CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

Vickers indentation using CSEM's Micro Scratch Tester (MST)

Introduction

The MST in its role as a scratch adhesion tester has already been widely used and accepted by industry for the qualitative measurement of thin film adhesion. By preventing the sample from moving, it is possible to use the MST as a conventional Vickers Hardness Tester, provided that a Vickers diamond indenter is used in place of the usual Rockwell diamond used in scratch experiments.

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

Figure 1 : Optical micrograph of a Vickers indentation into a standard steel reference sample (imprint diagonal, d = 51.2□m).

Methodology

Having installed a standard Vickers indenter on the MST (Fig. 2), the table speed should be set to zero in the Instrument Set-up menu. For indentations conforming to ASTM test method standard E48, the maximum force should be set to 10N in the same menu and loading must be specified as progressive.

After performing an indentation, the sample can be moved automatically under the integrated optical microscope in order to determine the imprint diagonal, from which the Vickers hardness can be acquired from appropriate tables. The length of the diagonal can easily be found by consecutively positioning the crosshair reticule at opposite corners of the imprint and reading the distance between them directly from the software.

Figure 2 : Close-up of standard Vickers indenter on the Micro Scratch Tester.

Conclusion

The indentation shown in Fig. 1 was carried out on a Vickers test sample giving a Vickers hardness of 707HV for a normal load of 10N.

The MST 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 thin films and coatings.

Notes

> For coated systems, the measured Vickers hardness value may vary if the indentation depth exceeds one tenth of the coating thickness, due to substrate effects.

> Scratching experiments should NOT be carried out with a Vickers indenter as severe damage to the tip will be incurred!

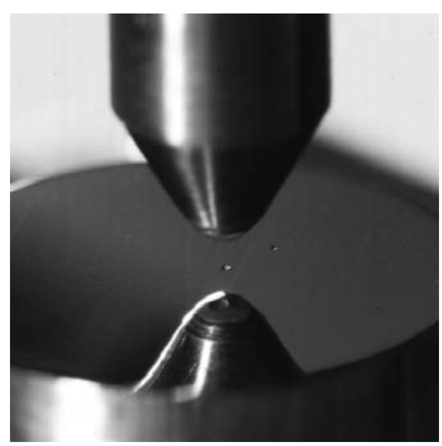

Figure 3 : Detail of Vickers indenter mounted above sample.

Abrasion testing of hard coatings with the Calowear Tester

Introduction

This study focusses on thin hard coatings dedicated to cutting tool protection. Although the most widely used coating for such an application is still TiN, new deposition techniques such as PA-PVD have introduced coatings with a broad range of mechanical properties. It is therefore essential to have an adequate method of testing such properties which is both simple and quick.

The Calowear Tester, whose principle is fully described in the autumn edition of this bulletin, provides a quantitative method for determining the abrasion resistance of both the coating and its substrate, such a parameter being of paramount importance in cutting tool applications.

Methodology

The theoretical background has been described elsewhere [1] and is based on a simple model for abrasive wear, equivalent to the Archard equation and assuming independant wear coefficients for the coating and the substrate. The equation is rearranged in order to get a linear plot of wear measurements made at different sliding distances. The abrasion coefficients of the substrate and coating are then extracted from the slope and intercept of the plot.

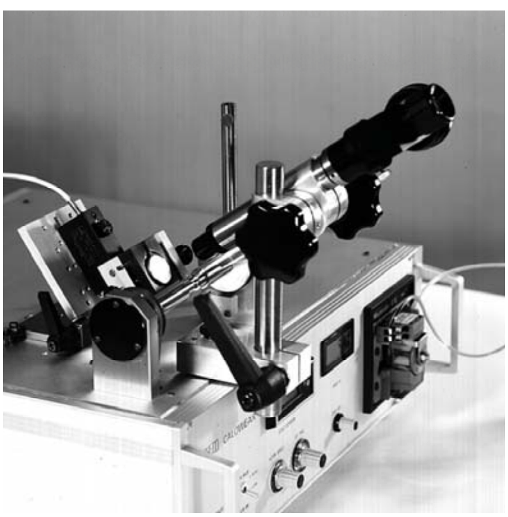

Figure 1 : The Calowear Tester, which provides an integrated optical microscope, normal force measurement, rev. counter and abrasive slurry mixer.

