

On Solid State Reaction of Multilayer Film of Al / Cu / Fe

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Abstract: Recent interest in nano-quasi crystalline Al-Cu-Fe powder is due to the unusual physical properties shown by this material. Report here new findings on multilayer film of Al/ Cu/ Fe deposited on glass substitute. During the course of investigation the composition variation across the thickness of this multilayer (at nano level) film Ar^+ ion sputtering has been observed the formation of alloy film. The composition of this film is found to be close to that reported for nano-quasi crystalline Al /Cu /Fe powder sample. The results of the X- ray photoelectron (XPS) and EDX studies are reported.

Keywords: Al/Cu/Fe ; thin film multilayer; nano quasi crystal ; XPS; EDX.

1. Introduction

Barua et al¹ have studied surface chemical properties of nano quasi crystalline powders of Al – Cu -Fe crystalline by the ball milling technique using XPS technique. It has been shown that composition of nano quasi crystalline phase is (Al: Cu: Fe) is 56.4: 23.4: 20.2. Quasi crystalline properties of these powder had been verified by X-ray diffraction and transmission electron microscopy studies². Earlier works on thin film of Al-Cu-Fe reported is, in true sense, not very thin³. It is now well established that film properties change with thickness of the film. I report here my findings of thin film of Al/Cu/Fe multilayers where thickness of each layer is 500 Å⁰. The aim of the present report is to show that ion sputtering causes the mixing and results in producing a stable alloy whose composition is close to the composition obtained for nano powders reported by Barua et al¹.

2. Experimental

Multilayer films of Al/Cu/Fe each layers of thickness 500 Å⁰ is deposited on glass substrate by physical vapor deposition technique. All the deposition is done at a pressure of 10⁻⁶ torr. Sequential deposition is done by breaking the vacuum in Nitrogen atmosphere before depositing the next layer. Thickness of each layer is determined by viewing the cross-section under SEM. XPS data are recorded in a VGESCA LAB MKII

Spectrometer using Mg K_α X ray source. X ray source was operated at 12 kv and 20 μA filament current. Wide scan and narrow scan spectra was recorded with a pass energy at 50 ev and 20 ev respectively. These settings gave the instrumental resolution as 1ev and 0.4 ev respectively. Quantitative estimation is done by measuring the areas under the narrow scan spectra using Shirley method of background selection. EDX measurements were done with JEOL JSM 5800.

3. Results and Discussions

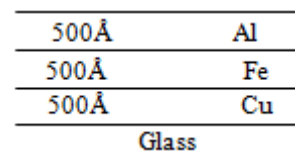


Figure 1: Gives the schematic diagram of the multilayer film studied.

Initial attempts were to measure the concentration across the thickness of the film. This is usually done by removing layers parallel to the surface by low energy Ar^+ ion sputtering. XPS studies are done at each of the freshly exposed surface. Thickness of layers removed could be determined by knowing the sputter rate of the elements. In the present investigation no attempt has been made to determine the precise value of the thickness. Normally, ion beam of uniform density is made to incident on the surface of the film (shown in fig.2) when the sample surface is at ground potential. This is normally done by earthing the sample surface by silver paint.

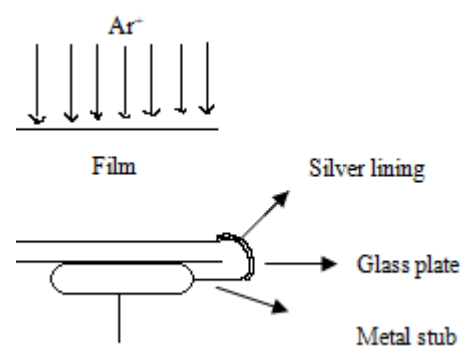


Figure 2

Since the substrate glass is non conducting the film surface in under study was made ground by connecting silver paste to the metal stub used for mounting sample for XPS studies. However because of improper grounding, it was observed that the film surface was etched unevenly. Figure 3. describes the nature of ion beam incident on the sample.

