CSM Instruments
Advanced Mechanical Surface Testing

CSM Instruments

APPLICATIONS BULLETIN

New Nano-Hardness Tester (NHT) for characterisation of MoS2 thin films

The technique

The NHT is especially suited to providing quantitative data on the hardness and modulus of sub-micron thin films and coatings, using an indentation method where a tip of known geometry is driven into the sample surface. The instrument itself can be seen in Fig.1 and features automated optical microscopic inspection before and after indentation.

The force on the indentor (all geometries can be used) is applied by an electromagnetic actuator, whilst the displacement is measured via a capacitive system, giving a force resolution of 10µN and displacement resolution <1nm.

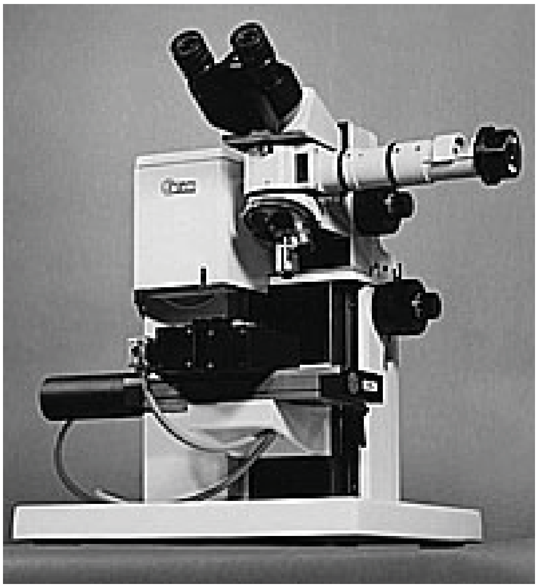


Figure 1 : The Nano-Hardness Tester (NHT)

Samples measured

For this study, a series of indentations were performed on an MoS_2/Pb multilayer that had been built-up by PVD magnetron sputtering, each layer having a thickness of 20nm. The total coating was of thickness 500nm.

Results

The measured Vickers Hardness, HV, was 11.2GPa and the Young's Modulus, E, was 332GPa for penetration depths in the order of 50nm. A typical force/displacement curve is shown in Fig. 2 for a larger indentation with depth 176nm. Further investigation was possible with CSM's AST positioning system which allows a specific sample area to be located with micron precision under a high resolution scanning force microscope. This made the location of a particular imprint very easy and gave valuable additional information as to the effects of the indentation and the subsequent material response.

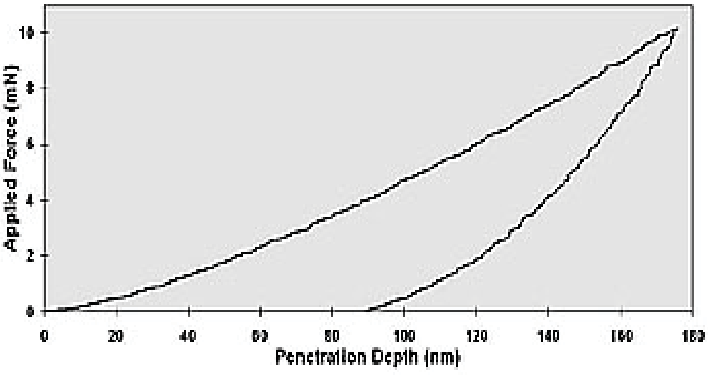


Figure 2 : Force/displacement curve for MoS2/Pb multilayer

A typical AFM image of a 10mN indentation is shown below in Fig. 3, this corresponding to the curve in Fig. 2. Although it was not possible to distinguish between each 20nm layer, the surface roughness around the imprint together with slight pile-up of material could be quantitatively measured.

Conclusion

The NHT has several advantageous features, in particular its differential measurement of the sample surface, made possible by a sapphire reference ring which allows exact relative positioning of the indentor tip. Thus the elasticity of the sample and holder is compensated, as is thermal drift during measurement. Full automation, together with optical observation make this an ideal instrument for better characterisation of thin films and coatings.

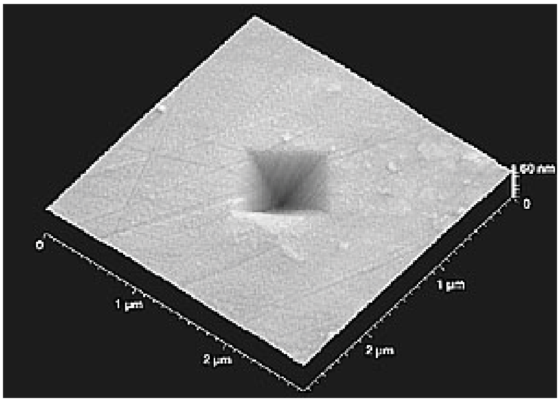


Figure 3 : AFM image of 10mN indentation on MoS2/Pb multilayer

Automobile bumper paint adhesion analysed using Micro Scratch Tester (MST)

Introduction

CSM Instruments has developed a new and unique method for testing the adhesion of a large variety of coatings on different substrates. The MST is based on the scratch adhesion test which is now widely accepted by industry for the qualitative measurement of thin film adhesion. It is rapid and simple, and allows direct comparison between similar coatings.

