The Mechanical Properties of Electroplated Cu Thin Films
Measured by means of the Bulge Test Technique

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ABSTRACT

The mechanical properties of freestanding electroplated Cu films were determined by measuring the deflection of Si-framed, pressurized membranes. The films were deformed under plane-strain conditions. The pressure-deflection data are converted into stress-strain curves by means of simple analytical formulae. The microstructure of the Cu films was characterized using scanning electron microscopy and x-ray diffraction. The yield stress, Young's modulus, and residual stress were determined as a function of film thickness and microstructure. Both yield stress and Young's modulus increase with decreasing film thickness and correlate well with changes in the microstructure and texture of the films.

INTRODUCTION

The mechanical properties of thin films are of great importance in many engineering applications such as integrated circuits and microelectromechanical systems, since they are directly related to device reliability [1]. In the past, much attention was focused on studying the mechanical properties of thin films of aluminum and its alloys as the material of choice for interconnects in integrated circuits. Recently, Cu has replaced Al in interconnects because it has higher electrical and thermal conductivity, and it has lower rates of electromigration [2]. Thus, the advent of Cu as an interconnect material makes it necessary to achieve the same level of understanding of its mechanical behavior as for Al thin films.

During the past decade, several experimental techniques have been developed for measuring thin film mechanical properties. There are two categories of experimental techniques for testing thin films. The first category focuses on films on substrates and comprises techniques such as nanoindentation and the substrate curvature technique. These techniques require minimal sample preparation, but film and substrate properties need to be decoupled. The other category concentrates on freestanding thin films. Techniques in this category require some sample preparation but it is usually straightforward to extract intrinsic film properties. The bulge test belongs to this category [3,4,5].

In the bulge test, a freestanding thin film is deflected by applying a uniform pressure to the film (Figure 1). The thin film membrane is initially flat. As the membrane deflects, the film experiences an in-plane strain. By measuring the pressure ($P$) and the deflection ($h$) at the center of the membrane, the in-plane stress and strain can be determined. Elastic, plastic, and time-dependent properties of the film can thus be obtained. One of the advantages of the technique is that strain can be changed quickly and isothermally, and that large strains can be imposed. For a
long rectangular membrane, the deformation can be taken as plane strain in the center part of the membrane. The in-plane stress and strain can then be calculated from the pressure-deflection data using the following simple formulae [4]:

\[ \sigma = \frac{Pa^2}{2ht} \quad \text{and} \quad \varepsilon = \varepsilon_0 + \frac{2h^2}{3a^2}, \]  

(1)

where \( t \) is the film thickness, \( 2a \) is the width of the membrane, as shown in Figure 1, and \( \varepsilon_0 \) is residual strain. Equation (1) is accurate for strains less than 1%.

In the work presented here, the mechanical behavior of electroplated copper films was investigated by means of the bulge test. The stress-strain curve, yield stress, and elastic modulus were measured as a function of film thickness. A careful analysis of the microstructure of the films makes it possible to correlate changes in the mechanical behavior with the microstructure of the films.

EXPERIMENTAL DETAILS

A. Experimental setup

A schematic of the bulge test used in this study is shown in Figure 2. The sample to be tested is mechanically clamped onto a sample holder and pressure is applied by pumping de-gased water into the cavity under the film with a syringe pump. The deflection of the film is measured by means of a laser interferometer with a He-Ne laser light source. The displacement resolution is half the wavelength of the light, i.e., 0.3164 \( \mu m \). The pressure is measured with a pressure gauge with a resolution of 0.1 kPa. A maximum pressure of 200 kPa can be applied. The experiment is controlled by computer via a data acquisition system.

![Figure 2. Schematic of bulge test apparatus](image)

B. Sample specification

The Cu films used in this investigation were obtained from Texas Instruments. The films were electroplated onto (100) Si wafers coated with LPCVD SiN\(_x\). A layer of TaN and a thin Cu seed layer were sputter deposited onto the SiN\(_x\) immediately prior to the electroplating process. Three different Cu film thicknesses were studied: 0.885 \( \mu m \), 1.805 \( \mu m \) and 3.015 \( \mu m \).

