CSM Instruments
Advanced Mechanical Surface Testing

Instruments

APPLICATIONS BULLETIN

Micro Scratch Tester (MST) for assessing the scratch resistance of SiO2 passivation layers

Introduction

Many commonly used metals and alloys, such as iron, titanium, and nickel, form passive films when exposed to oxidising environments. These passive films, which are often considered to be stable oxides, are exploited for their ability to significantly reduce corrosion and oxidation. Electrochemical testing of these materials and their various alloys is now common practice for assessing the corrosion resistance of a particular metal in a given environment. However, the mechanical properties of these passive films and the material on which the film has formed have not been thoroughly explored. This is partly due to the small scale of the films, which are generally oxide layers with thicknesses on the nanometer scale. With the silicon family of materials, passivation layers and their performance in service are a topic of increasing importance in todays' microelectronics and micromachining industries. This application note focuses on assessment of the scratch resistance of SiO2, SiN and SiC passivation layers. These types of coatings are ideal examples owing to their distinct and reproducible critical failure points.

Applications

The results presented in Fig. 1 summarise the four distinct failure points encountered when a SiN/SiC passivation layer is scratched with a progressive load range of 0 - 15 N and a diamond tip of radius 20 µm. The thickness of the coating is approximately 400 nm. The first critical force value of 3.26 N corresponds to damage along the sides of the scratch path. At 9.77 N first cracking occurs, after which continuous flaking occurs at 10.60 N. Shortly afterwards, the coating is seen to completely delaminate at a critical force of 11.74 N, causing substantial debris to be strewn around the scratch path (see Fig. 1 (d)).

In addition to scratch testing, passivation layers may also be characterised by indentation testing with a conducting tip [1] or by potentiostatic control of the sample [2]. The tip is loaded and the electrical resistance measured between the tip and the sample. For many systems, particularly metals, a sudden drop in resistance can be attributed to the breaking of the oxide film and the resultant contact between the two conductors. Future work is hoped in adapting potentiostatic control to scratch testing. SGS Thomson Microelectronics R &D Center in Carrollton, Texas is acknowledged for providing the samples featured in this application note.

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2. D. F. Bahr, J. C. Nelson, N. I. Tymiak and W. W. Gerberich, J. Mater. Res., 12 (1997) 3345-3353

