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The effect of grain and pore sizes on the mechanical behavior of thin Al films deposited under different conditions

E. Ben-David,[a,*] M. Landa,[b] M. Janovská,[b] H. Seiner,[b] O. Gutman,[a] T. Tepper-Faran[c] and D. Shilo[a]

[a] *Department of Mechanical Engineering, Technion-Israel Institute of Technology, 32000 Haifa, Israel*
[b] *Institute of Thermomechanics, Academy of Sciences of Czech Republic, Dolejškova 5, 18200 Prague, Czech Republic*
[c] *Microsystems Department, R&D and Technology Center, MANOR A.D.T Div., Rafael Advanced Defense Systems, Israel*

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Abstract—This paper presents a comprehensive study of the relationships between deposition conditions, microstructure and mechanical behavior in thin aluminum films commonly used in micro and nano-devices. A particular focus is placed on the effect of porosity, which is present in all thin films deposited by evaporation or sputtering techniques. The influences of the deposition temperature on the grain size, pore size and crystallographic texture were characterized by X-ray diffraction and scanning electron microscopy. The mechanical behavior of the films was investigated using four different methods. Each method is suitable for characterizing different properties and for testing the material at different length scales. Nanoindentation was used to study hardness at the level of individual grains; resonant ultrasound spectroscopy was used to measure the elastic moduli and porosity; and bulge and tensile tests were used to study released films under biaxial and uniaxial tensions. Our results demonstrate that even low porosities may have tremendous effects on the mechanical properties and that different methods allow the capture of different aspects of these effects. Therefore, a combination of several methods is required to obtain a comprehensive understanding of the mechanical behavior of a film. It is also shown that porosity with different pore size leads to very different effects on the mechanical behavior.

Keywords: Thin films; Mechanical properties; Tensile tests; Bulge tests; Porosity

1. Introduction

Thin metallic films are commonly employed in Micro/Nano Electro Mechanical Systems (MEMS/NEMS) [1] and are frequently subjected to various mechanical constraints, which may result in plasticity, wear, creep or fatigue [2,3]. Thus, the design of more reliable and sophisticated thin-film-based devices relies on the ability to characterize and adjust the mechanical properties of thin films. A characteristic trait of thin films is that specimen dimensions become comparable with the characteristic length scales that govern the mechanical behavior. Therefore, specimens at the micrometer and submicrometer scales often exhibit a mechanical behavior that may be different from the mechanical behavior of bulk specimens; this phenomenon is often referred to as the “size effect”.

Another important characteristic trait of thin films is that their mechanical properties are strongly dependent on the microstructure, which, in turn, is determined by deposition conditions and further by fabrication processes [4,5]. For example, Espinosa et al. [6] reported significant brittleness and hardness in Au films with thicknesses below 0.5 μm; however, this was not been observed by Emery and Povirk [7], who studied Au films in the same range of thicknesses and used the same deposition method, but with different substrates and fabrication processes.

In pure elemental metallic films, as are commonly used in MEMS and NEMS applications, there are two microstructural characteristics that strongly affect the mechanical properties, i.e. the grain size and porosity. Grain size has a well-known effect on the yield stress and ductility through the Hall–Petch effect [8]. The effect of porosity has been extensively studied in materials produced from powders (e.g. ceramic materials produced by sintering) [9,10] and glasses [11,12]. However, this effect has rarely been studied and is often overlooked in cases of thin metallic films. Previous studies have focused on the porosity [13] and pore size [14] in thin metallic films, but have not considered the influence of porosity on the mechanical properties of the films. In fact, pores are formed in almost all deposition techniques of thin metallic films, and in particular in evaporation and sputtering, which involve phase transformations from vapor to solid [15]. These pores can be extremely small (down to approximately 1 nm) and high in density (approximately 1×10^{17} cm^{-3}) [15–17], which makes them difficult to observe using conventional microscopy.

It is generally argued that porosity results in a decrease in both strength and ductility, and is therefore considered to be

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[*] Corresponding author; e-mail: bderan@tx.technion.ac.il

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undesirable. More specifically, the porosity can affect the mechanical behavior of metallic materials in two different ways. First, pores induce local stress concentrations, which may lead to the initiation of cracks [18,19]. Second, an inhomogeneous distribution of pores results in the development of paths with high pore content, which are high-probability regions for flow localization and fracture [20,21]. It is expected that the first effect will be more prominent in cases of large pores separated by long distances, while the second effect will be more prominent in cases of small pores separated by short distances. Thus, both the overall porosity and the pore size play important roles. In addition to these two effects, elemental gases located within the pores, such as oxygen and hydrogen, may induce local corrosion, which would affect the mechanical properties [22].

