Mechanical Characterization of Ceramic Particles

Using a Nano Indenter G200

Application Note

Jennifer Hay
Agilent Technologies

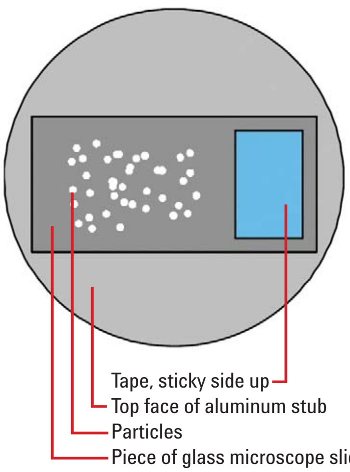

Figure 1. Schematic of the experimental setup.

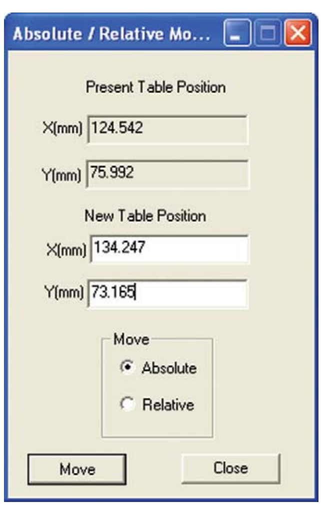

Figure 2. "Absolute/Relative Move" window.

Introduction

This application note presents the results of nanomechanical tests in which an Agilent Nano Indenter G200 was used to measure the force and displacement while crushing single ceramic particles. Two types of particles were provided for testing.

Experimental Setup and Procedure

Pieces of glass microscope slides (approximately 1 cm x 2 cm) were mounted to an aluminum stub using Crystalbond, which is a low-melting-temperature wax. After the stub and glass cooled to room temperature, a toothpick was used to deposit particles on the slide. The toothpick was inserted into the container and then tapped lightly at several points around the glass to deposit particles.

A 40 μm-diameter, flat-ended punch was used to crush the particles. It was found in preliminary testing that particles tended to adhere to the end of the punch after each test. To remove particles after each test, a small piece of Scotch tape was placed, sticky side up, on one end of the glass. After each test on a particle, a test was done on the tape to remove any residual debris. The setup is shown in Figure 1.

Ten particles of each type were tested. All tests were performed on a G200 Nanoindenter with a 40x microscope objective. The test method used was "Load, Disp, and Time". The distance (above the glass) from which the indenter began "looking" for the particle was 10 μm in the case of the first particle and 20 μm in the case of the second particle. The approach velocity was 50 nm/sec.

Once the indenter makes contact with the particle, a loading rate of 3.33 mN/sec is used to reach a peak load of 100 mN (30 sec loading). After the test is defined, the user must only hit the green arrow on the "Test" page to repeat it on each individual particle.

A third-party screen-capture software package, FullShot, was used to grab images before and after each test. Movement between the particles and the tape was facilitated by the "Absolute/Relative Move" function in Agilent's nanoindentation software. This window, shown in Figure 2, is obtained by right-clicking on the video display. Within the window, the user simply enters the (general) coordinates for the target location, selects the "Absolute" option, and then clicks "Move".

Figure 3. Image of residual impression of 40 μm-diameter punch.

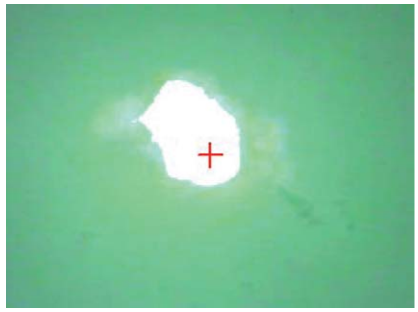

Figure 4. Before image of particle.

Figure 5. Post-test image of the particle shown in Figure 4.

These tests were run in individual mode, rather than batch mode, with the following procedure:

1. Center crosshairs over a particle to be tested. Verify that the sites for the two "surface-finding indents" are also clear.
2. Grab a "before" screen shot of the particle.
3. Test the particle.
4. Grab an "after" screen shot of the particle.
5. Center crosshairs over any clean location on the tape.
6. "Test" the tape in order to clean the indenter. (The results from this test may be deleted from the sample file immediately after the test is complete.)
7. Return to step 1 if more tests on this particle type are desired.
8. Save the NanoSuite sample file.

