Research on Polycrystalline Films for Micro- and Nano-Systems

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Abstract— **Polycrystalline films are used in a wide array of micro- and nano-scale devices, for electronic, mechanical, magnetic, photonic and chemical functions. Increasingly, the properties, performance, and reliability of films in these systems depend on nano-scale structure. In collaborative research with a number of SMA Fellows, Associates, and students, our group is carrying out research focused on probing, modeling and controlling nano-scale structural evolution during both vapor-phase and solid-phase polycrystalline film formation. In particular, high-sensitivity in-situ and real-time stress measurements are being used to study atomic scale forces and to characterize structure formation and evolution at the nano-scale. In other collaborative research, the affects of controlled structure and multi-film architectures on properties, such as piezoelectric characteristics and electromigration-limited reliability, are being explored. Through these interrelated activities, basic principles of the science and engineering of nano-scale materials are emerging.**

Index Terms—**interconnects, MEMS, stress, thin films**

I. INTRODUCTION

Patterned polycrystalline films are basic functional units in a wide array of micro- and nano devices and systems. These patterned films serve as device elements in microelectronic devices and carry current among devices in microelectronic circuits. Polycrystalline films are also used in information storage and in micromagnetic devices for reading and writing information. A single high-performance integrated circuit contains *kilometers* of wiring patterned from polycrystalline films [1], and the densest magnetic storage media consist of patterned polycrystalline elements with dimensions of order 10nm or less [2]. Microelectromechanical devices and systems based on the use of polycrystalline elements are under development for hundreds [3,4] of applications, including many in microphotonics, defense and security systems, and biotechnology.

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In all of these increasingly diverse and demanding applications, the properties, performance, and reliability of polycrystalline elements are governed by the details of the atomic-level processes that occur during their assembly, subsequent processing, and use. In research programs at MIT and in Singapore, we are carrying out basic studies of structure and stress evolution during formation and processing of polycrystalline films. We are also carrying out research on the reliability of polycrystalline films in specific applications, especially as interconnects in integrated circuits. Our goal is to develop experimentally validated models and methodologies for design and optimization of the structure and properties of polycrystalline device and system elements for micro- and nano-systems.

II. CO-EVOLUTION OF STRESS AND STRUCTURE DURING VAPOR-PHASE FORMATION OF POLYCRYSTALLINE FILMS

In vapor deposition, polycrystalline films form through adsorption of atoms on a substrate to form adatoms, clustering of adatoms to form stable single-crystal islands, growth of islands until they impinge on other islands, and coalescence of islands to form continuous films. The average island size at impingement, d_i, is determined by the rate at which stable clusters form (the island nucleation rate I), the rate of island growth G, and the size of the zone around a growing island from which adatoms are collected for growth of the island, δ [5]. These kinetic parameters are, in turn, affected by a number of parameters, but are particularly strongly affected the substrate temperature T_{sub} and the deposition flux, F_{dep} [6]. As continuous films form, the crystal or grain size can further evolve through coarsening processes involving surface self-diffusion or grain boundary motion. Structure evolution can also occur during post-deposition heating, and can be strongly affected by the stress state of the film and by pattering. In the past, we have carried out extensive experimental and theoretical research on structure and stress evolution during post-deposition and post-patterning processing of polycrystalline films. In recent studies we have focused on structure and stress evolution in the very earliest stages of film formation.

In earlier work, in-situ stress measurements have been employed in studies of stress evolution during Volmer-Weber (island) growth of polycrystalline films deposited using evaporative deposition [7-14]. Figure 1 schematically

