Temperature-Dependent Yield Properties of Passivated Aluminum Thin Films on Silicon Wafers

by

Edison C. Chu

Submitted to the Department of Materials Science and Engineering in partial fulfillment of the requirements for the degree of Master of Science in Materials Science and Engineering at the MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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Abstract

A systematic set of stress–temperature measurements using the laser-scanning wafer curvature technique is performed to examine the effect of passivation thickness and passivation material on stress evolution and yield properties of passivated Al films during thermal cycling for twenty cycles. A modification of Stoney’s equation for multiple films on a substrate is utilized to separate the individual stress response of the passivated Al film from experimental measurements of the average stress in the combined film structure of a passivated Al film and the average stress in single films of the passivation materials. Measurements of the stress–temperature response in unpassivated Al films indicate that film strength increases with decreasing film thickness, in agreement with work by other authors. The addition of a passivation layer to an Al film is observed to alter the stress–temperature response in the Al film. These effects include: (i) suppression of the compressive stress drop observed in some unpassivated Al films during thermal cycling, and (ii) a significant increase in the number of thermal cycles required to attain saturation in the stress hysteresis loop, and hence, in microstructural evolution. A preliminary microstructural study shows 3 to 4 grains present through the thickness of a passivated Al film after twenty thermal cycles, indicating that grain growth is suppressed by a passivation layer and suggesting that current theories of the strength of thin films based on a dislocation loop spanning the film thickness may not be valid for passivated films prior to microstructural stabilization. The tensile and compressive flow stresses of passivated Al films are affected by the thickness and type of passivation and change with thermal cycling. The effects of hillock suppression and constrained grain growth in passivated Al films may contribute to the observed differences in flow stress from unpassivated Al films.

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Chapter 1

Introduction

1.1 Stress Development in Aluminum Interconnects

As the semiconductor industry continues to move towards faster and denser integrated circuits through the reduction of device and interconnect dimensions, yield from manufacture and reliability during service have become increasingly limited by failure due to stresses which develop in the metal interconnect lines. These stresses are induced by the large differences in expansion or contraction which develop between the metal interconnects and surrounding materials during changing thermal conditions. During fabrication, an integrated circuit undergoes a number of processing steps each of which occurs at an elevated temperature. Initially, diffusion or implant processes are used to modify the silicon substrate to define the active device structures. These processes are then followed by the deposition and patterning of one or more metallization layers, each separated by a dielectric layer for electrical isolation, to connect the devices. A final passivation layer is then deposited to provide chemical, electrical and mechanical protection. A schematic of such an integrated circuit is presented in Figure 1.1 [1]. Currently, integrated circuits with device dimensions as small as 0.35 μm are being manufactured in industry. Aluminum and aluminum alloys, deposited by sputter deposition, are the most widely used interconnect materials due to their low resistivity, good adherence to oxidized silicon and ease of processing. Common dielectric and passivation materials include silicon dioxide (SiO₂) and silicon nitride (SiN), deposited
by low pressure chemical vapor deposition (LPCVD) or plasma-enhanced chemical vapor deposition (PECVD). SiO$_2$ is often used as an insulating film between metallizations because of its high breakdown strength and reproducible dielectric constant. SiN is widely used as a final passivation layer due to its high hardness, high dielectric constant relative to SiO$_2$, and low permeability to moisture and mobile metal ions. As an integrated circuit is subjected to repeated thermal cycling during thin film deposition processes, stresses develop in the Al interconnects as a direct result of the mismatch in thermal expansion coefficients between the aluminum interconnect material, the silicon substrate, and the dielectric or passivation materials.

![Diagram of layers in an integrated circuit](image)

**Figure 1.1:** A representative schematic of the layers in an integrated circuit. Mismatch due to differences in thermal expansion coefficients between the metallization and surrounding materials results in the generation of stresses in the metal lines during thermal cycling.

Several stress-related phenomena in metal lines have been found to cause the failure of integrated circuits during fabrication and service. During any heating stage of fabrication, the development of large compressive stresses may result in the plastic
deformation of Al and the generation of hillocks, or bumps, on the surfaces of metal films or interconnects [2-3]. These hillocks can cause short circuits if they grow large enough to crack a surrounding dielectric layer and come in contact with an adjacent metal line. Hole formation, induced by tensile stresses in metal films, has also been observed [4-6]. Another reliability problem is stress voiding, which has been found to occur in passivated aluminum lines after processing at elevated temperatures [7-11]. When cooled, interconnects have been found to develop a highly tensile stress state due to the large thermal mismatch with the substrate and surrounding passivation, which may lead to voiding in the metal lines from constrained stress relaxation and failure by the creation of an open circuit. Electromigration is a reliability problem that is known to occur in interconnects carrying electric current during service. Due to the high current density in interconnect lines, metal atoms diffuse in a direction opposite to the electric current as a result of momentum transfer from electrons. Electromigration may cause the development of a stress gradient in the interconnect, leading to failure by voiding [12-14]. Electromigration resistance has been correlated to the yield strength of the interconnect material [15], and as such, it is important to characterize the yield properties and stress state of Al thin films and interconnects during and after thermal processing.

With the goal of eliminating stress-related failures during fabrication and service and the trend towards smaller integrated circuit dimensions, it is apparent that a characterization of the yield properties and stress variation in Al thin films as a function of thickness during thermal cycling is required. Since actual integrated circuits consist of up to 8 or more levels of metal and dielectric layers, it is most important to study the behavior of Al thin films under such passivated states and repeated thermal cycling processing conditions. However, to the author’s knowledge, a systematic study of the effect of a passivation layer on the stress development in Al thin films on Si wafers has not been performed for more than one thermal cycle.

The objective of this work is to systematically study the effect of a passivation layer on the stress development in Al thin films on Si wafers that occurs with repeated thermal cycling. Si wafers with varying Al film thickness, passivation material type and
passivation film thickness are subjected to up to 20 thermal cycles. It is the goal of this work to characterize the yield strength of passivated Al thin films during repeated thermal cycling, so that integrated circuits, materials, and processes may be better designed for increased yield and reliability during fabrication. It is hoped that subsequent electron microscopy analyses of dislocation structures and microstructures on the specimens tested in this experimental work will provide a fundamental micromechanistic understanding of the yield behavior of confined thin metal films.

1.2 Overview

Chapter 2 provides some background on the development of stresses in thin films, a description of the wafer curvature technique used to measure stresses in thin films during thermal cycling, some explanations for the high strength of thin films, and a review of previous work performed on the yield strength and stress–temperature behavior of aluminum thin films during thermal cycling. Chapter 3 presents the materials and experiments used for this study. Chapter 4 provides the experimental results and a discussion of the yield strength and stress behavior of unpassivated aluminum thin films during thermal cycling, as well as the behavior of single films of the passivation materials deposited on silicon wafers. Chapter 5 includes a description of the modification of Stoney’s formula for multiple films on a substrate used to separate the Al film deformation in a trilayer structure and the experimental results and a discussion of the stress behavior of passivated aluminum films on silicon wafers subjected to thermal cycling. Lastly, Chapter 6 presents a summary of the conclusions and findings of this work and provides some suggestions for future research in this area.
Chapter 2

Background and Literature Review

This chapter first presents a basic overview of the mechanics and origins of stresses in thin films, and how these stresses induce elastic bending in the substrate. A description of the wafer curvature technique is given, which allows the study of the plastic deformation and yield properties in thin films during thermal cycling. Next, several explanations are given for the high strength of thin films. Finally, a review of the previous work performed on the yield strength and stress development of unpassivated and passivated aluminum thin films during thermal cycling is presented.

2.1 Mechanics of Stress in Thin Films on Substrates

Consider a thin film separated from a substrate, assuming that the thickness of the substrate is much greater than that of the film, as shown in Figure 2.1. If the film, in a stress-free state with the same dimensions as the substrate, is attached to the substrate, the system will remain in a stress-free state. However, if the dimensions of the film change relative to the substrate, then forces must be applied to the film to equate the dimensions at the interface when the film is attached to the substrate. For example, if the film shrinks relative to the substrate, tensile forces must be applied to attach the film to the substrate. For a film on a wafer, the forces required to maintain compatibility at the interface induces a biaxial stress state in the film. In order to produce stress-free surfaces, the principle of superposition can be used to remove the external force by applying an equal
and opposite force to the end of the film. In order to account for the uncompensated end moments that result, the film-substrate combination will bend elastically, as shown in Figure 2.1. For the case of a film in a compressive stress state, the film-substrate combination will bend in the opposite direction. More detailed and comprehensive treatments of the mechanics of thin films on substrates may be found in the literature [16-18].

Figure 2.1: Dimensional mismatch between the film and substrate is accommodated by the development of a tensile stress in the film and the elastic bending of the substrate [19].