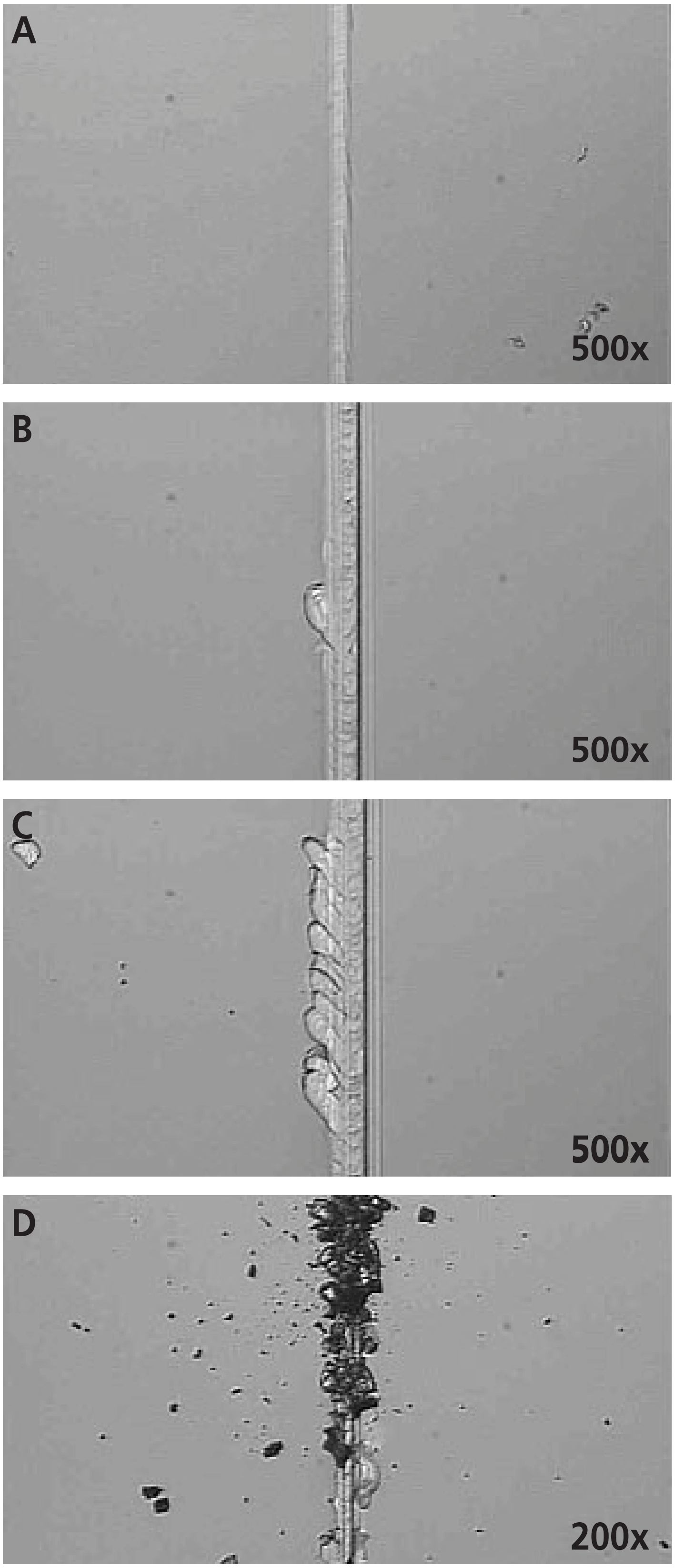

Figure 1 :Progressive load scratch data for an SiN/SiC composite layer which exhibits four distinct and reproducible critical failure points, namely (a) first damage, (b) first cracking, (c) continuous cracking and (d) full delamination.

No. 6 January 1998

High-Vacuum Tribometer for MoS2 alloy characterisation

Introduction

Although more expensive than oils and greases, solid film lubricants are a rapidly growing field despite their specialist requirements in mechanical design and coating application. In certain contamination-sensitive industries, such as food processing or textile manufacture, solid lubrication is often the only alternative available.

The most common solid lubricants include MoS2, graphite, PTFE and DLC, even though their friction and wear mechanisms remain complex and not fully understood. Recent improvements in coating technologies, especially plasma-assisted physical vapour deposition techniques (PAPVD), have enabled MoS2 to be successfully deposited for a wide range of uses. Historically, the most important of these is probably space applications, i.e., under vacuum conditions, where wide operating temperatures prevent the use of oils whose viscosity can vary greatly. In industrial applications, the lifetime of an MoS2 film can be improved by adding other materials which preferentially oxidise (e.g., Ni or Pb), by increasing the grain size and by reducing film porosity in order to prevent substrate corrosion.

