

Combined opto-mechanical measurements with the transparent indenter's tip

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Abstract. Transparent indenter's tip proposed elsewhere allows to observe sample surface before, after and during an indentation measurements. Such an ability allows not only to select the indentation with the real-time optic image and observe residual imprints, but also conduct spectroscopic measurements. Current work shows an examples of the tip application, in particular the possibility to observe a cracks during a scratching.

1. Introduction

Instrumented indentation technique allows to measure surface local values of hardness and local elastic modulus from the load-depth data without the need to use an optical microscope. The method has been used for several decades. Modern equipment allows to combine this method with high-temperature measurements [1], conduct an experiments within electron microscopes [2], detect acoustic emission [3], use an indenters along with another analytical equipment, in particular combine raman spectroscopy with an indentation. However, in most of the work the collection of raman spectra were performed only during the indentation of a transparent spectra in the location close to the sample edge, which allowed to perform indentation and spectroscopy measurements in the perpendicular directions. The transparent indenter – objective suggested elsewhere [6, 7] allows to overcome the limitation.

2. Indenter – objective scheme

Detailed description of the construction was given in the work [6]. Indenter-objective is made out of the diamond cylinder that has both ends faceted in the shape of the Berkovich pyramid in the way that each face on one end has a parallel face on the opposite end. For the Berkovich-shape pyramid this parallelity constrain means that the pyramids on the ends are rotated 60 degrees versus each other (Fig.1). This way faceted indenter has the following property. Parallel light beam incident along the indenters axis undergoes double refraction and maintains its parallelity after passing through the described tip. However, propagated beam is split into three sectors which are lineary shifted vs their original line of propagation. The visible through the indenter picture also split into three parts. An example of the testing structure TGZ-2 surface reconstructed image is shown on fig.2.

Analogous measurements of the sample surface can be conducted not only during the indenter's positioning but as well as during an indentation and after it. That gives a possibility to measure



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indentation contact area, which in turn allows to determine hardness directly according to the residual imprint, without need to perform additional movement beneath an optical microscope.

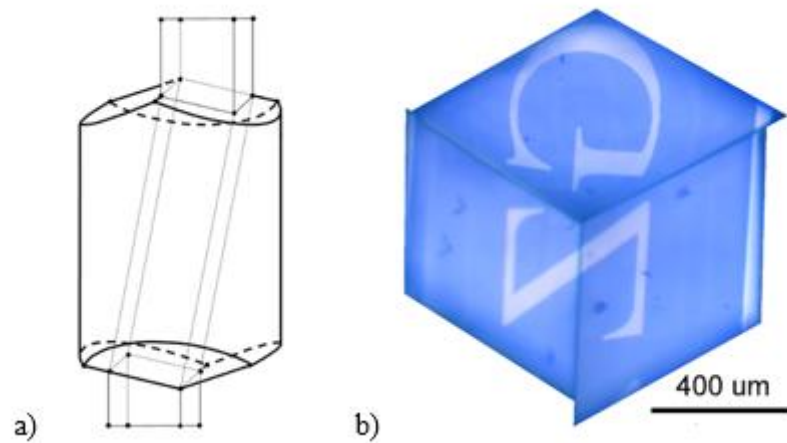


Figure 1. a) Transparent indenter-objective and beam optical path b) reconstructed image of the TGZ testing grating surface

Another example of the transparent indenter-objective application is an imaging of the surface during the scratch testing. Figure 2 shows an example of picture taken during the scratching made over the explosion-weld metals border. This case corresponds to the plastic surface deformation and scratch width can be used to calculate sample's hardness.

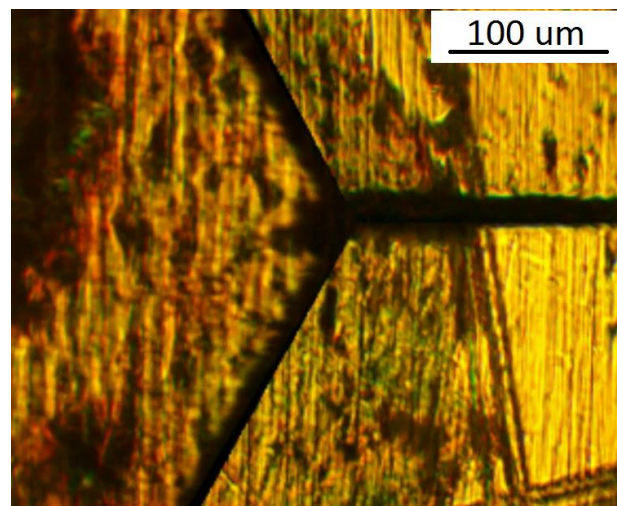


Figure 2. Image of explosion welded metals boarder taken during the scratching with a 300 mN load.

Scratching on the fused silica can show a different situation. At small load scratch appears to be mostly plastic, but after the load reaches a critical value cracks reach the surface so that the surface “explodes”. Figure 3 shows corresponding set of pictures that were taken during the scratching of fused silica surface with an increasing load from 1mN up to 1N. As it can be seen from the figure 2 a) when load is relatively low on can observe plastic deformation on the sample's surface, as the load increases crack reaches the surface scratch explodes and further deformation, which is shown on the figure 2c, occurs in a brittle manner.