2.2 Origins of Stress in Thin Films on Substrates

As described above, stresses in thin films on substrates can be considered to develop due to the accommodation of a dimensional mismatch between the film and substrate. The total stress in a thin film deposited on a thick, rigid substrate consists of the sum of contributions from an intrinsic stress, $\sigma_i$, and a thermal stress, $\sigma_{th}$:

$$\sigma = \sigma_i + \sigma_{th}.$$  \hspace{1cm} (2.1)
2.2.1 Intrinsic Stresses

Intrinsic stresses develop during the deposition or growth of a thin film on a substrate. These stresses may develop from nonequilibrium film growth, grain growth, defect formation, structural mismatch between film and substrate, densification or phase transformations which produce strains due to volume changes or atomic rearrangement which are constrained by the substrate. Intrinsic stresses are highly dependent on the film deposition conditions, and are generally irreversible. The origins of intrinsic stresses have been reviewed by Doerner and Nix [19] and Kinosita [20]. In soft, low melting point materials such as aluminum, the intrinsic stress is usually minimal, as the high atomic mobility allows the relaxation of stresses during film deposition. However, in hard, high melting point materials which are deposited at relatively low temperatures, the intrinsic stresses can be extremely significant. For example, the compressive stress in plasma-enhanced chemical vapor deposited silicon nitride (Si$_3$N$_4$H) can exceed 1 GPa [21-22].

2.2.2 Thermal Stresses

The thermal stress contribution is a result of the difference in thermal expansion or contraction between the film and substrate induced by heating or cooling from the film deposition temperature. During any variation in temperature from the film deposition temperature, a strain is induced in a thin film on a thick substrate due to the difference in thermal expansion coefficients. The strain in the film, $\varepsilon_f$, can be approximated (if the film and substrate thermal expansion coefficients vary similarly with temperature) as:

$$\varepsilon_{f,\text{thermal}} = (\alpha_s - \alpha_f)\Delta T,$$

(2.2)

where $\alpha_s$ and $\alpha_f$ are the thermal expansion coefficients of the substrate and film respectively, and $\Delta T$ is the temperature change from the film deposition temperature. From Hooke's law, while the film deforms elastically, the average biaxial stress induced in the thin film, $\sigma_f$, due to constraint by the substrate is:
where $E_f$ and $\nu_f$ are the elastic modulus and Poisson's ratio of the film, respectively. If the yield strength of the film is exceeded, plastic deformation will occur. Disregarding the intrinsic stress contribution, the film and substrate are free from thermal stresses at the film deposition temperature, and no substrate curvature is present. In the case when $\alpha_f > \alpha_s$ (such as an aluminum film on a silicon substrate), a tensile stress will be induced in the film when the system is cooled and the substrate will bend elastically, as in Figure 2.1. Likewise, when the system is heated, a compressive stress will be induced in the film when the temperature exceeds the deposition temperature, causing the substrate to bend in the opposite direction. The total amount of substrate bending is due to contributions from intrinsic and thermal stresses.

### 2.3 Stoney’s Equation

Stoney [23], in 1909, first derived the equation which allows the calculation of the total average biaxial stress in a thin film, $\sigma_f$, bonded to a thick, rigid substrate from the bending of the substrate:

$$\sigma_f = \frac{E_s}{6(1-\nu_s)} \frac{t_f^2}{t_f} \left( \frac{1}{R} - \frac{1}{R_0} \right),$$

where $E_s$, $\nu_s$, and $t_s$ are the elastic modulus, Poisson's ratio and thickness of the substrate, respectively, $t_f$ is the thickness of the film, $R_0$ is the radius of curvature of the bare substrate, and $R$ is the measured radius of curvature. From the thin film approximation, the stress in a thin film is determined only by the mismatch between the film and substrate, so that multiple films deposited on the same substrate can each be treated separately [24-30]. Each film contributes independently to the total substrate curvature giving, for the case of two films on the same substrate,
\[ \sigma_{f1}t_{f1} + \sigma_{f2}t_{f2} = -\frac{E_s t_s^2}{6(1-v_s)} \left( \frac{1}{R} - \frac{1}{R_0} \right), \]  

(2.4)

where the subscripts 1 and 2 denote the two different films. Stoney’s formula is the basis of the wafer curvature technique, which allows the determination of the total average stress in a thin film through substrate curvature measurements during thermal cycling.

### 2.4 Wafer Curvature Technique

The substrate curvature technique [24,31-32] offers a simple and accurate method of measuring the average biaxial stress in thin films on substrates during variations in temperature. This technique measures the elastic bending of the substrate that is induced due to the thermal mismatch which develops between a film and substrate with changes in temperature. A schematic of a wafer curvature system is shown in Figure 2.2. During a measurement, a laser beam is scanned and reflected across the surface of a wafer using a rotating mirror and lens system. The reflections are continuously monitored by a position sensitive photodiode (PSD), which measures the deflection of the reflected beam. For a perfectly flat wafer, all reflected beams converge to a single point. For a wafer which exhibits constant curvature, the change in position of the reflected beam as the laser is scanned across the wafer surface is constant and proportional to the wafer curvature. The average biaxial stress in the film can then be calculated using Stoney’s formula, Equation (2.4). As seen from Stoney’s formula, a knowledge of the mechanical properties of the thin film is not needed to determine the thin film stress; only the substrate properties and film thickness need to be known. These curvature measurements can be performed quickly and continuously in-situ during thermal cycling, so that the film stress can be determined as a function of temperature. For many metal films on silicon substrates, strains large enough to induce yielding in the metal may be generated, so that the plastic deformation behavior may be studied. At any given temperature after film yielding, the flow stress, or the stress at which the film would deform plastically at that temperature, may be obtained.
Mirror

Lens

Furnace

Substrate

Substrate with Film

Figure 2.2: A schematic of a laser-scanning wafer curvature measurement system. The deflection of the reflected laser from the incident beam is proportional to the curvature of the wafer.

2.5 The Strength of Thin Films

It has been well established that the mechanical behavior of polycrystalline thin films on a rigid substrate differs considerably from the behavior of bulk materials. Thin films typically possess significantly higher yield strengths than their bulk material counterparts. For example, the yield strength of an Al thin film can be several hundred MPa, while the tensile strength of annealed bulk Al is typically around 70 MPa. In addition, Al thin films display much higher flow stresses at elevated temperatures than bulk Al. The high strength of thin films on substrates has been attributed to the unique microstructure, reduced dimensions and constraint by a rigid substrate.

Several deformation mechanisms have been proposed to account for the plastic deformation behavior in thin films. These include grain boundary and lattice diffusion [33-34], dislocation climb [35], and dislocation glide [36-38]. These mechanisms for stress relaxation in thin films have been reviewed by Murakami et al. [39] and Koleshko et al. [35]. Models involving dislocation glide have been developed to explain the high
strength and the thickness dependence of strength in thin films. The grain size of thin films has been found to be limited by the thickness of the film. The microstructure of an as-deposited film consists of very fine grains, while an annealed film usually consists of one grain through the thickness of the film [25,40-42]. Since the motion of dislocations is impeded by the presence of obstacles such as grain boundaries, yield strength is expected to increase with decreasing grain size and thus, decreasing film thickness. Griffin, et al. [43,44] have observed in experiments involving the bulge test that the Hall-Petch relation, i.e. an inverse-square root dependence of yield strength on grain size, holds in free-standing Al alloy thin films. Chaudhari [36], Nix [37] and Thompson [38] have each developed models of dislocation slip which account for the high strength in thin films by considering the motion of a dislocation pinned at the interfaces of the film. Thompson calculated the flow stress of a polycrystalline thin film by equating the work done in driving a dislocation loop nucleated at the film surface through a cylindrical grain with the energy required to create a dislocation loop along the two sides and the bottom of the grain. The models of Chaudhari and Nix take into account the additional resistance to dislocation motion due to constraint by both a rigid substrate and the presence of a native oxide or other passivation layer at the film surface. Chaudhari minimized the total energy of the system, consisting of the strain energy, dislocation line and dislocation interaction energies, and the surface energy, to determine the maximum strain that can be supported by a film on a substrate. Nix’s model, based on a model for the movement of threading dislocations in epitaxial films developed by Freund [45], proposed the nucleation and motion of a dislocation pinned at the film-substrate and film-native oxide interfaces, shown in Figure 2.3. His derivation equated the work done by the stress in the film with the energies of the dislocations left at the two film interfaces to obtain an expression for the minimum biaxial stress required to move the dislocation in the film. Venkatraman [46] has observed this mechanism in Al-0.5%Cu films during in-situ TEM heating experiments. All three models predict that the film stress varies inversely with the film thickness. The models of Chaudhari and Thompson also indicate a critical thickness below which yielding will not occur and that the yield stress varies inversely with grain size.
Figure 2.3: Schematic of the movement of a dislocation pinned at both film interfaces in an oxidized thin film on a rigid substrate, resulting in the creation of misfit dislocations at the two interfaces [38].