The five coatings tested in this study were all produced by Physical Vapour Deposition (PVD) and consisted of TiN, TiCN, TiAlN, CrN and DLC deposited onto 440C steel and 100Cr6 substrates. An AISI 52100 steel ball was used with a diameter of 25.4mm and the abrasive slurry was a suspension of SiC particles with a mean size of 3µm (particle concentration = 750 g/l).

Results

Fig. 2 shows the abrasion rate of each coating on each substrate, whereas Fig. 3 shows the abrasion rate of each substrate. Both sets of data were determined from the same series of measurements. An interesting observation is that the DLC had a much smaller abrasion resistance than the titanium or chromium based coatings. TiCN is the tested coating having the lowest wear rate, this corresponding well to its superior performance in service. CrN had an average abrasion resistance, better than DLC but worse than the titanium based coatings. However, CrN has other practical advantages, namely its ability to deform with its substrate without cracking.

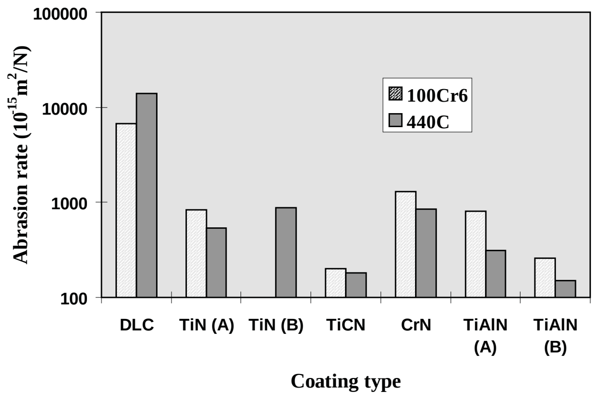

Figure 2 : Abrasion rates of various hard coatings.

Overall it can be seen that the abrasion rates obtained for 440C substrates are generally smaller than those on 100Cr6 substrates. Although the applied methodology is supposed to decouple the effects of the coating from those of the base material, it seems that further investigation is needed, especially regarding the influence of the substrate hardness which can change due to the deposition process.

Conclusions

The results presented confirm the ability of the Calowear Tester to rapidly distinguish between different types of coatings and depositions, making it an ideal instrument for quality control and materials research. Further studies still need to be carried out in order to investigate, for example, the role of the abrasive particle hardness and thus to optimise the Calowear process for each class of tested materials.

References

(1) K. L. Rutherford and I. M. Hutchings, A micro-abrasive wear test with particular application to coated systems, Surface and Coatings Technology, 79 (1996) 231-239

(2) K. L. Rutherford and I. M. Hutchings, Micro-scale abrasive wear testing of PVD coatings on curved substrates, Tribology Letters, 2 (1996) 1-11

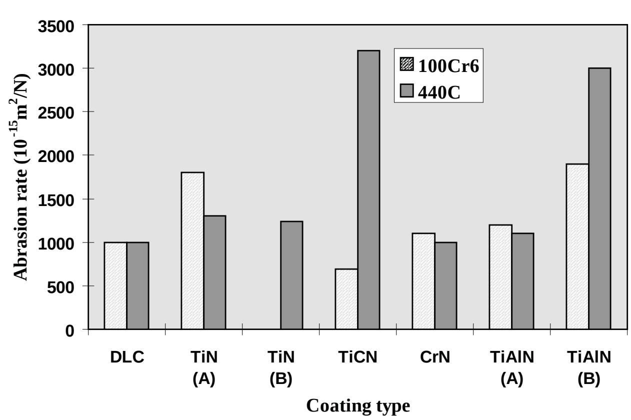

Figure 3 : Abrasion rates of the substrates.

Quality control of IC bonding pads with the Nano Hardness Tester (NHT)

Introduction

In todays integrated circuit (IC) industry, the main cost consideration is the time-to-market and to this end adequate quality control is fundamental during the development phase, allowing design and process engineers to evaluate the functionality of a new device before it reaches the final testing stage.