illustrates the broad classes of behavior that have been observed. Stress evolution during film formation and thickening is illustrated to the left of the dashed line, and stress evolution after deposition has been terminated is illustrated to the right of the dashed line. The classifications of behavior "LM" (low atomic mobility) and "HM" (high atomic mobility) are based on studies of Abermann and co-workers [7, 13] and the behavior classified as "ULM" (ultra-low atomic mobility) is based on more recent research [14-15]. Materials with LM behavior are generally characterized by copious nucleation and limited growth so that the grain size at coalescence is very small (often much less than 10nm). However, as these films thicken, growth occurs epitaxially on the grains already nucleated, and columnar grain structures develop. When growth is terminated, a slight tensile evolution is often observed. Materials that exhibit this behavior are usually thought to have limited (but finite) adatom mobilities. Materials showing HM behavior also often coalesce at film thicknesses of 10nm or less, but generally have sufficient adatom and grain boundary mobilities that equiaxed grains with in-plane grain sizes comparable to the film thickness develop even as the film thickens. When growth is interrupted, significant tensile evolution can occur in these systems. ULM materials are characterized by such low adatom mobilities that amorphous films are formed. When growth is terminated, no further stress evolution occurs. Materials that display LM behavior can display HM behavior at higher deposition temperatures [7, 16], and materials that display HM behavior at room temperature can display LM behavior when deposited at sufficiently low temperatures [17].

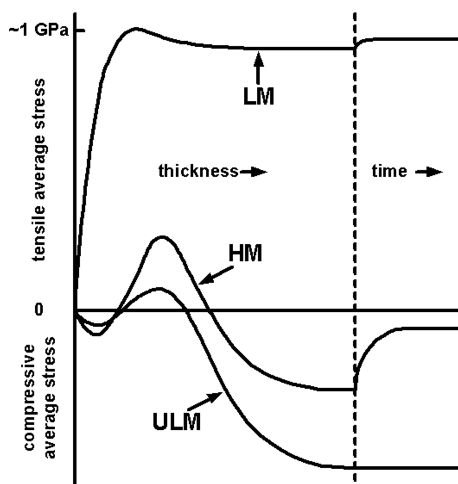

Fig. 1. Schematic diagram of three types of behavior observed in in-situ average stress measurements (average thickness = stress-thickness/thickness) during evaporative deposition of polycrystalline films. Left of the dashed line shows behavior during deposition at a constant rate (so that the ordinate is film thickness) and right of the dashed line shows evolution after growth is interrupted so that the film thickness is constant and the ordinate is time.

Focusing on materials displaying HM behavior, it has been established through experiments in which growth is interrupted for TEM observation that the compressive stress that develops in the earliest, pre-coalescence stage of growth, goes through a maximum and begins to increase as significant island coalescence occurs [10, 16]. In materials that demonstrate HM behavior, the tensile stress rises to a maximum and the stress subsequently decreases as deposition is continued, often resulting in a compressive residual stress. In work carried out in the past year we have focused on the very earliest stages of growth of polycrystalline films, since these are the stages that set the template on which further growth proceeds, and because the properties of films with nano-scale dimensions will be dominated by these processes. We have carried out both experimental and modeling work on the pre-coalescence compressive stress and the tensile stress that develops during coalescence.

It is thought that the tensile stress evolution that occurs during coalescence when islands strain to close the gap between them and form grain boundaries [10, 18, 19]. For very small island sizes at coalescence, this can lead to the very high tensile stresses consistent with LM behavior. The tensile stress that develops under conditions for HM behavior, may be more limited due to larger island sizes at coalescence and due to relatively rapid post-coalescence stress relaxation processes [10]. In previous years we developed models for the dependence of the coalescence stress as a function of the size of the coalescing islands. We carried out finite element analyses in which the total energy (both surface and strain energy) was calculated as a function of the length of grain boundary formed (the zipping distance). The equilibrium zipping distance for which the total energy is minimized could therefore be determined, and from this, the resulting stress could be calculated [10]. Calculated values were lower than previously calculated values [18, 19] by an order of magnitude, but were in better agreement with results from agreements and analytic models subsequently developed [20, 21]. Unlike existing analytic models, the finite element method can be readily modified to account for island-substrate contact angles other than 90^0. We find that changes in the contact angle change both the magnitude of the zipping stress (by as much as an order of magnitude) and the dependence of the zipping stress on the island size and coalescence [20]. This demonstrates the importance of film-substrate interactions in controlling both structure and stress evolution during the formation of polycrystalline films.

