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SEM Microstructure of Cooper Films in the SSC Beam Tube

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Objective

The objective of this work was to characterize the microstructure of the following copper films from Silvex using SEM.

(1) Cu-SI-1 Cu plated, 100μm, heat treated to 260°C for 1 hour

(2) Cu-SI-2 Cu plated, 100μm, heat treated to 190°C for 2 hours

(3) CuG-SI-1 Cu plated, 100μm, Au 2μm, heat treated to 190°C for 2 hours

Results

The three samples were characterized by SEM and X-ray diffraction method. The results are shown as below:

1. SEM Morphology

Figure 1-a, b and c show the free surface morphology of the three samples. As can be seen from these photos, the cloud structures were observed on the film surfaces. However, these clouds do not exactly show the grain size of copper or gold films. In fact, it shows the surface roughness caused by the columnar structure during film growth. This can be seen very clearly from figure 4-a, a cross section of sample CuG-SI-2. The surface roughness of this sample is exactly the same as we have seen from the free surface of the films.

We were unable to evaluate the grain size of the Cu-SI-1 and Cu-SI-2 samples from figure 1-a and b. However, the grain size of the CuG-SI-1 was evaluated and the SEM photograph of this sample is shown in figure 1-c. The large clouds of gold film corresponds to the surface roughness of copper film. The average grain size as defined from the grains of the gold layer within the clouds was 3μm.

Since the actual grain size and the porosity of copper films could not be observed from free surface, we had to look at the cross section of the films and understand the grain size change due to different heat treatments.

Figure 2-a shows the cross section of Cu-SI-1 sample which was heat treated at 260°C for 1 hour. The grain growth is more significant close to the surface and shows no preferred orientation. The average grain size of these random oriented grains is approximately 7μm and they are dense as show on figure 2-b. On the other hand, the grains near the stainless steel substrate show a preferred orientation along the film growth direction. It is obvious that the pore concentration near the stainless steel-copper interface is higher than that of random oriented grains near the surface.

Figure 3-a shows the sample heat treated at 190°C for 2 hours. It can be seen that the grains have a very strong preferred orientation and form a columnar structure. From figure 3-b we can see the large amount of porosity in this columnar structure.

Sample CuG-SI-1 with 2μm gold layer heat treated at 190°C for 2 hours has a structure similar to Cu-SI-1. For figure 4-a, it can be seen that the grains have random orientation and that the average grain size is about 7μm. The grains near the stainless steel-copper interface have preferred orientation along the film growth direction. Figure 4-b also shows that the grains are dense inside with pores on the grain boundary. As in Cu-SI-1, CuG-SI-1 has a higher pore concentration near the stainless steel-copper interface than near the surface.

2. X-Ray Diffraction

Figure 5 shows the X-ray diffraction patterns of Cu and Au films. For Cu-SI-1 and Cu-SI-2, the x-ray diffraction profiles are similar. We also observe the austenite structure of stainless steel peaks in these patterns which are observed due to the curvature of the sample which causes x-ray beam to hit the substrate. There are two peaks, $2\theta = 37.7^{o}$ and 37.3^{o}, that are the K_{β} peaks for stainless steel (110) and copper (111) respectively. For

the sample CuG-SI-1, the K_β peak of Cu (111) was not observed because of the presence of gold film and thereby decrease in intensity of copper lattice lines. However, The lattice lines of stainless steel still exist. According to these records, there is no significant change in the lattice structure of these copper films. When we look at the peak of Cu(111), for both heat treatment temperatures (190°C and 260°C), the FWHM (full width at half maximum) are around 0.4^{o}. This broad diffraction pattern is due to small grain size and crystal distortion compared to the high temperature treatment (>600°C), for which the FWHM is around 0.18^{o}.(1)

Conclusion

Since the temperatures of heat treatment are very low, the grain size obtained are small for these samples. From the SEM results, we conclude that the Cu-SI-2 sample has higher porosity than the other two samples. Moreover, to improve the columnar structure, either higher treatment temperature or a thin gold layer coating can be used.