IC bonding pads are of paramount importance in serving two functions, the first of which is to provide a defined contact with a test probe in order to test the continuity and basic functionality of a device, the second to provide a platform onto which connecting wires can be attached by thermosonic bonding. The latter is used to establish electrical contacts from a chip to the outer leads via very fine gold wires having a diameter down to 20µm.

In order to acertain whether a bonding pad is satisfactory or not requires a quantitative measurement of its Vickers hardness. The pad itself is produced by sputtering Aluminium onto the SiO_2 substrate creating a film of approximately 1µm thickness. Insufficient hardness of this film results in deep scrub marks (during probe testing) which then prevent a good bonding between the pad and the gold connecting wire. In addition, if the pad is too soft then substantial debris may be produced when the probe tip comes into contact with it, this being a very important consideration in such a particle sensitive environment.

Methodology

For this comparative study a selection of four wafers were chosen from different manufacturers and the Vickers hardness of each measured using the Nano Hardness Tester (NHT). Owing to the 1µm thickness of the Aluminium film, all indentations were performed with a maximum depth of 100nm in order not to have any substrate effects.

Fig. 1 shows one of the bonding pads after having made a series of eight nanoindentations. The scrub mark is also clearly visible. With the NHT's accurate positioning stages it was possible to make indentations within specific grains (Fig. 3) and thus prevent hardness variations due to grain boundary effects.

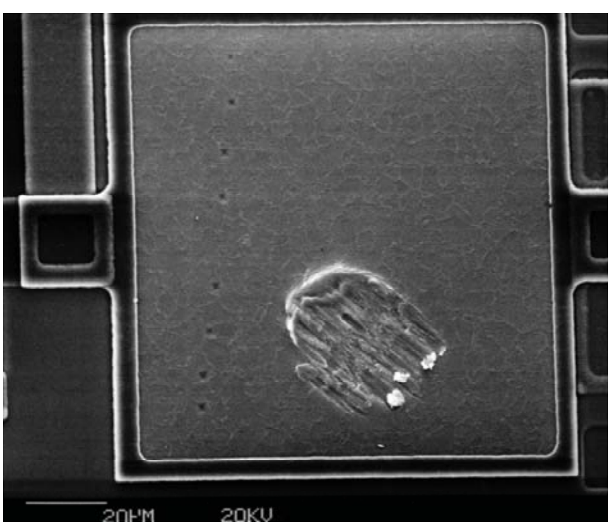

Figure 1 :SEM micrograph of one of the measured bonding pads showing the scrub mark (right) and a line of nanoindentations (left).

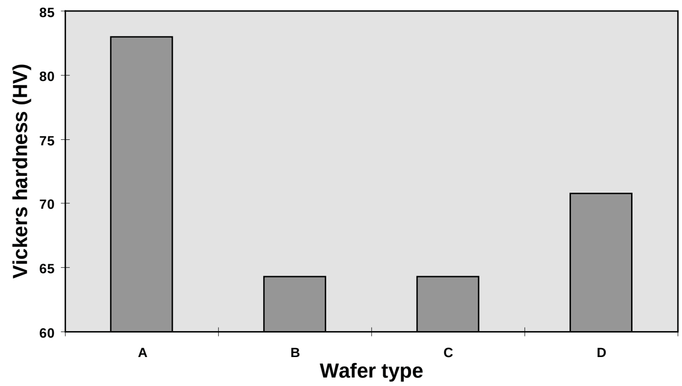

Figure 2 : Vickers hardness of bonding pads from four different manufacturers.

Results

The Vickers hardness of each bonding pad type is tabulated in Fig. 2, each value being averaged from several measurements. It can be seen that type A has the highest hardness and can therefore be considered as being 'good', whereas types B and C have much lower hardness and so are considered as 'bad'. Type D has an intermediate hardness value.

Conclusion

The NHT is a powerful analytical tool which is ideally suited to industrial quality control applications where short measurement times and automated operation are particularly important. In the case of IC bonding pads it has been shown that such a method is easily able to distinguish between different pad hardnesses and thus allow the choice of manufacturer to be made at an early stage in the development phase.

