-minute pause to minimize the stresses caused by the second phase transition. After this rest at constant temperature, the heating rate was again set to 20°C/min up to 1200°C. By plotting the curve with time on the X-axis, it is also possible to plot the temperature profile with time. The main difference between this and the previous curve is that during the rest at constant temperature, the contraction at 240°C and the expansion at 440°C reach completion during the pause at their corresponding temperatures.

The different heating cycle and the rest at constant temperature did not change the behavior of the material during the heat treatment, proving that these volume changes are not affected by time or by the speed of heating. Figure 6 (p. 20) shows the same result on the temperature scale,

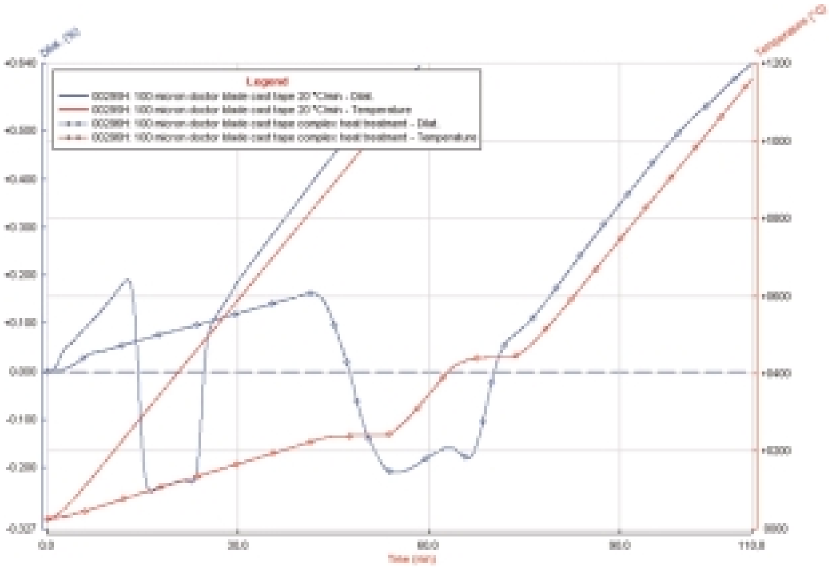

Figure 5. The thermomechanical behavior of the green ceramic tape with a different heating cycle, time based.

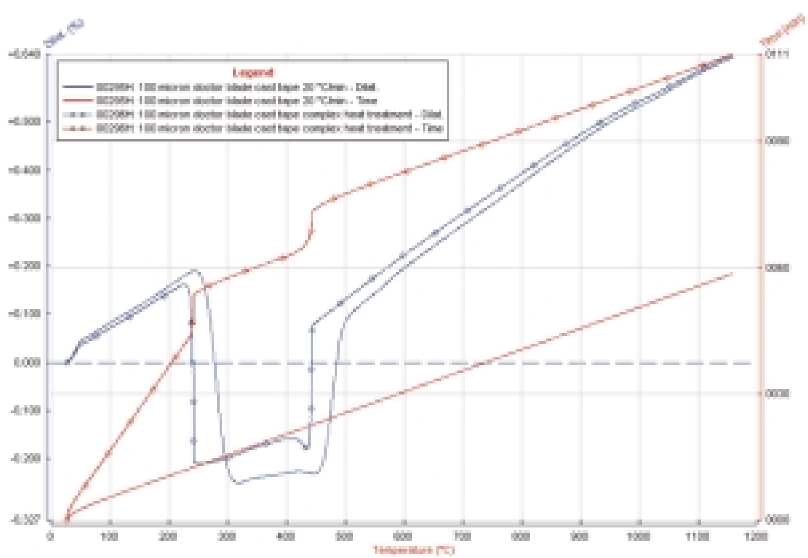

Figure 6. The thermomechanical behavior of the green ceramic tape with a different heating cycle, temperature based.

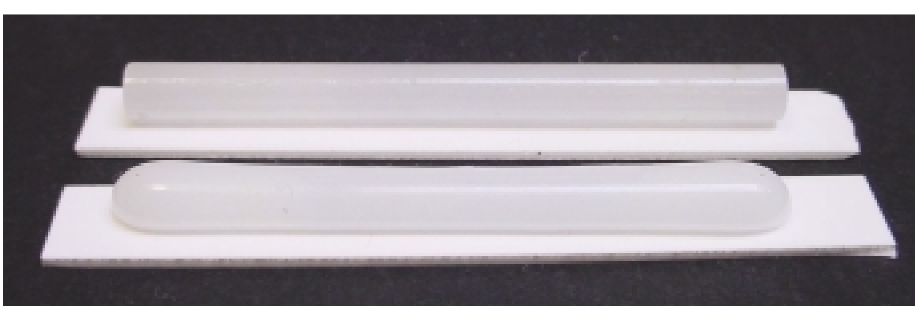

Figure 7. Glass rod specimens before and after the thermal expansion test. After the test, the edges of the specimen are completely rounded.

proving that the second heat treatment, which required twice the amount of time as the first treatment, did not change the actual behavior of the material.

The optical dilatometer can also be used to measure the thermal expansion of glass samples. Since the measurement is carried out without making contact, it is possible to overcome the softening temperature of the glass with no danger to the measuring system.

Figure 7 shows a glass sample before and after a test. Note that the edges of the specimen after the test are completely rounded. The rising section of the expansion curve after the glass transition temperature was much more prolonged compared to that of a push-rod dilatometer. The softening temperature measured with the optical dilatometer is the temperature at which the sample becomes liquid, and the surface tension makes the edges round.

After the glass transition, the curve continued to rise 230°C. The measured softening temperature was 200°C higher than the temperature measured with a push-rod dilatometer.

Accurate Analyses

The ability to carry out high-temperature and high-resolution measurements without making contact with the sample widens the field of research for ceramic and glass materials. Tests with the double-beam optical dilatometer have proven the instrument to be reliable, reproducible and extremely easy to use. Additionally, the measurement of the thermomechanical behavior of extremely thin specimens can be carried out even above the softening temperature, enabling ceramic and glass researchers to follow the actual behavior of materials during heat treatments.

Editor's note: Additional figures can be found with this article online at www.ceramicindustry.com.

For more information about the new double-beam optical dilatometer, contact Expert System Solutions, Via Virgilio 56/T, Modena 41100, Italy; (39) 59-886-0020; fax (39) 59-886-0024; e-mail info@expersystemsolutions.com; or visit www.expertsystemsolutions.com.