DETERMINATION OF MECHANICAL PROPERTIES FROM MICROCOMPRESSION TEST

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Abstract: *This paper describes a microcompression test of thin 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

One possibility how to perform mechanical tests on a very small scale is to prepare a small cylindrical specimen of micrometric size by focused ion beam and to execute a compression test using nanoindenter device equipped with a flat diamond punch. Such experiments are rather popular nowadays for the study of effect of specimen size on its mechanical behaviour (Nix et al. 2007).

We performed successfully such tests with the aim to determine elastic and plastic properties of thin films deposited on a substrate by PVD method (Kruml et al. 2009). In this paper, we tried to improve calculation method for determining Young modulus with higher precision.

2. Experiment

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 centers of large grains. The FIB milling procedure was optimized so that the final shape of Al part of the pillar is as close to the perfect cylinder as possible.

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.

3. Results

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

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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 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%. Quantitatively, influence of the substrate is described by coefficient C_1.

Second geometric factor which has influence on the elastic slope is conical shape of pillars. 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%. 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.

Tab. 1: Measured and calculated data.

Sample	crystal. orient.	$E_{exp,1}$ [GPa]	$E_{exp,2}$ [GPa]	E_p [GPa]	C_1	C_2	C_3	$\mathbf{E_{p,cor}}$ **[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**

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

$E_{exp,2}$ – 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,

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

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.

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