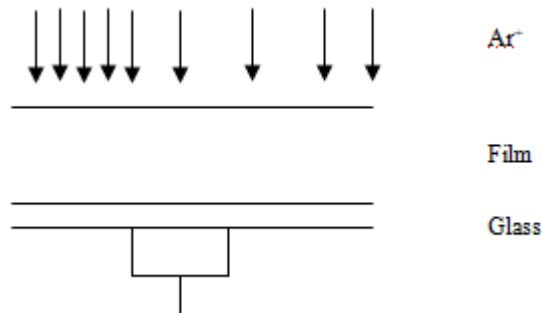


Figure 3

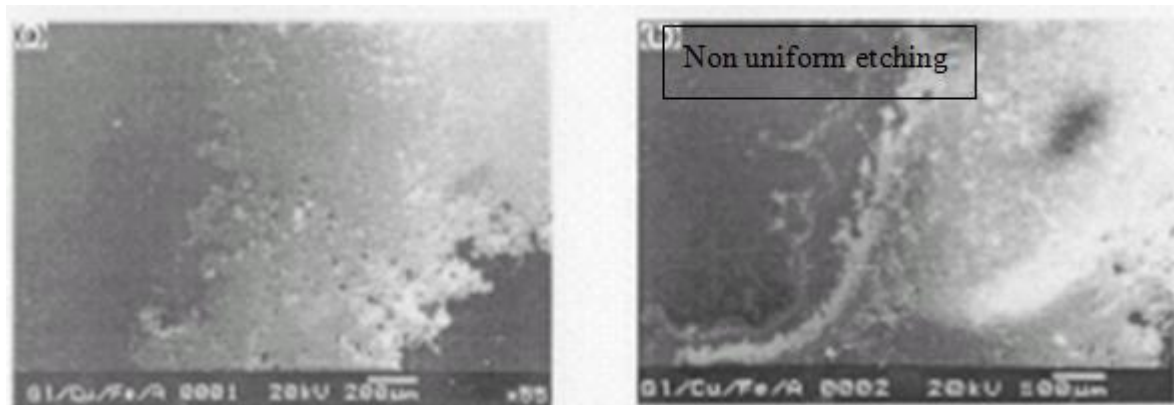
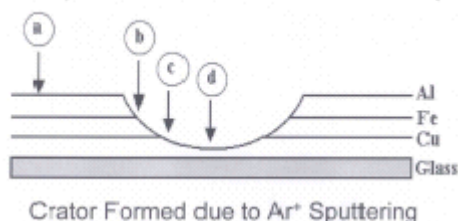


Figure 4 Figure 5

Figures 4. & 5. give the SEM micrographs of the crator formed due to non-uniform sputtering.

The film composition are measured by EDX. Table 1. gives the results of EDX measurements.

Schematic representation of Crater due to Ar⁺ Sputtering



Crator Formed due to Ar⁺ Sputtering

Figure 6

Figure 6. gives the schematic diagram of the crater and explain the geometrical nature. abcd are the regions where

Table 1: EDX composition

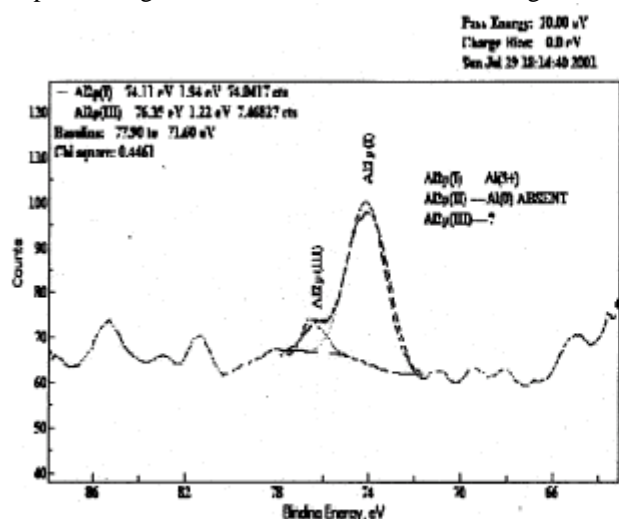
Locations	Al	Cu	Fe	Si
(a)	26	12	4.6	56
(b)	3.2	6.79	0.89	89
(c)	3	2.21	0.68	93
(d)	3.01	0.69	0.36	95

At (d) => Relation concentration of AL, Cu and Fe:

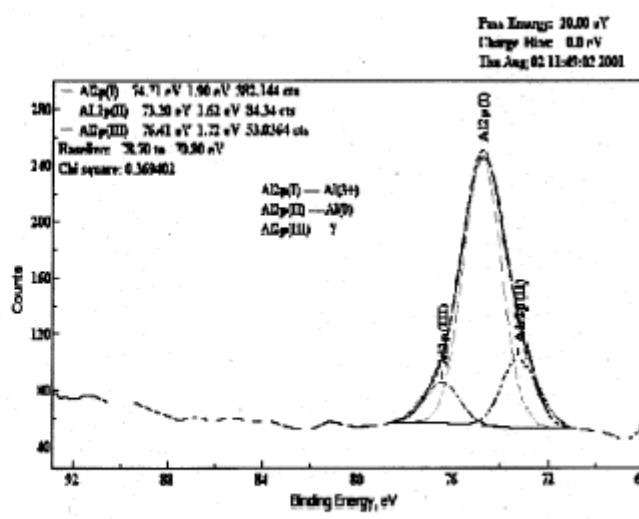
Al-74%

Cu- 16.9%

Fe-8.86%



Al 2p



Al 2p

Figure 7: (a & b)

Figures 7. (a, b) show the narrow scan spectra of Al 2p peaks from top of the film before the formation of the crater and Fig. 7b. gives the Al 2p peak after the crater is formed. These spectra also resolved to extract the chemical state of Al. Al 2p (I) component is due to oxide and Al 2p (III) component may be due to Cu 3p photoelectron line. It may also be due to the formation of some aluminum hydroxide. On comparing the spectra with 7b., it is observed that there are three distinct states. Oxide [Al 2p (I)], metallic [Al 2p (II)] and the third component at higher B.E. which is also present in 7a. Since in the wide scan it is observed that the presence of Cu, the contribution may also be due to Cu 3p. It is revealed from the studies with nano quasi crystalline Al-Fe- Cu, that Al (II) component is due to Al, forming the nano quasi crystalline state (1). In EDX of samples, all the Al contents, it may be that the relative concentration shows under table 1., gives the total composition of Al, out of which a small fraction is forming the nano phase. It is attributed that the presence of Al (II) component is due to metallic Al forming a nano phase alloy.

4. Conclusion

Ion mixing of thin multilayer film may be another cause of obtaining nano phase.

References

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