Abstract

The effect of the surface roughness on nanoindentation results was investigated instancing a series of CrN thin films deposited by unbalanced magnetron sputtering. The arithmetic roughness (Ra) of the films ranged between 2 and 10 nm and was measured by atomic force microscopy. The measured surface topography was incorporated into a finite element model, which allowed simulating the indentation of an axisymmetric sample by a rigid spherical indenter. For the applied conditions it was found that plastic deformation could be neglected and thus purely elastic material behavior was assumed. For roughness values of Ra ≈ 2, 5, and 10 nm, 100 indents each were simulated. Subsequently, the software Elastica and the approach by Oliver and Pharr were used to evaluate Young’s modulus of the CrN thin films from the simulated load-displacement curves.

Under the applied conditions, the increasing roughness causes a reduction of the contact area and leads to an underestimation of Young’s modulus. The mean Young’s modulus of all simulated indents on the rough surfaces lies 5–14% below the Young’s modulus determined for a perfectly smooth surface. This deviation seems to be independent of Ra, although the data scatter increases significantly with increasing roughness. Additionally, an influence of the lateral extension of the surface texture on the data scatter was observed which is not accounted for in roughness measures such as Ra.

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1. Introduction

Surface roughness can play an important role in the determination of mechanical properties from nanoindentation experiments, especially at low indentation depth. In order to calculate hardness or Young’s modulus from indentation data, precise knowledge of the contact area is needed. The contact area is not measured directly in the indentation experiment but it is usually calculated from the depth of contact and the indenter geometry. This implies the assumption that the contacting surfaces are completely smooth and in continuous contact at all points within the contact depth. Hence, samples for nanoindentation experiments should be very smooth and the indentation depth should exceed the arithmetic roughness by far (e.g. \( h > 20 \times Ra \) for hardness measurements with a Vickers indenter [1]). These requirements may pose a problem for the indentation of thin films, because the indentation depth is very limited in order to avoid a strong influence of the substrate. Under such circumstances, the surface roughness may cause a significant deviation of the actual contact area from the contact area according to the indenter geometry. Consequently, the surface roughness is a potential source of error for the analysis of nanoindentation data, especially for spherical indenters with large radii and small loads [2].

The analytical solution for elastic contact between a smooth spherical indenter and a flat sample is included in the Hertz theory [3]. Surface roughness can only be incorporated if the dimensions of the surface asperities are much smaller than the contact dimensions, which is not the case here [4]. To the best of our knowledge, little research relevant to the contact conditions in nanoindentation of typical PVD deposited thin films has been published. Experimentally, the problem has been tackled very recently by Qasmi and Delobelle, who investigated the
influence of roughness on the precision of Young’s modulus and hardness determination using nanoindentation with a Berkovich indenter [5]. Their findings agree with an earlier work of Bobji et al. who investigated the influence of surface roughness on the scatter in hardness measurements of rock material [6,7]. They found that the scatter decreases with higher loads and softer materials and that it can be described with a power law relation as a function of the penetration depth and roughness. It is the aim of this work to investigate by finite element modeling (FEM) the influence of the surface roughness on the Young’s modulus determined from nanoindentation with a spherical indenter for a typical hard coating.

2. Experiments

CrN coatings were deposited by unbalanced reactive magnetron sputtering in a commercial Oerlikon Balzers RCS Rapid Coating System. Chromium targets and a mixture of argon and nitrogen at a constant total pressure of 1 Pa were used. The substrate holder was situated at a distance of 22 cm in front of the target and no rotation was used. The coatings were deposited to a thickness of 3 μm on Si (100) wafers at a temperature of 350 °C and a nitrogen partial pressure of 0.25 Pa. Adjusting the bias voltages between −40 V and −120 V, coatings with a surface roughness Ra ranging between 2.6 nm and 10 nm could be synthesized.

The surface roughness was measured by atomic force microscopy (AFM) on a DI Dimension 3100 device. To evaluate the arithmetic roughness, an area of 10×10 μm was scanned with 512×512 sampling points.

