How to deal with roughness in nanoindentation measurements

Relevant for: NHT^3, Hit 300, UNHT^3, MCT^3

Nanoindentation is often used for measurement of thin layers and small volumes of material. On such materials low indentation depth is required to obtain correct results. However, roughness of the measured surface can negatively affect the results. In this application report we show the effect of surface roughness on the nanoindentation results and methods to minimize it.

Figure 1 – The Hit 300 nanoindentation tester.

1 Introduction

The nanoindentation technique has primarily been developed for use on thin layers and coatings because it allows measurements of layers with micrometer and sub-micrometer thickness [1–4]. The demand came from the thin film industry and research because hardness of thin films and coatings provides fast estimation of their quality. Since then, the method has found wide use also in other areas of material research such as biomaterials or metallurgy where hardness of different phases or grains reveals information about their microstructure [5–7]. With the growing demand for measurement of surface and near-surface properties the surface morphology became an important factor. Many thin films and coatings are smooth with roughness below 1 nm. However, there are many thin films whose surface roughness is in tens and hundreds of nanometers. The same applies for metallic samples: some are perfectly smooth but many samples exhibit considerable surface roughness. In nanoindentation the depth involved in the mechanical response is often in the tens and hundreds of nanometers, which is in the same order of magnitude as the surface roughness. The effects of surface roughness are mentioned in the ISO 14577 standard which specifies that the R_a of the measured surface shall be less than 5% the maximum indentation depth. In real life, this criterion is sometimes difficult to achieve and it is therefore good to know the effects of roughness on the nanoindentation results [8].

In this application report we demonstrate the influence of roughness on the hardness results on two typical nanoindentation samples: 4.2 µm thick AlTiN hard coating and stainless steel. Both materials were available with different surface states: in the as-deposited state (AlTiN) and in a polished state (stainless steel). The surface roughness was then modified by either roughening with sand paper (stainless steel) or by polishing with 3 µm diamond paste (AlTiN).

1.1 Materials and methods

The surface roughness of the AlTiN sample was measured in the as-deposited (non-polished) state and then after 1 minute, 3 minutes, 6 minutes, 10 minutes and 15 minutes of polishing with 3 µm diamond paste. The thickness of the coating was measured after each polishing step by Calotest. Hardness in the as-deposited state as well as after each polishing step was measured using the Hit 300 Anton Paar nanoindentation tester with 50 mN maximum load in a 4x3 indentation matrix with 100 µm spacing.

The stainless steel sample, with surface polished with 1 µm diamond paste, was roughened progressively with 2000, 1000, 600 and 240 sand paper (the higher the paper number, the finer the paper). After each roughening step the R_a of the surface was measured and the surface was observed in optical microscope. The hardness of the sample was measured with the Hit 300 Anton Paar nanoindentation tester using a 3x3 indentation matrix with maximum load 10 mN and spacing 100 µm between indentations.

Average hardness value and its standard deviation was calculated from all indentation curves. Only indentation curves obviously deviating from the main results (outliers) were excluded from the statistical analysis. Coefficient of variation (CoV, standard deviation divided by the average value) was calculated for each sample and each surface state.

In addition, the results obtained on the AlTiN coatings were verified on a 4.5 µm thick TiN coating.

2 Results

2.1 Stainless steel

The indentation curves obtained on steel polished with 1 µm diamond paste and subsequently roughened with 1000 and 240 sand paper are shown in Figure 2. The scatter of the curves increases with decreasing (rougher) sand paper number.

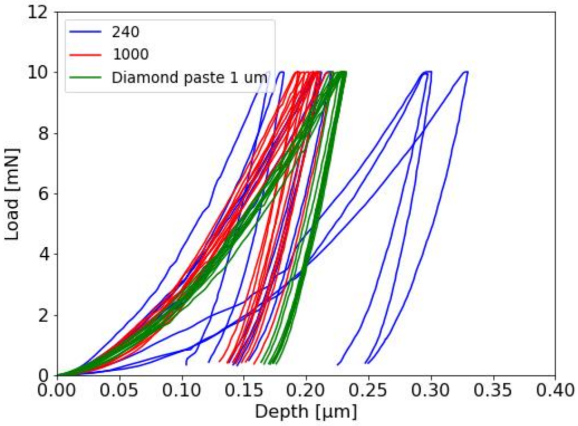

Figure 2 - Indentation curves at 10 mN on steel with different surface states.