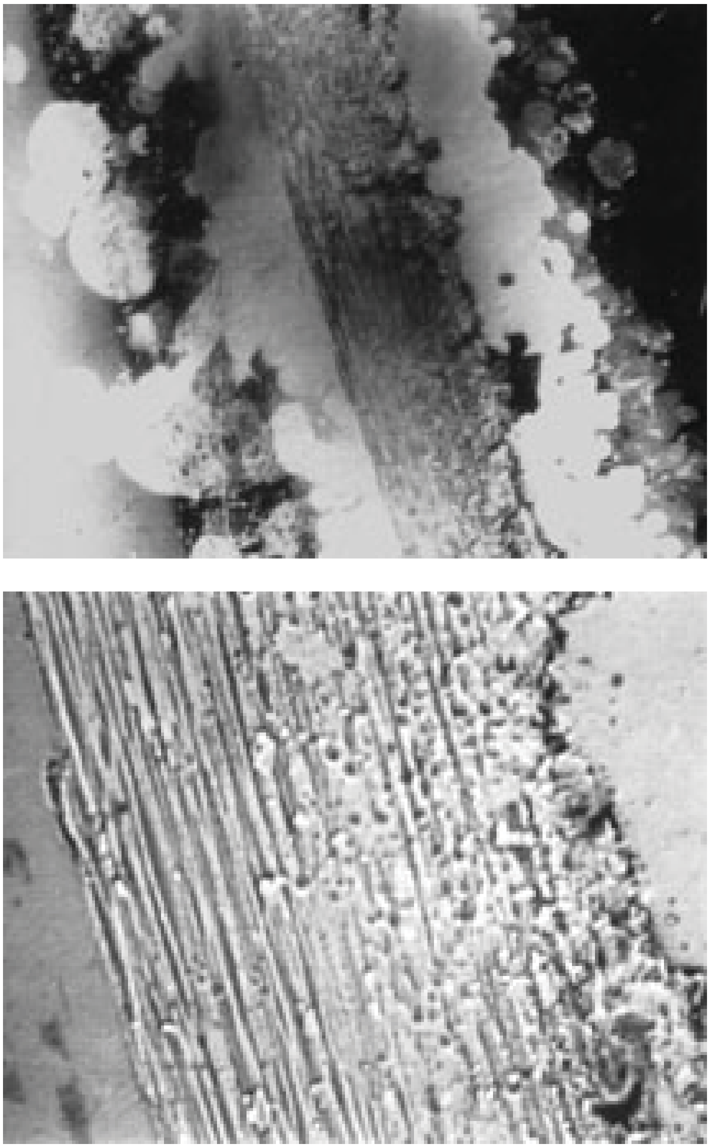

Figure 1 : Optical micrographs of wear track produced on an MoS2-coated bearing race tested to failure under vacuum conditions.

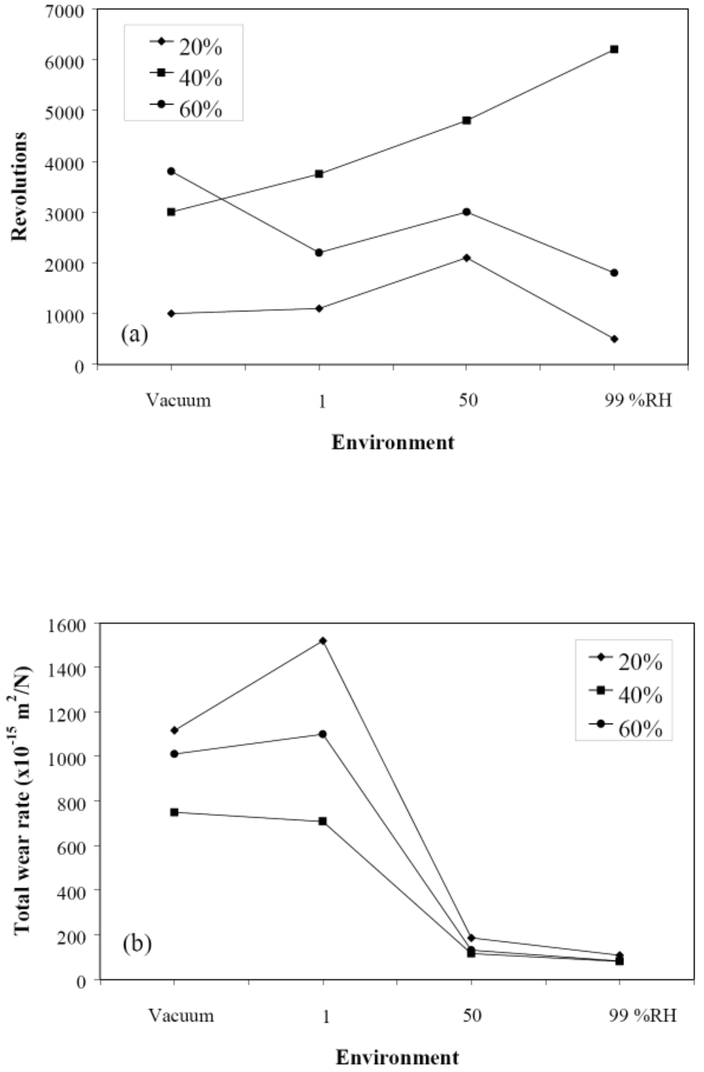

Figure 2 : Comparison between revolutions (up to m £ 0.3) (a) and total wear rate (b) of a Ni-Cu-P alloy containing different concentrations of MoS2, for various operating environments.