The finite element technique for calculation of the coalescence stress outlined above is based on continuum concepts. However, the island sizes at the nano-scale regime at which coalescence often occurs, might be expected to be affected by atomistic details of structures and kinetic processes. To explore the validity of use of continuum models at this atomistic length scale, we have also carried out molecular dynamic simulations of the coalescence process. The results of these studies are described in a separate paper in included in the proceedings [22].

In parallel with modeling studies of the coalescence processes, we are also carrying out experimental studies of coalescence in collaboration with Dr. Mark Yeadon of IMRE. We are studying Ni deposition on both single crystal MgO and amorphous Si_3N_4 substrates. The studies are carried out in MERLION at IMRE, which allows observation during deposition of films in a transmission electron microscope. By comparing observed behavior as a function of substrate

differences and changes in deposition conditions with simulated behavior, it is expected that the relative roles of elastic zipping, and diffusive filling of grain boundaries can be quantified, so that a better understanding of the origins and limits of coalescence stresses will be developed.

The origin of both the pre-coalescence, as well as the post-coalescence compressive stress observed in HM materials is less clear. Abermann *et al* proposed that both states result from a Laplace pressure associated with the surface stress of the islands [23], and Cammarata *et al.* suggested this stress is "locked-in" as the islands grow to larger sizes, due to traction at the island-substrate interface [24]. It is important that a large component of the compressive stress observed in the post-coalescence regime *reversibly* relaxes during growth interruptions [11]. Spaepen has suggested that the component of the post-coalescence compressive stress that reversibly relaxes during growth interruptions results from the trapping of adatoms in non-equilibrium states at the surface of the growing grains [25]. More recently, Chason *et al.* proposed that the post-coalescence compressive stress is associated with the trapping of adatoms in non-equilibrium states in the grain boundaries formed during film growth [26].

To study stress evolution in the pre-coalescence regime in greater detail, we have developed two new methods for making high-sensitivity in-situ stress measurements. First, we designed and fabricate a microelectromechanical device to make stress measurements during evaporative deposition and growth interruptions of Cu films. This device functions through piezoresistance measurements during deposition on (110) oriented single-crystal silicon cantilevers [27]. The cantilevers were patterned from bonded silicon-on-insulator structures and resistors and piezoresistors were formed in the thinned bonded (110) wafer using otherwise conventional microfabrication technology. The in-plane piezoresitivity for (110) layers is highly anisotropic. This allows temperature-compensated piezoresistance measurements through comparisons with resistors with no strain dependence (in a Wheatstone bridge structure). Forces associated with deposition on the beam can be electrically monitored through changes in the piezoresistance of the beam. This allows sensitive in-situ measurements based on electrical output from the deposition system. Unlike curvature-based measurements, this technique is free of critical alignment issues and can also be used when films are deposited on both sides of the beam (as in chemical vapor deposition processes).

A second technique developed for high sensitivity in-situ stress measurements was based on measurements of cantilever deflection using detection of motion of a reflected laser beam (Fig. 2) [28]. Motion detection was accomplished via differential measurements of currents in a closely spaced diode-pair, in much the same way that highly sensitive displacement measurements are made in atomic force microscopes. For in-situ use, the laser and diodes were placed in small, pressure-tight chambers that were rigidly mounted with the cantilever and mirrors on a common frame to create a single unit that could be placed fully within the deposition system. This reduces sensitivity losses associated with alignment drift and vibration. This 'atomic force sensor' allows in-situ measurements of cantilever tip deflections with a resolution of less than 1nm.

