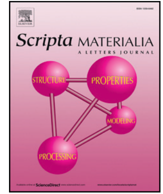
Scripta Materialia 130 (2017) 143–147

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat



Regular Article

Extracting flow curves from nano-sized metal layers in thin film systems

D. Kozic [a,*], V. Maier-Kiener [b], R. Konetschnik [c], H.-P. Gänser [a], T. Antretter [d], R. Brunner [a], D. Kiener [c]

[a] Materials Center Leoben Forschung GmbH, Leoben, Austria
[b] Department Physical Metallurgy & Materials Testing, Montanuniversitaet Leoben, Austria
[c] Department Materials Physics, Montanuniversitaet Leoben, Austria
[d] Institute of Mechanics, Montanuniversitaet Leoben, Austria

ARTICLE INFO

Article history:
Received 12 October 2016
Received in revised form 7 November 2016
Accepted 8 November 2016
Available online xxxx

Keywords:
Multilayer thin films
Finite element analysis
Constitutive modeling
Nanoindentation
Residual stresses

ABSTRACT

In this work the plastic deformation behavior of nanocrystalline 500 nm thick tungsten and copper films deposited on a silicon substrate is investigated. Nanoindentation experiments utilizing a spherical tip are used to determine the mechanical response of the film stacks. The data is used as input for an inverse optimization routine coupled to finite element simulations in order to determine the flow curves of the individual materials. We elaborate on how locally resolved residual stresses, microstructure, and external dimensions determine and influence the flow behavior and compare the results to the corresponding bulk materials.

© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

The microelectronics industry is driven by the idea of continuously reducing the dimensions of components and devices. Aiming at cost efficient components, the types and arrangement of thin film materials have to be well considered. Sub-micron thin films are commonly exposed to internal loading from residual stresses and their mechanical behavior is controlled by the external and internal dimensions. Experiments and modeling are the tools of choice for thin film characterization, when aiming to understand how and why multi-material systems fail in service. It is well known that materials at very small dimensions behave differently from their bulk counterparts [1–6]. The topics of material properties and especially the strengthening mechanisms of materials on the micron level and below have been intensely addressed in the last few decades. Two main areas of interest have been: (I) single-crystalline materials, where the controlling material parameter is the external dimension [1,2,7,8] and (II) polycrystalline materials, where the strength of a material can be improved by decreasing its grain size [9–11].

Concerning (I), the size effect on the plastic deformation in single-crystalline thin films can be described by the constrained movement of dislocations within the film [12–15]. In principle, the yield strength depends on the thickness of the film, which is directly related to the extension of at least one dislocation loop which has to fit into the film. The strengthening effect on a material by reducing the dimensions of single-crystalline specimens has also been observed experimentally, *e.g.* by tensile and compression testing [1,2,16,17] or bending beam experiments [7,18]. Regarding (II), the deformation properties of polycrystalline materials are determined by dislocation pile-up at grain boundaries [4,19]. As the thin film thicknesses for microelectronic components range between a few nm and a few μm, the strongest and most promising materials are nanocrystalline [9,20]. For example, Wang et al. [21]. have shown that the yield strength of a nanocrystalline tensile sample with an average grain size of 30 nm is almost twice the one of an ultrafine grained specimen where the grains measure 200 nm.

The majority of the work discussed above focuses on determining the deformation properties of single-crystalline materials on the micron level, or of polycrystalline materials containing a large number of nano-sized grains. Recently, attention has turned to the intermediate topic of thin film materials which occupy only a few grains over their thickness. Interestingly, studies [4,22,23] have shown that the strength of a material can be controlled by altering its microstructure while decreasing the external dimensions of the sample. Controversially, some researchers are reporting a strengthening effect [22], and others have observed a weakening of miniaturized nanocrystalline nickel pillars [23]. Therefore, a general consensus about the size effect on the strength of materials like these has yet to be established.

