::: Application Report

Anton Paar

Advanced Mechanical Surface Testing

Applications of indentation, scratch, tribology and coating thickness measurements in automotive industry

Part I: Indentation and scratch testing

Introduction

Anton Paar develops and manufactures precise instruments for characterization of mechanical properties of surfaces of many types of materials. The whole portfolio of Anton Paar instruments can be used in various sectors, such as (list is not exhaustive):

- Automotive
- Biomedical
- Cutting tools (hard coatings)
- Decorative coatings
- Electronics
- Polymeric materials
- Advanced ceramic & metallic materials

The aim of this application report is to demonstrate the use and capabilities of our instruments in the **automotive sector**. Although almost all of Anton Paar instruments can be employed in the automotive industry, here we will focus mainly on the following ones:

- Nanoindentation Tester (NHT³)
- Ultra Nanoindentation Tester (UNHT³)
- Revetest Scratch Tester (RST)
- Micro Scratch Tester (MST³)
- Nano Scratch Tester (NST³)

The first part of this application report provides an overview of selected applications of the above listed instruments for testing of hardness and elastic modulus, adhesion and scratch resistance. All applications in this report are based on the testing of different automobile components. The second part of this application report will focus on wear and friction properties and measurements of thickness of coatings.

Instrumented indentation

Nano Indentation Tester

Comparison of the brittleness behavior of two Cu-Pb alloys on bearing

Instrumented Indentation Testers (IIT) are high precision instruments used for the determination of mechanical properties of thin films, coatings and bulk materials. Properties such as hardness, elastic modulus, elastic and plastic work and creep can be determined on almost any type of material: soft, hard, brittle or ductile. As the automotive industry is using many advanced materials with surface treatments (DLC coatings for pistons and other low friction components, hard coatings for contacting parts, decorative coatings, etc.), the IIT is an important tool in characterization of the quality of these coatings.

An interesting application of the nanoindentation technique is comparison of the brittleness of two bearing components made of steel with different Cu-Pb coating alloys. The instrumented indentation tests were carried out using the NHT in order to characterize the brittle behavior of the Cu-Pb coatings.

The nanoindentation analysis focused on the following characteristics:

- the amount of plastic work of the indentation (W_{plast}, Figure 1),
- the ratio of elastic work to the total work of indentation (η_{IT}, Figure 2),
- the elastic modulus (E_{IT}, Figure 3).

In addition, Revetest scratch tests were done on the same samples in order to completely characterize the brittleness of these coatings.

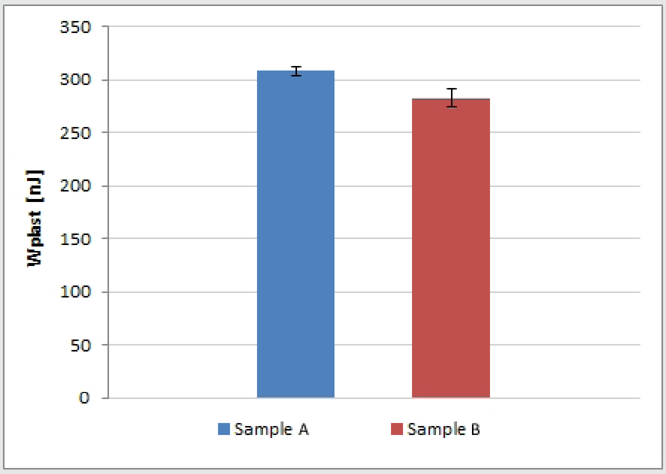

Figure 1 : Chart of the plastic work of indentation – W_{plast}.

