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## Mechanism of adhesion improvement in ion beam mixed $Cu/SiO_2$

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Adhesion enhancement in metal/ceramic combination is very important in practical application such as micro-electronics. Many techniques are employed to enhance the adhesion between metals and ceramics. Recently ion beam mixing metal/ceramic combination at relatively low temperature. This technique can be used to alter many factors critical to the adhesion such as interfacial roughness, structural barriers, or interfacial stress without modifying the bulk substrate properties, and it can lead to a strong bonding in nonthermodynamic equilibrium conditions. Thus this technique has been used to improve the adhesion significantly even in non-reactive immiscible metal/ceramic systems.

$Cu/SiO_2$ system is known to be a typical non-reactive combination since this system has a relatively high positive heat of reaction more than +20kcal/g.at. In this system, the interfacial reactions may not occur during thermal processing and the copper may coalesce to form an island structure, which lead to the weak adhesion. In this case, the ion beam mixing may be the most appropriate technique to improve the adhesion.

We employed the ion beam mixing technique to enhance the adhesion in $Cu/SiO_2$ system. A combined Rutherford backscattering spectroscopy(RBS), grazing angle X-ray diffraction(GXRD), X-ray photo-electron spectroscopy(XPS) and scratch test results are reported in this study to describe the mixing phenomena, the formation of intermediate layer, and the origin of adhesion enhancement in ion beam mixed $Cu/SiO_2$ system.

The commercially available fused quartz($SiO_2$) substrates were first lightly etched in dilute HF and thoroughly rinsed in deionized water before electron beam evaporation of copper in an oil free vacuum of $2*10^{-7}$ Torr. The thickness of Cu layer was chosen to match with the mean projected range of 80 keV $Ar^+$in Cu layer(35nm). Ion beam mixing was carried out at room temperature, 550K, and 650K. The ion does ranged from $1*10^{15}$ $Ar^+/cm^2$to $2*10^{16}Ar^+/cm^2$ at a typical ion flux of 1.5 $\mu A/cm^{22}$. The working pressure in the target chamber during the ion beam mixing was $2*10^{-6}$ Torr. The amounts of intermixing induced by ion mixing were evaluated with RBS. For RBS observation, the target was tilted to an angle of $60^0$ from the incident direction of 2.4 MeV $He^+$, and the energy of the backscatterd He was analyzed at a laboratory scattering angle of $170^0$ with a detector of 14keV energy resolution.

The adhesion of the Cu films was measured using a standard scratch test. Scratch tester equipped with a $120^0$ Rockwell C diamond indenter with a tip radius of 200μm. This instrument was equipped with an acoustic emission detector. The critical load was measured from the acoustic signal resulting from the coating fracture event which was confirmed by the optical microscope observation. The scratching rate and load rate were 10 mm/min and 10N/mm, respectively.

The formation of intermediate layer at the interface was investigated with GXRD and XPS, GXRD analyses were performd with incident angles ranging from $0.1^0$ to $0.4^0$ to obtain structural information in the ion mixed layer. The XPS data were obtained with Mg Kα radiation and depth profiles were obtained by rastering a 5 keV $Ar^+$ beam over a 5mm diameter surface. We have taken the XPS core level lines of Cu $2p_{3/2}$ (932.4 eV), and Cu $L_3M_{4.5}M_{4.5}$ AES line(334.5eV) to get chemical information in the mixed layer. The pass energy of the hemispherical analyzer were 17.5 eV for the high-resolution studies of the core level, and 44.75 eV for the low-resolution survey analysis.

In summary, a thin Cu layer (35nm) deposited on $SiO_2$ has been mixed with 80 keV $Ar^+$ at room temperature, 550 K and 650 K. Interfacial properties of irrated samples were investigated with Rutherford backscattering spectroscopy, grazing angle X-ray diffraction, X-ray photo-electron spectroscopy and scratch test. Adhesion of Cu film was improved by a factor of 3 at a dose of $1.5*10^{16}Ar^+/cm^2$ by the ion beam mixing at room temperature, while the high temperature ion beam mixing enhanced the adhesion by a factor of 5. The ballistic mixing plays a role on the improvement of adhesion for the room temperature ion mixing and the creation of $Cu_2O$ phase induced by ion beam mixing contributes to the enhancement of adhesion at high temperature.