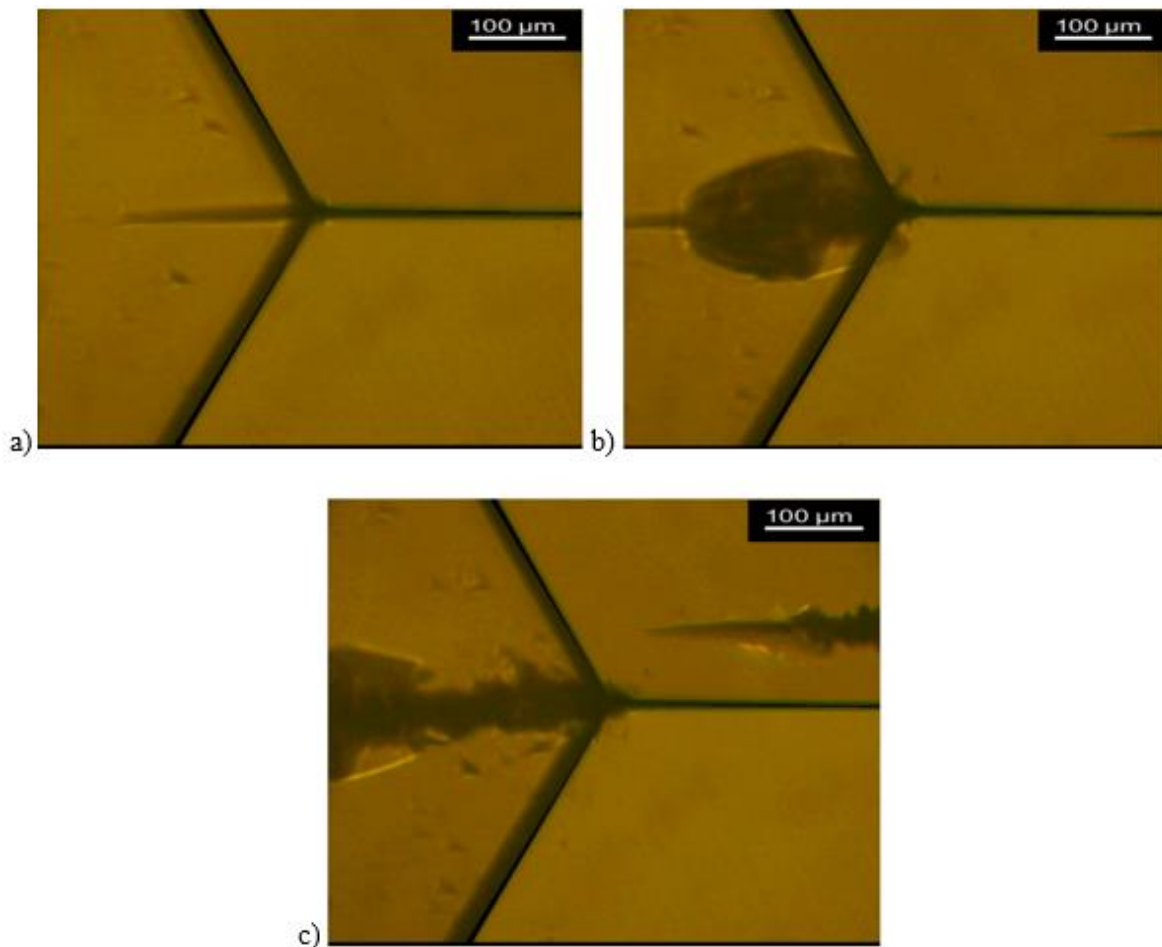


Figure 3. (a) plastic deformation of fused silica surface (b) fused silica surface at the moment of cracking

Such kind of in-situ surface optical measurements can be particularly useful for the adhesion measurements. In these measurements the determination of coating delamination moment plays an important role as it defines the critical load, which is used for further adhesion energy evaluation [8].

3. Raman spectroscopy during indentation measurements

An ability to obtain optical images during indentation directly through the tip leads to possibility to combine mechanical testing with optical spectroscopy measurements, in particular obtain Raman spectra simultaneously during an indentation [7]. Corresponding measurements were performed with the help of the special module designed to fit in the Raman spectrometer. The module allows applying and measure the load up to the force of 50 N. Measurements were performed on silicon sample's (100) surface. Two spectra collected on free and 50N loaded surfaces are shown on the figure 4.

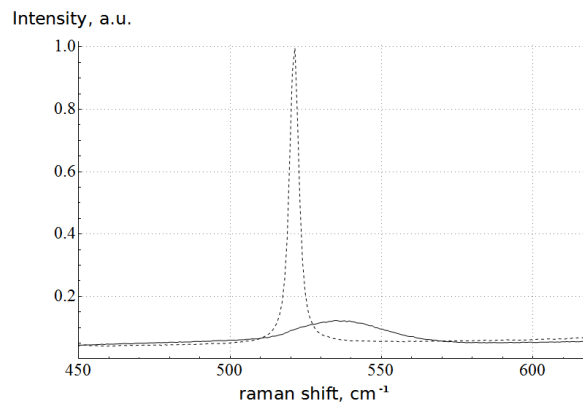


Figure 4. Shift of Si-I line under the load of 50N.

As it can be seen from the figure 3, when the load is applied Si-I line is shifted right to 20 cm⁻¹. 50N spectra also have a peak at 376 cm⁻¹, which are transform to series of peaks corresponding to the Si-III phase.

4. Conclusions

Transparent indenter-objective allows to obtain optical image of the surface, define deformed area surface during an indentation and afterwards. The indenter allows to conduct simultaneous indentation measurements and collect Raman spectra of the indented surface during its loading and unloading cycle.

Acknowledgments

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