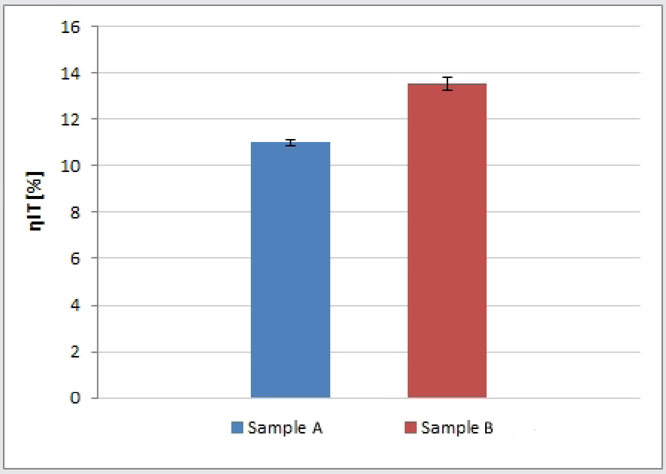

Figure 2 : Chart of the ratio of elastic work to the total work of indentation – η_{IT}.

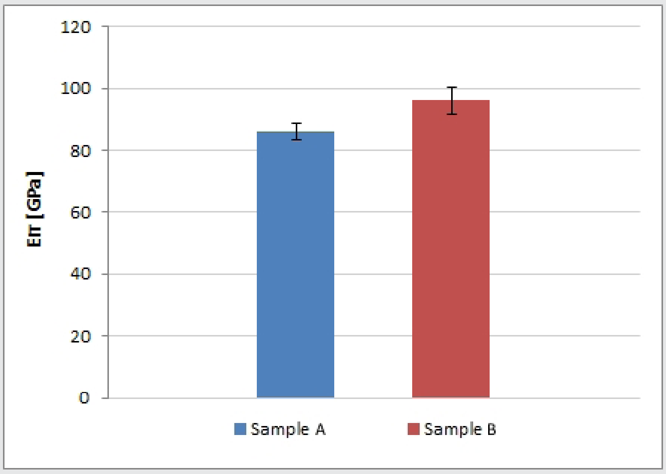

Figure 3 : Chart of the Elastic Modulus E_{IT}.

The experimental conditions for this application were: Berkovich indenter, linear loading, maximum load of 300 mN, loading and unloading rate of 1500 mN/min and pause at max load of 30 s.

The results show that sample B behaves more elastically than sample A. This was confirmed by both higher values of plastic energy W_{plast} (Figure 1) and ratio of elastic energy to total energy of indentation η_{IT} (Figure 2). Furthermore, Figure 3 shows that sample B has a higher elastic modulus, which is usually associated with more elastic behavior. Comparison of both characterization techniques (scratch and instrumented indentation) allowed a better comparison of the deformation behavior of the two tested coatings than when only one technique would be used. As stated previously, Figure 1-3 show that sample B behaves more elastically (i.e. it is very likely also more fragile). This is in agreement with the results found during the scratch tests, where sample B exhibited more fragile-like behavior than did sample A.

Ultra Nanoindentation Tester

Evolution of mechanical properties of a polymeric material with graded properties: case study on tires

Some applications require even better resolution and sensitivity than the Anton Paar TriTec Nano indentation tester to properly measure properties of thin or graded layers. In such cases the Anton Paar TriTec Ultra Nanoindentation Tester can be used. The unique patented feature of the Ultra Nanoindentation Tester (UNHT) is the active top referencing, which ensures unmatched thermal stability. Together with excellent force and displacement resolution, the UNHT is an ideal tool for testing of very thin layers and for long term creep measurements.

With the development of surface treatments, some polymers are treated to improve their mechanical properties near the surface. The depth of the affected layer has then to be determined. An example of this treatment is treatment by plasma which can be used for increasing surface hardness and stiffness of car tires. In the present application, the surface of polymeric material was treated by ion implantation in order to improve its mechanical properties. The depth affected by the treatment was ~400 nm.

The goal of this study was to study the evolution of the mechanical properties by indentation from the top surface and determine at which depth the properties of the base material are affected by the ion implantation. For this, the so-called Continuous Multi Cycle (CMC) indentation tests were carried out. The CMC procedure consists of applying cyclic loading-unloading indentations with increasing forces without losing contact with the surface (see Figure 4 for schematic illustration of the indentation procedure).

