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Application of nanoindentation technique to extract properties of thin films through experimental and numerical analysis

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One of the major topics in novel microelectronics are thin film materials – especially their mechanical properties. Accurate description of such materials is necessary in order to assess their reliability and to predict failures in electronic devices. The mechanical attributes of thin films can be determined using the nanoindentation test. However, with this equipment it is only possible to obtain estimates elastic parameters: Young's modulus and hardness of the thin layer. In the paper, it is demonstrated that with a support of numerical methods the plastic behaviour of the material can also be extracted. The numerical FEM model of the nanoindentation test was elaborated and numerical optimization algorithms were applied. The goal was to examine the elastoplastic behaviour of the investigated thin film, which is the aluminium layer in this case. Various numerical material models were used in order to decently extract the material properties of the investigated thin layer.

Keywords: *nanoindentation; finite element method; thin film*

1. Introduction

The indentation technique was introduced for the first time in the 19th century. It was primarily used to measure hardness H of the material. Historically, the indentation was done by indenters of various shapes, thus leading to a variety of tests and analytical definitions of hardness. The indentation technique, called nanoindentation [1], was introduced in 1992 to examine mechanical properties such as elastic moduli E of investigated materials. Currently, the nanoindentation technique is used in microelectronics to examine properties of thin layers, which is an important issue in reliability analysis.

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2. Nanoindentation

The nanoindentation technique [2] allows assessing Young's modulus of the indenting material. It requires a very precise tip shape, e.g. use of the Berkovich tip, and therefore assures high spatial resolutions [3] (Fig. 1a). It is based on an the analysis of real time load displacement during indentation, which identifies a loading and an unloading curve, as shown in Fig. 1b [4].

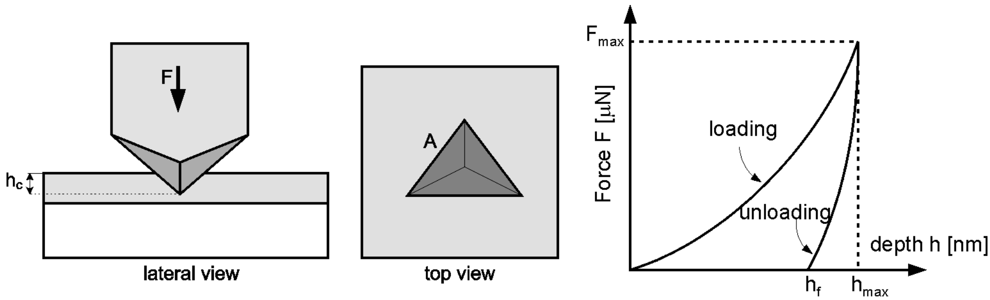

Fig. 1. The Berkovich nanoindentation test (a) and its result – the loading/unloading curve (b); $F/A = H_B \approx F/(24.56h_c)$, A – contact area, F – indenting force, H_B – hardness, h_c – contact depth

Taking into account the unloading nanoindentation curve it is possible to define and estimate the following elastic material properties [3, 5]:

- stiffness S

$$S = \left.\frac{dF}{dh}\right|_{F=F_{\max}} \tag{1}$$

where $F_{\max}$ is the maximum force during the loading and unloading indentation processes,

- hardness H

$$H = \frac{F_{\max}}{A_c} \tag{2}$$

where A_c is the contact area,

- effective modulus of elasticity E_{eff}

$$E_{\mathrm{eff}} = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} \tag{3}$$

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

2.1. Software tool

In order to perform the requisite numerical analysis, appropriate software had to be developed. The main aim is to model the elastoplasticity of the material, using:

• Analytical algorithms, based on theoretically justified formulae. The benefit of analytical algorithms is that elastic properties such as Young's modulus, can be evaluated accurately. Unfortunately, the drawback is that it cannot be used to study the plasticity behaviour, except the yield strength in some specific cases.

• Optimization analysis combining numerical analysis with optimization algorithms. The goal is to determine the best data fit curves for the experimental measurements of nanoindentation. The data fit curves depend on the selected plasticity model. The benefit of such an approach is that the elastoplastic behaviour of the material can be described with reasonably good accuracy.

3. Results and analysis

The properties of a large number of materials used for microelectronic packaging can be precisely described by mathematical models of elasticity or elastoplasticity. Typical stress–strain models for elastic and elastoplastic materials are presented in Fig. 2.