Application

Some typical examples of tests performed using the CSM high-vacuum tribometer are presented in Figs. 1 and 2. This instrument has the advantage of allowing a very wide range of possible test conditions, i.e., from a high vacuum to a completely humid environment. The results shown in Fig. 2 compare the effect of different MoS2 concentrations on the total wear rate and the number of revolutions required to cause coating failure. Additions of MoS2 are often used as particles in a hard film matrix such as Ni-Cu-P, this composite-type coating providing both a wear-resistant surface and some solid lubrication, with new pockets of lubricant being exposed as the film wears down.

Dr. P. Voumard of the CSM Tribo Coatings group is acknowledged for providing the results shown in Fig. 2.

Combined NHT/SFM for characterisation of Silicon materials

Introduction

The silicon family of materials are particularly interesting samples when studied via nanoindentation techniques. Pure silicon exhibits a rather unique behaviour when indented with a sharp indenter, this being characterised by a distinct discontinuity or pop-in during unloading. Such a phenomenon is observed in each of the [100], [110] and [111] orientations and has been widely published [1-2]. The load below which the pop-in disappears is generally in the range 5-20 mN and the hysteresis is thought to be due to a pressure-induced phase transformation from the normal diamond cubic form to a denser b -tin structure.

Application

Recent work [3] has shed some light on the processes occurring in and around an indented silicon surface. This has been achieved using the Nano Hardness Tester (NHT) combined with the Scanning Force Microscope (SFM) objective. The high resolution imaging capability of the latter can provide significant additional information about the indentation process and can be correlated to the conventional load-displacement data obtained.

Fig. 1 shows two sets of Vickers indentation data for p-type silicon under different loading conditions. Fig. 1 (a) shows the radial cracking which occurs at high loads (generally > 200 mN), whereas Fig. 1 (b) shows the drastic effects of increasing the load in 75 mN steps over four load-unload cycles. Such multicycle indentation causes substantial uplift of material around the residual imprint as a result of lateral cracking below the indenter, in addition to the radial cracks propagating away from the corners of the imprint. In contrast to normal metals, the energy absorbed by silicon is significant on each cycle and obviously contributes actively to the observed cracking; for example, in Fig. 1 the maximum load in (b) is 50 mN less than that attained in (a) but the SFM images show greater cracking in the former than in the latter.

Clearly, such cracking phenomena cannot be seen with conventional optical microscopy, so the SFM is an important addition to a nanoindentation instrument if the true response of the material to one or several load-displacement cycles is to be directly quantified. It is also capable of calculating the residual contact area, from which the true hardness value can be verified.