The nucleation and growth of hillocks, or protrusions from a film surface, have been observed to occur during the heat treatment of Al thin films on Si substrates. Hillocks are believed to form due to plastic deformation under compressive stresses by a mechanism which involves the diffusion of atoms along grain boundaries and the film-substrate interface [2,47]. Hillocks have been suppressed in Al films through the addition of a stiff, high strength passivation layer, which prevents the localized plastic deformation which leads to hillocking [48-50].

2.6 Literature Review

The stress development during thermal cycling of unpassivated Al thin films on Si substrates has been studied extensively. Sinha and Sheng [51] were the first to observe that the final room temperature tensile stress in Al films after thermal cycling varies inversely with film thickness. Several other studies have confirmed that tensile flow stresses in Al films during and after thermal cycling increase with decreasing film thickness [25,46,52-55]. Venkatraman [46,52-54] has performed experiments to separate thickness and grain size effects on strengthening in Al thin films on Si substrates. Utilizing a laser reflow technique and a technique involving the growth and dissolution of a barrier anodic oxide to systematically vary the grain size and film thickness in aluminum films, it was determined that decreasing film thickness and decreasing grain
size each lead to higher film strengths. Most studies have confirmed that the hysteresis of
the stress-temperature curve of Al thin films on silicon substrates measured during
thermal cycling essentially saturates after the second thermal cycle (i.e. additional
thermal cycling beyond the second cycle produces stress-temperature curves identical to
the second thermal cycle) [25-26,30,51,55,56]. This reflects a stabilization in the
microstructural evolution of the film and has been attributed to the cessation of
recrystallization, recovery and grain growth which occur during the first thermal cycle.

There has been comparatively little work performed on the stress development in
passivated Al thin films during thermal cycling, and most conclusions have been based
only on experiments involving only a few thermal cycles. Additionally, contradictory
views about the influence of passivation layers on the thermal stress behavior of
aluminum thin films have been presented. Sinha and Sheng [24] concluded that the
deformation of Al thin films was not affected by the presence of a SiN passivation film.
Venkatraman et al. [57] observed very little difference between the stress-temperature
(second cycle) curves obtained for a 0.5 μm thick Al-0.5% Cu film with and without a
500 Å passivation layer. Flinn and Chiang [58-60], using an X-ray diffraction technique
on Al-Si films passivated by tensile, low stress and compressive oxynitride films, have
concluded that the high intrinsic stresses in a passivation layer do not influence the stress
in the metal. Using the modification of Stoney’s formula described earlier to determine
the stress in passivated metal films, other studies have found that the deformation
behavior of Al alloy films during thermal cycling is altered by the confinement of a SiO₂
passivation layer [28-29]. Moske et al. [29], using thermal cycling experiments on Al (2
at.% Cu) passivated by SiO₂, have concluded that confinement by a passivation layer
increases the yield stress of the metal layer and that the measured total average stress over
both films of a passivated film is significantly less than the stress predicted from Stoney’s
equation for multiple films using separate Al and SiO₂ films measurements. Andersen et
al. [29] observed that passivated Al alloy films exhibited less plastic deformation (larger
range of the elastic limit) and both higher tensile and compressive flow stresses during
one thermal cycle when compared to unpassivated films. In contrast, Ho et al. [61] and
Yeo et al. [62] found that a 0.8 μm Al-1 wt.% Cu film passivated with a 0.6 μm SiO₂ film
exhibited more plastic deformation (smaller range of the elastic limit) and a lower final
tensile flow stress during one thermal cycle than an unpassivated film. In the only work
to examine the effect of a passivation layer beyond several thermal cycles, Shen and
Suressh [56] have found that the addition of a SiO$_2$ passivation layer on an Al thin film
bonded to a Si substrate significantly increases the number of thermal cycles required to
attain the saturation of curvature–temperature measurements.
Chapter 3

Materials and Experiments

3.1 Materials

All materials used in this study were prepared at MIT Lincoln Laboratory (Lexington, MA). In order to accommodate a systematic study, 22 different wafers were prepared. Substrates consisted of (100) oriented silicon wafers, which were 520 μm thick and 100 mm in diameter. A 185 Å thick oxide layer was thermally grown on all substrates prior to film deposition in order to prevent interdiffusion between layers and epitaxial growth. Pure Al films were applied by sputter deposition at room temperature, while silicon dioxide (SiO₂) and silicon nitride (SiN) films were produced using plasma-enhanced chemical vapor deposition at 300 °C. Ten bilayer wafers were produced, each consisting of a single film of Al, SiO₂ or SiN on a Si substrate. The Al film thicknesses deposited were 0.19, 0.63, 1.00, and 1.93 μm. The SiO₂ and SiN film thicknesses deposited were 0.11, 0.52 and 0.99 μm. The bilayer wafers will be referred to as Si-Al, Si-SiO₂, and Si-SiN for silicon substrates with single films of Al, SiO₂, and SiN, respectively. Twelve trilayer wafers were prepared, each consisting of an Al film passivated by either a SiO₂ or SiN layer. Trilayer wafers possessed an Al layer with thickness of either 0.19 or 1.00 μm, passivated by either a SiO₂ or SiN film of one of the three thicknesses listed above. The trilayer wafers will be referred to as Si-Al-SiO₂ and Si-Al-SiN for Si substrates with aluminum films passivated by SiO₂ and SiN, respectively. Table 3.1 presents representative bulk mechanical properties for each of these materials for comparative
purposes. Actual thin film properties, especially materials deposited by plasma-enhanced chemical vapor deposition, depend strongly on the processing parameters. Table 3.2 presents a matrix of all wafers that were prepared for this study.

<table>
<thead>
<tr>
<th></th>
<th>(100) Si</th>
<th>Al (bulk)</th>
<th>SiO₂</th>
<th>SiN</th>
</tr>
</thead>
<tbody>
<tr>
<td>E (GPa)</td>
<td>130</td>
<td>70</td>
<td>72</td>
<td>150</td>
</tr>
<tr>
<td>ν</td>
<td>0.28</td>
<td>0.33</td>
<td>0.16</td>
<td>0.25</td>
</tr>
<tr>
<td>α (10⁻⁶/°C)</td>
<td>3.0</td>
<td>23.6</td>
<td>0.55</td>
<td>1.0</td>
</tr>
<tr>
<td>σ_y</td>
<td>--------</td>
<td>70</td>
<td>-------</td>
<td></td>
</tr>
</tbody>
</table>

**Table 3.1**: Representative mechanical properties of materials used in thermal cycling experiments [59].

<table>
<thead>
<tr>
<th></th>
<th>Bilayer Wafers</th>
<th>Trilayer Wafers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># Al (μm) SiO₂ (μm) SiN (μm)</td>
<td># Al (μm) SiO₂ (μm) SiN (μm)</td>
</tr>
<tr>
<td>1</td>
<td>0.19 ----- -----</td>
<td>11 0.19 0.11 -----</td>
</tr>
<tr>
<td>2</td>
<td>0.63 ----- -----</td>
<td>12 0.19 0.52 -----</td>
</tr>
<tr>
<td>3</td>
<td>1.00 ----- -----</td>
<td>13 0.19 1.07 -----</td>
</tr>
<tr>
<td>4</td>
<td>1.93 ----- -----</td>
<td>14 0.19 ----- 0.11</td>
</tr>
<tr>
<td>5</td>
<td>------ 0.11 -----</td>
<td>15 0.19 ----- 0.52</td>
</tr>
<tr>
<td>6</td>
<td>------ 0.52 -----</td>
<td>16 0.19 ----- 0.99</td>
</tr>
<tr>
<td>7</td>
<td>------ 1.07 -----</td>
<td>17 1.00 0.11 -----</td>
</tr>
<tr>
<td>8</td>
<td>------ ----- 0.11</td>
<td>18 1.00 0.52 -----</td>
</tr>
<tr>
<td>9</td>
<td>------ ----- 0.52</td>
<td>19 1.00 1.07 -----</td>
</tr>
<tr>
<td>10</td>
<td>------ ----- 0.99</td>
<td>20 1.00 ----- 0.11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>21 1.00 ----- 0.52</td>
</tr>
<tr>
<td></td>
<td></td>
<td>22 1.00 ----- 0.99</td>
</tr>
</tbody>
</table>