The maximum penetration depth at 10 mN was approximately 0.2 µm – which is comparable to the R_a value of the surface after the 240-sand paper roughening. The scatter of the indentation curves was clearly reflected in the scatter of the hardness (H_{IT}). Figure 3 shows hardness values as a function of surface roughness expressed by the R_a. While average H_{IT} was only slightly affected by surface roughening (the observed increase can be attributed to work hardening), the standard deviation clearly increased with increasing R_a. The variation of H_{IT} was less than 3% for the lowest R_a of ~7 nm whereas for the highest R_a (177 nm) the variation of H_{IT} was more than 40% (Figure 4). To compare the surface with different roughness visually, an optical image of the surface after each roughening step was taken with the same magnification. Figure 5 shows the surface morphology of the stainless steel surface after 2000, 1000, 600 and 240 sand paper roughening. The 240 paper roughening resulted in the highest surface roughness which progressively decreased with increasing sand paper number.

The above-mentioned effects of surface roughness on nanoindentation results were demonstrated on hardness. Similar effects were observed also on the elastic modulus values: the roughest surface resulted in ~30% CoV whereas the polished surface resulted in ~5% of CoV. Lower variation of elastic modulus compared to hardness is due to the fact that the elastic modulus is related to the slope of the unloading curve (contact stiffness) which is less affected by the surface roughness than the maximum depth which is used for calculation of the contact area and the hardness value.

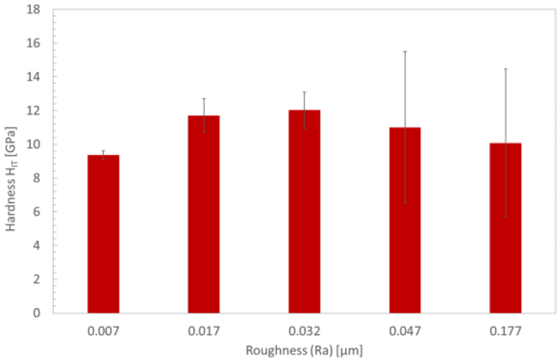

Figure 3 - Hardness and its variation as a function of surface roughness R_a.

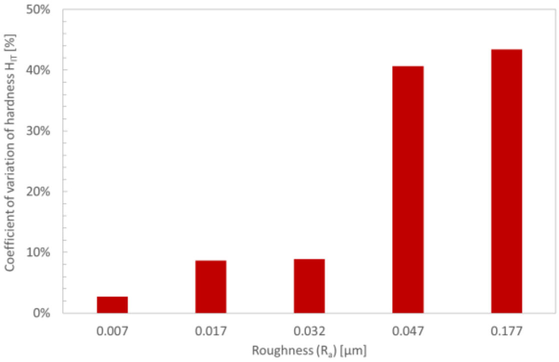

Figure 4 – Coefficient of variation of hardness as a function of surface roughness R_a.

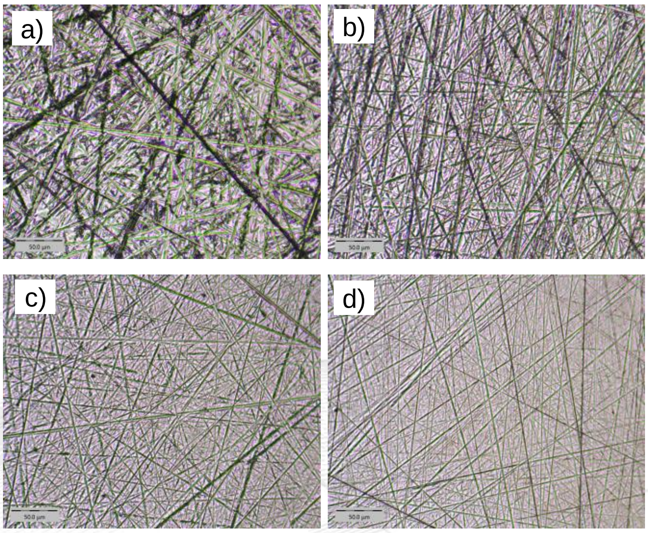

Figure 5 - Comparison of surface morphology after roughening with a) 240, b) 600, c) 1000 and d) 2000 paper.

