

## A COMPARISON OF DIFFERENT MEASUREMENT METHODS OF MECHANICAL PROPERTIES OF Al THIN FILMS

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### Abstract

The paper compares two different methods for testing of metallic thin films: microcompression test and nanoindentation. Microcompression test is one possibility how to perform mechanical tests on a very small scale. This method requires preparation of a small cylindrical specimen (micropillar) of micrometric size by FIB and execution of a compression test using nanoindenter device equipped with a flat diamond punch. Stress-strain curves of the thin films were obtained from such tests. Nanoindentation tests were then conducted to compare the results on the same films. Two different metal thin films - AlCuW, AlCuSi with thickness 2  $\mu\text{m}$  and grain size 3.8  $\mu\text{m}$  in average were prepared by PVD method. In this paper, we announce the results of measurements, a comparison of the results obtained by each method and identify advantages and limitations of the methods.

**Keywords:** Microcompression, nanoindentation, mechanical properties, Young modulus

### 1. INTRODUCTION

Mechanical properties of thin films are generally not easy to be measured. There are two frequently used methods - nanoindentation and microcompression techniques. Each of them have varying outputs and different advantages and disadvantages.

Nanoindentation continuously measuring load force on specimen and displacement of indenter is by far the most popular method. It is easy to be carry out. Indentation modulus and hardness can be measured, nevertheless this method is not suitable for measuring of plastic properties of thin films such as yield stress, ultimate stress or strain hardening coefficient [1].

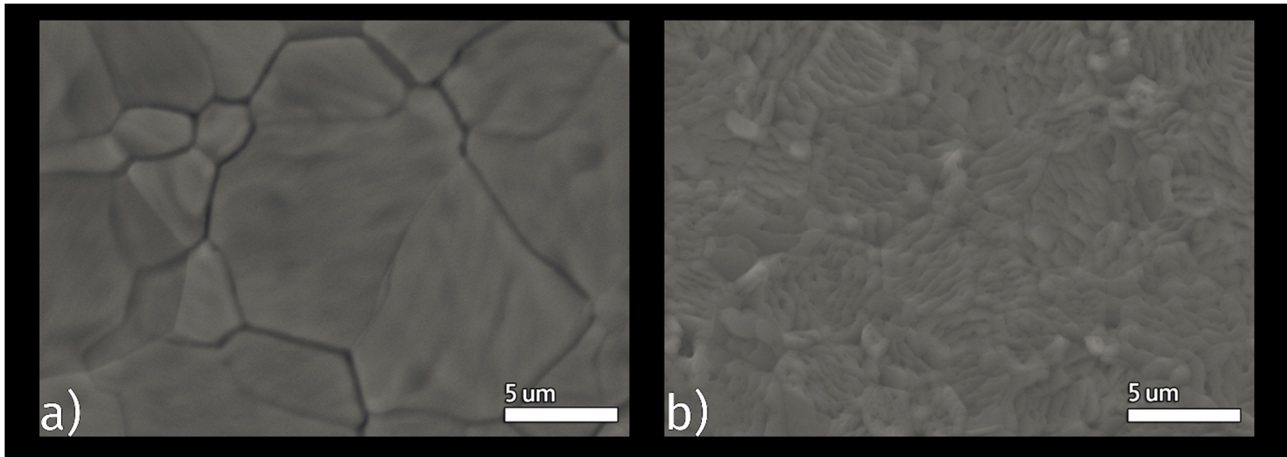
Microcompression test is the second possibility how to perform mechanical tests on a very small scale. This method is based on the preparation of small cylindrical specimen of micrometric size by FIB and execution a compression test using nanoindenter device equipped with a flat diamond punch. Such experiments are used e.g. for the study of effect of specimen size on its mechanical behaviour [2].

In this paper we performed successfully such tests with the aim to determine elastic and plastic properties of thin films deposited on a substrate by PVD method [3, 4]. Experimentally measured data from micro-compression tests need correction to obtain undistorted material properties. In past, we tried to improve calculation method for determination of Young modulus [5].