A scratch test on the sample induces controlled stress and strain to the surface coating via a diamond stylus from which the mechanical response can be measured by simultaneously recording friction transients, acoustic emissions and changes in surface morphology. Subsequent Raman mapping of the scratched region can provide additional information on the local stress and strain distribution, together with the molecular composition of any tribochemically produced residues.

In this study, the effectiveness of such an instrument is demonstrated for a selection of coated car bumpers kindly provided by a major car manufacturer. The objective was to compare the adhesive properties of three different bumper types having the same paint coating.

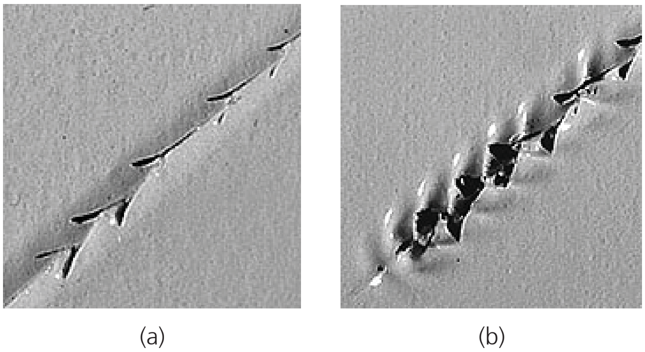
Experimental

It was found that the standard Rockwell C diamond stylus with tip radius 200mm was not sharp enough to generate good results. Thus, a hard metal tip was used which had a radius of 10m m, owing to the soft nature of the paint coating, to scratch three different samples. Raman mapping was then carried out to investigate stress and strain distributions across each scratch.

Figure 1 : Two consecutive scratches performed with maximum force 10N showing both failure modes and buckling of the paint coating.

Results

The three samples all showed two main failure modes, namely partial delamination of the paint coating (at critical force Lc 1) followed by total delamination (at critical force Lc 2). The magnitude of such critical forces gave an idea of the coating/substrate resistance to failure. For example, a poor bumper material had an Lc 1 of 1.0N and an Lc 2 of 2.7N, whereas a good bumper material had an Lc 1 of 3.4N and an Lc 2 of 6.8N for a total loading up to 10N. Two typical scratches can be seen in Fig. 1 over their full length, whereas Fig. 2 shows detail of the material response to partial and total delamination.



(a) (b)

Figure 2 : Detail of the two main failure modes seen on all three samples; (a) partial, and (b) total delamination.

With a good bumper material, Raman spectra confirmed that the stress and strain distribution was identical for the same area before and after scratching. In contrast, a bad bumper material had significant variation of stress and strain after deformation, i.e. a stress build-up in the coating/substrate system. This helped to explain the mechanisms occurring at an interfacial level. A good bumper plastic is elastic and is thus able to relax after loading and therefore prevent failure of the paint coating. The bad bumper material lacks such a capability and accumulates residual stress leading to premature failure of the paint coating.

Conclusion

This study has shown the great benefit of combining two very different, yet mutually beneficial, analytical techniques for a more complete characterisation of a coating/substrate system of major industrial importance. The Micro Scratch Tester is paving the way for a new generation of specialised testing instruments that can provide routine quality control of production-line coatings and surface treatments, as well as being used in a smaller scale research environment for the development of better materials and processes.

Relevant Publications

C. Julia-Schmutz and H. E. Hintermann; Microscratch testing to characterise the adhesion of thin layers, Surf. & Coat. Tech., 48 (1991) 1-6

D. Vaughn, B. G. Frushour and W. C. Dale; Scratch indentation, a simple adhesion test method for thin films on polymeric supports, J. Adhesion Sci. Tech., 8 (1994) 635-650

P. J. Burnett and D. S. Rickerby; The scratch adhesion test: an elastic-plastic indentation analysis, Thin Solid Films, 157 (1988) 233-254

K. Ajito, J. P. Sukamto et al.; Strain imaging analysis of Si using Raman microscopy, J. Vac. Sci. Tech. A, 13 (1995) 1234

Using CSM's Revetest (RVT) scratch-tester for Rockwell indentation

Introduction

The Revetest has already proved itself as a very reliable and popular instrument for the characterisation of hard-coated materials having a typical coating thickness exceeding 3m m. Such coatings can be organic or inorganic and include PVD, CVD, thermal, self-lubricating, decorative and magnetic, to name but a few. Substrates include metals, alloys, semiconductors, glasses, and ceramics.

