18th International Conference
ENGINEERING MECHANICS 2012
Svratka, Czech Republic, May 14 – 17, 2012

pp. 1435–1440
Paper **#299**

DETERMINATION OF MECHANICAL PROPERTIES FROM MICROCOMPRESSION TEST

M. Truhlář[1], T. Kruml[2], I. Kuběna[3], K. Petráčková[3], L. Náhlík[3]

Abstract: *This paper describes a microcompression test of Al - 1.5 wt. % Cu thin film deposited on Si substrate. Microcompression combines the sample preparation with the use of ion focused beam (FIB) with a compression test carried out using nanoindenter. Cylindrical specimens (pillars) were prepared using FIB. The diameter of pillars was about 1.3 μm and their height was about 2 μm (equal to the film thickness). Stress-strain curves of the thin film were obtained. The results depend on crystallographic orientation of pillar. The paper is focused to an attempt to determine as precisely as possible Young modulus of the film using experimental data and finite element modelling.*

Keywords: ***microcompression, thin film properties, focused ion beam, Young modulus, FEM modelling***

1. Introduction

Measurements of mechanical properties of thin film are not generally easy to be done. There are few methods for measuring of plastic and elastic properties of materials. Each of them has some weaknesses (Nix, 1989). This paper is aimed on recently developed microcompression experiments (Uchic & Dimiduk, 2005; Kruml et al., 2009; Kuběna et al., 2009; Kuběna & Kruml, 2009). This method combines preparation of specimen using focused ion beam (FIB) and compressive test using nanoindentation device. Combination of the two techniques enables one to fabricate micrometric cylindrical specimens with the axis normal to film plane, attached by bottom to the surface. Such specimens are referred in the literature as pillars.

In previous experiments it was found that pillars with diameter under 1 μm show a significant increase in yield strength. This can be explained by the fact that in such small volumes no longer any mobile dislocations are present and the stress required for plastic deformation is determined by stress which is required for activation of dislocation sources and not by stress needed for dislocation movement (Nix et al., 2007). Nowadays such experiments are rather popular. The majority of the effort is focused on effect of specimen size to the mechanical behaviour of material. Generally, it is found that “smaller is stronger”.

Microcompression experiments on pillars made from Al thin films have already been performed in our laboratory several times (Kruml et al. 2009; Kuběna et al., 2008; Kuběna et al., 2009; Kuběna & Kruml, 2009). The main goal is to use the technique for determining mechanical properties of thin films. In this paper, we tried to improve calculation method for determining Young modulus with higher precision from the microcompression data.

---

[1] Mgr. Michal Truhlář; Institute of Physics of Materials AS CR, Žižkova 22, 61662 Brno, Czech Republic and Masaryk University, Faculty of Science, Kotlářská 2, 61137 Brno, Czech Republic, truhlar@ipm.cz

[2] Prof. Mgr. Tomáš Kruml, CSc.; Institute of Physics of Materials AS CR, Žižkova 22, 61662 Brno, Czech Republic, kruml@imp.cz

[3] Bc. Klára Petráčková; Ing. Ivo Kuběna; doc. Ing. Luboš Náhlík, Ph.D.; Institute of Physics of Materials AS CR, Žižkova 22, 61662 Brno, Czech Republic and Brno University of Technology, Faculty of Material Engineering, Technická 2, 61669 Brno, Czech Republic, klara.petrackova@seznam.cz, kubena@ipm.cz, nahlik@ipm.cz

2. Experiment

The studied material was a thin film of Al – 1.5 wt. % Cu of chemical composition and 2.06±0.05 μm of thickness, prepared by Physical vapour deposition (PVD) method in the ON Semiconductors company. The Al film was composed of relatively large grains with the average diameter in the plane parallel to the film surface of 3.8±0.3 μm. Such large grains are a consequence of a relatively high substrate temperature during deposition (340°C). Substrate for the chemical deposition was <100> Si monocrystalline wafer. The EBSD analysis showed a very strong preferential <111> orientation of the normal to the film surface. In between Al layer and Si substrate there was an intermediate thin film of W – 10 wt. % Ti and thickness about 0.14±0.04 μm. This embedded layer improves the adhesion of Al layer and is used also as a diffusion barrier. The sandwich was prepared on Varian 3190 sputtering system at the ON Semiconductor company.