**Table 3.2**: Matrix showing materials and film thicknesses on each wafer used for thermal cycling experiments.

### 3.2 Experiments

The yield behavior of Al thin films was studied through *in-situ* measurements of curvature with changes in temperature using the laser-scanning wafer curvature technique, as described in Chapter 2, during thermal cycling experiments. The system
utilized in this study is commercially available as the Flexus F2900 (Tencor Instruments, Mountain View, CA), which provides an automated, computer-controlled interface to allow *in-situ* curvature measurements over a temperature range from 20 to 900 °C. This system is sensitive to changes in stress with an accuracy of 1 MPa for a 1 μm film on a 525 μm silicon substrate. Each wafer was thermally cycled in air between 25 and 450 °C at nominal heating and cooling rates of 10 °C/minute. Since cooling was performed in air, the actual cooling rate achieved was less than 10 °C/minute as the wafer neared room temperature. At this heating and cooling rate, creep and stress relaxation are insignificant, as shown in ref. [56]. The Si-Al wafers were subjected to three thermal cycles to allow for curvature saturation, while all other wafers were subjected to at least 20 thermal cycles. After the thermal cycling tests were completed, films were removed from the Si substrates through a wet etching process. SiO₂ films were removed using Timetch (Transcene Company, Inc., Rowley, MA), containing ammonium fluoride, which etches at a rate of 1.5 Å/second at room temperature. SiN films were removed using Transetch-N (Transcene Company, Inc., Rowley, MA), a special reagent derived from ortho-phosphoric acid, which etches at a rate of 125 Å/minute at 180 °C. Al films were removed utilizing a solution consisting of 77% phosphoric acid, 20% acetic acid and 3% nitric acid. The curvature of the bare Si wafers were then measured at room temperature, so that the absolute stress in the thin films could be determined.
Chapter 4

Thermal Cycling of Bilayer Materials:
Results and Discussion

This chapter presents the results and discussion of the stress measurements during thermal cycling experiments performed on the bilayer wafers consisting of single films of either Al, SiO$_2$ or SiN on a silicon wafers.

4.1 Aluminum Films on Silicon Wafers

A plot showing the average biaxial film stress as a function of temperature is presented in Figure 4.1 for three thermal cycles for a 1 $\mu$m thick Al thin film on a Si wafer measured using the wafer curvature technique. At room temperature, a small tensile stress is present in the film, made up of the intrinsic stress developed during film growth and the thermal stress from cooling due to any substrate heating that may have occurred during film deposition. Upon heating during the first cycle, the film stress decreases and demonstrates a region of linear dependence with temperature indicative of elastic behavior. The slope of this elastic region is given in the following manner by the derivative of Equation (2.2):

$$\frac{d\sigma_f}{dT} = \frac{E_f}{(1 - v_f)} (\alpha_s - \alpha_f).$$  \hspace{1cm} (4.1)
For Al thin films, this slope is equal to \(-2.15 \text{ MPa/°C}\) as calculated from the material properties from Table 3.1. Dashed lines possessing this slope are superimposed on the elastic regions of deformation in Figure 4.1. The curve crosses the zero stress axis at 50 °C, indicating that slight substrate heating did occur during film deposition. At 90 °C, the film stress begins to deviates from elastic behavior and a large drop in the compressive film stress begins to occur at a temperature of approximately 125 °C and a stress level of 90 MPa. With continued heating, the film softens until the maximum temperature of 450 °C is attained. Upon cooling, the film stress increases and displays another linear region as the film deforms elastically in tension. In this elastic region, the deformation due to compressive stresses during heating is reversed, and the total stress in the film becomes tensile in nature. Deformation is elastic until yielding of the aluminum occurs in tension at about 400 °C. As the film continues to plastically deform, the measured film stress increases with decreasing temperature, since the yield strength of the film increases with decreasing temperature. After completion of the first cycle, the tensile stress in the Al film is significantly higher than the stress at the beginning of the cycle.

The stress drop observed during the first thermal cycle has generally been attributed to recrystallization and grain growth [26,40]. Recrystallization, driven by the energy stored during plastic deformation from the film deposition process, results in the development of dislocation and strain-free grains and is followed by grain growth until the grain size is on the order of film thickness. Stress relaxation occurs during recrystallization due to the rearrangement and removal of dislocations, while grain growth leads to lower stresses due to a decrease in the grain boundary area which can restrict dislocation motion. It has also been suggested that hillocking contributes to this drop in stress, since the stress drop occurs over a small temperature range while grain growth is observed to occur over a wide temperature range, and the extent of grain growth could not account entirely for the significant decrease in stress [46,57,63]. Venkatraman [46] has observed that hillocks and grain growth both initiate at the temperature coinciding with the stress drop.
Figure 4.2 shows the stress development during the first thermal cycle for Al film thicknesses of 0.19, 0.63, 1.00 and 1.93 μm. The compressive stress drop occurs at nearly the same temperature and film stress for all of the Al film thicknesses, except for the thinnest film, where a discernible stress drop is not observed. In a study by Ericson et al. [47], it was observed that hillock nucleation in Al thin films occurred quickly at a specific temperature and continued to grow upon further heating. It was also observed that hillock density and average hillock size increased with film thickness and that very few and small hillocks were observed for the thinnest film (0.25 μm). Chaudhari [64] has calculated a critical film thickness of 0.3 μm based on grain boundary diffusion below which hillocks are not expected to form in lead films. These results would suggest that the stress drop would be larger for thicker films, and that for very thin films there may be little or no drop in stress due to the small extent of hillock formation and development. The results shown in Figure 4.2 correlate with this theory and indicate that hillocking may play a more significant role than grain growth in the stress drop observed during the first thermal cycle. The softening which occurs after the stress drop is most likely due to a combination of grain growth and hillock growth.

As heating begins during the second cycle, a linear elastic region is again observed. However, a significantly smaller stress drop occurs than in the first thermal cycle, indicating that the microstructure of the film has stabilized and that recrystallization and grain growth were completed during the first cycle. The decrease in stress is due to the decrease in yield strength with increasing temperature. The film begins to plastically deform in compression during the second cycle at a temperature of approximately 160 °C. With additional heating, the compressive stress level decreases slightly with temperature due to the decreasing yield strength of Al with increasing temperature. The stress development during cooling of the second cycle is similar to that of the first cycle. Additional thermal cycling past the second cycle produces essentially the same stress–temperature behavior as the second thermal cycle. The “saturation” of this stress–temperature behavior beyond two cycles indicates the stabilization of the film microstructure and the attainment of the ultimate grain size. The stress level after yielding of the film during either heating or cooling is equal to the flow stress of the film,
which is the stress at which the film would begin to yield at a stated temperature. A comprehensive mechanistic and microstructural report of the various regions in a stress-temperature measurement during thermal cycling of Al and Al alloys is given by Gardner and Flinn [40].

Figure 4.3 shows the stabilized stress–temperature curves for all four Al film thicknesses studied. Plastic deformation initiates at higher temperatures and stress levels as the film thickness decreases during heating, and lower temperatures and stress levels as the film thickness decreases during cooling. Thus the extent of plastic deformation that is exhibited over this temperature range decreases with decreasing film thickness, indicating that film strength increases with decreasing film thickness. The thinnest Al film, 0.19 μm, shows very little plastic deformation, and softening is not yet observed in the compressive stress region as in the three other samples. In the two thinnest specimens, it is noted that a steep increase in the tensile flow stress is seen at a temperature near 175 °C. This additional hardening region has been observed mainly in Al alloy films, for which the behavior is attributed to precipitation hardening [40]. However, since the phenomenon is also observed in pure Al films, precipitation hardening cannot be the only explanation. The reason for this behavior may involve the increased interaction of dislocations at lower temperatures [40] or a shift in the dominant deformation mechanism from dislocation glide to diffusional creep [65].

The tensile and compressive flow stresses are plotted at various temperatures as a function of film thickness in Figures 4.4 and 4.5, respectively. The tensile flow stress is observed to increase with decreasing film thickness at a specified temperature, which is the trend which has been well established for the variation of strength of thin films. The tensile stress data of previous studies of the variation of strength with film thickness is also included in Figure 4.4 for comparison. The results correlate well with the results of other authors. The compressive flow stress is also seen to increase with decreasing film thickness, as shown in Figure 4.5. For each of the three thickest films, the compressive flow stress decreases at higher temperatures due to the decreasing film yield strength with increasing temperature. The difference in magnitude of flow stress between the thickest and thinnest samples is much more significant in tension than in compression. In
compression the difference in flow stress at 450 °C is 147 MPa, while in tension the difference in flow stress at 35 °C is 245 MPa. Thus, the thermal cycling experiments performed on Al thin films corroborate that the strength of thin films in both tension and compression increases as film thickness decreases.