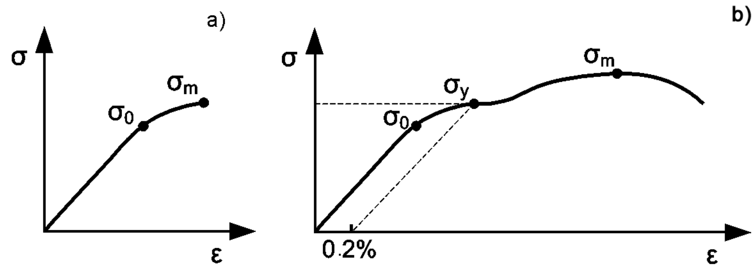

Fig. 2. The stress–strain curves for elastic (a) and elastoplastic (b) models

The research presented here focused on the mathematical description of the elastic and elastoplastic behaviour of specific materials. The above can be achieved by:

• analytical analysis, which uses the strict mathematical dependences in order to assess basic elastic properties of a material,

• numerical modelling, which uses finite element modelling (FEM) to describe the indentation loading and unloading curves, based on data obtained during the indentation tests.

3.1. Experimental setup

A hysitron triboindenter system, equipped with a Berkovich diamond indentation tip, was used for the experimental indentation procedure. The calibration of the system and of the area function $A(h)$ was undertaken on a fused silica sample, based on the method of Oliver and Pharr [1]. Aluminium thin layers on a silicon substrate were indented with indenters of various loads and depths. The aluminium layer, 1 μm thick, was fabricated in an evaporation process. The loading and unloading curves for each sample were determined experimentally, by performing nanoindentation. One of the goals was to identify a mathematical model that would accurately describe the relationship between the indentation depth and the indentation force.

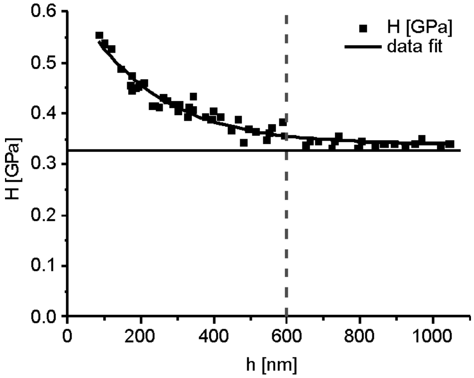

Fig. 3. Hardness *H* versus indentation depth *h* for aluminium thin layers

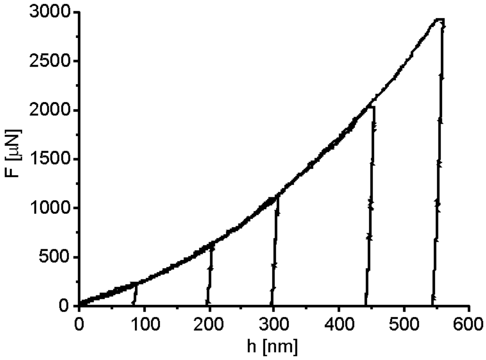

Fig. 4. Selected experimental results of loading and unloading indentation experiment for a thin layer of aluminum on silicon substrate

Classical analysis can describe elastic properties of a material such as the relationship between hardness *H* and indentation depth *h*, as shown in Fig. 3. As can be observed, material hardness should, and does, depend on the indentation depth. Despite the data scatter, hardness is clearly observed to decrease monotonically with respect to the indentation depth. Furthermore, the variation in the data measurements is clearly greater for thinner layers than for thicker ones. In fact, according to Fig. 3, the hardness measurements exhibit strong variation for indentation depths below 600 nm but are almost constant for indentation depths above 600 nm. This phenomenon is probably influenced by the presence of the substrate itself. In general, for indentation depths above 600 nm, the variations in the hardness data over the whole 1 μm thick layer are "flattened" by the interaction with the substrate. This clearly indicates the depth dependence of hardness and probably of other mechanical properties too such as Young's modulus or Yield stress for small indentation depths, e.g. below 600 nm in case of 1 μm aluminium layers.

In order to verify the most appropriate material model and corresponding properties for low indentation depths, a number of nanoindentation experiments were made using aluminium thin layers on silicon substrate. The selected results for the indentation depths lower than 600 nm are given in Fig. 4.

The experiments were then analysed numerically. In fact, the experimental results were the starting point to the numerical analysis. In order to extract the material properties, a global optimization algorithm based on simulated annealing with defined constraints technique was applied. In fact, the authors tested a number of optimization algorithms from local methods to global ones of which the simulated annealing seemed to be the most efficient and accurate.