The aim of experiment was prepare perfect cylindrical specimens with a diameter of about 1.3 μm with height determined by film thickness. Pillars were prepared in the FEI Quanta 3D FEG DualBeam™ system and Tescan Lyra 3 FEG microscopes. To ensure that the whole pillar is single crystalline were pillars produced in centres of large grains. The FIB milling procedure was optimized (Kuběna et al., 2009) so that the final shape of Al part of the pillar is as close to the perfect cylinder as possible.

The FIB milling was performed in several steps. First, faster milling by higher ion currents (~5 nA) was used for removing the majority of material in outer diameters, whereas the fine final milling of the central circular zone of about 3 μm in diameter was performed at low ion current (50 pA).

Loading of the pillars was carried out in compression using the MTS Nanoindenter XP machine equipped with a diamond flat punch of 20 μm in diameter. The diameter of the removed zone by FIB was chosen to be 25 μm, to ensure that the punch will not touch any other object except the pillar. During the deformation, it was ensured that the face of the flat punch was parallel to the upper face of pillars. The tests were carried out at nominal constant loading rate of 0.001 mNs^{-1}. At the onset of the deformation, pillars were therefore loaded in pure compression. Several unloading cycles were done with the purpose to measure elastic slope of the curve and to calculate Young modulus from these data.

Continuous stiffness measurement technique was used for measuring of indentation modulus and hardness as function of indentation depth. The indentation depth was chosen as 2 μm. It is know, that indentation modulus and hardness depend on indentation depth especially in case of thin film deposited on surface. A representative value of Al thin film was determined from plateau witch was found in dependences of indentation modulus and hardness on indentation depth.

From the microcompression experiments an equivalent of macroscopic compression curve is obtained. Such stress-strain curve provides information about yield stress, stress at chosen strain level or work hardening rate. For calculation of Young modulus, finite elements modelling must be used.

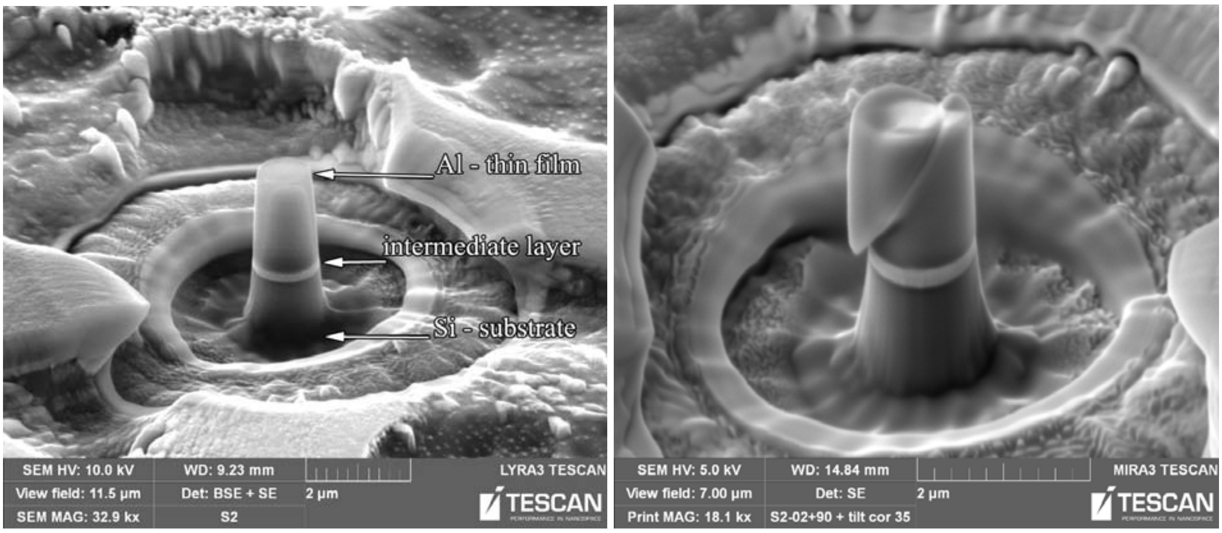

Fig. 1: The microcompression pillar S2 (a) before deformation; (b) after deformation

3. Results

3.1. Microcompression test

Prepared pillar from previous experiment is shown in Figure 1a. It was prepared using optimized process, (Kuběna et al., 2009). It is obvious that its geometry is close to the ideal shape, the minimum possible taper angle, smooth transition of the sample into the surrounding substrate and optimum depth of removed zone. These parameters have the greatest impact on homogeneous stress distribution in the sample. In this case the tapper angle is only 4 degree, substrate surface around pillar is also partially removed and substrate around pillar is not too rough. Pillar after microcompression test is show in Figure 1b. The obvious slip bands are likely produced during plastic bursts during deformation.