4.2 Silicon Dioxide Films on Silicon Wafers

Figure 4.6 shows the plots of average biaxial film stress as a function of temperature for the 0.11, 0.52 and 1.07 μm thick SiO₂ films on Si wafers over twenty thermal cycles measured using the wafer curvature technique. The stress in the 1.07 and 0.52 μm films is compressive over the entire cycling range, while the stress in the 0.11 μm film is always tensile. These values of stress are in the ranges of stress that have been observed for PECVD SiO₂ films [66-67]. Film stress is observed to become more tensile with decreasing film thickness. The average intrinsic stress in each film is approximately equal to the stress at the deposition temperature of 300 °C. The intrinsic stress values are 320, -64 and -106 MPa for the 0.11, 0.52 and 1.07 μm films, respectively. A plot showing the stress–temperature behavior of only the 1.07 μm thick SiO₂ film is presented in Figure 4.7 to describe the salient features. The SiO₂ film is under a compressive stress of 142 MPa in its as-deposited state. The stress which develops during thermal cycling of the SiO₂ film is elastic and no plastic deformation occurs. Since the thermal expansion coefficient of SiO₂ is less than that of Si, the stress in the SiO₂ film increases during heating and decreases during cooling. From the material properties from Table 3.1 and equation 4.1, the slope of the stress-temperature curve should equal 0.21 MPa/°C. From Figures 4.6 and 4.7, the slopes are observed to be about 0.15 MPa/°C, indicating that either the biaxial modulus is lower or the thermal expansion coefficient is higher than those used in the calculation. If the biaxial modulus from the properties listed in Table 3.1 is taken to be correct, the thermal expansion coefficient of this sample would be 1.25 x 10⁻⁶/°C.

It appears from the thermal cycling data that a structural change occurs in the film with continued thermal cycling, since the stress–temperature curve over twenty cycles
does not follow the same linear behavior over twenty thermal cycles, as seen in Figure 4.7. At the end of the heating phase of the first thermal cycle for the 1.07 μm SiO₂ film shown in Figure 4.7, there is a deviation from linear behavior and a slight decrease in stress, so that the total film stress decreases and becomes more compressive. Upon completion of the first thermal cycle, the compressive film stress is larger than the initial as-deposited stress. Figure 4.8 shows a plot of the SiO₂ film stress at 35 °C during the heating phase of each thermal cycle for the three SiO₂ film thicknesses, revealing a systematic increase in compressive film stress as the number of cycles increases for the two thickest films. The stress remains approximately constant over all cycles for the thinnest film. For the 1.07 μm film, the stress decreases for several thermal cycles, after which the stress essentially remains constant at a compressive stress of 164 MPa.

Changes in the stress of SiO₂ films have been previously observed, and have been attributed to the release or absorption of water from the film during thermal treatments. Most studies on the changes in stress in PECVD SiO₂ films have indicated that the stress becomes more tensile with heating [32,67], in contrast to the results of this study. McInerney and Flinn [68] attributed the change in stress during heating to the release of water that had been trapped in the film during the deposition reaction, which leads to a volume decrease and more tensile film stress. It should be noted that in previous studies, very significant increases in stress, up to 300 MPa, were observed during heating, while the decrease in stress observed in this study was found to be only 10 MPa during the first cycle for the 1.07 μm film and 19 MPa for the 0.52 μm film. Bhushan and Muarka [65] have observed a shift in stress towards the compressive direction during heating of SiO₂ films deposited using atmospheric-pressure CVD and LPCVD. It is generally acknowledged that the observed stress behavior of SiO₂ films is highly dependent on the specific deposition conditions during processing and ambient conditions during testing. For the purposes of this study, the thermal cycling measurements indicate that the film stress for SiO₂ films is essentially elastic in nature and may shift over a very small stress range over twenty thermal cycles.
4.3 Silicon Nitride Films on Silicon Wafers

Figure 4.9 shows plots of the average biaxial film stress as a function of temperature for the 0.11, 0.52, and 0.99 μm thick SiN films on Si wafers measured using the wafer curvature technique over twenty thermal cycles. The stress in the 0.99 and 0.52 μm films is always compressive over the entire cycling range, while the stress in the 0.11 μm films is initially compressive and becomes tensile after about 5 thermal cycles. These magnitudes of stress are typical of the values observed in other studies [66, 69-70]. Film stress is observed to increase with decreasing film thickness. The average intrinsic stress values, at the deposition temperature of 300 °C, are -327, -572, and -635 MPa for the 0.11, 0.52, and 0.99 μm films, respectively. A plot showing the stress–temperature behavior for only the 0.99 μm thick SiN film is presented in Figure 4.10. The film is under a compressive stress of 665 MPa in its as-deposited state. Upon heating during the first thermal cycle, the film deforms elastically, as expected. Since the thermal expansion coefficient of SiN is less than that of Si, the stress in the SiN film increases during heating and decreases during cooling. The calculated slope of the elastic interaction, using the properties listed in Table 3.1, is equal to 0.4 MPa/°C. From Figure 4.9, the slope of the stress–temperature curve for each film is closer to 0.15 MPa/°C, indicating either a lower biaxial modulus or higher thermal expansion coefficient than the values used for the calculation. If the biaxial modulus from the properties listed in Table 3.1 is taken to be correct, the thermal expansion coefficient of this sample would be 2.25 x 10⁻⁶/°C. At a temperature near 250 °C, the stress begins to deviate from linearity, and the compressive stress decreases. At 450 °C, the stress in the film is about 123 MPa larger than the initial stress. Upon cooling, the stress initially increases slightly, and then decreases back towards the line of elastic interaction. At 35 °C after the completion of the first thermal cycle, the film stress has increased by about 75 MPa. This stress increase with heating has been commonly observed and has been attributed to the diffusion of hydrogen from the SiN film [32, 69-71]. SiN films deposited by plasma-enhanced chemical vapor deposition typically possess a large amount of hydrogen, typically from 5 to 30 atomic percent, depending on the deposition conditions [69].
presence of hydrogen in the form of Si-H or N-H bonds can be considered as point defects, which must be accommodated by an expansion in the local lattice [71]. The surrounding lattice restrains the expansion, and therefore a compressive stress is generated in the film. The diffusion of hydrogen out of the film during heating relieves this compressive stress, and the compressive film stress decreases. The stress at 35 °C during each thermal cycle is plotted in Figure 4.11 for all three SiN film thicknesses, showing the continued increase in film stress with thermal cycling. The rate of decrease in compressive stress is nearly identical for the 0.99 and 0.52 µm SiN films, while the rate for the 0.19 µm film is significantly larger. It appears that a limiting stress would eventually be achieved with additional thermal cycling for each of the three thicknesses, corresponding to the complete removal hydrogen from the film.
Figure 4.1: A plot of the average biaxial film stress as a function of temperature for a 1 μm Al thin film on a 520 μm Si wafer during three thermal cycles measured using the wafer curvature technique. Solid markers indicate heating; open markers indicate cooling. The dashed lines possess a slope of \(-2.15\) MPa/°C indicating the elastic interaction expected as calculated from the properties of Al and Si.
Figure 4.2: Stress—temperature curves during the first cycle for four Al film thicknesses on Si wafers. Solid markers indicate heating; open markers indicate cooling. For the three thickest films, a drop in stress during heating occurs at nearly the same temperature and stress level. The stress drop is not observed in the thinnest film.
Figure 4.3: Stress–temperature curves of the stabilized cycles for four different Al film thicknesses. Solid markers indicate heating; open markers indicate cooling. The extent of plasticity exhibited over the temperature range decreases with decreasing film thickness, indicating that film strength increases with decreasing film thickness.
Figure 4.4: A plot of the tensile flow stress as a function of $1/\text{thickness}$ at various temperatures for Al thin films from this study and from previous work performed by other authors. Tensile flow stress increases as film thickness decreases.
Figure 4.5: A plot of the compressive flow stress as a function of $1/\text{thickness}$ at various temperatures for Al thin films. Compressive flow stress increases as film thickness decreases.
Figure 4.6: A plot of the average biaxial film stress as a function of temperature for three SiO₂ films of thickness 0.11, 0.52 and 1.07 µm during the first two thermal cycles measured using the wafer curvature technique. The dashed lines possess a slope of 0.15 MPa/°C indicating elastic interaction between the film and wafer.
Figure 4.7: The stress-temperature behavior of the 1.07 μm SiO$_2$ thin film for selected heating phases during twenty thermal cycles. The dashed lines possess a slope of 0.15 MPa/°C indicating elastic interaction between the film and wafer.
Figure 4.8: A plot of the film stress at 35 °C during each cycle for the three SiO₂ film thicknesses measured over twenty thermal cycles. The results show a systematic decrease in film stress over the first few thermal cycles and a compressive limiting stress with continued cycling for the two thickest films.
Figure 4.9: A plot of the average biaxial film stress as a function of temperature for three SiN films of thickness 0.11, 0.52 and 0.99 µm during the first two thermal cycles measured using the wafer curvature technique. The dashed lines possess a slope of 0.15 MPa/°C indicating elastic interaction between the film and wafer.
**Figure 4.10:** The stress–temperature behavior of a 0.99 μm SiN thin film on a Si wafer for selected cooling phases during twenty thermal cycles. The dashed lines possess a slope of 0.15 MPa/°C indicating elastic interaction between the film and wafer.
Figure 4.11: A plot of the film stress at 35 °C during each cycle for the three SiN film thicknesses measured over twenty thermal cycles. The results show the systematic increase in film stress with thermal cycling for all three films.
Chapter 5

Thermal Cycling of Passivated Al Films: Results and Discussion

This chapter presents the results and discussion of the thermal cycling experiments performed on Al films passivated by SiO\textsubscript{2} or SiN. First, plots showing the average biaxial stress over the combined film thickness of the passivated Al structures as measured from the wafer curvature technique are examined and compared. Next, the method based on a modification of Stoney’s equation for extracting the individual stress in the Al film from the trilayer stress measurements is described. The calculated stress development during thermal cycling in the Al films of Si-Al-SiO\textsubscript{2} and Si-Al-SiN wafers is then examined and compared to the stress in unpassivated Al films during thermal cycling.

