Application of nanoindentation technique in microelectronics

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Abstract: *Nanoindentation technique is widely used in microelectronics in order to define mechanical properties especially in reference to thin layers. In the above paper some numerical modeling and experimental results along with the extracted elastic and/or elasto-plastic mechanical properties are presented in reference to selected microelectronic materials as e.g. fused silica and aluminium.*

1. Introduction

Indentation technique was for the first time introduced in XIX century. Primarily, indentation technique was used to measure material hardness *H*. In history the indentation was done by indenters of various shapes thus leading to a variety of tests and analytical definitions of the hardness value. Currently, the indentation technique was enhanced in order to assess additionally basic mechanical properties, which was elastic modulus *E* of the investigated material. The above indentation method was introduced in the year 1992 and was named as nanoindentation [1].

2. Indentation vs. Nanoindentation Technique

2.1. Indentation

Indentation technique is used to measure an important material property, which is hardness *H*. Hardness in traditional sense is defined as material resistance to deformation.

$$H=\frac{F}{A} \tag{1}$$

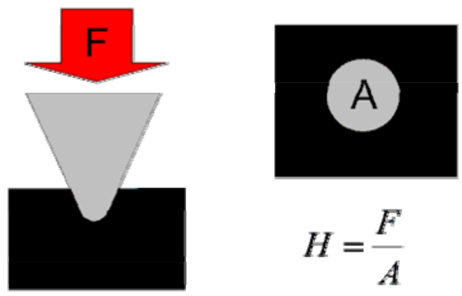

Fig.1. The indentation technique.

Generally hardness is measured by indenting the material surface with a known force *F* using an indenter of a selected shape. The left indentation print of the indenter on the material surface is used as a measure of hardness. In history the material hardness was usually measured by loading an indenter of specified geometry onto the material and measuring the dimensions of the resulting indentation. The measurement was done by indenters of various shapes thus leading to variety of tests and analytical definition of hardness value. The most important indentation tests are as follows:

- Brinell indentation test was proposed in 1900 as a first indentation standard hardness assessment method. It is done by a spherical indenter, typically of diameter $D=10$ mm.
- Vickers indentation test was developed in 1920. It is done by a diamond pyramidal shape indenter

- Rockwell indentation test in comparison to the above tests is based on comparing the penetration depth in reference to selected material, defined as reference material. Indentation can be done either by cone or spherical indenter shape
- Knoop indentation test is suitable to measure hardness at micro-scale. It is done by diamond ground pyramidal indenter shape, which has a ratio between long and short diagonal about *7:1*
- Berkovich indentation test is suitable for hardness measuring at micro and nano-scale. It is done with a diamond three sided pyramidal shape indenter. Berkovich test is commonly used in nanoindentation technique in order to evaluate elastic and elasto-plastic material properties

2.2. Nanoindentation

Currently the indentation technique was enhanced in order to address problems referring to nano-scale. The above technique is referred to as nanoindentation [2]. It is an improvement of the traditional indentation technique and in comparison to it allows assessing the Young's modulus of the indenting material. It requires very precise tip shape, e.g. Berkovich tip and therefore assures high spatial resolutions (Fig.2). It is based on real time load-displacement analysis during indentation, which leads to loading and unloading part as shown in Figure 3 [3].

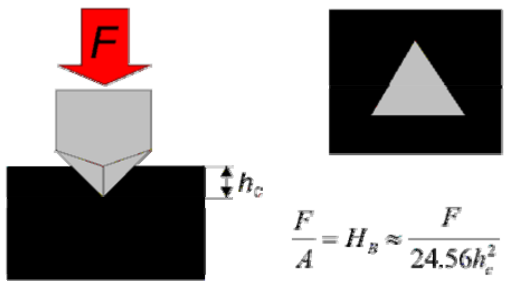

Fig.2. The Berkovich test.

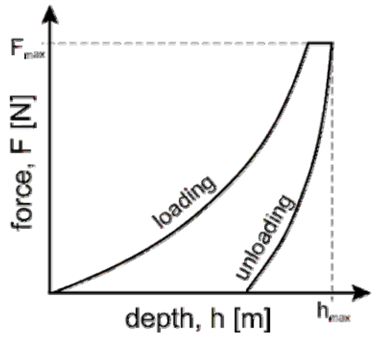

Fig.3. The shape of loading and unloading curve.

Taking into account the unloading nanoindentation curve it is possible to define and estimate the following elastic material properties [4]:

- stiffness S

$$S = \frac{dF}{dh}\bigg|_{F=F_{max}} \quad (2)$$

where F_{max} is a maximum force during loading and unloading indentation process.