3. FEM simulation

The indentation process was simulated with a nonlinear static analysis, taking into account large displacement effects but ignoring all time-dependent material behavior. The physical setup modeled consists of a spherical indenter with a radius of 50 μm on a cylindrical sample with 100 μm radius and 350 μm axial extension. The sample size has been chosen large enough to approximate the material behavior of a semi-infinite half-space under the applied conditions. 4-node bilinear axisymmetric elements are used to mesh the sample. The smallest elements measure 0.1×0.1 μm and are situated in the area of contact. Away from the center of contact, the mesh is gradually coarsening. The boundary conditions prohibit the movement of nodes on the rotational axis in a radial direction and the movement of nodes at the bottom of the mesh in an axial direction. The top 3 μm of the sample represent the CrN coating with a Young’s modulus $E$ of 323.6 GPa and a Poisson’s ratio $\nu$ of 0.25. The material below the coating is Si substrate ($E$=164.4 GPa, $\nu$=0.224). The diamond indenter is assumed to be rigid. A peak load of 15 mN is used which corresponds to a maximum indentation depth of 33 nm for the smooth sample.
An indication that the influence of plasticity is negligible is the complete reversibility of the simulated loading-unloading curves. Additionally, a simulation with elastic-perfectly plastic material properties was conducted assuming a yield strength of 8.5 GPa for CrN. The equivalent plastic strain was found to be below 0.02% and occurred locally at the asperities. The resulting load-displacement curve was virtually identical to the curve for purely elastic material behavior. Thus the error assuming purely elastic material behavior should be very small.

A comparison to the Hertzian solution of the contact problem was used to check the validity of the finite element model. Since the Hertz contact equations are designed for homogeneous materials, the complete finite element model of the sample was assigned to the properties of CrN or Si, respectively. Fig. 1 shows that there is very good agreement between the simulated and the Hertzian load-displacement curves for these two cases. Therefore, the model is assumed to be a valid representation of the indentation process. Furthermore, Fig. 1 contains the load-displacement curve for the sample consisting of a 3 μm thick CrN coating on a Si substrate. The deviation of the curve for the thin film from the bulk CrN indicates the effect of the substrate on the indentation behavior.

The surface roughness for the finite element model was taken from experimental AFM data. Line scans of the AFM surface profiles were then interpolated at the top surface node positions of the sample mesh and these nodes were repositioned in axial direction according to the measured surface profile. The effect of this interpolation on the surface roughness of the profiles remains below 0.1 Ra. However, it has to be kept in mind that the model does not resemble 3-dimensional surface roughness due to its symmetry, which is a necessary compromise in order to keep the model size within feasible limits.

4. Results and discussion

A series of samples with increasing roughness amplitude (Ra=2.6, 5.4, 11.1 nm) and two samples with similar amplitude (Ra=2.6, 2.8 nm) but different frequency were investigated. For each sample, 100 indents at different positions were simulated.

Comparing the simulated load-displacement curves for different surface roughness values in Fig. 2 it is evident that the data scatter increases with the roughness. A large data scatter due to high surface roughness is a problem frequently encountered when trying to measure the mechanical properties of thin films with nanoindentation. As the next step, these load-displacement curves were analyzed in order to determine the Young’s modulus of the thin films.

The analysis of the load-displacement curves was performed with the software Elastica [8] and with the approach according to Oliver and Pharr [9], but fitting a second order polynomial to the unloading curve instead of a power law. To check the sensitivity of the results to the fitting method all curves were evaluated using the upper 20% and the upper 80% for curve fitting. The difference in mean Young’s modulus was less than 2% (~2 GPa) and all results shown here were evaluated fitting the upper 80% of the unloading curve. Using these two methods, the corresponding Young’s modulus was calculated for each load-displacement curve and the resulting mean value and scatter are shown in Fig. 3. It can be seen that the mean Young’s modulus is consistently lower when evaluated with the Oliver and Pharr approach than when evaluated with Elastica. This is due to the effect of the softer substrate on the indentation result. A strong substrate effect was already indicated by the
position of the load-displacement curve of the coated sample between the curve for pure coating and pure substrate material in Fig. 1. While this effect is considered in the analysis with Elastica, it is not included in the Oliver and Pharr approach and therefore lowers its mean value. However, this effect is widely known [10–12] and shall not be the focus of this work.