This application note comes in response to a growing need amongst clients to not only perform scratch tests with their Revetest, but also have the option of carrying out indentation of their thick film for comparison and quality control purposes.

Principle

The standard stylus used for scratch testing is a Rockwell C diamond having a tip radius of 200m m. This stylus is pressed onto the sample surface with a normal force FN whilst the sample itself is displaced horizontally at constant speed. Therefore, by blocking such movement, it is possible to apply the normal force to a stationary sample and subsequently measure the diameter of the spherical imprint, from which the Rockwell hardness can be calculated. However, it should be noted that without having an indentor with a certified tip radius, only comparative hardness studies can be made, although in many industrial applications this is sufficient.

In addition, the measurement of the imprint diameter only provides the residual depth of indentation and not the true depth of indentation. If there is appreciable elastic recovery after removal of the load then there may be an appreciable difference between the residual and true depths of indentation.

The Rockwell test method is divided into the following categories, namely regular, superficial, light-load, micro and macro, depending on the load range used and the geometry of the indentor. Owing to the low-load capability of the Revetest, the superficial method has been chosen as the most appropriate, this being valid for a 15kg load as described by the ASTM test method E18.

Methodology

Having positioned the sample correctly under the indentor, the movement of the sample translation stage is blocked by switching the table selector to stop. The load range selector must be switched to the 200N position and the maximum indentation load to 150N (i.e. 15kg). A series of indentations can now be performed on the sample in question using progressive loading. The sample is then moved manually under the optical microscope (with calibrated eyepiece) to determine the imprint diameters.

For a Rockwell indentor of known radius, r, we can measure the imprint diameter, d, and thus calculate the depth of indentation, x. These parameters are shown schematically in Fig. 1, leading to the following equations:

r = x + y ....(1)

By Pythagoras, (d/2)2 + y2= r2.............(2)

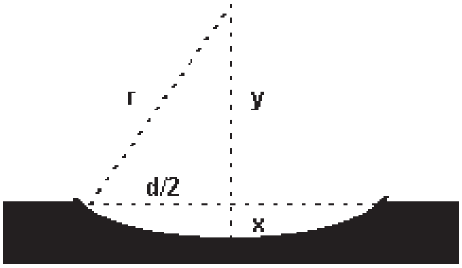


Figure 1 : Schematic representation of a spherical indentor imprint and the corresponding parameters.

The height, y, can be calculated from equation (2) and substituted into equation (1) to find the indentation depth, x. For a type C Rockwell indentor, the superficial Rockwell hardness, HC, is given by:

HC = 100 - x/2

For example, a Rockwell indentation can be performed on a Vickers test sample of known hardness and thus the Rockwell hardness can be verified via ASTM conversion tables. The indentation shown below in Fig. 2 was made in a Vickers test sample with HV of 777kg/mm2. The corresponding Rockwell hardness was found to be 91.5 using the Revetest and this value was then confirmed via such tables.

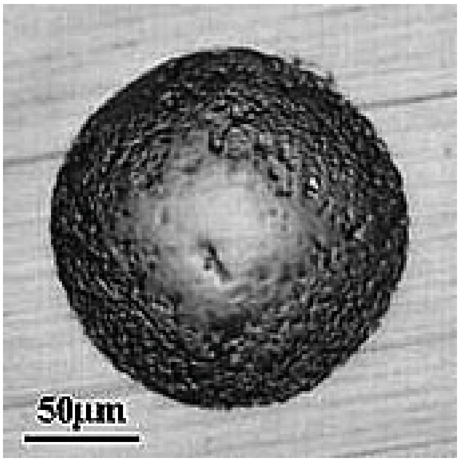


Figure 2 : Optical micrograph of a Rockwell C indentation into a standard 440C steel Vickers test sample (imprint diameter, d = 150mm).

Conclusion

The Revetest has been shown to provide accurate hardness data in addition to its scratch-testing capability, thus rendering it a far more versatile analytical tool for the characterisation of thick films and coatings. Future work will include use of this instrument for hardness determination using alternative indentor tips, e.g., Vickers, Knoop, etc, so as to offer further possibilities for such an instrument in its more common industrial environment.

Hard coating characterisation with the Revetest (RVT) scratch tester

Introduction

In parallel with the Calowear study of hard coatings, this application concentrates on characterisation of hard coatings using the Revetest scratch adhesion test. The three coatings chosen consist of TiN, TiCN and ZrCN, all of which are deposited by CVD and find use as cutting tool overcoats for minimising wear.