- hardness H

$$H = \frac{F_{max}}{A_c} \quad (3)$$

where A_c is the contact area

- effective modulus of elasticity E_{eff}

$$E_{eff} = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \quad (4)$$

where parameter E_{eff} relates to deformation of both materials: specimen and indenter.

3. RESULTS

A large number of materials applied in microelectronic packaging can be precisely described by elastic or elasto-plastic material models. The typical stress-strain behaviour for elastic and elasto-plastic material models are presented in Figure 4 and 5.

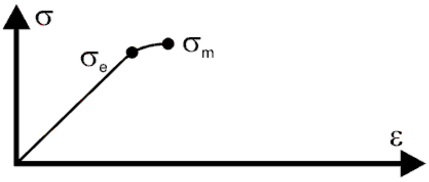

Fig.4. The stress-strain curve for elastic model.

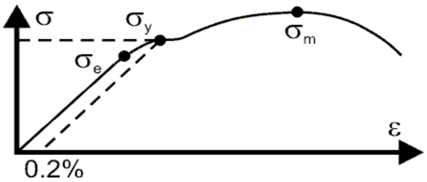

Fig.5. The stress-strain curve for elasto-plastic model.

The presented research activities focused on extraction of elastic and elasto-plastic material model and corresponding material properties. In fact the above can be achieved by:

- analytical analysis, which uses the strict mathematical dependencies in order to assess the basic elastic material properties
- numerical modelling, which uses finite element modelling (FEM) of the indentation experiment in order to predict the indentation loading and unloading curve

3.1. Experimental Results

The nanoindentation experiment of loading and unloading curve was done for fused silica and aluminium. One of the goals was to recognize the dependence of extracted material properties on indentation depth and the same indentation force value. The selected measurement results are given in Figure 6 and 7.

Fig.6. Selected experimental results for fused silica

Fig.7. Selected experimental results for aluminum

3.2. Analytical Analysis

Pure elastic material model behaviour is "located" at the vertex of the unloading part. According to the current knowledge concerning nanoindentation technique, it was recognized that the unloading part can be approximated by the power law formula, given by:

$$F = A_0 \left(h - h_f\right)^m \quad (5)$$

The main problem is that the real surface profile is not equivalent to indenter shape due to pile-up and sink-in phenomena. Therefore certain assumptions are to be taken, which lead to the following final set of analytical equations [5]:

- stiffness S:

$$S = \frac{df}{dh} = A_0 m \left(h - h_f\right)^{m-1} \quad (6)$$

- contact depth h_c:

$$h_c(S) = h_{max} - \frac{2}{\pi}(\pi - 2)\frac{F_{max}}{S} \approx h_{max} - \varepsilon \frac{F_{max}}{S} \quad (7)$$

where ε can change in the range from *0.72* up to *1.0* depending on the indenter shape.

- contact area A_c can be evaluated either by simplified or advanced equation. The simplified equation is given by:

$$A_c(h_c) \approx 24.56 h_c^2 \quad (8)$$

while the advanced equation is based on the so-called area function and is given by:

$$A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16} \quad (9)$$

- hardness H:

$$H(A_c) = \frac{F_{max}}{A_c} \quad (10)$$

- effective elastic modulus E_{eff}:

$$E_{eff}(S, A_c) = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} \quad (11)$$

In case of analytical extraction of elastic properties through nanoindentation data it is possible to assess roughly the yield stress as being directly correlated with hardness value and given by the following equation

$$\sigma_y \approx \frac{H}{C} \qquad (12)$$

where coefficient C can change in the range *2.5-3.0*. The final results of analytical analysis for Young's modulus E and hardness H are given in Figure 8 and 9 along with 95% confidence limits. As can be noticed result scatter for Young's modulus E of aluminium is much higher than that of fused silica, which may be due to ductile behaviour of aluminium vs. brittle behaviour of fused silica.

Fig.8. The extracted results for fused silica

Fig.9. The extracted results for aluminium

3.3. Numerical Modeling

A number of problems in contemporary engineering can be solved by numerical modelling. There are a number of modelling techniques, but the presented analysis was done with FEM method using ANSYS v.11. In fact FEM models of nanoindentation experiments are able to ensure high accuracy as to the loading as unloading part, which is mainly due to the application of different material behaviour due to thermo-mechanical loading. One of the benefits of FEM models is their ability to analyze the loading and unloading response due to different material models and corresponding properties as e.g.: elastic, elasto-plastic with and without hardening effect and even viscoelasticity. Additionally, it is possible to simulate the nanoindentation either using 2D or 3D models. Basing on a number of numerical experiments it was found out that 2D axisymmetric numerical models are precise enough to simulate the nanoindentation experiment. The figure below shows an example of 2D and 3D FEM models of the nanoindentation experiment.

