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Ion-Etch Characteristics of Epitaxial Copper on Sapphire

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For the first time a face-centred-cubic metal film has been epitaxially grown on sapphire. Film crystallinity and ion-etch characteristics were studied using back reflection X-ray diffraction and scanning electron microscope techniques. Single-crystal copper films have been deposited on basal plane sapphire substrates in the temperature range 240 to 375° C. The films exhibited bulk metal resistivity. Ion-etching studies of the films, using argon ions, have demonstrated superior quality, resolution and vertical etch profile of interconnection transmission lines over those of polycrystalline or less crystalline films.

1. Introduction

The successful growth of single-crystal films of copper on the basal plane of sapphire ($\alpha-Al_2O_3$) was recently reported [1, 2]. This was the first instance of the epitaxial growth of a face-centred-cubic metal on sapphire. The presence of a twin relationship in the copper film was demonstrated. The epitaxy was shown to be:

$$(111)_{Cu} \parallel (0001)_{\alpha-Al_2O_3}$$
$$[2\bar{1}\bar{1}]_{Cu} \parallel [2\bar{1}\bar{1}0]_{\alpha-Al_2O_3}$$

These films exhibited the bulk metal resistivity of 1.69×10^{-6} ohm-cm.

The interest in producing single-crystal films of copper on sapphire was motivated particularly by the need for low resistivity interconnections

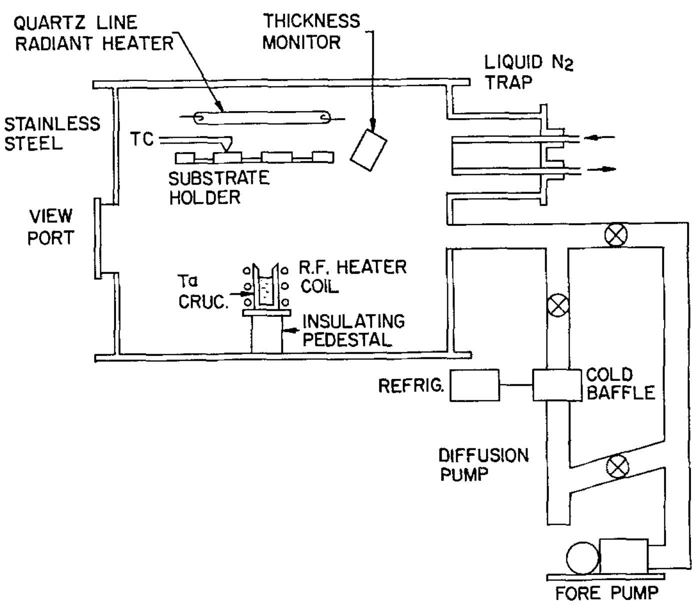

Figure 1 Schematic diagram of vacuum system used to deposit copper films.