During microcompression test force and displacement of flat punch and time was continuously recorder. This data were recalculated on values of engineering stress and engineering deformation according to equations

$$\sigma = \frac{F}{S_0} \tag{1}$$

$$\varepsilon = \frac{\Delta l}{l_0} \tag{2}$$

where S_0 is the initial cross-section of the pillar, l_0 is initial height of pillar.

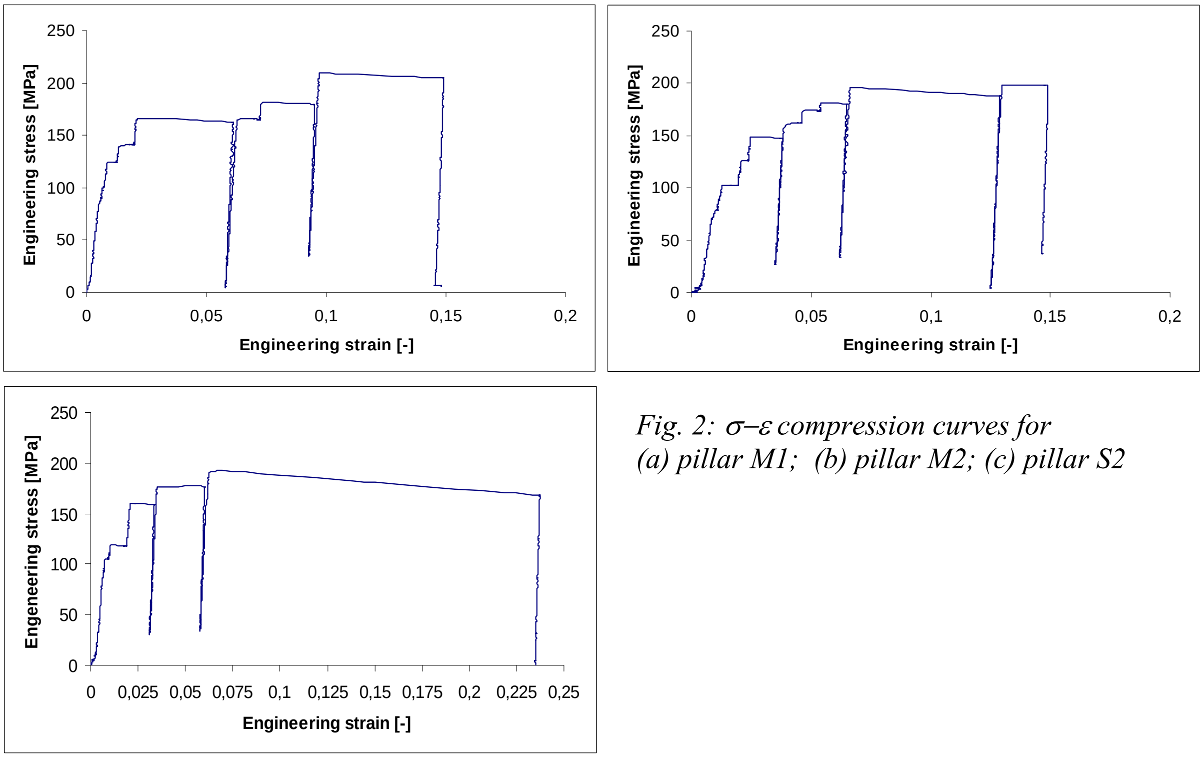

Fig. 2: σ–ε compression curves for (a) pillar M1; (b) pillar M2; (c) pillar S2

Actually, compression tests were carried out on monocrystalline pillars, therefore results strongly depend on crystallographic orientation of pillars. The crystallographic orientation was measured by EBSD followed by Schimd factor m determination. Subsequently, it was possible to recalculate σ–ε coordinates to τ–γ coordinates by equations $\tau = \sigma.m$ and $\gamma = \varepsilon / m$. Example of σ–ε compression curves for three different pillars are shown in Figure 2.

Obviously, the shapes of compression curves in Figure 2 are completely different from typical curve of the bulk materials. The deformation behaviour of pillars consisted of sudden jumps of fast plastic deformation called plastic bursts in the literature. Firstly, stress increases and pillars are deformed only elastically. After reaching of stress necessary for activation of a dislocation source, tens or hundreds dislocations loops propagate through pillar and disappear on free surface of pillars. It results in plastic bursts and in fast deformation of pillars as long as the dislocation source is active. When the dislocation source is exhausted, pillars are deformed again only elastically until other dislocation source is activated. The strain hardening was not observed.