Using the atomic force sensor, we have recently characterized stress evolution during and after growth interruptions in the *pre-coalescence* regime [28]. As illustrated in Figures 3a and 3b, we find that, as in the post-coalescence regime, a large component of the compressive stress reversibly relaxes during growth interruptions. We have further found that, as also observed in the post-coalescence regime, the magnitude of the reversibly relaxed stress decreases when deposition is carried out at a lower deposition flux. When stress vs. thickness plots for depositions with pre-coalescence growth interruptions are compared with those for continuous depositions, the onset of the tensile rise associated with island coalescence is seen to be delayed until higher nominal film thicknesses are reached. We believe that this is an indication of the effects of island coarsening associated with growth interruptions. We also observe a striking asymmetry in the kinetics of relaxation at the beginning of a growth interruption, and the build-up to the steady state compressive state once growth is resumed. We find that the steady state stress is recovered after deposition of less than a tenth of a monolayer. This has led us to postulate that the reversible component of the steady state compressive state is associated with the presence of a relatively large number of adatoms during deposition compared to the number that would be present in equilibrium. During growth interruptions the *excess* adatom population present during deposition is accommodated at lattice sites in new or existing clusters, via relatively slow diffusive processes. Resumption of growth quickly leads to steady state adatom population that is the sum of the excess and equilibrium populations. This postulate is consistent with the observed kinetic asymmetry of the stress build-up and relaxation.

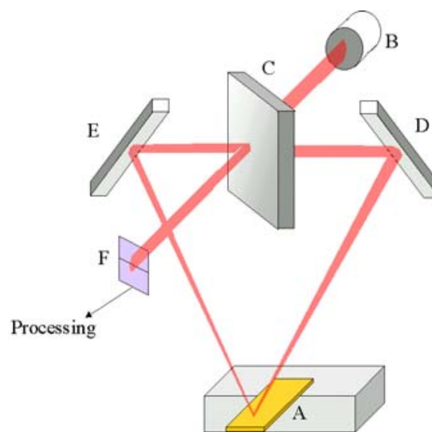

Fig.2. Schematic diagram of an atomic force sensor for high sensitivity in-situ stress measurements. A laser beam (B) is reflected from the backside of the tip of a cantilever (A) while a film is deposited on the other side. The beam is reflected to a diode pair (F), and the path length is increased using mirrors (C, D, and E). Motion of the cantilever tip leads to motion of the reflected spot at the diode pair so that differential measurements of diode currents allows for sensitive measurements (with a resolution of less than 1nm) of the tip deflection. The laser, diodes, cantilever, and mirrors are all rigidly mounted on a common frame, and the entire frame is immersed in the vacuum.

Implicit in this explanation is that individual adatoms cause a compressive stress in the substrate, and that the collective stress associated with a sub-monolayer population of adatoms can be measured using the atomic force sensor in the experiment described above. It has also been argued earlier that the attractive force between adatoms and bulk atoms can cause a displacement of surface atoms as shown in [29-30]. The resulting elastic field can be described as a force dipole, with the resultant displacement field characterized by the elastic Green's tensor for a half-space. The closed-form solution for the radial displacement field of an isotropic surface due to a force-dipole is [29, 31-32]: $u(r) = A (1-\nu^2) / (\pi E r^2)$, where r is the in-plane distance from the point source, ν is the Poisson's ratio, E is Young's modulus, and A is the magnitude of the force-dipole associated with the adatom-surface couple. We have estimated 'A' for homo-adatoms using an embedded atom potential for Cu, and determined that at a coverage of 0.1ML the stress in the plane of the surface was more that 1 GPa [28], as observed in the experiments outlined above.

