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Short communication

# A new method to extract elastic modulus of brittle materials from Berkovich indentation

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ABSTRACT

This work describes a method to reliably measure the elastic modulus of brittle materials from Berkovich indents under the continuous stiffness mode. It involves depositing a metallic film at the surface of the tested material to absorb the inelastic damage caused by the penetrating indenter. The Zhou-Prorok model was employed and rearranged to decouple the film and substrate contributions. This converted it into a hyperbolic form that approached an asymptote as the indenter approaches the film/substrate interface. This asymptote was described by a simple linear approximation whose slope directly revealed the substrate's elastic modulus. The method enabled the elastic behavior of the brittle ceramic to be assessed without the indenter penetrating it. In fact, this method can assess properties of an unknown film/substrate composite as long as one has an estimate of the film thickness and the film can appreciably plastically deform.

## 1. Introduction

Depth sensing indentation has long been a standard method to rapidly assess mechanical properties of bulk materials as well as thin films and nanoscale structures. It is adept at measuring fundamental material properties such as elastic modulus owing to its direct relationship with the unloading stiffness [1–6]:

$$S = \frac{dP}{dh} = E\frac{2}{\sqrt{\pi}}\sqrt{a}, \tag{1}$$

where $S$ is the unloading stiffness, $E$ is the elastic modulus and $a$ is the contact area. However, direct indentation of hard, brittle materials becomes complicated by the formation of cracks and other defects during indenter penetration [7–10]. These defects hinder assessing the material's elastic modulus as they consume the stored elastic energy imparted into the material from the loading segment. Consequently, the measured unloading stiffness is lower than expected resulting in a smaller calculated elastic modulus. This work reports on a method to overcome this issue by employing a metallic film to absorb the inelastic damage of the penetrating indenter while transferring the load to the brittle material.

## 2. Experimental

Gold was chosen for the film material owing to its ease of deposition and capacity for plastic deformation. Single crystal (0001) aluminum oxide was used as the substrate due to its tendency for indentation crack generation [11]. The Zhou-Prorok model for thin film indentation was employed as it has been shown to be adept at predicting indentation elastic properties of film/substrate composites [12,13]. This model, leveraged from the King modified Doerner and Nix empirical function [4,14], has the basic form of the inverse rule of mixtures with weighting factors that modulate the interplay between film and substrate:

$$\frac{1}{E} = \frac{1}{E_f}(1 - \Phi_s)\cdot\left(\frac{E_f}{E_s}\right)^{0.1} + \frac{1}{E_s}\Phi_f \tag{2}$$

Here, $E$ is the composite modulus, $E_f$ and $E_s$ are the elastic moduli of the film and substrate respectively. The weighting factors applied to both the film and substrate, $\Phi_f = e^{-\nu_f(t/h)}$ and $\Phi_s = e^{-\nu_s(t/h)}$, account for the effects of lateral restraint that the film has on the substrate and vice versa. Here, $\upsilon_f$ and $\upsilon_s$ are film and substrate Poisson's ratios, $t$ is the film thickness, and $h$ is displacement into the film. The ratio of $E_f/E_s$ raised to the 0.1 power accounts for the initial substrate effect on the indentation data. A comparison of this model with other models was given in prior work [12,13].

The composite elastic modulus as a function of penetration depth was measured on an MTS Nanoindenter XP with a Berkovich diamond tip using the continuous stiffness measurement. The testing frequency was set as 45 Hz with a harmonic displacement target of 2 nm. All testing was performed under 0.05 nm/s of thermal drift rate. For a statistical representation of the indentation data each sample was indented with a 5 × 5 array of indents with 100 μm spacing in the x and y

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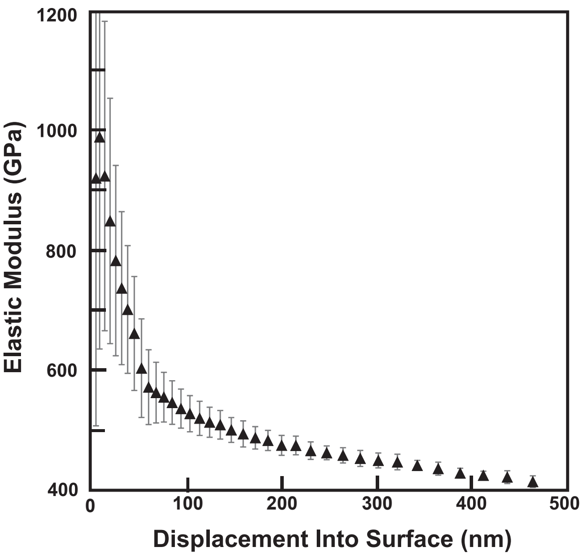

**Fig. 1.** Direct indentation of an $Al_2O_3$ single crystal (0001) illustrating that no clear consistent value of elastic modulus can be identified.

directions. Scanning electron micrographs of the indents were generated on a JEOL JSM 7000F and the focused-ion-beam (FIB) sectioning of indents was accomplished on a TESCAN LYRA3 FIB-FESEM. The gold films employed in this work were deposited by a Denton Discovery 18 sputtering system. A ten nanometer titanium layer was first deposited to adhere the gold film to the substrates. The unit was operated under DC power with substrate rotation using 100 W of power and 25 sccm of Ar flow [12,13,15–19]. Film thickness was measured on cross-sections with electron microscopy.