### 2. EXPERIMENT

Two different metallic thin films - AlCuW (14.0 wt. % W; 1.5 wt. % Cu; bal. Al) and AlCuSi (1.5 wt. % Cu; 0.5 wt. % Si; bal. Al) with thickness  $2.06 \pm 0.05 \mu\text{m}$  are studied. The Al films were composed of relatively large grains with the average diameter of  $3.8 \pm 0.3 \mu\text{m}$  in the plane parallel to the film surface (**Fig. 1**). Such large grains are a consequence of a relatively high substrate temperature during deposition (340 °C). The EBSD

analysis (**Fig. 2b**) showed a very strong preferential  $\langle 111 \rangle$  orientation of the normal to the film surface. The films were deposited on  $\langle 100 \rangle$  Si monocrystalline wafer. An intermediate W-10%Ti layer was present between the wafer and the film. This sandwich was prepared on Varian 3190 sputtering system at the ON Semiconductor company.



**Fig. 1** Comparison of the surface of thin films studied in this paper: a) AlCuW with obvious grain boundaries, b) AlCuSi with rough surface and indistinct grain boundaries

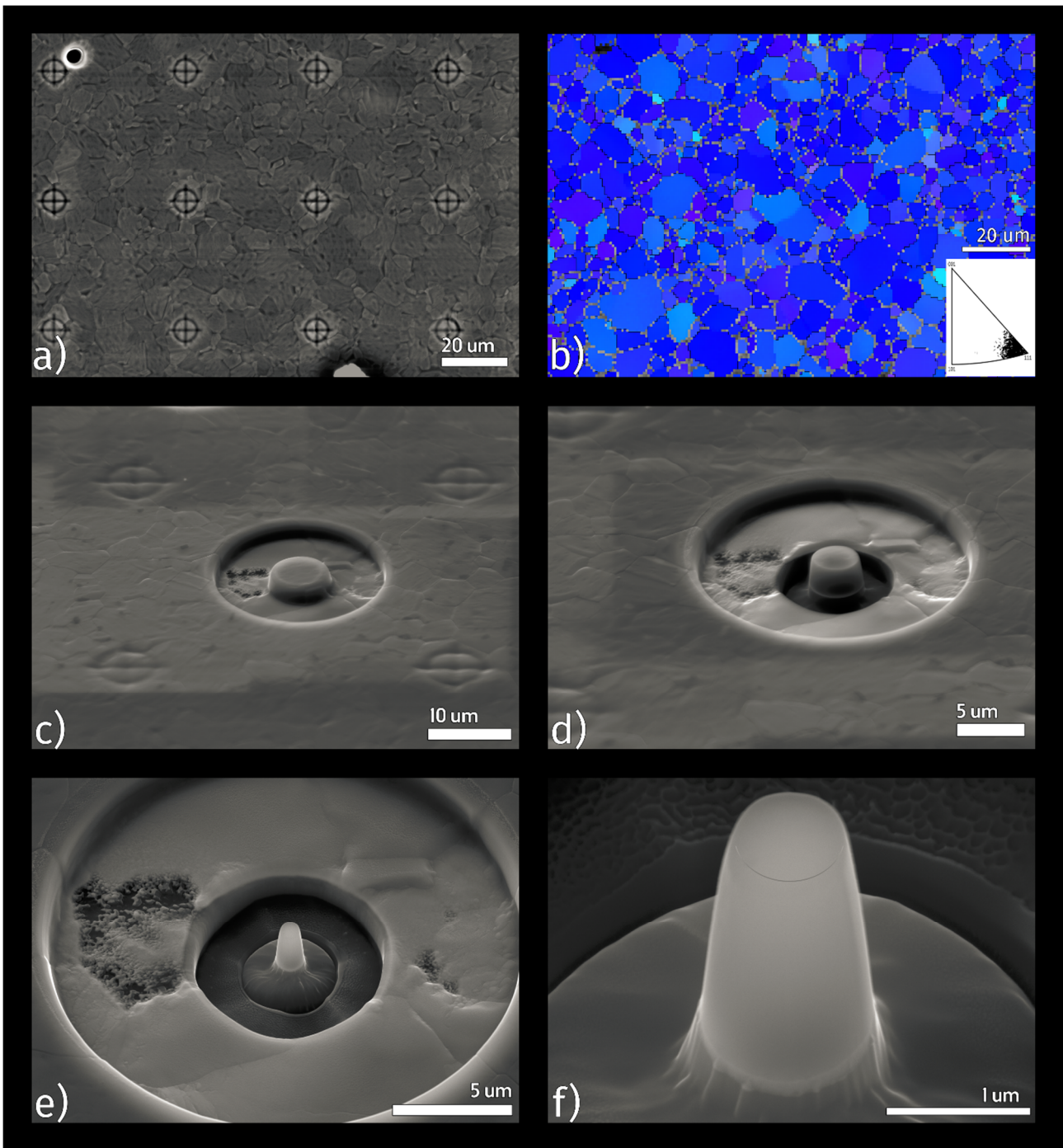
## 2.1 Microcompression test

The microcompression specimens (micropillars) were prepared in the Tescan Lyra 3 FEG microscope. The aim was to obtain cylindrical specimen with a diameter of  $1.0 \mu\text{m}$  and a height determined by the film thickness. Micropillars were produced in centers of large grains, to ensure that the whole micropillar is single crystalline. The FIB milling procedure was optimized [6] and modified so that the final shape of the micropillar is as close to the perfect cylinder as possible.