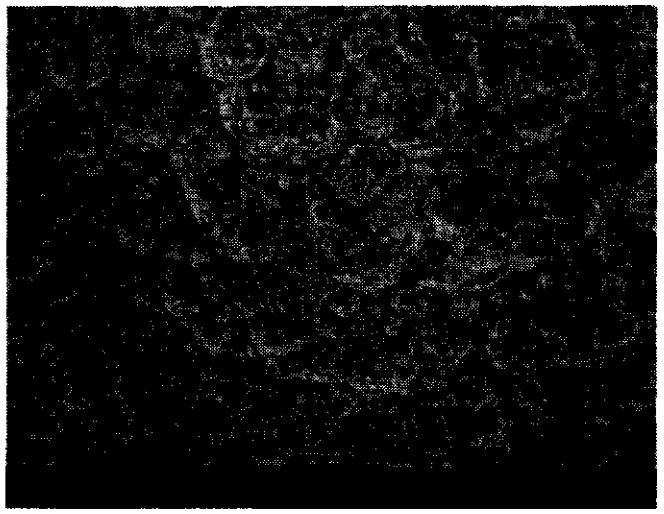
Future Work

1. The three specimens from Silvex will be heat treated at higher temperature to study the grain structure change.
2. Optimization of the processing conditions for CVD copper films on stainless steel tubes.
3. Investigation of the relationship between photodesorption and the chemistry and microstructure of the copper layer.

4. Setup of a simple electron desorption experiment with a Residue Gas Analyzer (RGA).
5. Identification of facilities to co-extrude a copper tube inside a stainless steel tube.
6. Study the effect of heat treatment on the adhesion of Cu layer with stainless steel.
7. Investigation of the densification of copper films on stainless steel for possible solutions to further reduce porosity.
8. Exploration of the other production and treatment techniques to reduce the photodesorption.

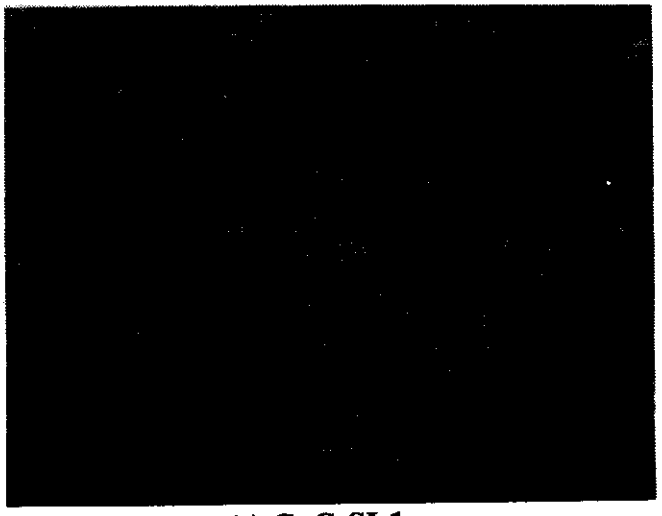
Reference

1. The progress report "Improvement of Microstructure and Chemistry of Copper Films in The SSCL Beam Tube", G.Q. Lu, S.B. Desu, W.T. Reynolds and A.O. Aning, August 6,1993.



