High-speed Mechanical Properties of CoorsTek TTZ Zirconia and Single-Crystal Alumina

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Abstract

In this work, we used standardized and advanced instrumented indentation to measure the mechanical properties of two common technical ceramics: CoorsTek TTZ zirconia and single-crystal alumina. By means of standardized indentation, we measured the Young's modulus of the TTZ to be 249±9.7 GPa and the Vickers hardness to be 1600±65 kg/mm2; we likewise measured the Young's modulus of the alumina to be 435±14 GPa and the Vickers hardness to be 2820±100 kg/mm2. By means of a proprietary indentation technique (**NanoBlitz 3D**) we generated mechanical-properties maps of the TTZ which illuminate the mechanical distinction between the primary cubic zirconia phase and an inter-granular silica phase.

Introduction

Advanced technical ceramics have many uses in modern life, from medical implants to cutting tools. Their suitability for certain applications derives largely from their mechanical strength. However, mechanical properties can depend significantly on the manufacturing process by which the final part is produced. For example, in its product literature, CoorsTek advises that "The designer should recognize that exact properties may vary according to product configuration..." [1]. Therefore, it is quite desirable to be able to measure mechanical properties directly on an as-manufactured part. Instrumented indentation is the ideal tool for such on-the-spot measurements, because it measures Young's modulus and hardness, without having to image the residual impression in any way.

Instrumented indentation is a development of traditional hardness tests such as Brinell, Rockwell, Vickers, and Knoop. Instrumented indentation (also called nanoindentation) is similar to traditional hardness testing in that a hard indenter, usually diamond, is pressed into contact with the test material. However, traditional hardness testing yields only one measure of deformation at one applied force, whereas during an instrumented indentation test, the force and penetration are measured during the entire time of force application and removal. The technique has now matured to the extent that it has been standardized by ISO [2] and ASTM [3]. Instrumented indentation defines hardness (H) as the applied force divided by the projected contact area, usually in units of GPa, but this definition of hardness can easily be converted to Vickers hardness (HVc) [2]. Further, the recovered displacement, sensed while indentation force is reduced, allows the calculation of the local Young's modulus (E) of the material.

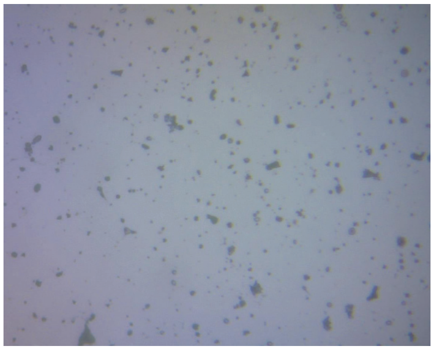

Figure 1. CoorsTek TTZ zirconia surface as prepared for instrumented indentation. Light phase is cubic zirconia; dark phase is inter-granular silica.

In this work, we use instrumented indentation to characterize two technical ceramics. The first is a toughened zirconia, CoorsTek TTZ. This ceramic has a grain size of about 40 micrometers with an inter-granular phase as shown in the microscope image in Figure 1. The light grains are cubic zirconia (ZrO_2), with substitutional MgO to promote the stability of the cubic structure. The inter-granular (dark) phase is predominately silica with other impurities; this inter-granular phase arises from impurities present in the raw material. The second ceramic is a single-crystal alumina (Al_2O_3). First, we use a standard test method to measure the Young's modulus, hardness, and Vickers hardness of both materials, and compare our results to reported properties for these materials. Then we use a proprietary indentation technique, Nanomechanics' **NanoBlitz 3D**, to generate surface maps of the CoorsTek TTZ. With this advanced indentation technique, each indentation takes less than 1 second.

1. "TTZ Zirconia Material Properties," URL: http://www.coorstek.com/materials/ceramics/zirconia_TTZ.php, last accessed 2016-03-21.
2. "Metallic Materials–Instrumented Indentation Test for Hardness and Materials Parameters," ISO 14577, International Organization for Standardization, Geneva, Switzerland (2002).
3. "Standard Practice for Instrumented Indentation Testing," E2546-07, ASTM International, West Conshohocken, PA (2007).

Experimental Method

The alumina sample was purchased as a 2 cm (diameter) disk, with the top and bottom faces optically polished normal to the c-axis of the crystalline structure. The disk was mounted in order to perform the indentation test on a polished face, in the c-axis direction. As received, the TTZ sample was too rough to be tested, so it was polished in preparation for instrumented indentation. A disk, approximately 2 cm (diameter) and 1 cm thick was rough ground with water using silicon carbide starting at 220 grit through 600 grit. The testing surface (top face of the disk) was then polished using 6µm and 1µm diamond on a medium nap cloth with an alcohol-based extender. Final polishing was done using 0.05µm colloidal silica on a low nap cloth, and polished in a vibratory polisher with 0.06µm colloidal silica and a medium nap cloth.