Micropillars were prepared in three steps (**Fig. 2**). In the first step (**Fig. 2c**) fast FIB milling was used. We removed material from annular zone with diameter from  $10$  to  $20 \mu\text{m}$ , to ensure that the punch will not touch any other object except the micropillar. The diameter of the diamond punch was  $10 \mu\text{m}$ . In the second step (**Fig. 2d**) we removed material using lower current of FIB to obtain area of the final diameter  $3 \mu\text{m}$ . Final step (**Fig. 2e**) is made in micro machining mode with slow circulating beam scan with low current and voltage.

Microcompression tests were conducted using Hysitron TI950 TriboIndenter. Compression tests were adjusted so that the maximum force of compression caused deformation of approximately  $1/10$  of thickness of the layer, i.e. about  $200 \text{ nm}$ . It was found that such maximum load forces is about  $200 \mu\text{N}$ . Microcompression tests were carried out at nominal constant loading rate of  $2.5 \mu\text{Ns}^{-1}$  and unloading rate  $25 \mu\text{Ns}^{-1}$ . Three partial unloadings to the half of load force were performed on forces  $50 \mu\text{N}$ ,  $100 \mu\text{N}$  and  $150 \mu\text{N}$  with the aim to measure elastic modulus. During the deformation, care was taken that the face of the flat punch was parallel to the upper face of micropillars.

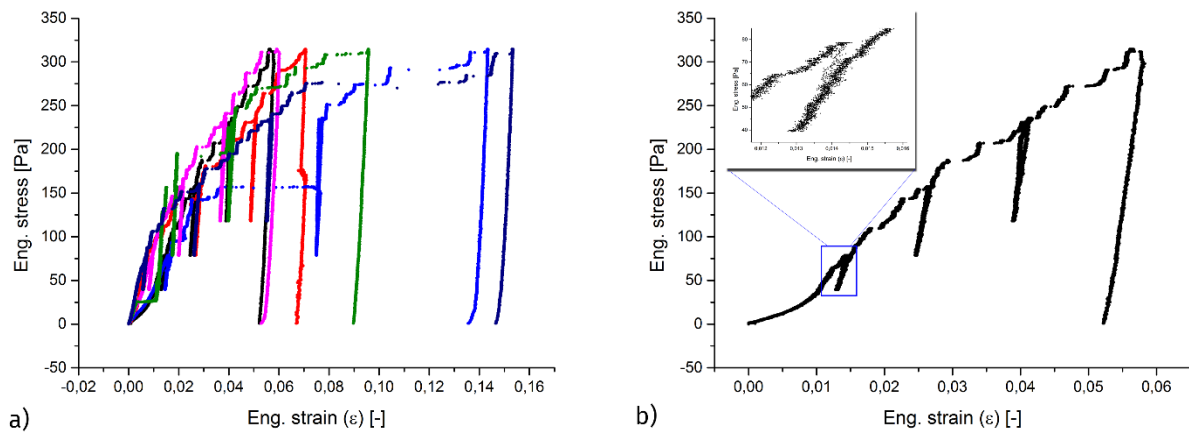
The results were analysed and stress-strain curves were plotted (**Fig. 3**). Young modulus were calculated from the slope of partial unloadings. Plastic deformation of micropillars is not regular, typical plastic strain bursts - fast plastic deformation events - were observed. Stress-strain curves (**Fig. 3**) contain horizontal parts which correspond to these bursts.