Grain size, pore size and overall porosity are strongly influenced by the deposition rate and temperature. Fast deposition rates and low temperatures (i.e. small diffusion lengths) result in small grains and high porosities, whereas slow deposition rates and high temperatures result in larger grains and lower porosities. Due to geometrical considerations, it is expected that the pore size will be comparable to the grain size. Thus, a deposition at a high temperature may result in a lower porosity but larger pore sizes, which may not necessarily be advantageous. The deposition temperature also has a strong influence on the film's residual stress [23].

Another factor that influences the porosity is the crystallographic orientation of the deposited film [24]. In face-centered cubic (fcc) metals, the most densely packed planes are the (1 1 1) planes, followed by the (1 0 0) planes and then the (1 1 0) planes. Therefore, for a given atom flux, grains oriented with the (1 1 1) face parallel to the surface grow more slowly than grains oriented along the (1 0 0) or (1 1 0) faces. A film with a strongly preferred (1 1 1) orientation is expected to have a lower porosity than a film with a strongly preferred (1 0 0) or (1 1 0) orientation. However, films without preferred orientations are expected to have grains which grow at different rates, which may increase the porosity.

There are numerous experimental methods for studying the mechanical behavior and properties of thin films. These methods can be classified and considered according to several different variables. One variable is the size of the probed volume; another, more important variable is the number of grains within the probed volume. Modulus mapping [25] allows the mapping of elastic properties at nanoscale resolutions, but only in a very thin region close to the surface. In nanoindentation [26], the probed volume is usually between tens and hundreds of nanometers in each direction, lengths which span a single or a few grains. When testing films with low porosities, this probed volume may contain just one pore or even no pores. All other techniques typically probe a large number of grains; local effects are thus minimized and the measurement can be considered a representative average of the material.

Another variable, which is important for the analysis of mechanical measurements, is the uniformity of the stress and strain fields. In tensile tests of dog-bone-shaped samples, the stresses and strains are approximately uniform and uniaxial. Thus, stress–strain curves can be plotted, and the mechanical behavior over entire elastic and plastic regimes can be directly observed. In other methods, such as nanoindentation, microbeam bending [27] and bulge testing [28,29], the stress and strain fields are multiaxial and non-uniform. As a result, the overall measured behavior represents integrations over regions which experience different stresses and respond in different (elastic or plastic) manners. In the purely elastic regime, these measurements can be analyzed by assuming a linear Hooke's law. However, as soon as the region yields and exhibits plasticity, the analysis becomes very complicated, especially in cases where the material response in the plastic regime is unknown. When considering the effect of porosity, different loading conditions (e.g. uniaxial vs. multiaxial) result in different pore stress concentration factors and different effects.

Another important variable is the appropriateness of the mechanical testing method for measuring specific properties. For example, nanoindentation is based on semi-phenomenological analysis rules [26], which provide rough evaluations of the Young's modulus and hardness. Tensile tests are suitable for measuring properties related to plasticity (e.g. the yield and ultimate stress values and the strain at failure) but have inherent difficulties in measuring the Young's modulus [30]. In particular, in test methods where the strain is determined by measuring the overall sample elongation, even a small compliance in the sample grippers or a minor slip of the sample with respect to the grippers may result in a significant underestimation of the Young's modulus. Methods based on resonant ultrasound spectroscopy (RUS) [31–33] and surface acoustic waves [34] are specifically designed for measuring the elastic properties of thin films. In these methods, the strain amplitude is on the order of 10^{-6} (for a comparison, the strain amplitude in nanoindentation is approximately 10^{-1}) and the measurement is not affected by rigid body motions. As a result, only elastic strains are measured. The methods provide better accuracy than other mechanical testing methods.

The above discussion indicates that there is no one mechanical testing method which is advantageous over all other methods. Instead, a combination of several methods must be applied to study different mechanical properties under different length scales. This statement is especially relevant when considering a comprehensive study of the effect of porosity.

In this paper, we present a comprehensive study of the influence of deposition conditions on the microstructure and mechanical properties of thin aluminum films. In Section 3, we characterize the grain size, pore size and crystallographic texture of films that have been deposited under different temperatures. In Section 4, we characterize the mechanical behavior of the films using four different methods, which characterize different properties at different length scales. Specifically, nanoindentation is used to study the hardness at the level of individual grains, RUS is used to measure the elastic moduli and estimating the amount of porosity, bulge tests are used to study released films under biaxial tension and tensile tests are used to study released films under uniaxial tension. As will be shown, porosity has various effects on the mechanical behavior and different methods are required for capturing different aspects of these effects.

2. Materials and methods

This study used thin aluminum film samples, which were e-beam evaporated from 99.999% pure aluminum pellets onto 4" silicon (1 0 0) substrates. The silicon substrates consisted of previously deposited external layers of 400 nm