5.1 Stress Evolution in Trilayer Structures During Thermal Cycling

The stress–temperature measurement obtained directly from the wafer curvature technique for a passivated Al film during thermal cycling provides a measure of the average biaxial stress over the combined thickness of the Al and passivation layers.
5.1.1 Si-1 μm Al-SiO₂ and Si-1 μm Al-SiN Wafers

The evolution of the average biaxial stress in the composite film structure for a Si-1 μm Al-1.07 μm SiO₂ and a Si-1 μm Al-0.99 μm SiN wafer during twenty thermal cycles are shown in Figures 5.1 and 5.2, respectively. It is evident that the addition of a passivation layer to a 1 μm Al film alters the measured stress behavior during thermal cycling. The average stress levels for both specimens are highly compressive, owing to the large compressive stress contributions from the passivation layers. Whereas the stress hysteresis curve of an unpassivated Al film saturates after the second thermal cycle, the addition of a passivation layer significantly increases the number of thermal cycles to saturation. The stress hysteresis curve for the Si-1 μm Al-1.07 μm SiO₂ wafer becomes narrower (the difference between the maximum stresses during heating and cooling decreases) with thermal cycling due to a downward shift towards higher compressive stress levels during cooling, while the stress level during heating shifts slightly towards lower compressive stresses. The elastic slope and elastic tensile stresses during cooling remain constant, while the slope of the stress–temperature curve after tensile yielding decreases with continued thermal cycling, indicating that the plastic deformation response of the Al film is altered. The hysteresis curve for this sample appears to be near saturation after twenty thermal cycles.

The hysteresis loop for the Si-1 μm Al-0.99 μm SiN wafer shown in Figure 5.2 shifts upward towards lower compressive stress levels during both heating and cooling with increased thermal cycling. The hysteresis loop also becomes narrower with increased thermal cycling, since the rate of compressive stress decrease is lower during cooling than for heating. The hysteresis loop for this sample has not saturated after 20 thermal cycles, but appears to be approaching saturation.

In all of the passivated 1 μm Al films, the significant compressive stress drop that was observed for an unpassivated 1 μm Al film during heating in the first cycle of an as-deposited film is not present in the hysteresis curve for the passivated film, indicating that the microstructural change that causes the phenomenon is suppressed in the presence of
the passivation layer. The direction of the shift in stress hysteresis loops observed for each passivation type is consistent with the shift towards higher compressive stresses for the SiO₂ films and the shift towards lower compressive stresses for the SiN films with thermal cycling as described in Chapter 4. The stress evolution described for each passivation type is qualitatively similar for the other passivation thicknesses on 1 µm Al films. With increasing SiO₂ and SiN layer thickness, the hysteresis curves become narrower, as expected due to the increased stress contribution of the stiff passivation layer. This is reflected in the plots of the first cycle for all three passivation thicknesses on 1 µm Al films shown in Figures 5.3 and 5.4, for SiO₂ and SiN passivation materials, respectively. For the 0.11 µm thicknesses of SiO₂ and SiN passivations, it was interesting to note a discernible increase in the slope of the stress-temperature curve during cooling. This behavior was previously observed only in the unpassivated 0.11 µm Al film, and was not observed in the unpassivated 1 µm Al film.

5.1.2 Si-0.19 µm Al-SiO₂ and Si-0.19 µm Al-SiN Wafers

The evolution of the average biaxial stress in the composite film structure for a Si-0.19 µm Al-1.07 µm SiO₂ wafer and a Si-0.19 µm Al-0.99 µm SiN wafer during twenty thermal cycles are shown in Figures 5.5 and 5.6, respectively. In these films, it is evident that the addition of a passivation layer also affects the stress development in 0.19 µm Al films. The stress hysteresis curve for the Si-0.19 µm Al-1.07 µm SiO₂ wafer shifts downward with increased thermal cycling, due to increases in the compressive stress during both heating and cooling, while the stress hysteresis curve for the Si-0.19 µm Al-0.99 µm SiN wafer shifts upward with increased thermal cycling, due to lower compressive stresses during both heating and cooling. The magnitude of the stress levels for both specimens are compressive, due to the large contributions to stress by the thick passivation layers. The hysteresis curves for both passivation types have not saturated after twenty thermal cycles, but appear to be approaching a limit. The hysteresis curves become narrower with increasing passivation thickness, as shown in the curves for the first cycle for the 0.19 µm films passivated by SiO₂ and SiN as shown in Figures 5.7 and
5.8, respectively. The difference between the maximum tensile and compressive stresses remains relatively constant for both passivation types, so that the stress-temperature hysteresis curves do not become narrower with increased thermal cycling. An interesting feature observed in the curve of the Si-0.19 μm Al-1.07 μm SiO₂ wafer is the steep slope decrease followed by a steep slope increase after yielding has occurred during the cooling cycle, which was not observed in the unpassivated 0.19 μm Al film. The steep rise in the stress during cooling of the unpassivated 0.19 μm Al film is apparently suppressed by the presence of a 0.99 μm SiN passivation layer. Again, the direction of the shift for both passivation types is consistent with the changes in stress observed for the single films of the passivation materials from Chapter 4.

5.2 Si-1 Al μm-1.07 μm SiO₂ Microstructure After Thermal Cycling

Figure 5.9 shows a cross-sectional transmission electron microscopy micrograph of the microstructure of a 1 μm Al film passivated by 1.07 μm SiO₂ film after being subjected to twenty thermal cycles. Three to four grains are observed through the thickness of the Al film, with a grain size of approximately 250 nm. It is evident that the Al film does not possess the columnar microstructure consisting of a single grain through the film thickness that is commonly observed in annealed and thermally cycled unpassivated Al thin films. This suggests that the presence of a passivation layer constrains the grain growth in Al thin films during the thermal cycling. This observation is consistent with the observed stress evolution with thermal cycling for a passivated Al film, which indicate that significantly more cycles are required to obtain a saturated hysteresis curve than the 2 to 3 required in an unpassivated Al film, corresponding to microstructure stabilization of the film. The observed microstructure also suggests that the models for thin film strength based on the assumption of a single grain or single threading dislocation across the film thickness is not valid for passivated aluminum films, as long as the microstructure is not columnar.
5.3 Determination of Al Film Stress in Trilayer Wafers

It is apparent that the addition of a passivation layer to an Al film alters the measured stress evolution. It has been noted that several significant changes are observed in the stress response of unpassivated Al films obtained directly from wafer curvature measurements, such as the suppression of both the compressive stress drop in 1 \( \mu m \) Al films and the steep slope increase of the stress curve during cooling of the 0.19 \( \mu m \) Al films. However, it is most important to separate the magnitude of the film stress present in only the Al film to clearly observe the effect of a passivation layer on the deformation behavior and to elicit the yield properties of Al thin films.

In Chapter 4, the stress development during thermal cycling in individual films of SiO\(_2\) and SiN on Si wafers was described. In order to extract the stress in the Al film in a passivated Al structure using the modification of Stoney’s formula for multiple films on a substrate presented in Chapter 2, it must be assumed that each film interacts with the Si wafer separately and that the stress in each film is independent of the stress in the other film. Given this assumption, the stress development in the passivation layer in a passivated Al structure is equivalent to the stress measured for a single film of the passivation material on a wafer. Following the analysis of Andersen et al. [28], the total average stress in the passivated Al structure, \(<\sigma>\), is calculated from a wafer curvature measurement during thermal cycling and is given by:

\[
<\sigma> = -\frac{E_s}{6(1-\nu_s)} \left( \frac{t_s^2}{(t_{Al}+t_p)} \right) \left( \frac{1}{R} - \frac{1}{R_0} \right),
\]

(5.1)

where the \( t_{Al} \) and \( t_p \) denote the Al and passivation film thicknesses, respectively. The total average stress in the passivated Al structure is also given, by the balance of line tension, by:

\[
<\sigma> = \frac{\sigma_{Al} t_{Al} + \sigma_p t_p}{t_{Al} + t_p}.
\]

(5.2)

The stress in the passivated Al film can then be determined by subtracting the average biaxial stress in the passivation material from the total average biaxial stress in the
passivated Al structure, each weighted by the film thickness, by rearranging Equation (5.2):

$$\sigma_{al} = \frac{\langle \sigma \rangle (t_{al} + t_p) - \sigma_p t_p}{t_{al}}. \quad (5.3)$$

Thus, the average stress in a passivated Al film may be determined by making two experimental measurements: the total average stress in a passivated Al film on a silicon wafer and the stress in a single passivation film on a silicon wafer. The stress development during thermal cycling is calculated by performing this calculation at each temperature. Figure 5.10 displays a plot which shows the experimental components and the resultant stress calculation for a 1 \(\mu\)m Al film passivated by a 0.99 \(\mu\)m SiN film.