In this work we are focusing on the material behavior of thin nanocrystalline films with a countable number of grains over the film thickness. The plastic material properties of the films in a multi-layer composite exposed to residual stresses are calculated. We determine the flow behavior of a thin tungsten (W) and copper (Cu) layer on a silicon (Si) substrate with (100) orientation by combining spherical nanoindentation experiments and finite element (FE) modeling. The investigations are performed on two stack configurations. One sample

* Corresponding author.
E-mail address: darjan.kozic@mcl.at (D. Kozic).

http://dx.doi.org/10.1016/j.scriptamat.2016.11.008
1359-6462/© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

consists of a stepwise fabricated W-layer and the second specimen is a W—Cu—W tri-layer system, both deposited on Si. The appropriate yield strength and hardening parameters for each individual thin film are determined by inverse optimization in combination with FE simulations.

The investigated samples were prepared at room temperature using physical vapor deposition on a Mantis Sputter System (Mantis, Thame, United Kingdom). The (100) oriented Si wafer has a thickness of 525 μm and each of the deposited W- or Cu-layers is approximately 500 nm thick with a globular grain structure. The grain size varies between 60 and 70 nm [24]. In order to guarantee a good adhesion of the first material on Si a 10 nm thick chromium seed layer was deposited on the Si-substrate before the deposition of the subsequent layers. The vacuum was never broken during fabrication, as the three sputtering targets were mounted in the same chamber. Due to their negligible thickness, the ultra-thin Cr-layers were not considered in the presented investigations. Further information about the deposition conditions and parameters can be found in [24].

Two different stack configurations were used in order to assess the W- and Cu-layer with respect to their flow behavior. To characterize the flow and hardening behavior of W, a specimen with a 1 μm thick W-layer on Si was manufactured. The W-layer was not fabricated in one single step. After depositing about 500 nm of W the process was interrupted for 4550 s before the rest of the layer was completed. A disruption between the two steps results in a variation of the residual stress state in the film as shown in [24]. For the second stack configuration, a Cu-layer was deposited between the two W-layers, resulting in a W—Cu—W tri-layer. Here, each layer has an approximate thickness of 500 nm.

In order to determine the flow behavior of the W and Cu films, we have performed nanoindentation experiments. The experiments were conducted at room temperature on a platform Nanoindenter G200 (Keysight Tec. Inc., Santa Rosa, *CA*, USA) equipped with a spherical diamond indentation tip (Synton-MDP AG, Nidau, Switzerland). The tip has a radius of ~10 μm. A standard load controlled operating scheme was applied to reach a maximum load of 100 mN at a preset loading time of 30 s.

The experimental results are used to calculate the plastic behavior of the W and Cu film through inverse optimization together with FE analysis. While under load, the two materials are assumed to follow the well-known Ramberg-Osgood [25] material model:

$$\varepsilon = \frac{\sigma}{E} + \alpha\left(\frac{\sigma}{\sigma_0}\right)^m, \tag{1}$$

where σ denotes the stress, σ_0 the yield strength and ε is the total strain in the model. The Young's modulus of the material is defined as E, and α as well as m represent parameters describing the hardening behavior. The two key parameters used in the optimization procedure to fit the simulated force-displacement curve to the experimental data are σ_0 and the inverse hardening parameter m. The Ramberg-Osgood material model is designed to give a continuously rising stress with increasing strain mainly depending on the hardening parameter. Therefore, this model is not able to predict an ultimate strength of the material before the point of fracture at high strains. However, for lower strains the model gives a reasonable prediction of the resulting flow stresses.

The substrate is assumed to be linear elastic. A summary of the elastic material behavior for W and Si was already given in a former article [26]. In the present case for W and Si, respectively, Young's moduli of 411 GPa and 170 GPa are used, and the Poisson's ratio for both materials is 0.28 respectively. Additionally, we use a Young's modulus of 130 GPa and a Poisson's ratio of 0.34 for Cu, as in [24].

For the simulations the commercial FE software package ABAQUS (Simulia, Dassault Systemes Simulia Austria GmbH) is used and Python scripting is utilized to perform the optimization and data processing. The FE simulations are coupled to an inverse optimization routine (details see below) in order to determine iteratively the Ramberg-Osgood hardening parameters by comparing the resulting force-displacement curves to the experimental results.

Fig. 1a shows the 2D axisymmetric model of the W—Cu—W tri—layer system on a Si substrate; the y-axis is the symmetry axis. The surface below the nanoindenter and the side face are unconstrained, allowing free movement of the material in lateral direction across the surface. The diamond indenter tip is assumed to be perfectly spherical. Unlike the material below the tip, sliding of the indenter in x-direction is restricted. This means that the tip is only allowed to move in indentation direction, parallel to the y-axis. The force controlled experiment is realized by applying a concentrated force at the tip, denoted by F in Fig. 1a. Finally, the bottom of the model is constrained in y-direction. For the material system 4-node bilinear (CAX4) elements are used. The element size close to the nanoindenter tip is held at a constant size of 50 nm in order to have an appropriate resolution of the region with meaningful deformation, as shown in Fig. 1b. The remainder of the model has a coarse mesh with a maximum element size of 500 nm ensuring sufficiently low computation times for the simulations.

Since residual stresses play an important role in such thin film stacks [27], they should be considered in the model. The locally resolved residual stresses within the thin films have been determined in earlier work by using the ion beam layer removal (ILR) method [24,26,28]. In brief, average compressive residual stress in the order of 1 GPa in the W films and a few 100 MPa tensile residual stresses in the Cu films prevail. For details the reader is referred to [24]. The locally resolved residual stresses for the W—Cu—W tri-layer stack are projected onto the FE model in Fig. 1b. The stresses act perpendicular to the symmetry axis and are prescribed by means of the user subroutine SIGINI. Depending on the distance from the origin in y-direction the residual stresses are induced at the integration points of the elements. The magnitude of the tangential stress is equal to the residual stress component. Thus, a biaxial stress state in the thin films is realized.

In order to find the appropriate material behavior, FE simulations and the above described Ramberg-Osgood stress-strain relation are coupled by an inverse optimization routine. Motivated by the original ideas of Levenberg and Marquardt [29,30], a bounded least-square optimization is used to assess the difference between the results from simulations and the target force-displacement data from the experiments.

Six nanoindentations were carried out on each layer system in order to verify the consistency of the experiments. In Fig. 2, the corresponding indentation curves are presented. As a consequence of the nanocrystalline nature of the probed materials, the experimental curves for the W—Si (solid lines) as well as the W—Cu—W—Si (dashed lines) stack configuration almost coincide and lie within 2% deviation. The maximum indentation depth for the W—Si specimen reaches approximately a fourth of the layer thickness. Moreover, the plastic indentation depth for the W—Cu—W—Si stack is about 20% of the total layer thickness of 1.5 μm. As 10% of film thickness were exceeded, potential substrate effects cannot be excluded [3,31]. However, because the indented volume contains a large number of grains, this effect should not be significant. The difference between the loading segments of the two stack configurations is explained by the softer Cu-interlayer in the W—Cu—W tri-layer system. Thus, the nanoindenter does not penetrate into the pure W-layer as deep as it does into the layer system with Cu. Similar effects have been observed in [32] where titanium and chromium interlayers have been introduced into a W/Cu/SiO_2/Si system.

In order to calculate an appropriate stress-strain relation for the W- and the Cu-layer, the problem is divided into two steps. As a first step the plastic behavior of W in the W—Si specimen is optimized. Secondly, the flow behavior of W is used to characterize the Cu film in the W—Cu—W—Si stack. In Fig. 3a the load-displacement data resulting from the inverse optimization of the flow and hardening behavior is presented. Each optimization in Fig. 3a is fitted to its equivalent indentation curve in Fig. 2 and the corresponding optimization parameters

Download English Version:

https://daneshyari.com/en/article/5443484

Download Persian Version:

https://daneshyari.com/article/5443484

Daneshyari.com