The testing parameters involved use of sphero-conical tip of 20 µm radius, maximum forces in the cycles varied between 10 and 250 µN (the maximum force in each cycle was increased in each following cycle), loading time of 0 s, unloading time of 10 s and pause of 30 s at maximum load in each cycle.

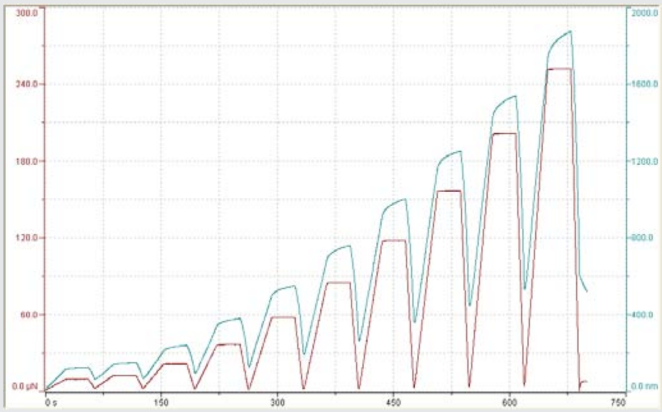

Figure 4 : Depth (green) and force (red) evolution during a CMC cyclic loading test (10 cycles).

As it can be seen in Figure 4, the cyclic indentation test is well suited for studying the effects of the surface treatment. The depth profile of elastic modulus obtained from the CMC indentation showed that in the depth of ~400 nm a change of the slope was observed. This change of slope of elastic modulus indicated that the polymer was affected by the ion implantation approximately to this depth (i.e. ~400 nm); measurements deeper in the material showed a constant value of elastic modulus.

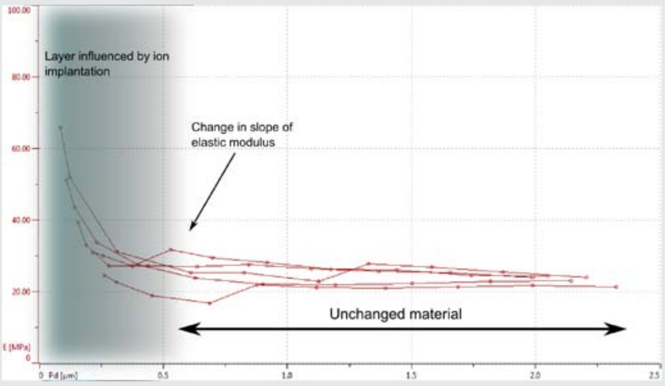

Figure 5 : Evolution of the elastic modulus as a function of depth during the cyclic indentation.

Such indentation measurements at very low forces were possible thanks to the UNHT thermal drift-free, active top referencing and its excellent force and displacement resolution.

Scratch Test

Revetest Scratch Tester

Differences in elastic behavior of galvanic coating of steel bolts

The scratch testers are instruments dedicated to characterization of adhesion and scratch resistance of thin films and coatings. This technique is widely used for testing of materials coated with hard protective coatings, materials coated with medium hardness coatings or for materials with softer, polymeric coatings. Anton Paar TriTec manufactures three types of scratch testers, which are dedicated for different applications:

- the Revetest Scratch Tester is most often used for adhesion testing of hard coatings with thickness in the micrometer range,
- the Micro Scratch Tester is used for materials with softer (metallic) and thinner coatings,
- the Nano Scratch Tester is suitable for testing of soft (polymeric) coatings or very thin coatings (hundreds of nanometers).

A great advantage of all Anton Paar scratch testers is the ability to record **Panorama** images (Pat. Pend.): this feature consists of recording the image of the entire scratch track and synchronization of this image to the recorded signal. This unique feature allows fast and efficient comparison of scratches on different samples even after tested samples are no longer present. It is also a great tool for archiving the results of the scratch tests.