3.2. Determination of elastic slope from experimental measurements

A methodology for evaluation of experimental data for given type of testing (Petráčková et al., 2012) has not been fully established yet. Since dependence of loading force on deformation of pillar is measured, following procedure was adopted:

- To determine a strain $\varepsilon(z)$ in direction of loading we consider height h of Al film (see Fig. 3). Then $\varepsilon(z) = u(z)/h$, where $u(z)$ is experimentally measured displacement applied on the top surface of the pillar.
- As a stress σ we consider force applied on top of pillar (loading force of nanoindenter) divided by mean value of area cut perpendicular to pillar axis through Al layer. The typical dependence of σ - $\varepsilon(z)$ is shown in Fig. 2

We assume that the elastic deformation of σ - $\varepsilon(z)$ curve is described by the unloading parts. From the first unloading, the elastic slope was determined. The elastic slope reflects the elastic deformation of the Al specimen, W – intermediate layer and Si substrate.

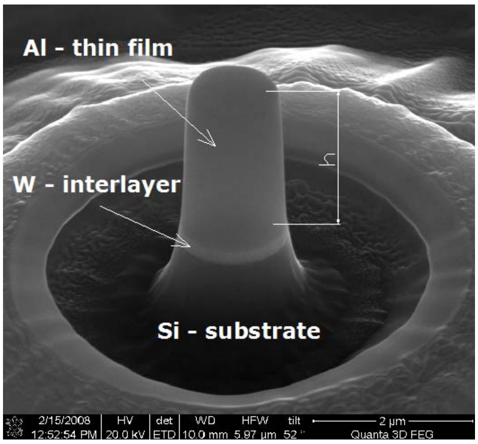

Fig. 3: Example of tested pillar. Geometry of the substrate part of the pillar, the tapper angle and the intermediate layer are taken into account for the Young modulus determination.

During the next loading cycle the sample is already partially deformed and has already a different shape than that used for numerical calculation. For this reason, data from first unloading are considered as the most reliable (see $E_{\mathrm{exp,1}}$ value in Table 1). The parameters of second unloading are presented for the comparison (see $E_{\mathrm{exp,2}}$ values in Table 1).

Three different pillars were measured and the resulting values of measured elastic slopes E_{exp} are shown in Table 1 for each tested pillar.

3.3 Numerical simulation for determination of Young modulus

Finite element method (FEM) was used for numerical simulation of microcompressive test and theoretical values of elastic slope were found (see E_p values in Table 1).

The data determined from experimental measurements (E_{exp1}) are slightly but systematically smaller than the values calculated with FEM. It is caused by the fact that the mechanical properties of all simulated components were taken from literature for the pure, bulk Al which may differ from the properties of thin film prepared by the PVD process.

Geometric factors of tested pillars such as conical shape of pillar, tungsten interlayer between studied Al layer and Si substrate, silicon substrate, etc. influence the measured elastic slopes. The influence of the three mentioned factors was numerically evaluated. Due to the linear elasticity, the influence of the individual factors on experimental data is proportional.

The most influencing geometric factor is the presence of Si substrate. It was estimated that it reduces the measured elastic slope during the microcompressive testing by almost 30%. That can be expected because the substrate creates a significant part of pillar and is also deformed during the testing. Quantitatively, influence of the substrate is described by coefficient C_1. Its values are presented in Table 1.

Tab. 1: Measured and calculated data.

Sample	pillar axis	$E_{\text{exp},1}$ [GPa]	$E_{\text{exp},2}$ [GPa]	E_p [GPa]	C_1	C_2	C_3	**$E_{\text{p,cor}}$ [GPa]**
M1	[3 5 9]	48.0	56.1	52.35	1.36	1.03	0.99	**66.6**
M2	[2 4 5]	45.6	52.3	56.30	1.22	1.07	0.99	**58.9**
S2	[1 2 3]	48.7	55.5	52.22	1.33	1.04	0.99	**66.7**

$E_{\text{exp},1}$ – elastic slope obtained by experimental measurements (first unloading),

$E_{\text{exp},2}$ – elastic slope obtained by experimental measurements (second unloading),

E_p – theoretical elastic slope obtained by numerical simulation of microcompressive test. As the input, elastic constants found in the literature for pure Al were used,

C_1, C_2, C_3 – correcting coefficients describing influence of substrate, conical geometry, and interlayer,

$E_{\text{p,cor}} = E_{\text{exp},1} \cdot C_1 \cdot C_2 \cdot C_3$, i.e. the values of Young modulus for given orientation of Al crystal lattice