2.2 AlTiN coating

While surface preparation of bulk materials is a well-controlled process, polishing of hard coatings is more delicate since only a small portion of the coating can be removed. The thickness of the remaining coating after polishing shall be measured in order to define reliably the maximum indentation depth at which the effect of the substrate is not influencing the results. When thickness measurement is not possible, a hardness depth profile (obtained by a Sinus mode) can be used to determine the maximum indentation depth on the polished coating.

A 3 µm diamond paste and polishing machine Le Cube (Presi, Eybens, France) was used for polishing of the AlTiN coating. The duration of polishing varied between 1 minute to 15 minutes: we observed that such polishing times lead to decrease in scatter of results – without significant removal of the coating. The indentation measurements after different polishing times are shown in Figure 6.

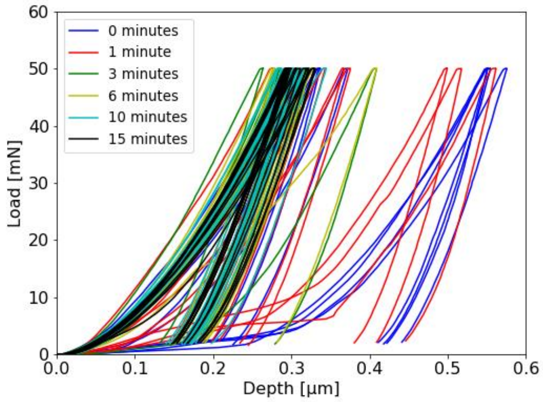

Figure 6 - Indentation curves at 50 mN on the AlTiN coating after different polishing with 3 µm diamond paste for different times.

Surprisingly, the surface roughness (R_a) remained at ~0.13 µm for all polishing times except the 15-minute polishing when the R_a value decreased to ~0.03 µm. However, the coefficient of variation has significantly decreased already after three minutes of polishing (see Figure 7 for details). This was a very encouraging outcome, nevertheless it had to be confirmed that the remaining thickness of the coating is sufficient for correct measurements. Calotest measurements at the as-deposited state and after each polishing step (Figure 8 and Figure 9) have shown that even after 10 minutes of polishing with the 3 µm diamond paste only about 0.1 µm of the coating was removed. This result confirms that fast polishing with diamond paste can be used for important improvement of the repeatability of the nanoindentation results – with only negligible removal of the layer.

Our results also indicate that the R_a value is not always a relevant parameter for assessing the repeatability of the measurements. A significant improvement of the repeatability (i.e. lower CoV) was observed despite only negligible decrease of the R_a value.

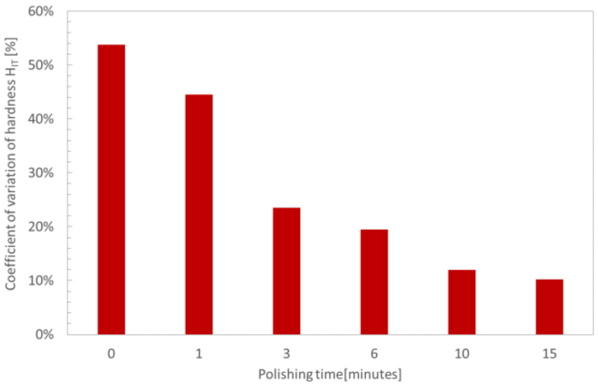

Figure 7 - Coefficient of variation of hardness as a function of polishing time.

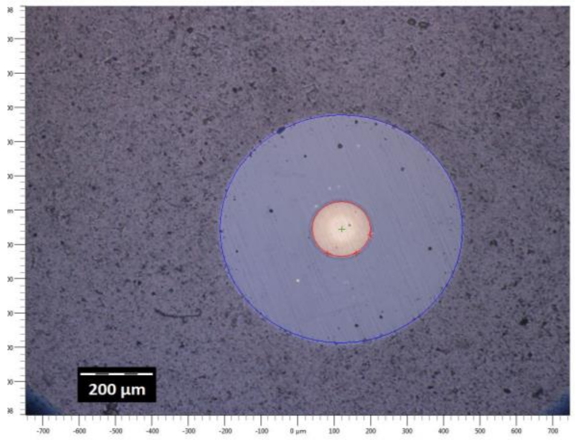

Figure 8 – Calotte on the non-polished AlTiN surface.