### 5.4 Stress Evolution in Passivated Al Thin Films During Thermal Cycling

From wafer curvature measurements providing the total average stress over the combined Al and passivation thickness in a trilayer wafer described in Chapter 5.2 and the average stress in a single film of passivation material in a bilayer wafer described in Chapter 4, the stress in the Al film was determined for all passivated Al film wafers, using the calculation method based on Stoney’s equation described above.

#### 5.4.1 Si-1 \(\mu\)m Al-SiO\(_2\) and Si-1 \(\mu\)m Al-SiN Wafers

The calculated evolution of the biaxial stress in the Al films in a Si-1 \(\mu\)m Al-1.07 \(\mu\)m SiO\(_2\) wafer and a Si-1 \(\mu\)m Al-0.99 \(\mu\)m SiN wafer during twenty thermal cycles are shown in Figures 5.11 and 5.12, respectively. The results indicate that the deformation behavior of 1 \(\mu\)m Al thin films is affected by the presence of a passivation layer. In the case of the Al film in the Si-1 \(\mu\)m Al-1.07 \(\mu\)m SiO\(_2\) wafer, the stress hysteresis curve becomes narrower with increased thermal cycling, due to decreasing tensile and compressive flow stresses. For the Al film in the Si-1 \(\mu\)m Al-0.99 \(\mu\)m SiN wafer, the hysteresis curve also becomes narrower due to decreasing compressive flow stress and an essentially constant tensile flow stress near room temperature with increased thermal cycling. For both of
these samples, the initial as-deposited stress calculated for the Al film is near the stress measured for the unpassivated 1 μm Al film. During heating in the first cycle, it is evident that the stress drop that was observed in an unpassivated 1 μm Al film does not occur with the addition of a passivation layer. This indicates that the stress drop has been effectively suppressed by the passivation layer during the first thermal cycle. The suppression of the compressive stress drop during the first thermal cycle is found to occur in the 1 μm Al films with both passivation types and all three passivation thicknesses examined, as exhibited in Figures 5.13 and 5.14.

Figures 5.15 and 5.16 show plots of the calculated tensile flow stresses at 35 °C and compressive flow stresses at 450 °C over twenty thermal cycles for 1 μm Al films passivated with varying thicknesses of SiO₂ and SiN, respectively. Figure 5.15 indicates that both the tensile and compressive flow stresses decrease with thermal cycling for all SiO₂ passivation thicknesses. The plot in Figure 5.16 also indicates an increasingly narrower hysteresis curve with thermal cycling due to the presence of a SiN passivation layer, produced by either a decrease in the tensile flow stress and a constant compressive flow stress with thermal cycling as for the 0.11 μm SiN passivation or a decreasing compressive flow stress and a constant tensile flow stress as with the 0.52 and 1.07 μm passivation thicknesses. Thus, in general, for the passivated 1 μm Al films, the stress hysteresis curve becomes narrower with thermal cycling due to a decrease in either the compressive or tensile flow stress or both. In comparison to the unpassivated Al film, the observed compressive stresses initially are generally higher and tensile stresses lower for Al films passivated by both passivation types.

Figures 5.17 and 5.18 show plots of the tensile flow stress at 35 °C and the compressive flow stress at 450 °C for passivated 1 μm Al films as a function of the passivation thickness after two and after twenty thermal cycles, respectively. Very little systematic behavior can be inferred after two thermal cycles, except that the flow stresses present in the 1 μm Al films passivated by SiO₂ are relatively independent of passivation thickness. After twenty thermal cycles, there is no change or lower values of the flow stresses in both tension and compression for all passivation thicknesses when compared to the levels after two cycles. For passivation by SiO₂, the tensile flow stress after twenty
thermal cycles indicates increases and the compressive flow stress decreases with increasing passivation thickness. For passivation by SiN, there are trends of decreasing tensile flow stress and essentially constant compressive flow stress with increasing passivation thickness.

These observations support the theory that hillocking plays more of a role in the compressive stress drop during the first cycle than grain growth. The development of hillocks due to compressive stress relaxation during a heating cycle may strengthen the film during the subsequent cooling cycle, since the growth of hillocks is not easily reversed. The suppression of hillocks in the Al film would also lead to higher compressive flow stresses, since stress relaxation during compression is more constrained. Thus, if the addition of a passivation layer suppresses hillocks, this would lead to higher compressive stresses and lower tensile flow stresses, or a shift downward in the stress hysteresis. Thus, it is theorized that all passivation layers effectively suppress hillocking causing higher compressive and lower tensile flow stress levels than the unpassivated 1 µm Al film.

In Section 5.2, it was observed that the presence of a 1.07 µm SiO₂ passivation layer constrains the grain growth in a 1 µm Al film. Constrained grain growth would lead to higher initial compressive and tensile flow stresses compared to an unpassivated Al film, due to the smaller grain size. With continued thermal cycling, the grain size continues to increase due to additional grain growth, leading to lower tensile and compressive flow stresses. The continuing grain growth during thermal cycling may contribute to the decrease in the difference between maximum compressive and tensile flow stresses which is observed in passivated 1 µm Al films. These theories and the results suggest that hillocking has more of an effect on strengthening in tension than constrained grain growth.

The shift towards more tensile stresses in the SiN passivation layers with thermal cycling may produce more of a constraint on grain growth with thermal cycling, while the opposite trend for the SiO₂ passivation layers may lead to a decreasing constraint on grain growth. This theory may explain the thickness effects in tension after 20 thermal cycles, where the Al tensile flow stress increases with increasing thickness for SiN passivation
and decreases with SiO₂ passivation, since a thicker SiN layer would restrict grain growth more and a thicker SiO₂ layer would restrict growth less.

5.4.2 Si-0.19 μm Al-SiO₂ and Si-0.19 μm Al-SiN Wafers

The calculated stress evolution in the Al film in a Si-0.19 μm Al-1.07 μm SiO₂ wafer and a Si-0.19 μm Al-0.99 μm SiN wafer are shown in Figures 5.19 and 5.20, respectively. The stress evolution displayed by both specimens are qualitatively similar to the behavior exhibited in the corresponding passivated 1 μm Al films. For both samples, the stress-temperature curve remains elastic over a significantly larger temperature range than the unpassivated film, indicating that the microstructural change which causes a slight relaxation in compressive stress during the first cycle for an unpassivated film is suppressed. For the 0.19 μm Al film passivated by a 1.07 μm SiO₂ film, the hysteresis loop shifts slightly downward towards more compressive stresses and saturates after a few thermal cycles. The hysteresis curves are shifted downwards from the unpassivated 0.19 μm film, corresponding to lower tensile and higher compressive flow strengths. For the calculated stress behavior in the Al film of the Si-0.19 μm Al-0.99 μm SiN wafer, the hysteresis loop during the first thermal cycle starts at a stress near the measured as-deposited stress of the unpassivated 0.19 μm Al film. The tensile flow stress at the end of the first thermal cycle is nearly equal to the flow stress measured for the unpassivated Al film. With continued thermal cycling, the hysteresis loop effectively shifts upward, leading to lower compressive and increasing tensile flow stresses. The hysteresis loop has not saturated after twenty thermal cycles, but appears to be approaching a limit.

Figures 5.21 and 5.22 show the calculated stress-temperature plots for the first thermal cycle for 0.19 μm Al films passivated by different thicknesses of SiO₂ and SiN, respectively. These results indicate that, for the first cycle, the compressive flow stresses are larger and the tensile flow stresses smaller than those measured during the first cycle of the unpassivated 0.19 μm Al film due to the significantly longer elastic range that is exhibited for all passivation thicknesses of both passivation materials.
Figures 5.23 and 5.24 show the compressive flow stresses at 35 °C and the tensile flow stresses at 450 °C over twenty thermal cycles for 0.19 μm Al films passivated with varying thicknesses of SiO₂ and SiN, respectively. For the 0.19 μm Al films passivated with SiO₂, the behavior is very systematic. Both the Al compressive and tensile flow stresses for each passivation thickness remain essentially constant over the twenty thermal cycles. The Al tensile flow stresses for all passivation thicknesses are lower than the measured unpassivated Al film, while the compressive flow stresses for the two thicker passivation layers are higher than the unpassivated Al film. In the case of the Al films passivated by SiN layers, the compressive and tensile flow stresses are observed to shift upwards significantly towards higher tensile flow stresses and lower compressive flow stresses over twenty thermal cycles for the two thicker SiN passivations. These are the only cases in which the tensile flow stress is enhanced by the presence of a passivation layer in comparison to the unpassivated film. For both passivation material types, the difference between the maximum tensile and compressive flow stresses remains relatively constant over twenty thermal cycles. This result may be due to an as-deposited grain size for Al that is similar to the 0.19 μm film thickness, so that the extent of grain growth is small. The flow stress levels in both tension and compression for all passivation thicknesses are higher for the 0.19 μm Al film when compared to the 1 μm Al passivated by SiN, as expected due to the smaller Al film thickness.