3.2. Numerical modelling

A number of problems in contemporary engineering can be solved by numerical modelling. There are a number of modelling techniques but the analysis presented here exploits the FEM method, using ANSYS v.11. In fact, FEM models and data analysis of nanoindentation experiments can accurately describe the loading and unloading stages. One of the benefits of FEM models is they can exploit different mathematical models of materials. For example, they are suitable for investigating elasticity, elastoplasticity, or even viscoelasticity, with or without the hardening effect. In addition, it can be used to simulate nanoindentation, either using 2D or 3D models. Based on several numerical experiments, it was found that 2D axisymmetric numerical models are precise enough to simulate nanoindentation. Figure 5a shows an example of a 2D FEM model, and in Fig. 5b corresponding experimental data are shown and the data fit curve provided by numerical analysis.

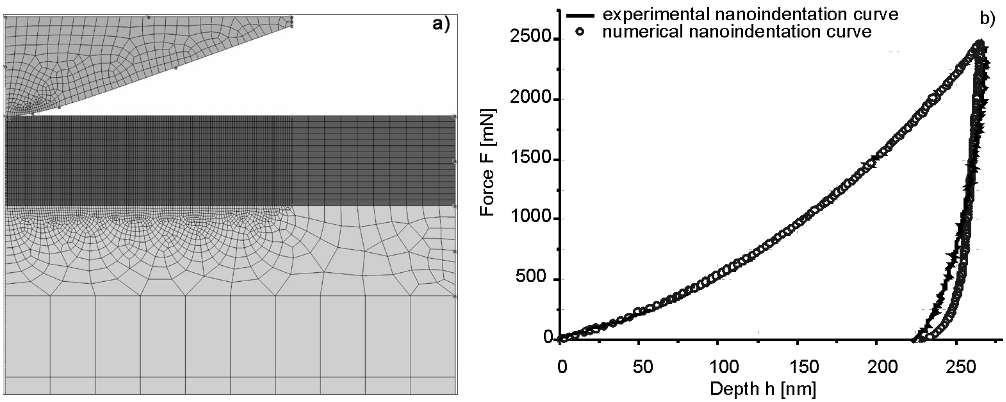

Fig. 5. The 2D numerical model of a nanoindentation test (a) and corresponding experimental and numerical results (b)

A numerical model with adjustable parameters was designed. In particular, it facilitates a systematic investigation of geometric features, for example thickness of a thin layer and indenter shape can be modelled by adjusting the area function coefficients. Thus, the numerical model can be adjusted to the actual geometry of the experimental indentation process. The results of numerical modelling are in very close agreement with the theoretical predictions.

As mentioned earlier, numerical analysis requires an optimization algorithm to find the best data fit, as shown in Fig. 5b. Many local and global optimization algorithms are available; the authors used simulated annealing, which, in their opinion, lead to the most efficient and accurate fitting [7]. The above procedure combining numerical modelling, experimental results and optimization algorithm finely allowed one to extract the most appropriate elastoplastic material model and corresponding properties.

Three models of elastoplastic materials were selected:

• bilinear response without hardening defined by two parameters: Young's modulus E and yield stress σ_y,

• bilinear response with hardening defined by three parameters: Young's modulus E, yield stress σ_y, and tangent modulus E_y,

• non-linear response with hardening, corresponding to the true stress–strain curve defined by the Ramberg–Osgood dependence [6]. Its three adjustable parameters satisfy the following equation:

$$\varepsilon = \frac{\sigma}{E} + K\left(\frac{\sigma}{E}\right)^n \tag{4}$$

where the total strain ε is a function of stress σ; it depends on Young's modulus E as well as on the constant K and exponent n which describe the plastic hardening of the material.

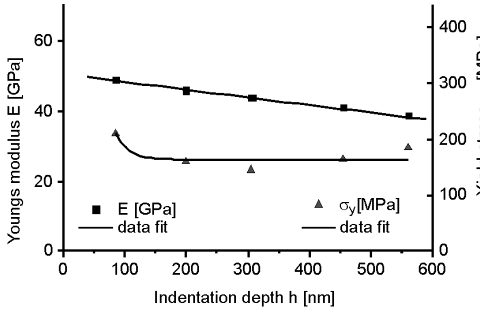

Fig. 6. Data fit curves for the bilinear model without hardening, showing Young's modulus E and Yield stress σ_y as functions of the indentation depth h in the case of a thin layer of aluminium

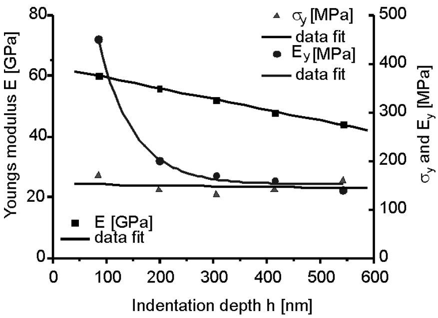

Fig. 7. Data fit curves for the bilinear model with hardening describing Young's modulus E, yield stress σ_y and tangent modulus E_y as functions of the indentation depth h in the case of an aluminium thin layer

The data-fit curves for the bilinear model of elastoplasticity without hardening, describing Young's modulus E and Yield stress σ_y in functions of the indentation depth h, are shown in Fig. 6. Young's modulus E decreases linearly upon increasing the indentation depth h, whereas the yield stress σ_y is observed to decay non-linearly for small depths, but then it levels off to a constant value of about 150 MPa for higher depths.