C. Sample preparation

The Cu membranes were prepared using standard micromachining techniques [4]. After Cu deposition, a layer of benzocyclobutene (BCB) was spin coated onto the Cu films to protect them during subsequent processing. Standard photolithography was used to define long rectangular windows (with aspect ratio 4:1) in the SiN\(_x\) coating on the backside of the substrate with the edges of the rectangles aligned with the \(<110>\) directions in the Si substrate. Freestanding rectangular membranes were created by using a potassium hydroxide based wet etch. Finally, freestanding copper films were obtained by removing the SiN\(_x\) and TaN using...
reactive ion etching and by stripping the protective BCB layer using an organic solvent. This sample preparation process flow can be applied to a wide range of thin films with minor or no changes. The size of the membranes is $2.4 \times 10 \ \text{mm}^2 \ (2a = 2.4 \ \text{mm})$.

**EXPERIMENT AND DISCUSSION**

A. **Experimental data**

![Stress-strain curves](image)

**Figure 3.** Stress-strain curves for copper films of different film thickness.

Stress-strain curves of copper films with three different thicknesses ($t = 0.885 \ \mu\text{m}$, $1.805 \ \mu\text{m}$ and $3.015 \ \mu\text{m}$) are presented in Figure 3. A maximum strain of approximately $0.8\%$ is obtained and the strain hardening behavior of the films is clearly shown. The residual stress in the films is on the order of $100 \ \text{MPa}$. The thicker films have a more gradual transition from elastic to plastic behavior compared to the thinner films.

The yield stress $\sigma_y$ is defined as the stress level at which the plastic strain reaches $0.05\%$. The plane strain modulus, $M = E/(1-\nu^2)$, where $E$ is Young’s modulus and $\nu$ Poisson’s ratio, is obtained from the slopes of the unloading curves. The yield stress and plane strain modulus are plotted as a function of the film thickness in Figure 4. The value of $M$ varies from 130

![Yield stress and plane strain modulus](image)

**Figure 4.** Yield stress and plane strain modulus of copper film of different thickness.
GPa for the thickest films to 156 GPa for the thinnest films, compared with 138 GPa for bulk polycrystalline Cu (based on a Young’s modulus of 121 GPa and Poisson’s ratio of 0.35 [6]). The yield stress of the thickest films is 225 MPa, but it increases to about 300 MPa for the thinnest film, compared to approximately 100 MPa for bulk polycrystalline Cu [7].

B. Microstructure

The microstructure of the Cu films was characterized using SEM and XRD techniques [8]. The average grain size of the Cu films is listed in Table 1.

<table>
<thead>
<tr>
<th>Film Thickness</th>
<th>Average grain size</th>
<th>Fiber orientation</th>
<th>&lt;111&gt;</th>
<th>&lt;110&gt;</th>
<th>&lt;100&gt;</th>
<th>&lt;112&gt;</th>
<th>&lt;023&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.885 µm</td>
<td>2.3 µm</td>
<td>Volume fraction</td>
<td>0.44</td>
<td>0.04</td>
<td>0.04</td>
<td>0.09</td>
<td>0.06</td>
</tr>
<tr>
<td>1.805 µm</td>
<td>3.8 µm</td>
<td>Volume fraction</td>
<td>0.32</td>
<td>0.05</td>
<td>0.09</td>
<td>0.04</td>
<td>0.06</td>
</tr>
<tr>
<td>3.015 µm</td>
<td>5.1 µm</td>
<td>Volume fraction</td>
<td>0.23</td>
<td>0.05</td>
<td>0.10</td>
<td>0.03</td>
<td>0.03</td>
</tr>
</tbody>
</table>

The crystallographic texture of the films was determined by means of x-ray diffraction. All Cu films had a pronounced <111> texture, typical for f.c.c metal films. In all cases, however, the following minor texture components were also detected: <100>, <110>, <112>, and <023> [8]. The volume fractions of the texture components as estimated from the x-ray peak intensities are listed in Table 1. The fraction of the <111> component decreases significantly with increasing film thickness.