Another important feature of Anton Paar scratch testers is the capability of evaluating both elastic and plastic behavior of materials, using the so-called **pre-scan** and **post-scan** procedure. This is possible by performing a three-pass procedure: by scanning the surface at low force before the scratch we obtain the original surface profile $\boldsymbol{P_f}$; the penetration depth during the scratch $\boldsymbol{P_{sc}}$ is obtained directly during scratching* and the residual depth $\boldsymbol{R_d}$ is obtained by low force scanning of the scratch track after the scratch ($\boldsymbol{R_{sc}}$). The true penetration depth $\boldsymbol{P_d}$ is then obtained as the difference between the original surface profile and the depth during the scratch:

$$P_d = P_f - P_{sc}$$

**note that most commercially available scratch testers offer only the Psc value which can be strongly affected by sample geometry and does not necessarily show the true penetration depth. The values of $\boldsymbol{P_d}$ and $\boldsymbol{R_d}$ are automatically calculated in the Anton Paar Scratch software.*

The residual depth $\boldsymbol{R_d}$ (elastic recovery) is calculated as

$$R_d = P_f - R_{sc}$$

The $\boldsymbol{R_d}$ shows the degree of elastic recovery of the material after the scratch.

The evaluation of the ability of the material to elastically recover after a scratch is also important in the automotive industry, whether it concerns hard coatings or soft polymeric paints. Elastic recovery after scratch damage is crucial for many galvanized parts such as steel sheets or bolts (see Figure 6). The vehicle's components tested for this application were steel bolts with three different, 7 µm thick, galvanized coatings.

Figure 6 : An example of galvanized steel bolt.

The test parameters used for this application were: 200 µm-radius diamond Rockwell indenter, progressive loading from 1 to 50 N, scratch length of 3 mm and scratching speed of 6 mm/min. The penetration and residual depth during the scratch tests was recorded.

Figure 10 and Figure 11 show respectively the penetration depth curves and the residual depth curves for the three samples tested.

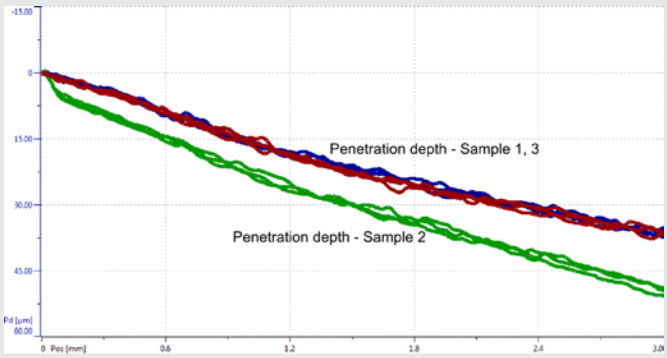

Figure 7 : Penetration depth curves for the three galvanized samples.

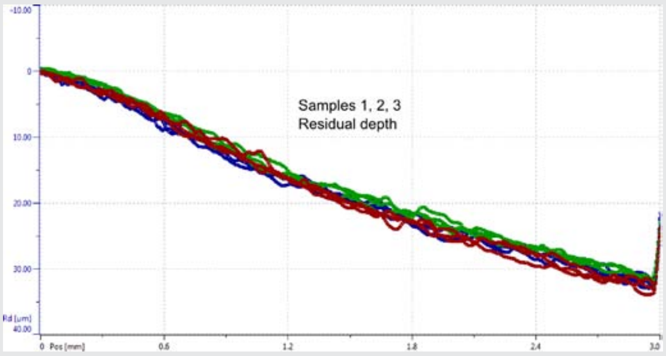

Figure 8 : Residual depth curves for the three galvanized samples.

By comparing the information of both curves (***P_d*** and ***R_d***) we could infer that the sample represented by the green lines (sample 2) had a more significant elastic recovery compared to samples 1 (blue lines) and 3 (red lines), despite Sample 2 having larger value of ***P_d***. Sample 2 is therefore resisting the scratch damage more than samples 1 and 3, which recover less after a scratch (the ***P_d-R_d*** value is smaller for samples 1 and 3). This important analysis of scratch and elastic recovery behavior of galvanized metallic samples could be performed thanks to the pre-scan and post-scan feature of the Revetest scratch tester.