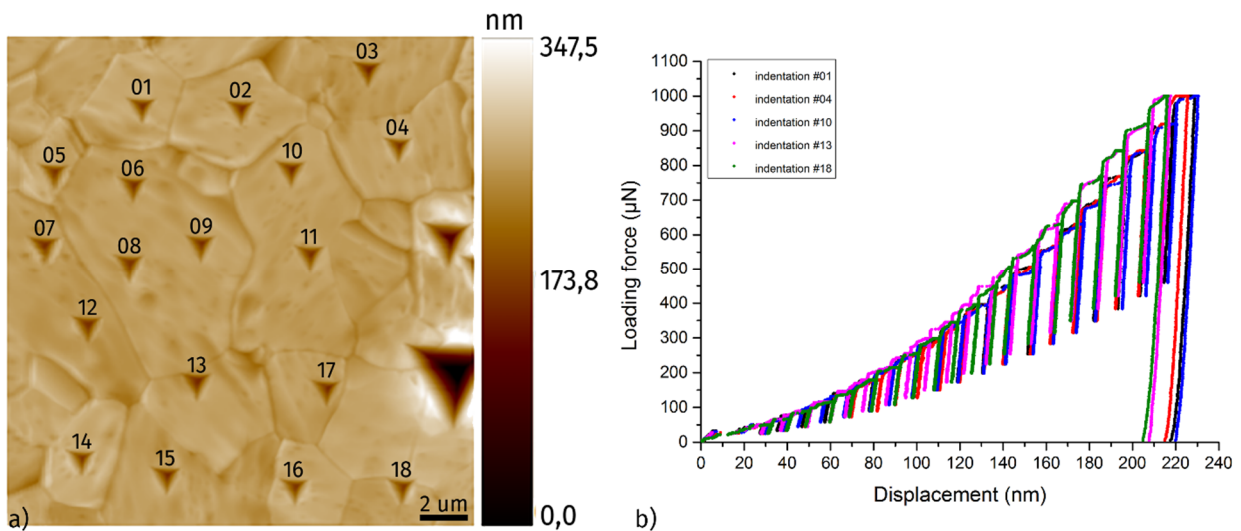
**Fig. 2** Representation of the methods for preparing of micropillar for microcompression test: a) surface of AlCuW thin films with orientation marks, b) EBSD map of the selected area, c) the first step of FIB preparation, d) the second step of FIB preparation, e) micropillar after the third and last step of preparation, e) detail of micropillar no.6 on AlCuW, view at an angle of 30 degrees

## 2.2 Nanoindentation test

Nanoindentation test were performed on the same Hysitron TI950 TriboIndenter. We used standard Berkovich diamond tip. Two different kinds of indentation tests were done: i) indentation with one unloading, ii) indentation with several partial unloadings (**Fig. 4b**). Maximum load force were 1 mN and first unloading was performed at a force 0.05 mN. Time of loading was 5 s with maximal force 1 mN. We obtained reduced Young modulus  $E_r$  and hardness of the two thin films.



**Fig. 3** Stress-strain curves of microcompression test for a) 6 micropillars on AlCuW, b) micropillar #6 on AlCuW with detail of the first partial unloading



**Fig. 4** Nanoindentation on AlCuW thin film: a) surface detail showing the locations of 18 nanoindentation positions, b) force-displacement curves of 5 selected nanoindentation - position no. 1, 4, 10, 13, 18

### 3. RESULTS

#### 3.1 Nanoindentation test

Average values of results measured in 18 nanoindentation test performed on both films are given in the **Table 1**. The Young modulus of the two materials  $E$  was estimated according Oliver & Pharr [7] using reduced modulus and assuming Poisson ratio of the film 0.3. Standard deviation of results comprises experimental scatter and also different crystal orientation for individual indentations.

**Table 1** Results of nanoindentation experiments on AlCuW and AlCuSi

AlCuW	$E_r = (100 \pm 20)$ GPa	$H = (760 \pm 120)$ MPa	$E = (96 \pm 18)$ GPa
AlCuSi	$E_r = (85 \pm 4)$ GPa	$H = (580 \pm 70)$ MPa	$E = (81 \pm 4)$ GPa

#### 3.2 Microcompression test

Six micropillars on each of the studied thin film were tested in compression. Conicity of micropillars ranged from 7° to 9°. Micropillar diameter on the top varied from 0.85 μm to 0.98 μm and final height of the micropillar was around 1.86 μm.

Elastic moduli were calculated from all partial unloadings (**Tables 2, 3**). It was observed that measured moduli are systematically lowest for the first unloading and highest for third unloading. Petráčková et al. [5] showed using detailed FEM analysis that measured elastic moduli  $E_m$  must be corrected to effect of substrate and imperfect shape of micropillar. The correction coefficient for this types of pillars is  $1.32 \pm 0.02$  [5]. The corrected values of Young modulus  $E_{cor} = 1.32 \times E_m$  and their average values and standard deviations are given in **Tables 2 and 3**.