Methodology

The three coating types, all deposited onto hardmetal substrates and of thickness 10µm, were tested using the Revetest with progressive loading from 0 to 130N. The load rate was set at 100N/min. and the table speed at 10mm/min. Optical microscopy (integrated in the instrument) was then used to investigate the critical points along each scratch and thus determine the critical loads at which first failure (partial delamination) and total failure of the coating occurs.

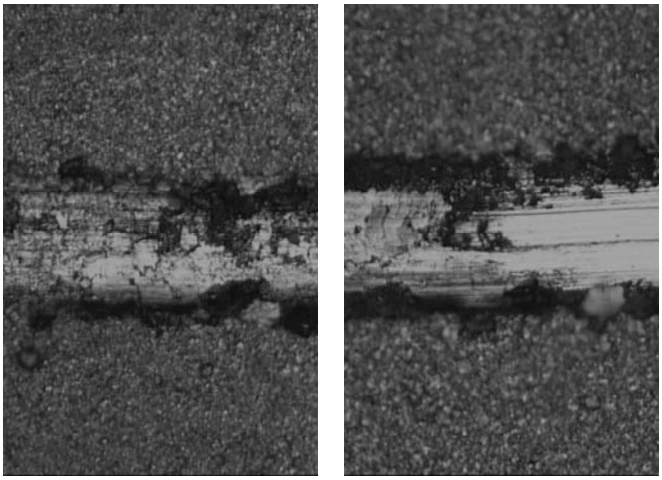


Figure 1: Optical micrographs showing partial delamination (left) and total delamination (right) for a scratch on a TiN coating deposited on a hardmetal substrate.

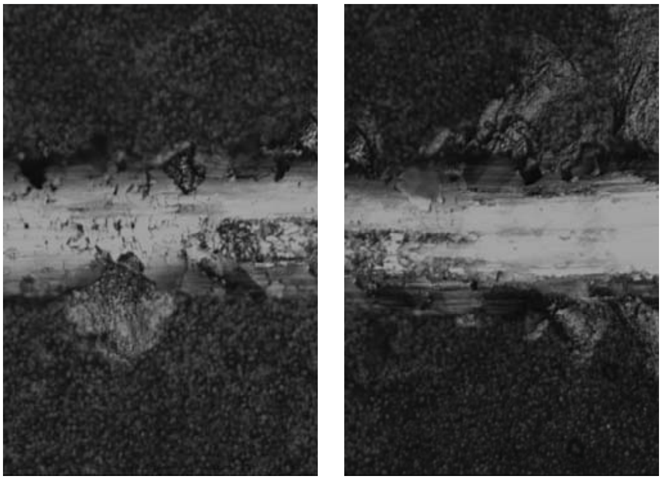


Figure 2: Optical micrographs showing partial delamination (left) and total delamination (right) for a scratch on a ZrCN coating deposited on a hardmetal substrate.

Results

The critical failure loads for the three sample types are summarised in Table 1. The TiN coating had the greatest scratch resistance, closely followed by the ZrCN, whereas the TiCN required a much lower load for total delamination to occur. Figs. 1 and 2 show the critical points for the TiN and ZrCN coatings respectively and it can be seen that although both materials have similar critical force values, their mode of failure is quite different. The TiN failure is characterised by cracking and delamination from the substrate, the ZrCN by flaking due to its more brittle nature.

Failure type	Critical load (N)		
	TiN	TiCN	ZrCN
Partial delamination	82.5	45.8	68.7
Total delamination	98.2	67.3	88.6

Table 1 : Summary of critical load values for the three tested coatings

Conclusion

The Revetest is shown to be an ideal tool for characterising hard coatings and distinguishing rapidly between different types of failure mode. This instrument has already proved itself in numerous fields, both industrial and scientific, as an efficient method of quality control and as a valid technique for development of new deposition techniques in research areas such as aerospace, tribology, electronics and optics.

Relevant Publications

(1) P. J. Burnett and D. S. Rickerby, The scratch adhesion test: an elastic-plastic indentation analysis, Thin Solid Films, 157 (1988) 233-254

(2) C. Julia-Schmutz and H. E. Hintermann, Microscratch testing to characterise the adhesion of thin layers, Surf. And Coat. Tech., 48 (1991) 1-6

CSM Instruments

This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

Editor Dr. Nick Randall

Should you require further information, then please contact:

CSM Instruments
Rue de la Gare 4
CH-2034 Peseux
Switzerland

Tel: + 41 32 557 5600
Fax: +41 32 557 5610
info@csm-instruments.com
www.csm-instruments.com

DISTRIBUTOR:

6210.11.96 CSM Instruments / Printed in Switzerland