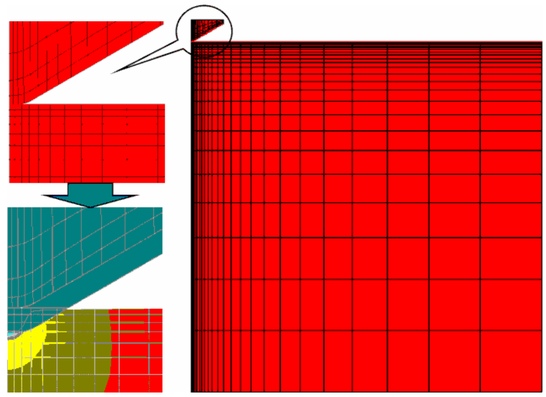

Fig.10. 2D numerical model of nanoindentation process

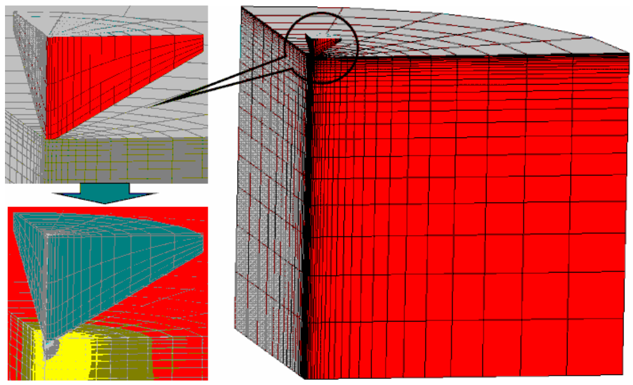

Fig.11. 3D numerical model of nanoindentation process

The results of numerical modelling are in a very close agreement with the analytically extracted data. Comparison between experimental and numerical modelling results for fused silica and aluminium are given on Figure 12 and 13.

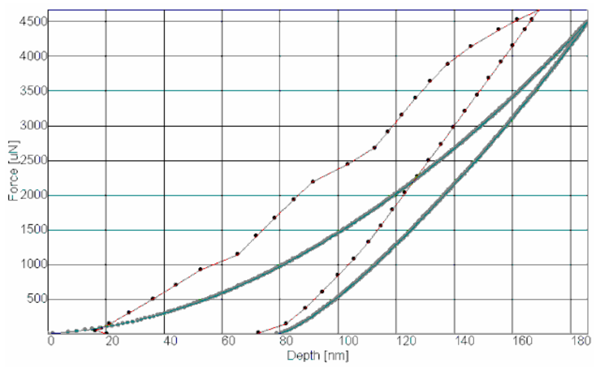

Fig.12. Experimental (line) and numerical (dots) results for fused silica

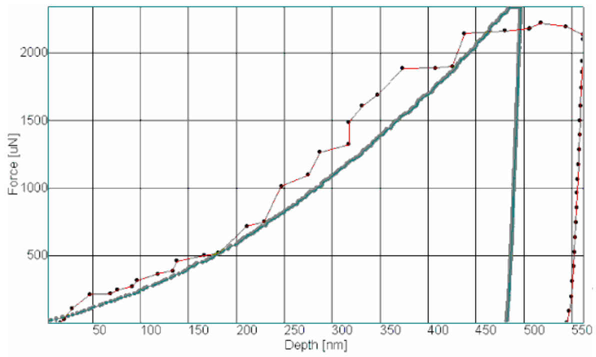

Fig.13. Experimental (line) and numerical (dots) results for aluminium

The disadvantage of analytical approach is inability of elasto-plastic material model assessment, e.g. either the material can be described by bilinear with or without hardening phenomena or nonlinear hardening, etc., which is given on Figure 13.

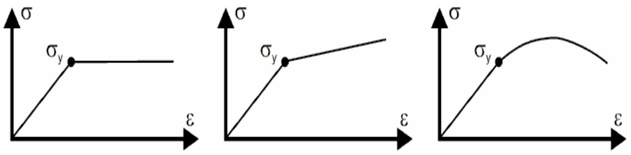

Fig.14. Different elasto-plastic material models

Due to a wide range of optimization methods there is a possibility of fitting numerical modelling and experimental results in order to assess the elasto-plastic material model and corresponding properties as Yield stress σ_y, which will be done as the continuation of the current project.

4. Conclusions

Current developments and trends in microelectronics are focused on thin layers and novel materials. This leads to application of different test and measurement methods, which are capable to measure basic mechanical properties of such materials on micro-scale. The current paper focuses on application of the nanoindentation technique in order to extract the basic elastic and elasto-plastic mechanical properties of fused silica and aluminium through analytical and numerical approaches.

Acknowledgments

The authors would like to thank the Wroclaw Centre for Networking and Supercomputing (WCSS) for the possibility of using the calculation equipment and the finite element software.

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