The data fit curves for the bilinear model of elastoplasticity with hardening, describing Young's modulus E, yield stress σ_y and tangent modulus E_y in functions of the indentation depth h, are given in Fig. 7. The model is characterised by monotonically decreasing functions of the indentation depth h. One of the advantages of the model is that the values for Young's modulus E are higher and more realistic. Unfortunately, similarly to the previous model, it seems to be inappropriate for accurate analysis of thin aluminium layers.

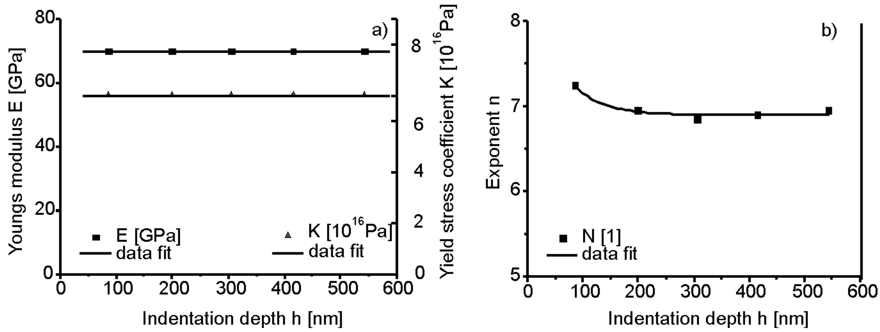

Fig. 8. Data fit curves for the non-linear, Ramberg–Osgood model without hardening, describing Young's modulus E, constant K (a), and the exponent n (b), as functions of the indentation depth h, for thin layer of aluminum

The data-fit curves corresponding to the Ramberg–Osgood model for an elastoplastic material, describing Young's modulus E, constant K and exponent n in functions of the indentation depth h, are given in Fig. 8. The results were obtained under the assumption that Young's modulus E is close to 70 GPa while the constant K is close to 7×10^{16}. The only adjustable parameter in that experiment was the exponent n. Putting all this information together, and substituting these values into the non-linear Ramberg–Osgood equation (Eq. (4)), leads to an elastoplasticity model for the material (Fig. 9).

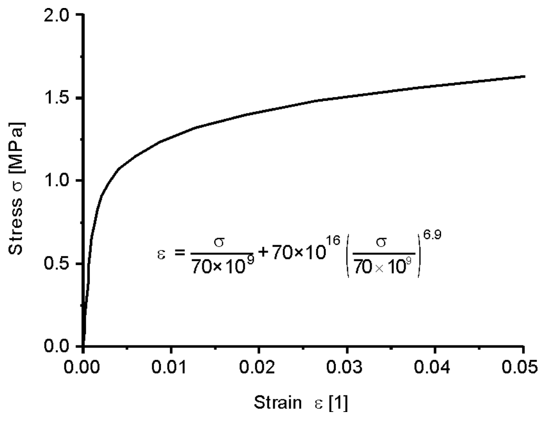

Fig. 9. The stress vs. strain curve for thin aluminium layers, according to the Ramberg –Osgood model, using average values for the model parameters E, K and n

Another benefit of the above model is that it implies plastic strain even at very small stress levels, making this approach more expressive than a bilinear elastoplasticity law, when it is applied in a Coffin–Manson type relation for life testing analysis.

4. Conclusions

Current developments and trends in microelectronics are focused on thin layers and novel materials. This led to application of various tests and measurement methods, capable of determining basic mechanical properties of such materials on both the microscale and the nanoscale. The project presented in this paper focuses on the nanoindentation technique. The specific aim was to describe, both numerically and theoretically, the dynamics of elasticity and elastoplasticity in aluminium. Several mathematical models were considered: the results of tests showed the Ramberg –Osgood model provided the most accurate description of elastoplasticity in the considered material. Furthermore, the Ramberg–Osgood model provides an accurate description of what occurs when very thin aluminium layers are subjected to nanoindentation; it shows, e.g., that the Young's modulus is of the order of 70 GPa, which corresponds well with aluminium bulk properties. The model is described by three variables, whose values appear to be almost constant over the investigated range of indentation depths h. Finally, it should be possible to exploit the Ramberg–Osgood model to predict the response of the material over a wide range of indentation depths.

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