Localised measurements at specific sites make this instrument an attractive solution to many other IC and other related industrial problems. However, the instrument is also of great benefit in a smaller scale research environment for the development of better materials and processes.

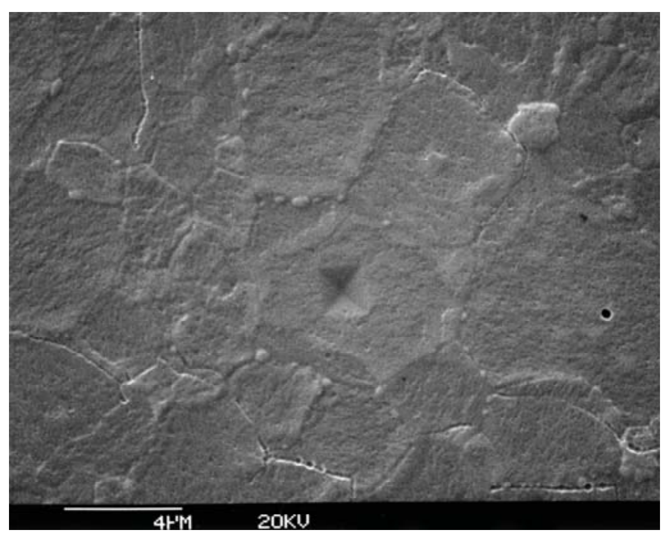

Figure 3 :SEM micrograph showing the sputtered Aluminium grain structure and the ability of the NHT to make a highly localised indentation within a specified grain.

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.

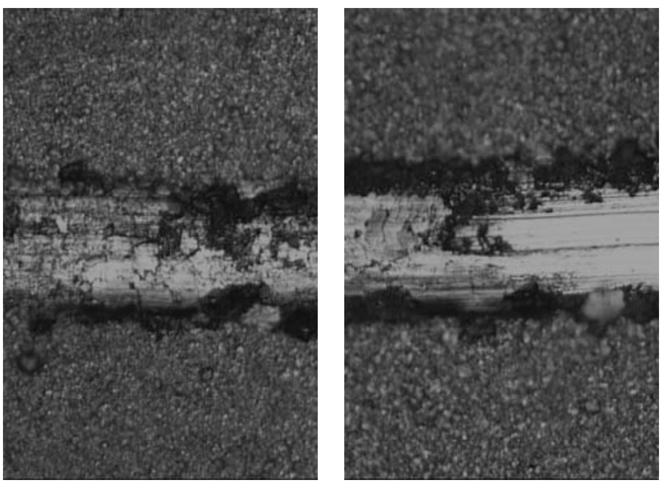

Figure 1: Optical micrographs showing partial delamination (left) and total delamination (right) for a scratch on a TiN coating deposited on a hardmetal substrate.

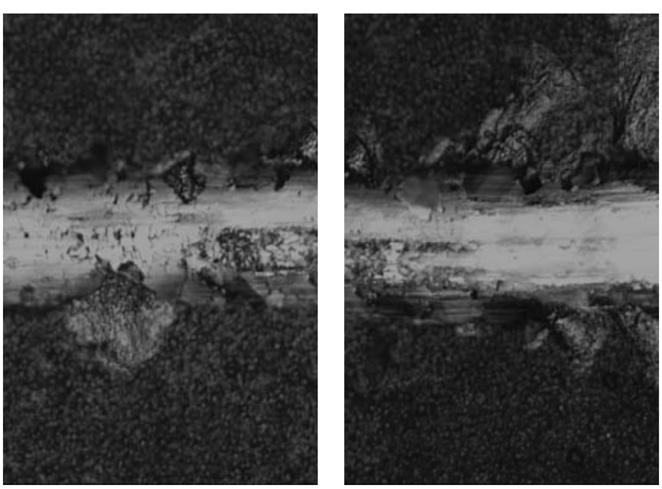

Figure 2: Optical micrographs showing partial delamination (left) and total delamination (right) for a scratch on a ZrCN coating deposited on a hardmetal substrate.

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

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

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