Nano Scratch Tester

Scratch resistance of automotive clear-coat

The Nano Scratch Tester (NST) is especially suited to characterize the adhesive and cohesive failure of thin films, soft and hard coatings with a typical thickness of 1 µm and below. Examples are thin and multilayer PVD, CVD, PECVD, paints and lacquers, protective films and many others. The Nano Scratch Tester can also be used for determination of elastic-plastic behavior of thick (~40 µm) polymeric coatings using the pre-scan and post-scan procedure. Typical example of such thick polymeric coating is clear-coat on car paints. The purpose of this special transparent coating is to resist scratching and to recover when a scratch is performed.

According to the definition, clear-coat is an automotive painting technique in which a coating of clear lacquer or other synthetic liquid is applied over the base in order to enhance its shine and durability. Hence, due to mainly aesthetics purpose, it is important to know its scratch resistance and the ability to recover elastically. The ability of the clear-coat to recover elastically is sometimes referred to as mar resistance and it is commonly used in characterization of polymeric coatings.

In the current application four different samples with automobile clear-coat coatings of approximately 50 µm thickness were provided in order to determine their critical load Lc1 (appearance of first failure). The following parameters were used for the Nano scratch tests: 2 µm-radius diamond sphero-conical indenter, progressive load from 1 mN to 30 mN, scratch length of 1 mm and scratching speed of 1 mm/min.

Figure 9 : Panoramic view of the four scratch tracks with their respective Lc1 (critical load) indicated.

As it can be seen in Figure 9, sample 4 showed the best performance (resistance to failure) among the four tested samples: sample 4 exhibited the highest value of Lc1 value.

Determination of critical loads of Indium tin oxide (ITO) coated glass for windshields

Nowadays, the industry is using more and more thin films for different purposes: decorative, durability, functional, and many others. These films are sometimes very thin, with thicknesses of several hundreds of nanometers or even less. The materials used in the composition of these films can be either organic or inorganic, which will then be of paramount importance at the moment of the test in view of the characterization.

Indium tin oxide (ITO) is one of the most widely used transparent conducting oxides because of its electrical conductivity and optical transparency, see Figure 10. It is also used in the automobile industry where it prevents formation of ice on the windshield. The presented study aimed at determining the adhesion of the ITO coating to windshield glass. The thickness of the ITO coating has to be kept relatively low as larger thicknesses would lead to opacity of the coating.

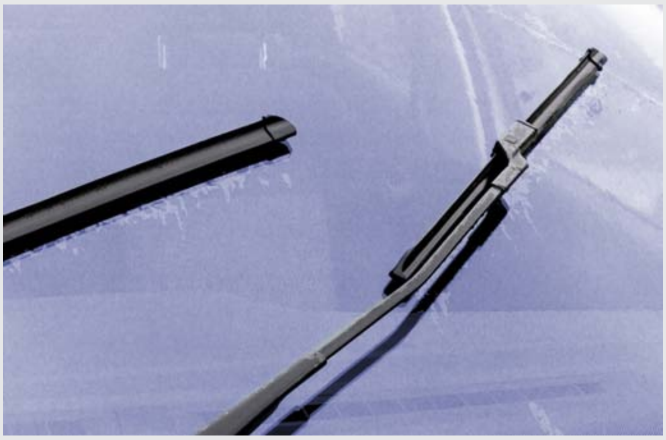

Figure 10 : An example of car windshield that can be coated with ITO for easier defrosting.

Therefore, in view of these restrictions, very sensitive technology is needed to obtain reliable results when testing adhesion of such thin films. This is the case of the Nano scratch tester (NST), which can apply very low loads and thus test very thin films. In addition – as with most of Anton Paar TriTec instruments – the NST can be equipped with a 100x magnification objective, which can visualize scratch tracks even on such thin coatings.