## 3. Results and discussion

The influence that defect generation has on measured elastic modulus when directly indenting a hard, brittle material is best seen in Fig. 1. Here the elastic modulus of single crystal (0001) aluminum oxide is plotted as a function of indenter displacement into the surface. Initially the measured modulus began at high value and rapidly decreases in the first 10–50 nm as Berkovich contact is established. The modulus then continued to decrease towards 400 GPa as the indenter approached the final indent depth. At no point is a clear consistent value identified. It is possible that the data could approach a consistent value if indented further. However, the commercial vendor reported a value of 472 GPa, which is consistent with literature values for bulk $Al_2O_3$ (0001) reported around 460–470 GPa [20,21]. It is readily apparent that the unloading stiffness was influenced by inelastic damage from the penetrating indenter. An electron micrograph of a typical indent from this data set is given in Fig. 2. Cracks radiating from the corners of the Berkowich indent are evident and an FIB cross-section shows they propagate downward into the material. The presence of these indentation generated defects obscures accurate extraction of elastic properties.

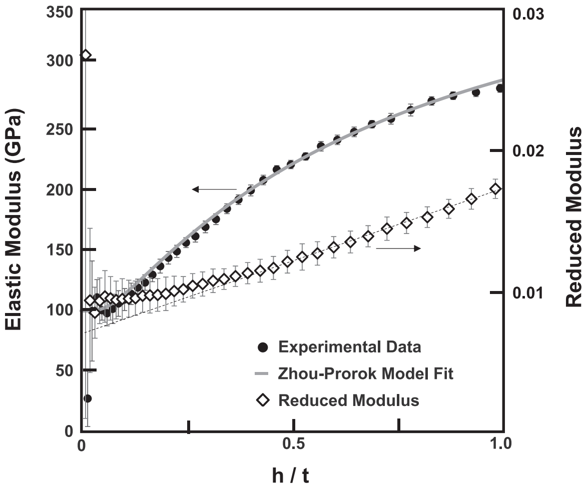

**Fig. 3.** Experimental indentation results of the Au/$Al_2O_3$ film/substrate composite and calculated reduced modulus.

The Zhou-Prorok model for thin film indentation can be leveraged with deposition of a metallic film to probe the properties of the substrate without indentation generated defects. A 475 nm thick gold film was deposited on the single crystal (0001) aluminum oxide substrate used above and indented to a depth of 900 nm, approximately two times the film thickness. The measured composite modulus is shown as a function of the depth/thickness ratio ($h/t$), closed circles ●, in Fig. 3. The modulus begins at approximately 100 GPa and increases continually as the substrate is progressively engaged with penetration into the composite. The solid gray line is the result of the Zhou-Prorok model using fitted values of $E_f = 80$ GPa, $\nu_f = 0.42$, $E_s = 475$ and $\nu_s = 0.226$. These values are very similar to literature values for both materials [20,21]. The agreement with the model when the indenter is within the film (between $h/t = 0$ and $h/t = 1$) is a good indication that the ceramic substrate is likely free of indentation generated defects. This was confirmed with a focused ion beam cross-sectioning of the indents, Fig. 4. Here, the indenter was halted at depth of 425 nm ($h/t = 0.89$) and the $Al_2O_3$ substrate did not appear to exhibit any damage in the three indents imaged.

The Zhou-Prorok model agrees with the composite data well enough; however, it relies upon estimating the ceramic's elastic modulus

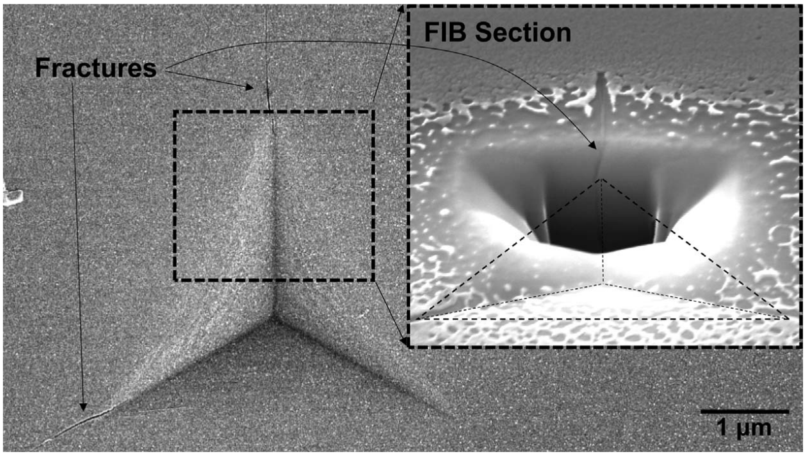

**Fig. 2.** Scanning electron micrograph of a residual indentation on the $Al_2O_3$ (0001) substrate with visible fractures highlighted by arrows. The inset is an FIB section revealing the fracture has propagated well into the material.