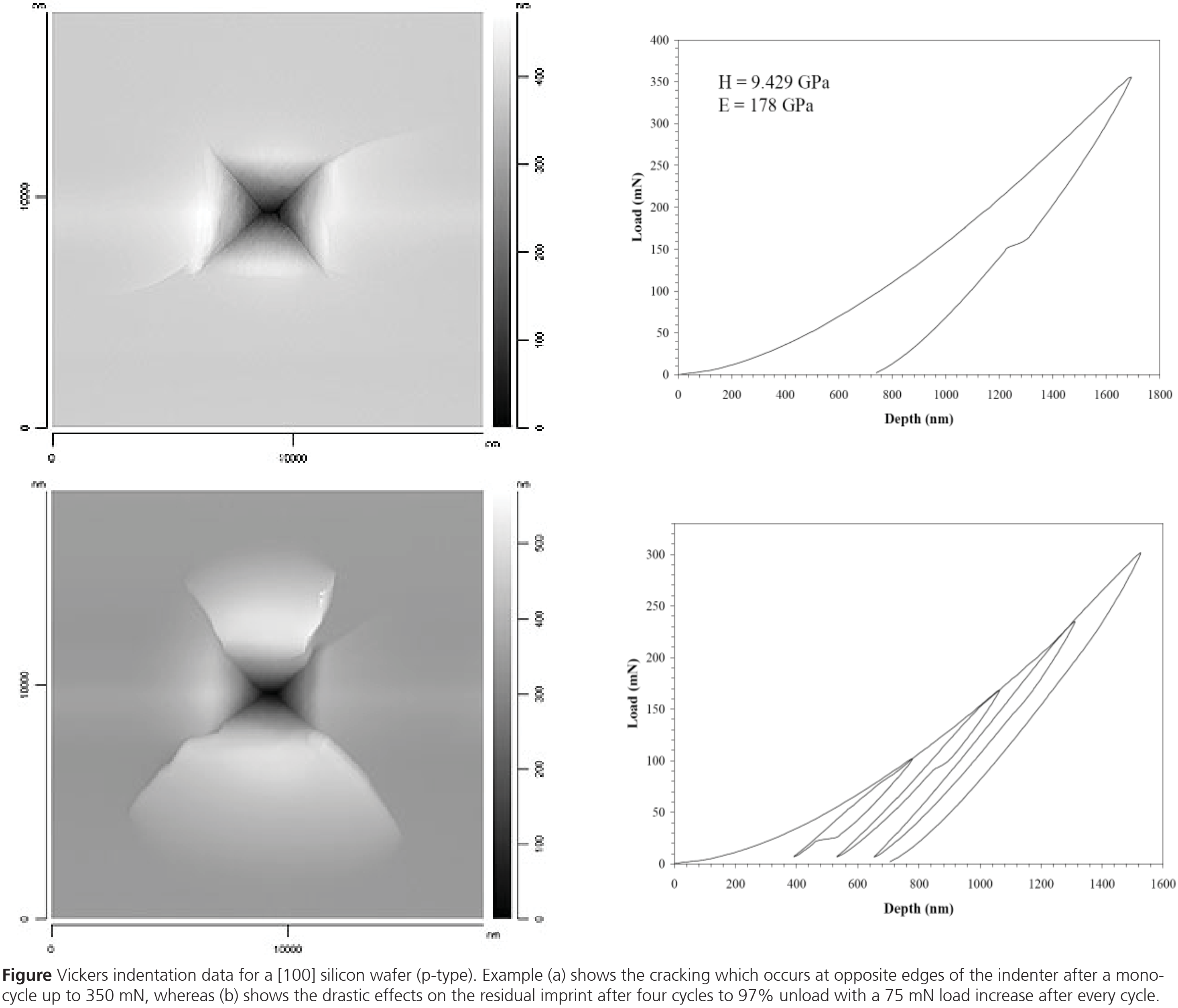

Figure Vickers indentation data for a [100] silicon wafer (p-type). Example (a) shows the cracking which occurs at opposite edges of the indenter after a mono-cycle up to 350 mN, whereas (b) shows the drastic effects on the residual imprint after four cycles to 97% unload with a 75 mN load increase after every cycle.