(a) Cu-SI-1

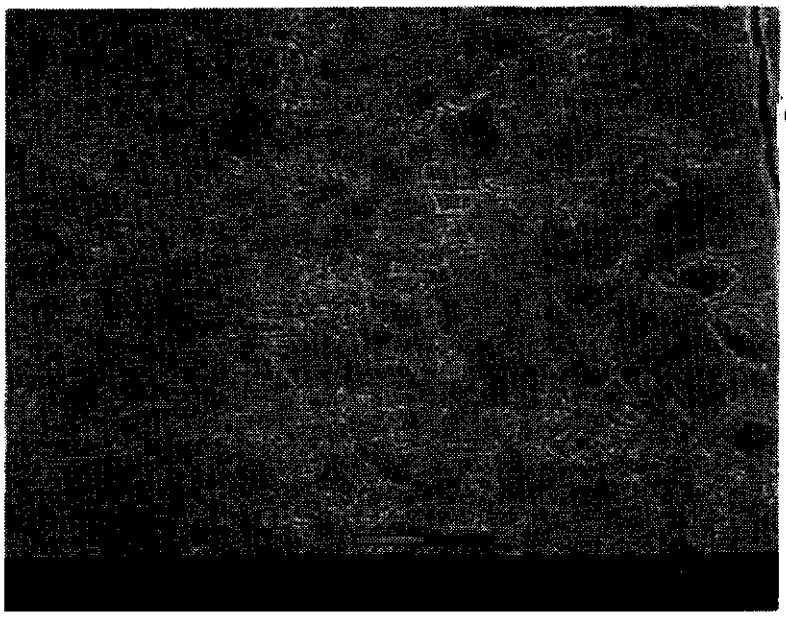
(b) Cu-SI-2



(c) CuG-SI-1

Figure 1. SEM free surface morphology/

260°C for 1 hour

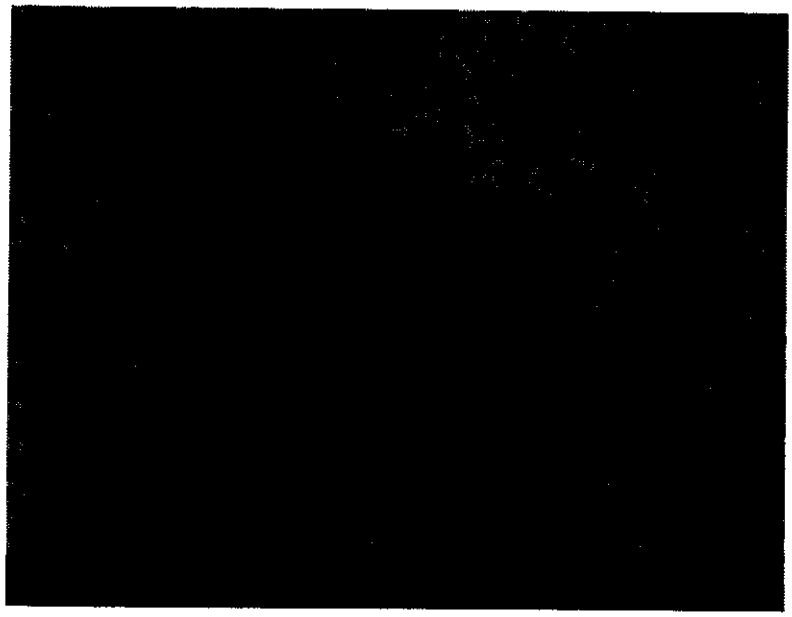


(a)

Figure 2. Cu-SI-1 SEM cross section.

190 °C for 2 hour

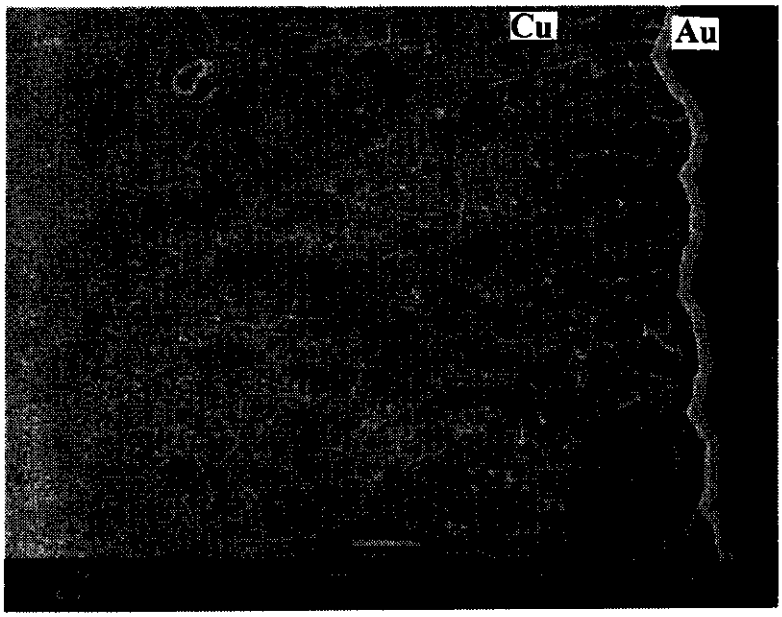
(a)



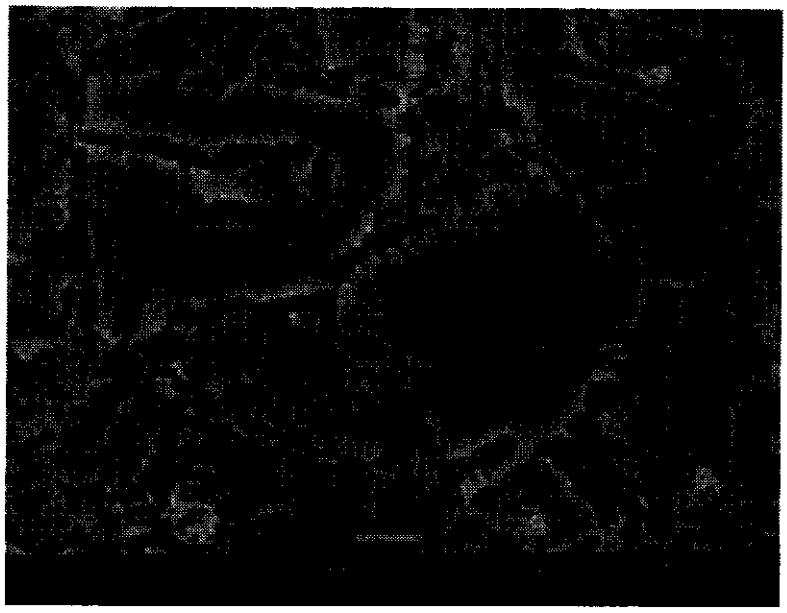
(b)

Figure 3. Cu-SI-2 SEM cross section.