Second geometric factor which has influence on the elastic slope is conical shape of pillars. The FIB procedure used for preparation of testing pillars does not allow producing the pillar as a prefect cylinder. With the optimized procedure (Kuběna et al., 2009), it can be ensuring that pillar shape is close to cylindrical one with the taper angle of 4°. Numerical expression of the influence on elastic slope is shown in table 1 as coefficient C_2. In this case the conical geometry of pillar influences data obtained by experiment of about 5%. This factor does not depend on material parameters of the pillar but only on the geometry. It means that multiplying experimental data by $C_2 = 1.05$ eliminates the influence of conical geometry with taper angle of 4 degree for any type of material tested.

Finally, it was estimated that existence of tungsten interlayer affects the elastic slope by about 1%. If the interlayer has the same thickness, negligible in comparison with the studied thin film, its influence is small and can be neglected. This holds for any combination of materials. Numerical expression of this effect is shown in Table 1 as C_3 coefficient.

The last column in Table 1 is the Young modulus of the Al film, for the given crystallographic orientation. It was calculated from the first elastic unloading and corrected for the three geometrical effects:

$$E_{\text{p,cor}} = E_{\text{exp},1} \cdot C_1 \cdot C_2 \cdot C_3 \tag{3}$$

It is visible that the E values are in a good agreement with the values for bulk Al, which varies from 64.1 GPa for <100> direction to 77.4 GPa for <111> direction. On the other hand, the E values of Al thin films prepared by PVD process reported in the literature varies significantly, from about 47 to 74 GPa (Chinmulgund et al., 1995). It can be explained by the fact that the deposited materials contain porosities and other defects. The high values of E found in this work shows that the tested film is of a very good quality.

4. Conclusions

In this paper, the methodology for evaluation of Young modulus of thin film from data obtained by microcompressive test was suggested. The influence of specific pillar geometry and presence of other phases (W – interlayer, Si – substrate) within the tested specimen can be calculated by finite element method and elastic slope obtained directly from experimental stress–strain curve can be corrected for these factors. The measured value of elastic slope was taken from the first unloading part of the curve. It was found that the elastic constants of the tested film are close to the ones reported for bulk Al which means that the film is of a good quality and did not contain significant amount of porosity.

Acknowledgement

This work was supported by Specific academic research grant of the Ministry of Education, Young and Sports of the Czech Republic provided to Brno University of Technology, Faculty of Mechanical Engineering FSI-S-11-11/1190

References

Chinmulgund, M., Inturi, R.B., Barnard, J.A.: Effect of Ar gas pressure on growth structure, and mechanical properties of sputtered Ti, Al, TiAl, and Ti_3Al film. *Thin Solid Films* 270, 260, (1995)

Kruml, T., Stranyanek, M., Ctvrtlik, R., Bohac, P., Vystavel, T. and Panek, P.: A new method for mechanical testing of thin films: application on Aluminum. *Journal of Material Research 24* (2009), 1353

Kuběna, I., Kruml, T.: Optimalizace přípravy mikrokomresních vzorků z tenkých vrstev pomocí fokusovaného iontovéhosvazku. In: *Víceúrovňový design pokrokových materiálů '09*,

Kubena, I., Kruml, T., Hutar, P., Bohac, P., Stranyanek, M., Ctvrtlik, R., Panek P. and Vystavel, T.: Measurement of mechanical properties of thin films by nanocompresion. In: *Vrstvy a povlaky 2008*, Liss, Roznov p R., 103, (2008)

Kuběna, I., Kruml. T., Vystavěl, T.: Příprava mikrokompresních vzorků z tenkých vrstev po-mocí fokusovaných iontových svazků. *SEMDOK 2009 Conference proceedings*, Súlov, 73, (2009)

Nix, W.D.: Mechanical properties of thin films. *Metallurgical Transactions A* 20 A (1989), 2217

Nix, W.D., Greer, J.R., Feng, G., Lilleodden, E.T.: Deformation at the nanometer an microme-ter length scales: Effect of strain gradients an dislocation starvation. *Thin Solid Films* 515 (2007), 3152

Petráčková, K., Kuběna, I., Truhlář, M., Náhlík, L., Kruml, T.: Estimation of mechanical properties of thin Al surface layer. *Applied Mechanics 2012*

Uchic, M.D., Dimiduk, D.M.: A methodology to investigate size scale effects in crystalline plasticity using uniaxial compression testing. *Materials Science and Engineering A* 400-401 (2005), 268