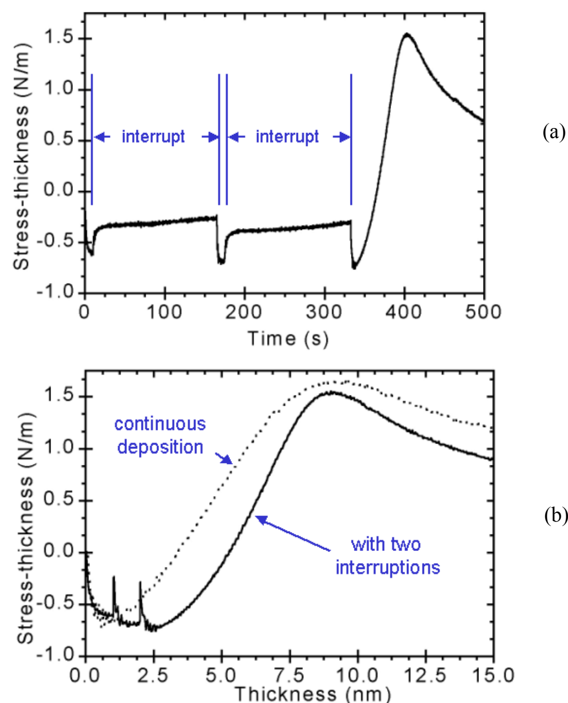

Fig. 3. Data obtained for Cu deposited on Pyrex at room temperature. This data was obtained using a device of the sort illustrated in Figure 3, and shows the effects of growth interruptions in the pre-coalescence regime. A large fraction of the pre-coalescence compressive stress also reversibly relaxes during growth interruptions. a) Shows stress versus time, and b) shows stress vs. thickness for the same experiment (solid line) as well as an experiment carried out without interruptions, but otherwise under the same conditions. During deposition, atoms are deposited at a constant rate so that thickness is proportional to time. Note in Figure a) that the recovery of the steady state stress occurs much more rapidly than the relaxation of stress at the beginning of a growth interruption [28].

If our interpretation of the results outlined above is correct, in-situ stress measurements coupled with models and simulations provide a capability with which adatom coverages during film growth and during growth interruptions can be quantitatively assessed. Studies of stress evolution during growth and growth interruptions coupled with ex-situ STM and in-situ TEM studies will also provide a detailed picture of island formation, coarsening and coalescence processes, that includes atomic kinetic information with spatial and temporal resolution that is not available through microscopy studies alone.

Work in this area has so far been carried out primarily by two MIT students supported in part by SMA. However, a new SMA Ph.D. student has recently begun work in this area, working in collaboration with Dr. Mark Yeadon.

III. Structure Evolution During Growth of Oriented Polycrystalline Films on Amorphous Substrates

Polycrystalline films often develop non-random crystallographic orientations. This can result from energy minimization (e.g., surface, interface, or strain energy) or due to growth velocity anisotropy during film formation or during post-formation structure evolution [33]. Usually, when films are deposited on amorphous substrates, this leads to films with restricted crystallographic texture (specific crystallographic directions normal to the plane of the film), but random in-plane orientations. However, it has recently been discovered ion-bombardment or directional deposition can lead to films with constrained in-plane orientations as well. Recent collaborative research has demonstrated that highly oriented films of PZT (with both a strong texture and in-plane alignment) can be grown on multilayer polycrystalline structures on amorphous silica [34]. Recent research focused on revealing the mechanism of this oriented growth is described in another paper in this proceedings [35].

IV. Structure Evolution During Volmer-Weber Growth of Epitaxial GaN on Si

The Volmer-Weber growth mechanism (island growth) described above in the context of polycrystalline film formation also occurs in epitaxial systems with poor lattice matching. We are studying the coalescence stage of CVD growth of GaN on Si, investigating the role of surface nano-topography in affecting the coalescence processes, misfit accommodation, and the defect density of the resulting polycrystalline film. This work involves an SMA Ph.D. student and will be described in detail in another paper in this proceedings [36].

V. Stress Evolution During Reactive Film Formation

Many important polycrystalline films are formed via solid-state reaction rather than vapor deposition. For example, the polycrystalline metal silicide films used as diffusion barriers to metal silicon contacts and as part of the gate electrode in metal-oxide-semiconductor field effect transistors are formed through reactions of metallic films with silicon. Polycrystalline film formation also proceeds through nucleation, growth and coalescence of crystalline islands. We are using stress measurements made during reactions of nickel

films with single crystal silicon substrates to correlate stress evolution with structure evolution. We are also carrying out experiments on Si wafers subjected to applied stresses in order to determine whether the rate of reaction is modified. This work involves an SMA Ph.D. student and will be described in detail in another paper in this proceedings [37].

