INTERFACE EFFECTS ON THE ADHESION OF THIN ALUMINUM FILMS

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ABSTRACT

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Differences in the adhesion and fracture toughness of aluminum films on sapphire due to the presence of controlled contaminants are being investigated. Adhesion is evaluated by use of nanoindentation and continuous scratch tests. A comparison was made of the properties of textured thin films of aluminum (178 to 1890 nm) that were vapor deposited onto (0001) oriented sapphire substrates. A very thin (10 Å) layer of carbon was deposited at the interface of selected samples prior to the vapor deposition of the aluminum. Spalling was observed during continuous scratch testing in specimens with carbon at the interface but not in specimens without carbon at the interface.

INTRODUCTION

Understanding the interaction between thin films and their substrates is being recognized as key to understanding the ultimate reliability of electronic devices which employ this structure. Very thin films of some materials are noted to display mechanical properties different than the bulk parent material. This behavior maybe due to structural defects, the behavior of dislocations in a confined volume, and/or the presence of residual stresses. How these affect the ultimate mechanical properties can be further influenced by the film thickness and the presence of impurities. To gain an understanding of the role of impurities at the interface, this study has developed a deposition chamber in which controlled amounts of contaminants can be introduced prior to physical vapor deposition (PVD) of films. Evaluation of the differences in mechanical properties of the variations of Al on sapphire substrates is being evaluated by use of nanoindentation techniques.

MATERIALS AND PROCEDURE

The effect of contaminants on the mechanical properties of thin films was investigated by introducing 8-10 Å of carbon on (0001) sapphire substrates prior to deposition of thin aluminum films. Prior to deposition, the (0001) substrate surfaces were polished to an optically flat finish and subjected to a mild HCl etch to remove metallic contaminants. This procedure was followed by a 1400 °C anneal in air, a chemical cleaning in ammonium fluoride, de-ionized water rinse and N₂ drying. The samples were then mounted into the analysis chamber of the deposition system.

The separate analysis chamber is connected to the deposition system with a base pressure of 2×10^{-11} torr. The substrate was heated to 250 °C and held for 1 hr to drive off moisture. Evolving species were verified as water vapor by an in-situ quadrupole mass spectrometer. Surface cleanliness was verified by Auger analysis before and after all processes. After characterization, the sample was moved to the ancillary chamber for film deposition. Film thickness was determined in-situ by a quartz crystal deposition monitor and subsequently verified using independent stylus measurements.

Aluminum thin films were deposited by PVD onto the smooth single crystal sapphire substrates at a background pressure of 5×10^{-9} torr. The substrates were not actively heated, but the temperature rose during deposition from an ambient 24 °C to 34 °C due to radiant heating from the source. Carbon was also sputter deposited on selected substrates using a low energy (2 Kev) ion gun as illustrated in Figure 1. The sputtering gas was Ar at a pressure of 5×10^{-5} torr and the sputter target was graphitic carbon. This method was chosen to permit very low deposition rates to be achieved in a controlled fashion.

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Figure 1. Schematic of Carbon Deposition Process at Aluminum-Sapphire Interface

Hardness and elastic moduli properties of the Al on sapphire were determined from indentations made using a Nanoindenter II[®] test system developed by Nano Instruments, Inc. A triangular (Berkovich) pyramid-shape diamond indenter tip is driven into the surface at a constant loading rate of 30 $\mu\text{N/s}$ to depths of 400 nm. Depth positioning of the indenter is determined by a capacitance displacement gauge with a resolution of 0.2 to 0.3 nm. A coil and magnet assembly, located at the top of the loading column, drives the loading column and indenter toward the sample with a force resolution of approximately 0.5 μN . Indentation loads and corresponding depths are continuously recorded throughout each test. Calibrations previously determined for the Berkovich indenter tip were used to calculate the area as a function of indenter depth. Use of this area function and the recorded depth of the indenter allowed calculation of the elastic moduli and hardness properties using the method of Oliver and Pharr [1-3].

The indenter can also be configured with a conical diamond indenter with a 0.6 mm tip radius and a 90° included angle that is simultaneously driven into the films at a rate of 15 nm/s and across the films at 0.5 mm/s. During each test, the normal and tangential loads, the indenter depth, and the lateral position are continuously monitored. From spallation events, the fracture energy and interfacial fracture toughness of the film can be calculated using the elastic approach of Venkataraman, et. al. [4, 5].

RESULTS AND DISCUSSION

All Al films deposited show a (111) texture using x-ray diffraction (XRD) analysis as shown in Figure 2. Hardness values are calculated for the films as shown in Figure 3 and are noted to decrease with increasing film thickness. Hardness values of 1-2 GPa for the 178 nm films are in good agreement with our previous work and that of other researchers [6, 7]. The decrease to hardness values of 0.4 to 0.5 GPa in the 1890 nm thick films approaches the strength of bulk aluminum.

Continuous microscratch testing resulted in spalling of the film with carbon at the interface as shown in Figure 4 indicating contaminants have an important contribution to film adhesion properties. Loads of 2.5 mN appear to trigger the spallations at indenter penetrations of 800 nm into the material, which is well past the interface of the 178 nm thick film. In films without carbon at the film interface, no evidence of spallation was observed.

Figure 2. XRD Analysis of aluminum films on sapphire.

Figure 3. Hardness of Aluminum Films on Sapphire

(a)

(b)

Figure 4. No spallation is observed in a 100 nm scratch into a 178 nm thick film of aluminum as shown in (a). Introduction of a 10 Å layer of carbon at interface results in spallation of the aluminum film as shown in (b).

Differences in the behavior of the films to standard indentation tests were also observed for both thicknesses as illustrated in Figure 5 for the 1890 nm film. Excursions or discontinuities in the loading portion were observed at loads of 0.2 mN in the films without carbon at the interface. The presence of carbon at the interface eliminated this phenomena suggesting that the discontinuities are due to interface effects.

Figure 5. Loading and unloading data from continuous indentation tests.

Similar trends have been observed in whisker pull out tests in which carbon layer formation has been reported to enhance the work of fracture properties [8]. Brennon [9, 10] cited extensive fiber pull out occurring in composites with a 10-40 nm layer of carbon present attributed to debonding within the carbon layer. The effect on shear strength was recently reported by Dehn, et. al [11]. A decrease in adhesion of copper to sapphire was observed by incorporating up to 110 nm thick carbon layers. Our studies indicate this debonding to occur with much thinner layers (10 Å) of carbon.

SUMMARY

- 1) A technique has been demonstrated to place a controlled amount of contaminant at interfaces of thin films.
- 2) Spallations occurred in the aluminum films with carbon at the interface but not in films without the carbon, indicating that contamination has important contribution to film adhesion properties.
- 3) Discontinuities in load vs. displacement data obtained from continuous indentation tests are likely due to interface effects.

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