Microcompression tests enables determining plastic properties of thin films. One possibility is to measure stress necessary to deform micropillar to 1 % plastic strain  $\sigma_{1\%}$ . This value is also given in **Tables 2 and 3**.

**Table 2** The results of measurements of mechanical properties of AlCuW thin film using microcompression testing including calculation of Young's modulus corrected to conicity of a micropillar and effect of the substrate

Micropillar no.	Miller index	1 <sup>st</sup> unloading $E_{cor}$ [GPa]	2 <sup>nd</sup> unloading $E_{cor}$ [GPa]	3 <sup>rd</sup> unloading $E_{cor}$ [GPa]	$\sigma_{1\%}$ [MPa]
AlCuW #1	[5 6 6]	45.56	67.22	84.69	190
AlCuW #2	[3 4 4]	42.08	62.91	77.97	154
AlCuW #3	[2 2 3]	40.96	61.23	83.58	159
AlCuW #4	[1 2 2]	31.83	55.74	77.90	180
AlCuW #5	[3 4 4]	34.57	61.02	82.52	194
AlCuW #6	[5 5 6]	33.51	53.57	81.42	147
		$E_{cor} = (38 \pm 5)$	$E_{cor} = (60 \pm 5)$	$E_{cor} = (81 \pm 3)$	$\sigma_{1\%} = (171 \pm 18)$

**Table 3** The results of measurements of AlCuSi thin films using microcompression testing

Micropillar no.	Miller index	1 <sup>st</sup> unloading $E_{cor}$ [GPa]	2 <sup>nd</sup> unloading $E_{cor}$ [GPa]	3 <sup>rd</sup> unloading $E_{cor}$ [GPa]	$\sigma_{1\%}$ [MPa]
AlCuSi #1	[4 5 5]	29.26	53.17	80.06	152
AlCuSi #2	[2 3 3]	34.07	64.39	81.40	183
AlCuSi #3	[5 5 6]	35.20	71.07	80.81	--
AlCuSi #4	[1 2 2]	41.71	62.34	80.40	184
AlCuSi #5	[4 4 5]	39.27	64.17	78.40	135
AlCuSi #6	[5 6 6]	43.93	69.73	83.81	137
		$E_{cor} = (37 \pm 5)$	$E_{cor} = (64 \pm 6)$	$E_{cor} = (81 \pm 2)$	$\sigma_{1\%} = (158 \pm 21)$

#### 4. DISCUSSION

The two presented measurement methods are complementary. Nanoindentation gives access to hardness and Young modulus of the films. The Young modulus is determined in condition of high triaxial stresses under the tip. This value might be influenced by the crystallographic orientation of the grain.

On the contrary microcompression provokes uniaxial stress in micropillars. Measured value thus corresponds to elastic modulus for crystallographic orientation of the micropillar. Measured values of  $E_{cor}$  depend on stress level when the unloading is done and the best agreement with the nanoindentation results was found for third unloading. We propose the following explanation. For small deformation, contact between the flat tip and upper surface of the micropillar is not perfect, mainly due to roughness of the film surface. This contact is improved with increasing deformation, therefore it is necessary to reach certain minimum value of strain for correct measurement of elastic modulus. Consequently, we consider values measured in the third unloading to be correct.

Due to the strong texture of tested films the scatter of  $E_{cor}$  is low; however it would be much larger in case of highly anisotropic material without texture. The agreement of Young modulus measured by both methods is quite good, taking into account the mentioned differences in the stress state.

Due to peculiar shape of stress-strain curves on such small specimens containing plastic bursts, the most important plastic characteristic of bulk materials, yield stress at 0.2 % plastic strain  $R_{p0.2}$ , suffers from high scatter. Stress at 1 % of plastic strain seems to be more representative characteristics of the yield stress.

## CONCLUSIONS

- Nanoindentation and microcompression test are complementary methods for the measuring of the mechanical properties of the thin films.
- Hardness and Young modulus measured in condition of triaxial stress state were measured by nanoindentation.
- Microcompression test enables measurement of stress-strain curves, i.e. determination plastic properties of thin films. Measured Young modulus corresponds to elastic modulus in specific crystallographic direction. Its determination is more complex: i) care must be taken of good alignment of tip and micropillar, ii) roughness of the film causes lower values for low strains and iii) FEM analysis is necessary for correcting measurements from the influence of the substrate and imperfect micropillar shape.

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