VI. Reliability of Polycrystalline micro-Elements

An important application of polycrystalline films is to provide electrical interconnections in integrated circuits. Films are patterned into lines with widths in the deep-submicron range, and lengths ranging from 10 to 1000 microns. These lines are used as wiring to connect the tens of millions of devices in a high performance integrated circuit. The total collective length of these wires in a single integrate circuit is of order 1km. As technology improves so that devices shrink, the number of devices per circuit, and hence the number of wires and the total wire length will further increase. This has occurred at an exponential rate over time, so that the required reliability per unit length of interconnect has increased at an exponential rate. In addition, as the wires themselves shrink, they must carry higher current densities. The chief reliability concern for interconnects is wear-out due electromigration-induced damage. The rate of electromigration-induced damage increases with increasing current density. Consequently, for a given interconnection technology, the reliability decreases at an exponential rate over time. These trends, an exponentially increasing reliability requirement and an exponentially decreasing reliability, drives continuing research and development of technologies that lead to interconnects with higher intrinsic reliability.

The increasing length of interconnect wiring has also lead to an increasing impact of wiring on the performance of integrated circuits. Delays in signal transmission among devices now dominate over delays associated with device switching. This has driven the semiconductor industry to work toward replacement of Al-based interconnects with a technology based on lower resistance Cu-based interconnects.

Electromigration is current-induced self-diffusion of metal atoms. It is this mass transport that causes stress changes that can lead to compressive or tensile failures. Cu has much lower grain boundary and lattice diffusivities than Al, and therefore, can, *in principle*, have much lower rates of electromigration and electromigration-induced damage. However, the reliability of integrated circuit interconnects depends on all aspects of the materials system used, not just the characteristics of the primary electronic conductor. Current Cu-based interconnection technology is based on a materials system in which diffusion at the interfaces of Cu interconnects occurs more rapidly than diffusion along grain boundaries. Copper's reliability advantage over Al is significantly reduced because of this [38]. Also, current Cu-based technology leads to conditions under which the stress required to initiate failure through void nucleation is an order of magnitude lower than for Al. Furthermore, because Cu is not capped by a refractory metal layer that can shunt current around a void, a much smaller void can cause failure in Cu than in Al [39-40]. In addition, because the thin refractory metal liners at the base of Cu-based vias do not reliably block electromigration, a much larger fraction of the kilometers of Cu interconnect are susceptible to electromigration-induced failure than in Al-based systems [40]. Finally, as diffusion barrier liners are made thinner, and low-k and low-modulus dielectrics replace high modulus dielectrics, leakage and extrusions become more likely. While Cu has the *potential* for providing the basis for a high-reliability metallization system, many aspects of the associated Cu metallization technology and materials systems must be changed to realize this needed reliability improvement.

We have a large SMA activity focused on assessing, modeling and improving the reliability of Cu-based IC interconnects. This program involves 2 SMA Ph.D. students and 2 MIT Ph.D. students, and involves 2 Singapore-based SMA Fellows and 1 MIT-based SMA Fellow. The work of the SMA students will be described in other papers in this proceedings [41-42]. The work of the MIT students closely parallels this work [40, 43-45]. These collaborative activities are focused on understanding the influence of film properties in multi-film and multi-material micro-structures in very complex micro-systems. Microelectronic systems are rapidly evolving toward the nano-scale regime in which a detailed understanding and control of the properties of nano-scale films and device element is crucial.

VII. Summary

Polycrystalline films and nano-elements are used in a very wide array of microelectronic, microelectromechanical, micromagnetic, and microphotonic devices and systems. Their performance and reliability in these systems depends critically on their structures and stress state. These characteristics develop in the earliest stages of film formation. As device dimensions continue to shrink into the nanoscale regime, it is becoming increasingly critical to probe, understand and control the nanoscale processes that occur during the assembly of nanoscale materials. Through collaborative research with SMA fellows and students we are developing experimental and modeling tools for the study of nanoscale structural evolution of materials. We are also exploring the influence of nanoscale structure and structure evolution on the properties and reliability of polycrystalline films used in complex micro-systems.

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