190 °C for 2 hour



(a)



(b)

Figure 4. CuG-Si-1 SEM cross section.

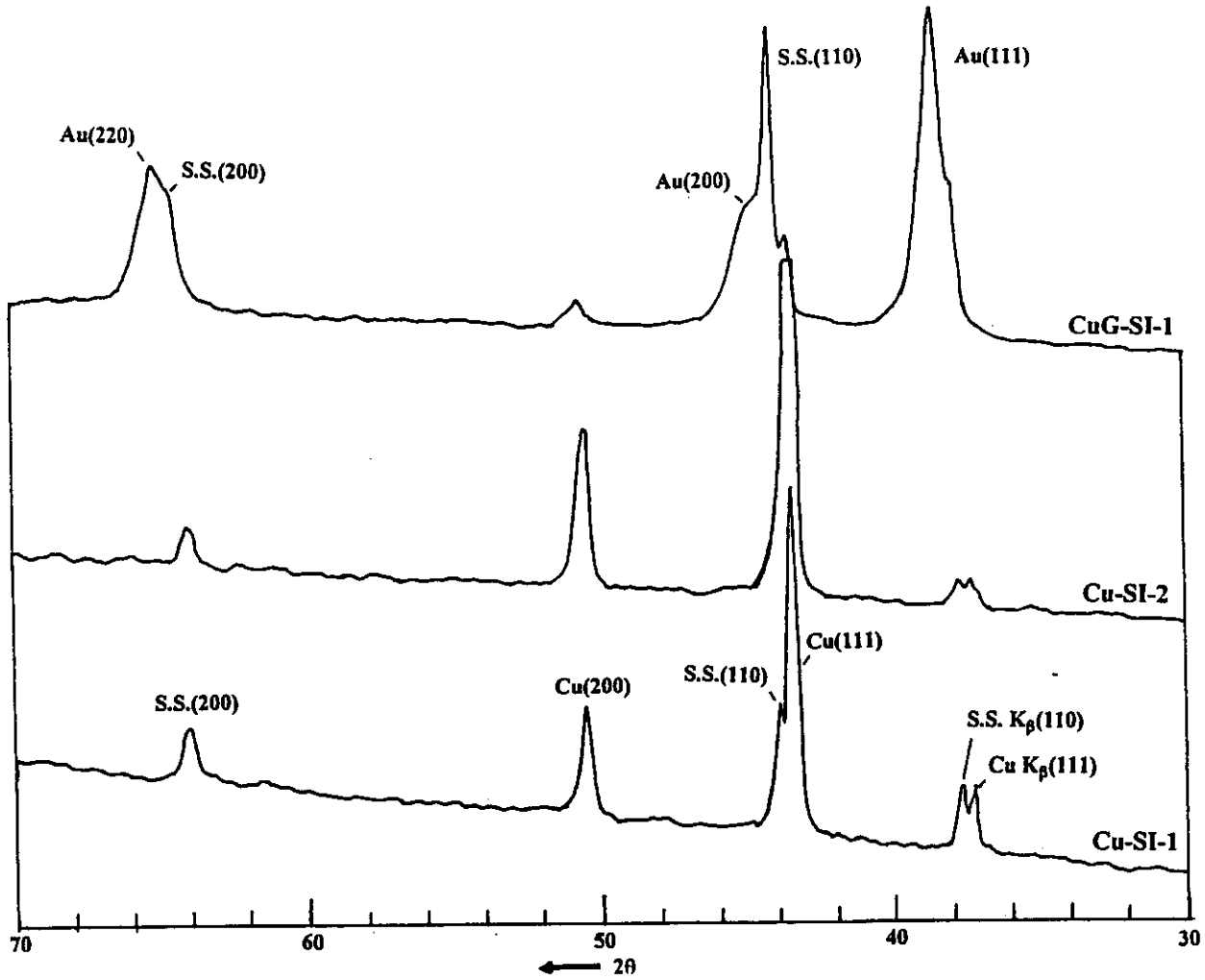


Figure 5. X-Ray diffraction profiles.