Surface roughness is extremely important in Instrumented Indentation Testing, the technique used by the Micro and Nano Indentation Tester (MHT/NHT), because the mechanical properties of the tested material are calculated on the assumption that the sample surface is flat. No surface is perfectly flat and every surface has a certain amount of roughness. This application note focuses on the roughness influence of copper on the mechanical surface properties measured by IIT.

Because the IIT technique uses the measured penetration depth to estimate the residual contact area and subsequently calculate the hardness, the surface roughness can have a significant influence on the resultant values. The contact between two bodies is not only controlled by the material properties (elastic modulus and hardness) but by topographical properties as well.

Two situations may arise in practice: firstly, when the indenter comes into contact with a peak, the non-uniform contact increases the localized stress at the points of contact, deforming the material to a greater depth at relatively low loads. This can result in a greater penetration depth and lower calculated hardness. Secondly, if the indenter comes into contact with a valley, the opposite phenomenon is observed, i.e. the true contact area is underestimated and consequently, the calculated hardness is overestimated.

Two copper samples with different average roughnesses Ra were investigated: the first one with a mirror polish (referred to as “smooth” in this application note) was measured with a profilometer at 5 nm Ra, the second with a sand paper finish (referred to as “rough”) was measured at 167 nm Ra. The first phase of testing was to perform 10 indentations at 10 mN maximum force on both samples using a Berkovich indenter. Fig. 1 shows optical micrographs of a typical indentation on each of the two measured sample. It can clearly be seen that the residual imprint on the smooth surface is clearly defined whereas on the rough surface it is deformed and unclear.

The force-penetration depth curves for both samples are shown on Fig. 2 confirming that these curves are more scattered on the rough surface than on the smooth one. The 10 curves obtained on the smooth surface are superimposed making obvious the homogeneity of the surface whereas on the rough surface the curves are scattered and shifted depending on whether the indentation was performed on a valley or on a peak.

Consequently, it can be seen that on a smooth surface the hardness is relatively constant whereas on a rough surface, the hardness is sometimes overestimated, sometimes underestimated. The instrumented hardness results shown in Fig. 3 demonstrate that the average value for both surfaces is quite close with 1338 MPa and 1405 MPa whereas the standard deviation is 11 MPa and 308 MPa (0.8% and 22% of the mean value) for the smooth and rough surfaces respectively.

Figure 1: Optical micrograph of residual indentation imprints at 10 mN force in copper samples with (a) smooth surface and (b) rough surface.

Figure 2: Force-penetration depth curves performed at 10 mN force on smooth and rough copper surfaces. 10 indentations tests have been performed on each surface showing that the curves are more scattered on the rough surface than on the smooth surface.

Figure 3: The average instrumented hardness results of 10 indentations performed on the smooth and rough samples. The average value for both surfaces is quite close with 1338 MPa and 1405 MPa whereas the standard deviation is 11 MPa and 308 MPa (0.8% and 22% of the mean value) for the smooth and rough surfaces respectively.
The aforementioned results confirm that it is imperative to know the condition of a surface before proceeding with an instrumented indentation test. The Ra value should be less than 5% of the maximum penetration depth as stipulated in the International Standard ISO 14577-4.

According to this standard and applying the 5% rule to the rough sample with a measured Ra of 167 nm, the maximum penetration depth should be at least 3340 nm (it was around 500 nm for the 10 mN force used previously). A second set of 5 indentations was then performed at 400 mN maximum force resulting in an average maximum penetration depth of 3780 nm. A residual imprint of one of these indentations is shown in Fig. 4 and has a much clearer outline.

Figure 4: Optical micrograph of an indentation imprint at 400 mN force in a copper sample with high roughness.

To compare both tests (10 mN and 400 mN), a normalized profile of the rough sample is presented with a schematic of the indenter in Fig. 5. It shows that for the 10 mN test, the surface cannot be considered as flat because the indentation imprint is small compared to the asperity size, which is not the case for the 400 mN test.

Figure 5: Normalized profile of the rough copper sample with a representation of the indenter for (a) 10 mN and (b) 400 mN maximum force. It shows that if the maximum penetration depth is higher than 20 times the roughness Ra, the surface can be considered as flat for such load.

Finally, the force-penetration depth curves for the rough sample shown in Fig. 6 confirm the better repeatability of the measurement if the maximum penetration depth is adapted to the roughness of the sample.

Figure 6: Force-penetration depth curves on the rough copper surface. The maximum force of 400 mN has been chosen so that the maximum penetration depth is higher than 20 times the roughness Ra.

In conclusion, roughness has a strong effect on the dispersion of results in Instrumented Indentation Testing and is an important factor to take into account when planning experimental parameters. Smooth and well-polished surfaces are therefore preferred for reproducible and reliable results. But it must be also pinpointed that polishing can introduce some residual stresses at the sample surface if the sample is not prepared adequately. Residual stress created by “hard” polishing can have some detrimental influence on the hardness evaluation at the nanometer scale.

An additional phenomenon which can influence the results of IIT are pile-up and sink-in effects. These produce variations in the true projected contact area where the contact area can be over- or underestimated. When indentations are performed deeper in materials, the effect of roughness will be less important but the pile-up or sink-in effect might increase.

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