As a general trend, an increasing data scatter with increasing Ra can be identified in Fig. 3. While for a roughness of Ra=2.6 nm, the standard deviation constitutes 27% of the mean value, it amounts to 58% of the mean value for a roughness of Ra=11.1 nm in Fig. 3a. The latter roughness value is not exceptionally high for sputter deposited coatings, but indeed the scatter seems unacceptably large for a meaningful determination of the Young’s modulus of this thin film. However, it can be seen in Fig. 3a and b that the offset of the mean Young’s modulus from the actual (input) value for the simulated coating seems independent of the surface roughness and the data scatter. All mean values of the rough samples lie below the value for the perfectly smooth sample (dashed lines in Fig. 3). No clear trend for the mean value with increasing roughness amplitude is visible. It remains within 8–14% below the value for the smooth sample for the data evaluated with Elastica and within 5–7% for the data evaluated according to Oliver and Pharr. Thus, the surface roughness leads to an underestimation of the Young’s modulus, but the amount of this deviation seems to be independent of the actual value of Ra. This indicates that the mean value of a sufficiently large number of indents can give a good approximation of the Young’s modulus of the coating, even if the surface is rough and the data scatter is high. On the other hand, it shows that there is a noticeable influence of the surface roughness on the measured properties also for surfaces with a smoothness beyond what can be reached with a simple polishing process (Ra=2.6 nm). The data scatter may be smaller for smoother surfaces, but the resulting mean value is not necessarily more accurate. In practice, this may mean though that a mean value of comparable quality can be reached with fewer indents for smoother surfaces.

Comparing the data for the two samples with similar roughness amplitudes (Ra=2.6, 2.8 nm) it appears that the frequency of surface elevations may also influence the measured properties. The high-spot count was chosen as a measure for the number of surface elevations per unit length [13]. Multiplication with the nominal contact radius yields the average number of surface elevations within the contact radius. The sample with Ra=2.6 nm has an average of 2 elevations within the contact radius and the sample with Ra=2.8 nm has an average of 3 elevations within the contact radius. In Fig. 3 it can be seen that the surface with more elevations within the contact area produces less data scatter.

Even for the highest roughness amplitude and high-spot count encountered here, the surface topography of the rough sample can be described best as slightly undulated related to the size of the indenter. Between 2 and 6 elevations are situated within the area of contact and their lateral extension exceeds their height by far. Fig. 4 exemplarily shows the simulated von Mises stress under maximum load for a sample with Ra=11.1 nm. Stress is accumulated at the surface elevations under load but due to their low height to width ratio, they are not completely flattened under the applied conditions. The contact area under maximum load is smaller than in the case for a perfectly smooth surface as can be seen in Fig. 5. This deformation behavior makes the rough coatings appear more compliant than a smooth coating and leads to the underestimation of Young’s modulus by nanoindentation. Greenwood et al. found that the Hertz theory is not likely to be in error by more than 7% if the ratio of indenter radius (R) and surface roughness (σ) to the contact radius given by the Hertz theory (a₀) is a₀/σ ≤ 0.05 [14]. This ratio is exceeded by a factor of more than 2 by all samples investigated here which may explain the larger scatter of the data analyzed with Elastica/Hertz compared to Oliver and Pharr.

5. Conclusions

For the indentation of CrN thin films with a spherical indenter in the elastic deformation range, the measured Young’s modulus tends to be underestimated. The amount of underestimation due to roughness lies between 5% and 14% depending on the analysis method. Within the investigated roughness range, this amount is independent of the arithmetic roughness Ra. This includes surface roughness values in the region of polished surfaces. For the chosen large number of indents per sample, the scatter shows no influence on the mean value. The scatter of the measured Young’s moduli increases with increasing Ra and shows a tendency to decrease with increasing high-spot count. A comparison to a 3-dimensional model and experimental values is planned for the future.

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References