Using Nanomechanics' **iNano**, we performed twenty indentations on each ceramic, in conformance with ISO 14577 (an international standard governing instrumented indentation). Individual indentations were separated by 40 micrometers. The CoorsTek TTZ was tested to a peak force of 25mN, and the alumina was tested to a peak force of 50mN. The load-time protocols are shown in Figure 2; the loading time was 20 seconds, followed by a dwell time of 2 seconds. The unloading rate was the same as the loading rate, but near the end of the unloading segment, the force was held constant for a period of 80 seconds in order to measure and compensate for the thermal expansion/contraction of the sample. (This technique of measuring and compensating for thermal drift is commonly used in instrumented indentation, because it improves the accuracy of the displacement measurement.) Each test took about 2 minutes.

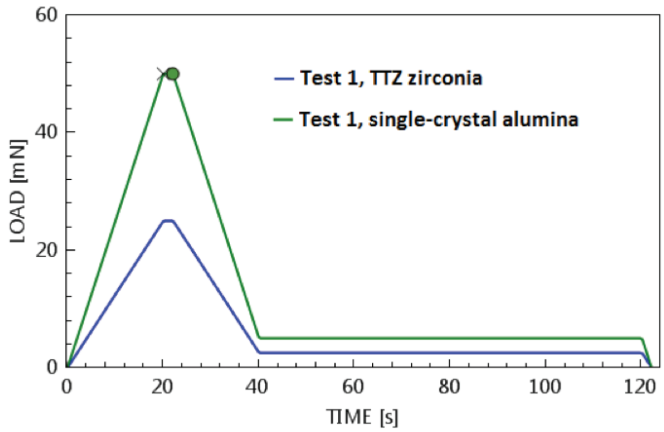

Figure 2. Load-time prescription for standardized indentations into TTZ zirconia (blue) and alumina (green).

Lastly, we used Nanomechanics' proprietary instrumented indentation technique (trade name: **NanoBlitz 3D**) to make a map of the surface properties of the CoorsTek TTZ. The test area was a square, 100 micrometers by 100 micrometers, incorporating both cubic zirconia grains and inter-granular silica. Within this area, an array of 50x50 indentations was prescribed, for a total of 2500 indentations. The peak force for each indentation was 20mN. The entire indentation array took about 30 minutes to complete.

Results and Discussion

Figure 3 shows two load-displacement curves, one for each material, which were measured in accordance with ISO 14577. These are the fundamental measurements from which Young's modulus, hardness, and Vickers hardness are calculated. Table 1 summarizes the calculated mechanical properties and compares these properties to reference values [1, 4-5]. Figure 4 shows the **NanoBlitz** indentation array on the CoorsTek TTZ as well as the maps of Young's modulus and hardness derived therefrom. The measured Young's modulus for the c-axis alumina (E=435 GPa) is less that the reference value of 499 GPa. However, alumina is highly anisotropic; in the a-b direction, the Young's modulus is only 403 GPa. Indentation testing is not entirely uniaxial; indentation along the c-axis is influenced by elasticity in the a-b direction. Consequently, the measured indentation

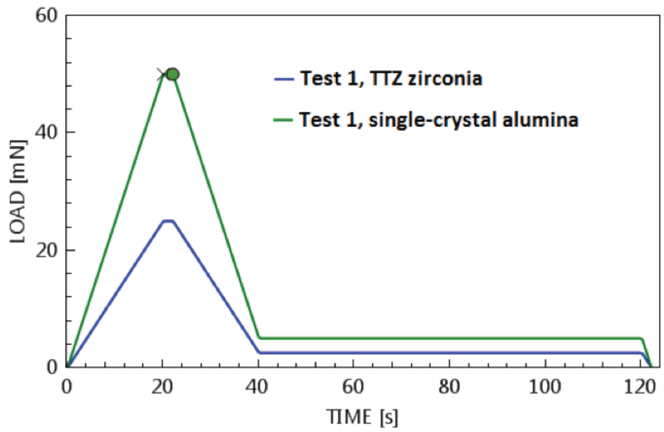

Figure 3. Load-displacement curves for two standardized indentations into TTZ zirconia (blue) and alumina (green).

1. "TTZ Zirconia Material Properties," URL: http://www.coorstek.com/materials/ceramics/zirconia_TTZ.php, last accessed 2016-03-21.
4. Simmons G. and Wang H., Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook, 2nd ed., M.I.T. Press, Cambridge, MA (1971).
5. "Sapphire (Al2O3) Specifications," URL: http://www.janis.com/Libraries/Window_Transmissions/Sapphire_Al2O3_TransmissionCurveDataSheet.sflb.ashx, last accessed 2016-03-21.

modulus is between that for the c-axis direction and that for the a-b direction. In their canonical paper on instrumented indentation, Oliver and Pharr report a value of 441 GPa for c-axis alumina [6].

Table 1. Summary of measured and reference properties.
§499 GPa is for the c-axis direction; E = 403 GPa perpendicular to the c-axis [5].

Measured by instrumented indentation (ISO 14577) N = 20					**Reference values**		
Material	Force	E (GPa)	H (GPa)	HVc (kg/mm2)	E (GPa)	Microhardness (kg/mm2)	Poisson's Ratio
Alumina (c-axis)	50	435±14	29.9±1.15	2820±100	499§ [5]	2200 (HV) [6]	0.23 [5]
CoorsTek TTZ	25	249±9.7	17.0±0.69	1600±65	200 [1]	1200 (HK 1kg) [1]	0.3 [1]

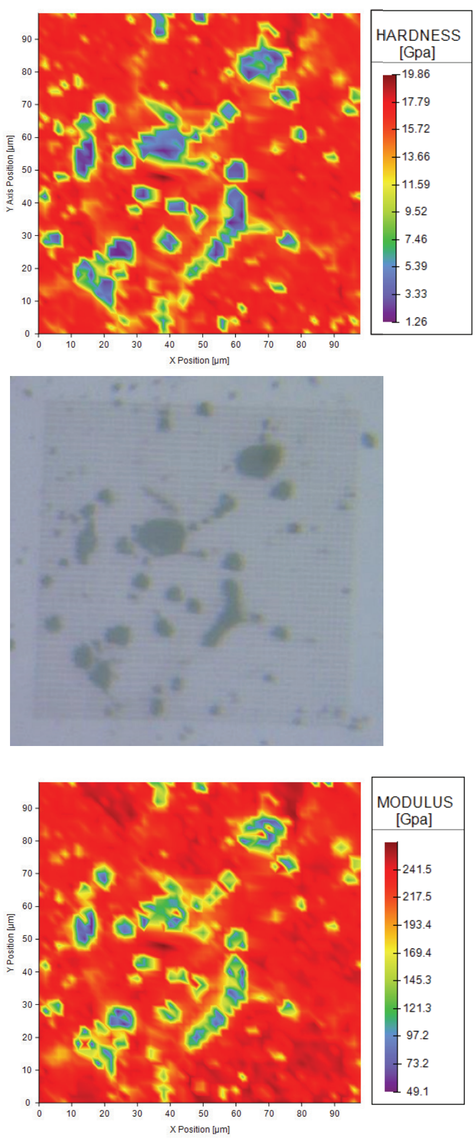

Figure 4. For CoorsTek TTZ, surface maps of hardness (top) and Young's modulus (bottom. The center image shows the indentation array (50 x 50) used to generate the mechanical-properties maps.

For the CoorsTek TTZ zirconia, the measured Young's modulus is significantly greater than what is reported for bulk. We assert that the discrepancy is due to the scale of the test. Our indentations were all deliberately placed in the cubic zirconia phase, and so the indentation modulus is that of cubic zirconia alone. By contrast, the bulk modulus includes the influence of both the cubic zirconia phase and the inter-granular silica phase, so it is not surprising that the bulk modulus is lower is less than the indentation modulus.

For both the alumina and the zirconia, the Vickers hardness values measured in accordance with ISO 14577 are significantly greater than reference values of microhardness. In crystalline materials, plastic deformation occurs by dislocation initiation and propagation, and thus depends on the scale of the test. Smaller indentations incorporate smaller volumes of material, which may not already include dislocations. Accordingly, crystalline materials are truly stronger at smaller length scales. This phenomenon is commonly called "indentation size effect". Therefore, we submit that if it were possible to do a traditional microhardness test at this scale (which it is not, because the residual impressions are too small) then the microhardness would be comparable to what we have measured. This is a distinct advantage of instrumented indentation: it provides access to hardness measurements at much smaller scales than traditional microhardness.

The maps of Young's modulus and hardness in Figure 4 give unprecedented insight into the microstructure of TTZ. Indeed, we see that the inter-granular phase has properties which we might expect for low-quality silica: E ~ 70 GPa and H ~ 5 GPa. We submit that these quantitative images are far more valuable for understanding and predicting the mechanical behavior of TTZ than what might be achieved with bulk testing methods (tension, compression, microhardness, et cetera).

1. "TTZ Zirconia Material Properties." URL: http://www.coorstek.com/materials/ceramics/zirconia_TTZ.php, last accessed 2016-03-21.
5. "Sapphire (Al2O3) Specifications." URL: http://www.janis.com/Libraries/Window_Transmissions/Sapphire_Al2O3_TransmissionCurveDataSheet.sflb.ashx, last accessed 2016-03-21.
6. Oliver, W.C. and Pharr, G.M., "An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments, Journal of Materials Research 7(6):1564-1583 (1992).

Conclusions

Nanoindentation is a valuable measurement tool precisely because volume affects microstructure, which in turn affects mechanical properties. In this work, small-scale testing provides unique insight into the mechanical behavior of two common technical ceramics. For both materials, we find increased strength for deformation at smaller length scales. For the CoorsTek TTZ, we find that the bulk Young's modulus arises from the mechanical influence of both the cubic zirconia and the inter-granular silica phase.

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