C. Strengthening mechanisms

Several mechanisms have been proposed to explain why thin metal films often support higher stresses than their bulk counterparts [1]. The effect of the substrate or passivating layers has been studied by Nix [1]. A simple dislocation channeling model indicates that the yield stress of a thin film, \( \sigma_y \), scales with the inverse of its thickness, \( 1/t \). This theory cannot, however, be applied to the present study since Cu does not form a strong passivating oxide. Nevertheless, a general trend of increasing yield stress with decreasing film thickness is observed experimentally (c.f. Figure 4).

The well-known Hall-Petch relationship, on the other hand, focuses on the strengthening effect of grain boundaries. In the Hall-Petch relation, the yield stress of a given material is proportional to \( d^{1/2} \), where \( d \) is its average grain size. Taking into account other strengthening mechanisms, the Hall-Petch relation can be written as:

\[
\sigma_y = \sigma_0 + k \sqrt{d},
\]

where \( k \) is the Hall-Petch coefficient, and \( \sigma_0 \) is the contribution from other strengthening mechanisms, such as impurities or dislocation interactions.

The average grain size of the Cu films is tabulated in Table 1. As expected, grain size increases with increasing film thickness. In Figure 5, the yield stress of the Cu films is plotted as a function of \( d^{1/2} \). A linear fit of the experimental data, shown as the dashed line in Figure 5, results in the following Hall-Petch relationship:

\[
\sigma_y = 97.0 + 305/\sqrt{d},
\]
where \( d \) and \( \sigma \) have units of \( \mu \text{m} \) and MPa, respectively. In Equation (3), the Hall-Petch coefficient is equal to 0.305 MNm\(^{-3/2}\), which is roughly three times larger than the coefficient of bulk copper \((k=0.112\text{MNm}^{-3/2} [9])\). This value agrees with results from earlier studies [2,10], where the yield stress of Cu thin films was determined through use of the substrate curvature technique. Given that the strains achieved by this technique are relatively small and that the yield stress in the bulge test experiments was defined at a plastic strain of 0.05%, this agreement is not surprising. In bulk materials, however, the yield stress is usually defined at somewhat larger strains to ensure fully plastic conditions.

In order to compare the current experimental results with those for bulk materials, the 0.2% yield stress of the Cu films is also shown in Figure 5. The solid line in this figure represents a fit of the Hall-Petch equation to these data:

\[
\sigma_y = 187 + 189/\sqrt{d},
\]

Equation (4) yields a Hall-Petch coefficient of 0.189 MNm\(^{-3/2}\), which is much closer to the coefficient for bulk copper. From Figure 5, it is clear that the Hall-Petch coefficient depends sensitively on the exact definition of the yield stress.

### D. Relation between film stiffness and crystallographic texture

As shown in Figure 4, the stiffness of the copper films has a strong dependence on film thickness. This is caused by the crystallographic texture of the films and the elastic anisotropy of Cu. With reference to Table 1, the <111> texture component is dominant in all films, and the volume fraction of this component increases with decreasing film thickness. Thus, one would expect the modulus of the Cu films to be a strong function of the volume fraction of the <111> fiber component. This is indeed so as can be verified in Figure 6.

The theoretical plane strain moduli of films with pure <111>, <100>, <110>, <112> and <023> fiber textures have been calculated from the elastic constants of bulk single crystal copper [6]. The results are listed in

![Figure 5. Yield stress of the Cu films as a function of grain size.](image)

![Figure 6. Plane strain modulus as a function of the volume fraction of the <111> texture component.](image)
Table 1. For comparison purposes, the plane strain modulus of polycrystalline Cu, as well as those of films with a pure (111) and (100) fiber textures are presented in Figure 7 together with the experimental data.

It should be noted that the unloading cycles in the stress-strain curves in Figure 3 do not show any hysteresis. This indicates that no anelastic deformation mechanisms are active in these films. This observation should be contrasted with the behavior of Cu films on substrates [11].

**Figure 7.** Plane strain modulus as a function of film thickness.

**SUMMARY**

The bulge test technique has been used to study the mechanical properties of freestanding electroplated Cu films. Both yield strength and stiffness of the films increase with decreasing film thickness. These observations can be attributed to changes in the film microstructure as the thickness is decreases. In particular, the strengthening is due to the grain size, which scales with film thickness. The enhanced stiffness is related to the increased volume fraction of the <111> fiber texture component.

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