MECHANICAL CHARACTERIZATION OF SMALL VOLUMES VIA NANOINDENTATION

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Abstract: The load-penetration depth data obtained using nanoindentation testing can be used in a procedure to compute important mechanical properties, such as Young modulus, stiffness and hardness of the studied systems. A series of nanoindentation tests were performed on thin CeO\textsubscript{x} films deposited on aluminium substrate and the calibrated data for the indenter tip geometry were used for processing the current measurements. The importance of regular calibration of the indenter tip for getting reliable approximation for the contact area at shallow indentation depths is depicted and discussed.

Keywords: mechanical properties characterization, nanoindentation, contact area calibration

1. Introduction

For the purposes of thin film nanoindentation performed with Agilent Technologies Nano Indenter G200 equipped with a diamond Berkovich tip (Figure 1) a calibration on a silica reference sample was performed to estimate the blunting of the indenter tip and to determine the contact area at shallow indentation depths, less than 250 nm. This calibration is important for systems of films thinner than one micron deposited on a more compliant substrate. The reason is that in this case for the determination of the film mechanical properties it is required to use nanoindentation data at depths less than 100 nm while the standard calibration of the contact area function is performed at more than ten times larger depths of penetration.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{berkovich_diamond.png}
\caption{Berkovich diamond 3-sided pyramid.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{indentation_modulus_hardness.png}
\caption{Indentation modulus and hardness}
\end{figure}

2. Materials

The hardness and Young's modulus are determined by means of nanoindentation experiments for two different samples being film-substrate systems obtained at different initial states and under different conditions for depositing the conversion CeO\textsubscript{x}/Cu films. The substrate for the first system (system 1) is Al 1050 treated with acetone before films deposition. The substrate for the second system (system 2) is also Al 1050 but in addition it was post-treated with 3 wt. \% NaPO\textsubscript{4}.

The results for the indentation modulus and the hardness for the substrates and the two systems of conversion CeO\textsubscript{x}/Cu films are shown in Figure 2. The loading program is depicted in Figure 3. It consists of four cycles of loading and unloading and follows the standard XP method: G-Series Basic Hardness, Modulus, Tip Cal, Load Control with maximum load of 0.93 mN and peak hold time of 20 s.
3. Indenter tip calibration

The geometry of the indenter tip plays important role in processing the data from nanoindentation tests as it determines the size of contact area whose value is used in the calculation of the hardness and the reduced modulus according to the Oliver and Pharr method [1]. The fit function is based on powers of the contact depth $h_c$ (the penetration depth at first full contact, Figure 4) with polynomial fitting coefficients to be calibrated against data on a material with known elastic modulus.

Figure 4. Schematic presentation in a cross section of the indentation test geometric characteristics.

In our case we used fussed silica whose mechanical properties are given and it has been proven to be homogeneous and with constant properties in depth.

4. Indenter tip calibration results

The expression for the contact area as a function of the contact depth reads:

$$A(h_c) \approx C_0 h_c^2 + C_1 h_c^4 + C_2 h_c^{1/2} +$$
$$+ C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16}$$

Therefore, we have to determine six fitting coefficients that are involved in the fitting function in a linear way (e.g. linear regression can be applied). Figure 5 presents the chart of the algorithm used to perform the indenter tip calibration against indentation data on reference fussed silica sample.

Figure 5. Chart of the contact area calibration algorithm.

The loading program for the nanoindentation tests used for the contact area calibration is shown in Figure 6.

Figure 6: Loading program for Silica Reference sample, maximum load of 3 mN

The results of the linear regression fit of the data obtained on fused silica in 2011 and 2016 are given in Figure 7 (default fit in this figure is understood the contact area function obtained in 2009 for maximum penetration interval from 1 um to 3 um).

Figure 7: Comparison of the calibrated contact area polynomial functions in years 2011 and 2016.
Conclusions
In the course of five years of using the particular Berkovich diamond tip there was a noticeable blunting of the tip that results in a different contact area for shallow depths. The more recently obtained area function has steeper slope than the former one thus indicating a larger contact area at identical $h_c$.

The actual area was used to correct the determined mechanical properties of Ce/Cu film on aluminium substrate. Calibration of the indenter tip coefficients is particularly important for very small penetrations less than 200 nm. The results reported here reveal the importance of careful and regular calibration of the contact area in case Oliver and Pharr method is to be used to process the nanoindentation test data for obtaining reliable values for the indentation modulus and hardness. Area tip calibration is of special importance for indents at depths below 100 nm.

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References: