Thin Solid Films 606 (2016) 45–50

Contents lists available at ScienceDirect

Thin Solid Films

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

The electro-mechanical behavior of sputter-deposited Mo thin films on flexible substrates

Tanja Jörg [a,*], Megan J. Cordill [b,c], Robert Franz [a], Oleksandr Glushko [b,c], Jörg Winkler [d], Christian Mitterer [a]

[a] Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben, Franz-Josef-Strasse 18, 8700 Leoben, Austria
[b] Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Montanuniversität Leoben, Jahnstrasse 12, 8700 Leoben, Austria
[c] Department of Materials Physics, Montanuniversität Leoben, Jahnstrasse 12, 8700 Leoben, Austria
[d] Business Unit Coating, PLANSEE SE, Metallwerk-Plansee-Strasse 71, 6600 Reutte, Austria

ARTICLE INFO

Article history:
Received 2 September 2015
Received in revised form 9 February 2016
Accepted 16 March 2016
Available online 19 March 2016

Keywords:
Molybdenum thin film
Magnetron sputtering
Flexible substrate
Electrical resistance
Fragmentation test
Film stress

ABSTRACT

The electro-mechanical performance of Mo thin films grown by dc magnetron sputter deposition on polyimide substrates was investigated. A series of 500 nm thick Mo films was synthesized at different discharge powers to evaluate the effect of deposition conditions on the structure–stress relationship and to correlate the intrinsic properties of the Mo films with their electro-mechanical response. Different in-situ fragmentation tests were performed to assess the crack morphology, the change in electrical resistance and the film stress during straining. A direct relation between the residual stress state of the Mo thin films and the discharge power was noticed as the stress changed from tensile to compressive with increasing discharge power. All Mo films showed brittle fracture when strained in tension and the critical crack onset strain correlated with the residual stress state. In-situ synchrotron diffraction experiments enabled the characterization of the fracture strength, which was unaltered by the discharge power and found to be approximately 1700 MPa for all Mo thin films studied.

© 2016 Elsevier B.V. All rights reserved.

1. Introduction

Flexible electronics are the basis for highly promising technologies with the potential to revolutionize the functionality of electronic devices and applications. In the last years, there has been substantial progress in this area and prototypes are already produced that can be bent, rolled and folded [1,2]. Most applications, such as displays, sensors or radio-frequency identification tags require active components like thin film transistors (TFTs). While organic materials are successfully applied as semiconductor materials within flexible TFTs, their electrical performance is often not sufficient to be used as electrical contacts. Due to their higher conductivity compared to organic materials, thin metal films are usually used as electrodes and interconnects [1–4]. Thus, the ability to bend and stretch metal thin films on polymer substrates without deterioration of their electrical properties is crucial to enable the full commercial exploitation of flexible electronics.

Mo thin films are used as conducting materials in many electronic applications, ranging from source/drain electrodes in TFTs to back contacts in $CuInGaSe_2$-based solar cells. This is based on their unique combination of properties, such as low electrical resistance, high thermal stability and chemical inertness, which makes Mo an excellent material for these devices [5–8]. Although the number of studies investigating the fracture properties of thin films on polymer systems is constantly increasing, including brittle layers like Cr [9,10], Ta [11], Ti [12], indium tin oxide [13–15], SiN_x [16] and SiO_x [17], there is limited information available about the behavior of Mo films on compliant polymer substrates. Bollero et al. [8] reported on electrical and optical properties of Mo films at varying sputter deposition conditions. Blösch et al. [18] applied similar films as back contacts in flexible solar cells and measured the resistivity and the residual stress of the Mo films. A detailed analysis of the electro-mechanical deformation behavior of Mo films on flexible substrates, however, seems to be absent in literature.

The aim of the current study is to determine the influence of deposition parameters on the deformation and fracture behavior of Mo thin films. Therefore, a series of films was synthesized on polyimide substrates by magnetron sputter deposition at different discharge powers. Fragmentation tests were used to observe the failure events in-situ under a laser scanning microscope during uniaxial tensile straining. Furthermore, in-situ fragmentation test were combined with electrical resistance measurements and synchrotron diffraction to correlate the stress evolution in the films with their electro-mechanical response during straining.

2. Experimental details

2.1. Thin film synthesis

Mo thin films were synthesized on 50 μm thick polyimide foils (UBE UPILEX-S 50S) and 380 μm thick Si (100) substrates, which were

* Corresponding author.
E-mail address: tanja.joerg@unileoben.ac.at (T. Jörg).

http://dx.doi.org/10.1016/j.tsf.2016.03.032
0040-6090/© 2016 Elsevier B.V. All rights reserved.

ultrasonically cleaned in ethanol prior to the deposition process. Plasma etching of the substrates and deposition of the Mo films was performed using an industrial-scale in-line dc magnetron sputter deposition system of type FHR Line.600 V [19] equipped with a rotating cylindrical Mo target (∅ 150 mm × 600 mm). The load–lock chamber was used to mount the substrates onto the carrier and after a pressure of less than 1×10^{-4} Pa was reached, a gate valve opened and the substrates were moved into the deposition chamber. The latter was evacuated to a base pressure of less than 3×10^{-5} Pa before the deposition process was started. During plasma etching, the carrier oscillated with a velocity of 20 mm/s for 12 cycles in front of a plasma, which was generated by a glow discharge between an asymmetric electrode configuration and powered by a radio-frequency power supply (13.56 MHz) with a discharge power of 0.8 kW and an Ar gas pressure of 0.32 Pa. For the deposition, the carrier was stationed in a central position opposite to the magnetron at a target-substrate distance of 75 mm.

The substrate temperature is an important parameter necessary to understand thin film growth conditions. Since no additional heating was applied to the system, the temperature increase due to the energy transfer from the plasma to the substrates was measured using a platinum resistance temperature detector (OMEGA Pt100) positioned on the carrier between the substrates. As shown in Fig. 1, the dc power strongly affects the substrate temperature.

A series of Mo thin films was deposited at different discharge powers between 0.5 kW and 10 kW at an Ar working gas pressure of 0.52 Pa. Since the sputter rate increases linearly with the power, the deposition time was reduced accordingly to deposit Mo films of 500 nm thickness (see Table 1). As a consequence of the reduced deposition time, the final substrate temperature was rather constant with a maximum value of about 150 °C. This value is small in relation to the high melting point of Mo being 2623 °C [20].

2.2. Thin film characterization

The microstructure of the Mo films was characterized by X-ray diffraction (XRD) in θ/2θ geometry using a Bruker-AXS D8 Advance diffractometer equipped with Cu-K_α radiation and parallel optics. Rietveld refinement [21] of the XRD patterns to obtain the crystallite size in the out-of-plane direction was performed using the TOPAS V4 software package [22] based on the fundamental parameter approach [23,24]. XRD characterization of residual stress and density was carried out utilizing a Rigaku SmartLab diffractometer equipped with a 5-circle goniometer, Cu-K_α radiation and a parabolic multilayer mirror in the primary beam. Residual stress measurements were performed according to the $\sin^2 \psi$ method by recording the Mo (110) diffraction peak in a ψ range of 0–80° at 10 different ψ angles. The in-plane biaxial residual stress was calculated according to [25,26]. The software ElastiX [27] was used to calculate the X-ray elastic constants for the Mo film from the

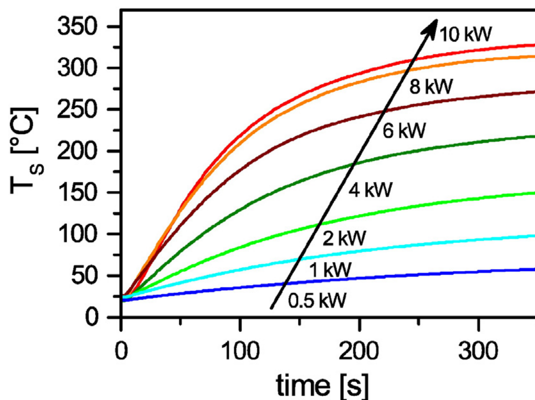

Fig. 1. Substrate temperature T_S as a function of plasma exposure time during deposition of Mo films at different discharge powers.

Table 1
Details of sputter deposition parameters at different discharge powers.

Power [kW]	Deposition rate [nm/s]	Deposition time [s]	Final substrate temperature [°C]
0.5	0.6	882	75
1	1.0	484	108
2	2.6	193	120
4	4.5	110	137
6	6.4	78	151
8	8.1	62	151
10	10.9	46	121

single-crystal elastic constants presuming the Hill model [25,28]. The density was determined by X-ray reflectivity (XRR) measurements of the Mo thin films grown on Si substrates, while all other film characterizations were conducted on polyimide substrates. The Leptos Bruker software was employed for fitting of the critical angle with a genetic algorithm [29,30].

The electrical resistance R of the Mo films was determined by four-point probe analysis with a Keithley 2000 multimeter. The electrical resistivity was then calculated according to:

$$\rho = \frac{R \times t \times W}{L} \quad (1)$$

where t is the film thickness, W the sample width and L the measured length between the contacts.

In-situ fragmentation tests were used to relate intrinsic properties like stress and electrical resistance with the mechanical deformation during straining. Uniaxial tensile tests were performed with a MTS Tyron 250® Universal testing machine, while recording the change in electrical resistance with a four-point probe as described in [31]. For each film, three samples (5 mm × 35 mm) were strained continuously to a maximum elongation of 15% with an initial gauge length and displacement rate of 20 mm and 5 μm/s, respectively. The critical failure strain was defined as the strain at which the measured resistance increased by 10% compared to the resistance of the unloaded sample according to [13]. To link the change in electrical resistance to the deformation behavior in the films, further straining experiments were performed with an Anton Paar TS600 straining device mounted under an Olympus LEXT OLS4100 scanning confocal laser microscope. The samples (7 mm × 35 mm) were strained stepwise with a loading rate of 10 μm/s up to 12% strain and surface images were taken at each step. The crack density was evaluated using a line intercept method and counting the number of cracks intersecting the line across an entire micrograph. Three lines were used for every surface image and the average crack density was calculated as ratio between the average number of cracks visible and the length of the micrograph. The corresponding crack spacing was determined as reciprocal value of the crack density. The failure strain obtained from this experiment was then compared to those from the electrical measurements. To investigate the internal stress development during loading, the same straining device was positioned inside the beamline KMC-2 at the synchrotron BESSY II in Berlin, Germany. A more detailed description of the method is given in an earlier publication [32]. The in-situ diffraction tests were performed with a synchrotron X-ray beam with a wavelength of 0.177 nm and a diameter of 300 μm. Upon loading, diffraction patterns of the (110) Mo peak were continuously measured at four different ψ angles using a VÅNTEC-2000 detector from Bruker-AXS with an exposure time of 5 s. The Mo films were strained continuously with a loading rate of 2 μm/s to a maximum strain of 12% and after a holding time of 5 min unloaded to 0 N. Prior to and after the straining experiment, the residual stress state of the Mo films was analyzed from the (110) reflections of a high resolution scan using 10 different ψ angles. Again, the $\sin^2 \psi$ method was employed to calculate the stress from the lattice strain, applying a Mathematica routine and the Hill model.

Download English Version:

https://daneshyari.com/en/article/1664024

Download Persian Version:

https://daneshyari.com/article/1664024

Daneshyari.com