Results & Discussion

The results and images for one particular test are reviewed in this application note. Figure 3 shows the residual impression of the 40 μm-diameter punch. This image can be used to determine the size of tested particles. (All screen shots were obtained at exactly the same resolution to facilitate such quantitative image analysis.)

Figure 4 shows a "before" image of a particle; Figure 5 is the post-test image of the same particle. In this case, most of the remains of the particle adhered to the end of the punch. Figure 6 shows the force-displacement data that were acquired during contact between the punch and the particle.

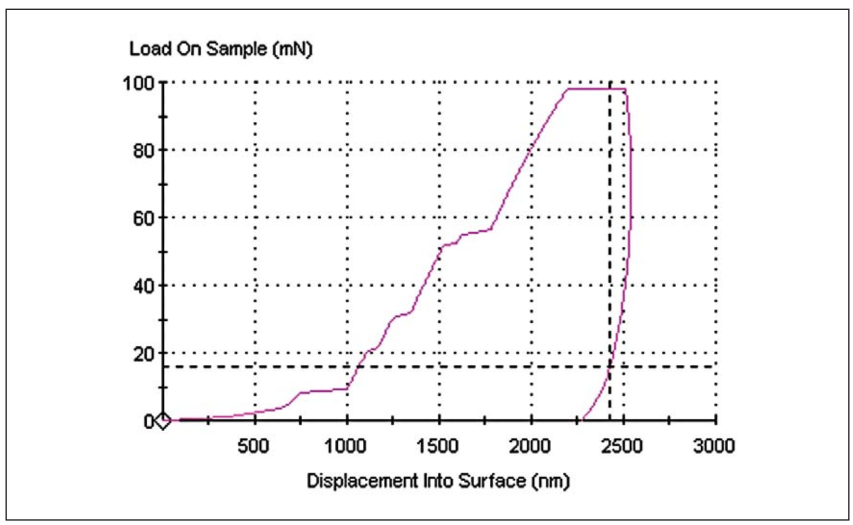

Figure 6. Force-displacement data acquired during contact between the punch and the particle.

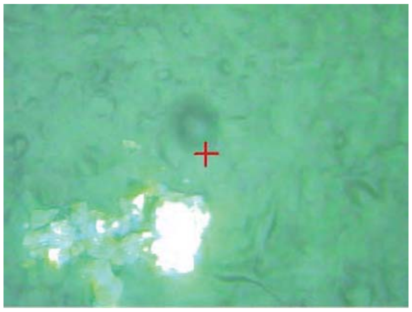

Figure 7. Image showing residual material left on tape from test.

Finally, Figure 7 shows the residual material from this test that was left on the tape. Near the center of the image is the residual impression from the first prescribed test, showing the indenter to be clean. But before the first prescribed test, two "surface-finding" tests are done. These preliminary tests allow the instrument to determine the precise elevation of the test surface. Most of the material was left on the tape as a result of these preliminary tests, which were performed below and to the left of the first (and only) prescribed indent.

The challenge with this type of testing is to analyze the force-displacement data to yield results that allow meaningful differentiation between materials that are, indeed, different. For example, it may be useful to calculate the area under the force-displacement trace, as this is a measure of the work involved in deforming the particle. Furthermore, it is probably a good idea to normalize this work by the volume of the particle.

For future testing, a larger-diameter punch (~200 µm) would be useful.

Technology and Applications

The Nano Indenter G200 is powered by electromagnetic actuation to achieve unparalleled dynamic range in force and displacement. The instrument's unique design avoids lateral displacement artifacts, while software compensates fully for any drift in force. Using the G200, researchers can measure Young's modulus and hardness in compliance with ISO 14577. Deformation can be measured over six orders of magnitude (from nanometers to millimeters).

Applications of the G200 include semiconductor, thin films, and MEMs (wafer applications); hard coatings and DLC films; composite materials, fibers, and polymers; metals and ceramics; and biomaterials and biology.

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