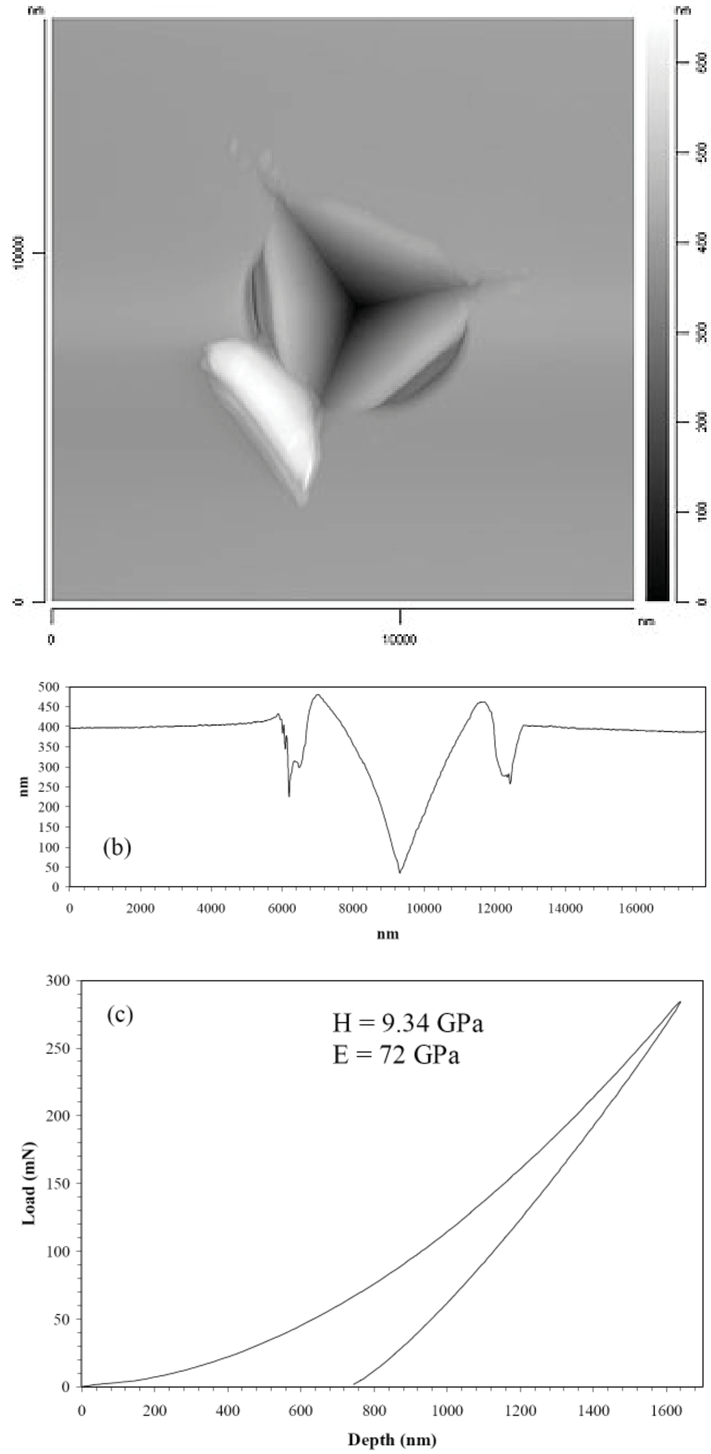

Figure 2 : Berkovich indentation data for fused silica showing the cracking effect at high loads (i.e., 300 mN).

Fused silica is a member of the silicon family which exhibits a very large elastic recovery during unloading, as depicted in the load-displacement curve of Fig. 2 (c). For relatively low loads (not shown) the residual imprint has quite an unusual appearance suggesting that the sides of the indentation are elastically recovered during unloading, whilst the corners are not. With a Berkovich indenter, additional plasticity may well be caused by the stress concentration at its edges, meaning that the indenter is able to permanently mark the position of the corners of the indentation at maximum load.

If the applied load is increased, for example to 300 mN as shown in Fig. 2, the load-displacement curve has the same shape and the calculated hardness and modulus remain the same as for a low load indentation. However, the SFM image and corresponding cross-sectional profile show that the residual imprint is quite different at high applied loads; subsurface median and lateral cracking causes uplift of the sides of the imprint and a large flake of material is also visible at one corner of the indentation. This characteristic material response is highly reproducible and such large flakes of uplifted material are always found over a corner, indicating that lateral crack propagation is obviously higher at zones where stress concentrations are higher.

Conclusion

This study has focussed on indentation into Si and SiO2 (fused silica), both such materials being of great importance to the semiconductor and microfabrication industries. A better knowledge of the inherent deformation modes, the response to localised compression and the ability of the material to dissipate its absorbed energy, whether via cracking or by phase transformation, are all topics which require further research.

Although both Vickers and Berkovich indenter geometries have been utilised, the additional possibility of using accurately-ground spherical-tipped indenters will allow a more global characterisation of such materials, especially in terms of their stress distribution characteristics.

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