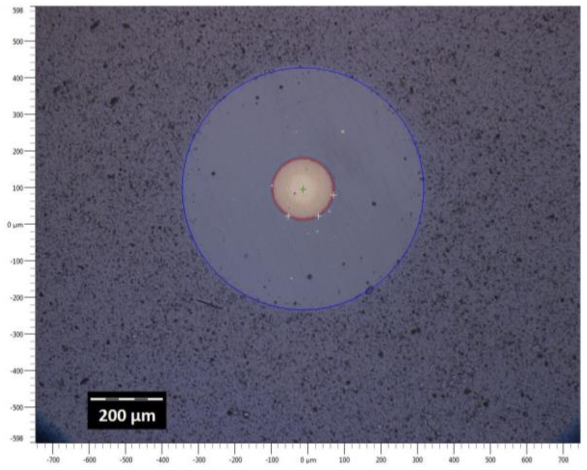

Figure 9 – Calotte on the AlTiN surface after 10 minutes polishing with 3 µm diamond paste.

2.3 TiN – verification of AlTiN polishing results

The effects of polishing of the AlTiN hard coating had to be confirmed on another hard coating. For this we used a TiN coating with 4.5 µm thickness and we polished it with 2500 sand paper and 1 minute with 6 µm and 3 µm diamond paste. Polishing with the 2500 sand paper resulted in removal of ~1 µm of the coating but polishing with the 6 µm and 3 µm paper resulted in removal of less than 0.5 µm, leaving approximately 4 µm thick TiN coating on the steel substrate. The CoV was 23% for the as-deposited state and it decreased to 8% for the 2500 sand paper and 11% for the 6 µm diamond paste. All indentations were done using the Hit 300 with 10 mN maximum load to ~0.13 µm maximum depth. These results suggest that polishing with the 6 µm diamond paste for 1 minute is the most efficient procedure since it removes only a small fraction of the coating while the standard deviation decreases two times. Polishing with 3 µm paste removed even less of the coating but did not lead to significant decrease of the coefficient of variation.

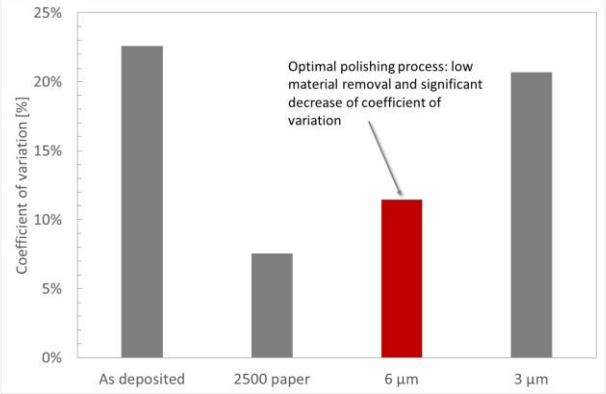

Figure 10 – Coefficient of variation for the TiN coating after different polishing procedures. The 6 µm polishing is the best compromise between layer removal and standard deviation.

2.4 Nanoindentation of hard coatings – polishing with diamond paste or with Calotest?

Calotest has long been recommended for quick local polishing of hard coatings in order to improve the repeatability of nanoindentation measurements. The greatest advantage of Calotest polishing is that the depth of the calotte shows directly how much of the coating thickness has been removed in the calotte. The main drawback of this method is that all indentations have to be made in the relatively small center of the calotte – which requires precise positioning.

Polishing with diamond paste eliminates the necessity for precise positioning. The problem is that we a priori do not know how much of the coating thickness has been left. However, with Calotest we can easily measure the remaining thickness of the coating and choose the maximum indentation depth for safe indentations without substrate influence. Since the entire surface of the sample has been polished, the precise positioning is no more required. A hardness of elastic modulus depth profile (Sinus measurement) can be used for assessment of the maximum indentation depth. An important improvement of the measurement repeatability can therefore be achieved relatively easily.

3 Summary

Due to shallow indentation depths, correct measurement of hardness and elastic modulus of thin coatings and small phases requires appropriate surface roughness. Even microscopic roughness can strongly affect the results of hardness and elastic modulus. This is reflected in large variation of the results. Here we have demonstrated how metallographic polishing leads to decrease in the variation of results on stainless steel, AltiN and TiN coatings. In the case of hard coatings, which is the main application for NHT^3 and Hit 300 instruments, already a 1-minute polishing results in substantial improvement in the repeatability of the results. It was also shown that such fast polishing process removes only a small portion of the hard coating and the nanoindentation can be done without the influence of the substrate.

4 References

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