In this study two samples of ITO coated glasses with thicknesses of 100 nm (0.1 μm) were provided in order to analyze the differences between them in terms of adhesion of the system coating – substrate. A series of scratch tests together with Panoramic imaging was performed in order to determine the adhesion of these two types of coatings. The parameters used for testing of this material were diamond sphero-conical tip with 10 μm radius, progressive loading from 0.5 mN to 100 mN, scratch length of 500 μm and scratching speed of 1000 μm/min.

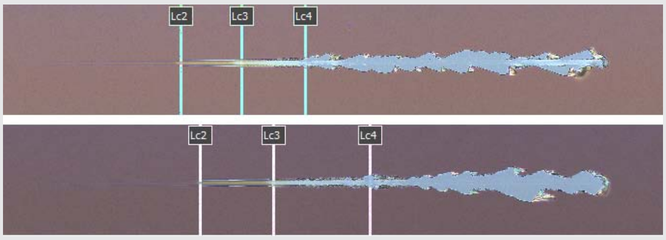

Figure 11 : Panoramic view of the scratch tracks of the two samples. Vertical lines indicate the critical loads. Sample 1 – top, Sample 2 – bottom.

Figure 14 shows the Panorama images of the scratch tracks. It can be seen that sample 2 showed higher critical loads than sample 1. This means that the coating on sample 2 has better adhesion (better resistance to scratching) than does sample 1.

Comparison of the brittleness behavior of two Cu-Pb alloys on bearing

This application of the Revetest scratch tester is complementary to the nanoindentation analysis of the same Cu-Pb coating pre-sented in the section dedicated to the indentation testing (see Figure 1). The goal was to compare the brittleness of two bearing components made of steel with different Cu-Pb coating alloys (see Figure 12 for an example of such bearing).

Figure 12 : Automobile bearing component.

Due to the lack of catastrophic failure, adhesion of ductile coatings is often difficult to determine using the conventional method of critical loads. The critical loads on these coatings are not determined visually as the plastic deformation is predominant in the failure mechanism and no abrupt change in scratch morphology is observed. Furthermore, the probability of a failure event (critical load) in a brittle material is higher than in a ductile material, which usually means that more sounds are likely to be emitted during the test (acoustic emission). Acoustic emission can therefore be used for determination of critical load – although optical inspection will remain the determining factor in critical load definition.

Figure 13 shows an example of acoustic emission signals obtained using the Revetest scratch tester. The difference between the two tested samples could be detected in this way, indicating a more brittle behavior for the coating 1 (represented by the green lines) than for coating 2, where less acoustic emission events were recorded.

The test parameters used in this application were: 100 μm-radius Rockwell diamond indenter, progressive loading from 1 to 150 N, scratch length of 1 mm and scratching speed of 2 mm/min.

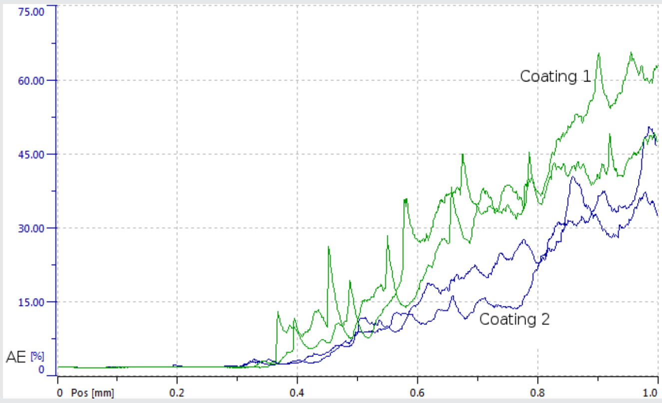

Figure 13 : Acoustic emission curves of the two samples. Two tests per sample were carried out.

The second part of this application report will focus on the applications of tribology and coating thickness measurements in the automotive industry.

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