Figures 5.25 and 5.26 show the Al tensile flow stresses at 35 °C and the compressive flow stresses at 450 °C as a function of the passivation thickness after 2 and 20 thermal cycles, respectively. For the second thermal cycle, the flow stresses for the Al films passivated with SiN increase in both compression and tension with increasing thickness. For the Al films passivated with SiO₂, the tensile flow strength decreases with increasing thickness, while the compressive flow strength increases with increasing thickness. These trends essentially remain the same after twenty thermal cycles, except at lower tensile and compressive stress levels. The most significant change after twenty thermal cycles is the high tensile flow stress of the Al films passivated with SiN, which increase to values higher than the unpassivated 0.19 μm Al film. The flow stresses in both tension and compression for the 0.19 μm Al films passivated with SiO₂ decrease with thickness.
after twenty thermal cycles. The trends in the tensile flow strength observed after the 20 thermal cycles are identical to those seen in the passivated 1 μm Al films, and tend to correlate with the theory presented.
Figure 5.1: A plot of the average stress evolution in the combined film structure of a 1 μm Al film passivated by a 1.07 μm SiO₂ film during twenty thermal cycles measured using the wafer curvature technique. The stress behavior appears to be near saturation after 20 thermal cycles.
Figure 5.2: A plot of the average stress evolution in the combined film structure of a 1 µm Al film passivated by a 0.99 µm SiN film during twenty thermal cycles measured using the wafer curvature technique. The stress behavior appears to be approaching saturation after 20 thermal cycles.
Figure 5.3: A plot of the change in average stress over the combined film structure as a function of temperature during the first thermal cycle for 1 μm Al films passivated with three different thicknesses of SiO₂.
Figure 5.4: A plot of the change in average stress over the combined film structure as a function of temperature during the first thermal cycle for 1 µm Al films passivated with three different thicknesses of SiN.
Figure 5.5: A plot of the average stress evolution in the combined film structure of a 0.19 μm Al film passivated by a 1.07 μm SiO₂ film during twenty thermal cycles measured using the wafer curvature technique. The stress behavior appears to be near saturation after 20 thermal cycles.
Figure 5.6: A plot of the average stress evolution in the combined film structure of a 0.19 μm Al film passivated by a 0.99 μm SiN film during twenty thermal cycles measured using the wafer curvature technique. The stress behavior appears to be approaching saturation after 20 thermal cycles.
Figure 5.7: A plot of the change in average stress over the combined film structure as a function of temperature during the first thermal cycle for 0.19 μm Al films passivated with three different thicknesses of SiO$_2$. 
Figure 5.8: A plot of the change in average stress over the combined film structure as a function of temperature during the first thermal cycle for 0.19 μm Al films passivated with three different thicknesses of SiN.
Figure 5.9: A cross-sectional transmission electron micrograph showing the microstructure of a 1 μm Al film passivated with 1.07 μm SiO₂ after 20 thermal cycles.
Figure 5.10: A modification of Stoney's equation allows the separation of the stress in the Al film from experimental measurements of the average film stress over the combined film structure of a passivated Al film and the film stress in a single passivation layer. This plot shows the calculation of the stress in a 1 µm Al film passivated by a 0.99 µm SiN layer.
Figure 5.11: The calculated stress evolution in a 1 μm Al film passivated by a 1.07 μm SiO₂ layer. The measured stress evolution of an unpassivated 1 μm Al film over three thermal cycles is included for comparison.
Figure 5.12: The calculated stress evolution in a 1 μm Al film passivated by a 0.99 μm SiN layer. The measured stress evolution of an unpassivated 1 μm Al film over three thermal cycles is included for comparison.
Figure 5.13: A stress–temperature plot of the first thermal cycle for 1 μm Al films passivated with varying thicknesses of SiO₂. The plot displays the suppression of the compressive stress drop that is exhibited in the curve for an unpassivated 1 μm Al film.
Figure 5.14: A stress–temperature plot of the first thermal cycle for 1 μm Al films passivated with varying thicknesses of SiN. The plot displays the suppression of the compressive stress drop that is exhibited in the curve for an unpassivated 1 μm Al film.
Figure 5.15: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) over twenty thermal cycles for 1 µm Al films passivated with various thickness of SiO₂.
Figure 5.16: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) over twenty thermal cycles for 1 µm Al films passivated with various thicknesses of SiN.
Figure 5.17: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) for passivated 1 μm Al films as a function of the passivation thickness during the second thermal cycle.
Figure 5.18: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) for passivated 1 µm Al films as a function of the passivation thickness during the twentieth thermal cycle.
Figure 5.19: The calculated stress evolution in a 0.19 μm Al film passivated by a 1.07 μm SiO₂ layer. The measured stress evolution of an unpassivated 0.19 μm Al film over two thermal cycles is included for comparison.
Figure 5.20: The calculated stress evolution in a 0.19 μm Al film passivated by a 0.99 μm SiN layer. The measured stress evolution of an unpassivated 0.19 μm Al film over two thermal cycles is included for comparison.
Figure 5.21: A stress-temperature plot of the first thermal cycle for 0.19 μm Al films passivated with varying thicknesses of SiO₂.
Figure 5.22: A stress–temperature plot of the first thermal cycle for 0.19 μm Al films passivated with varying thicknesses of SiN.
Figure 5.23: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) over twenty thermal cycles for 0.19 μm Al films passivated with various thicknesses of SiO₂.
Figure 5.24: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) over twenty thermal cycles for 0.19 μm Al films passivated with various thicknesses of SiN.
Figure 5.25: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) for passivated 0.19 μm Al films as a function of the passivation thickness during the second thermal cycle.
Figure 5.26: A plot showing the tensile flow stress at 35 °C (positive stress) and compressive flow stress at 450 °C (negative stress) for passivated 0.19 μm Al films as a function of the passivation thickness during the twentieth thermal cycle.
Chapter 6

Conclusions and Future Work

6.1 Conclusions

Based on the analysis of the experimental measurements performed in this study on unpassivated and passivated Al thin films subjected to thermal cycling, the following conclusions have been made:

- Hillocking may play a more significant role than recovery, recrystallization and grain growth in the compressive stress drop commonly observed during heating in the first cycle of as-deposited Al films.

- Both the tensile and compressive flow strengths of unpassivated Al thin films increase with decreasing film thickness and are of magnitudes larger than the corresponding yield properties of bulk Al, in agreement with the work of other authors.

- Structural changes in films of SiO$_2$ and SiN cause shifts in the measured elastic stress-temperature response for these films during thermal cycling. The stress in SiO$_2$ films is observed to shift towards the compressive direction, while the stress in SiN films shifts towards the tensile direction.

- The addition of a passivation layer to an Al film on a Si wafer alters the stress-temperature response of the Al film during thermal cycling:
  1. The number of thermal cycles required to attain saturation in a passivated Al film is significantly increased to 20 or more thermal cycles.
2. The compressive stress drop observed in unpassivated 1 μm Al films is suppressed.

- A preliminary microstructural study shows that 3 to 4 grains are present through the thickness of a passivated Al film after twenty thermal cycles, indicating that grain growth is suppressed and suggesting that current theories of the strength of thin films based on a threading dislocation loop pinned at the film interfaces may not be valid for passivated films prior to microstructural stabilization.

- The tensile and compressive flow stresses for passivated Al films are affected by the thickness and type of passivation and change with thermal cycling.

- The effects of hillock suppression and constrained grain growth in passivated Al films may contribute to the observed variation in flow stresses with passivation thickness and thermal cycling.

### 6.2 Suggestions for Future Work

The results and conclusions of this work provide a basis for further study of the strength and yield properties of passivated Al thin films. Suggestions for future work include:

- Detailed and systematic observation and characterization of the microstructural evolution during thermal cycling with emphasis on the changes in grain size and shape and dislocation morphology and motion in passivated aluminum thin films, with the goal of correlating the microstructural changes with the stress-temperature behavior and trends measured in this work,

- Examination of the effect of a passivation layer on the stress during thermal cycling in Al alloys and other materials, such as copper, which are commonly used in the microelectronics industry and which may experience additional phenomena which affects strength, such as precipitation hardening, and
• Extension of the investigation of the evolution of yield properties and microstructure during thermal cycling to passivated aluminum line structures, which may incur the additional considerations of a triaxial stress state and anisotropic yield properties.
Bibliography