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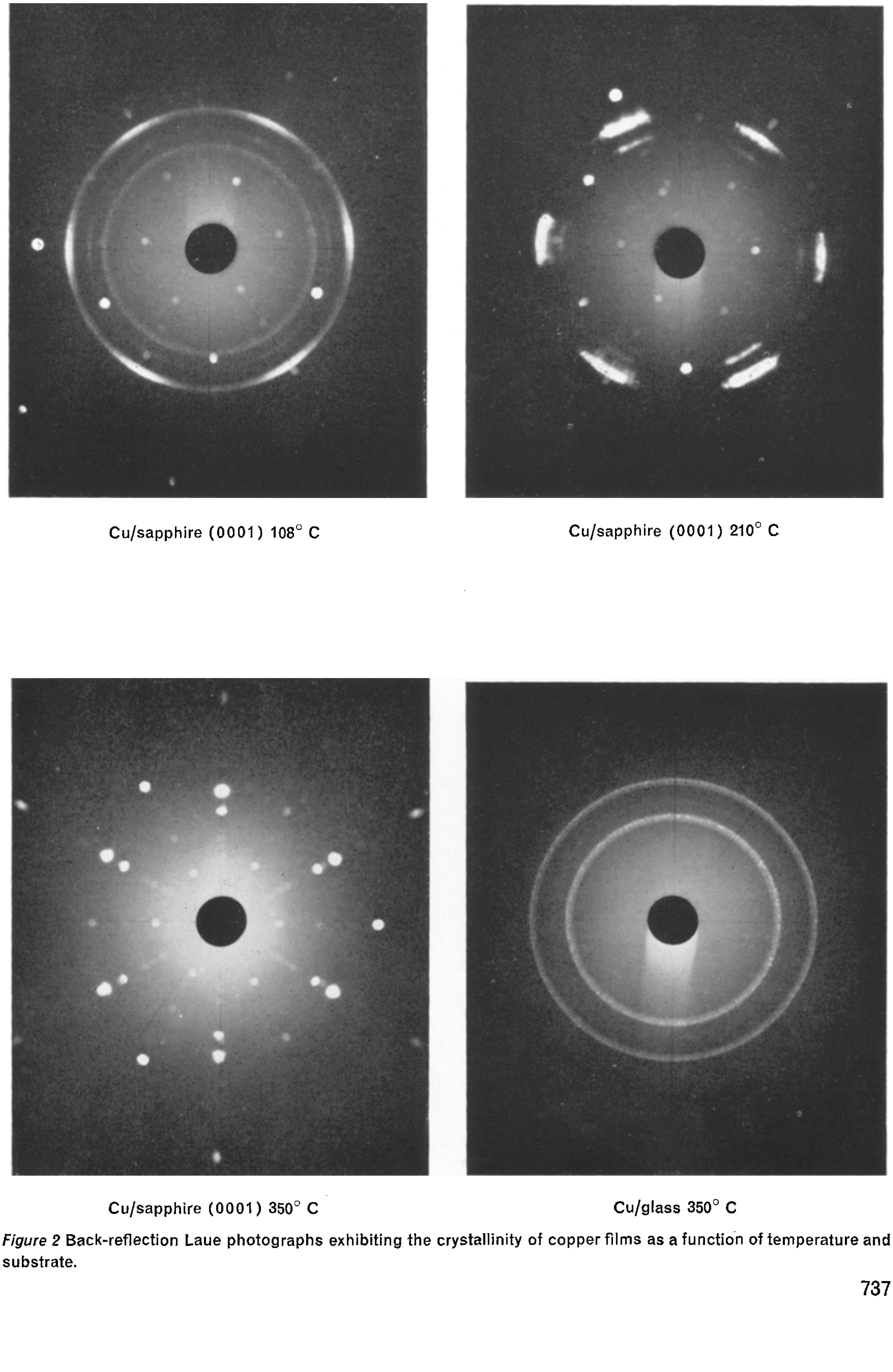

Cu/sapphire (0001) 108° C

Cu/sapphire (0001) 210° C

Cu/sapphire (0001) 350° C

Cu/glass 350° C

Figure 2 Back-reflection Laue photographs exhibiting the crystallinity of copper films as a function of temperature and substrate.

and transmission lines in microelectronic technology. As the speed of the circuits increase, the time delay due to circuit interconnection becomes comparable to the circuit delay itself. As a consequence, the trend has been to reduce the delay time by increasing the packing density which, in turn, demands narrow interconnection lines and the use of high conductivity metals. Hence copper which, except for silver, has the lowest resistivity of the metals, was chosen. Most interconnection transmission lines in current microelectronic technology use polycrystalline aluminium films of higher bulk resistivity.

It is well known [3, 4] that ion-etching or sputtering is a function of, among other variables, the crystallographic orientation of the metal grains, the rate being proportional to the atom density normal to the incident ions. Thus single-crystal films would be particularly amenable to ion-etching techniques for producing high resolution transmission lines with good vertical etch profiles [5].

2. Experimental

2.1. Film Deposition

A schematic diagram of the vacuum system used to obtain the films is given in fig. 1. The films were vacuum-evaporated by conventional RF induction-heated tantalum crucibles at substrate temperatures, much lower than those in chemical reaction methods, e.g. pyrolytic decomposition of metal hydrides or reduction of the halide. The films were prepared from five 9+ pure copper pellets in a stainless steel, silicone oil diffusion vacuum system employing a liquid nitrogen trap, capable of providing a vacuum of 1 to 5×10^{-6} torr. The source-to-substrate distance was 10 in. and the deposition rate was of the order of 100 Å sec^{-1}. The substrates were radiantly heated by GE Quartzline lamps.

The substrates* were 1 in $\times$ 0.010 in. wafers cut from Verneuil grown sapphire boules. They had a 1 μin. mechanically polished surface on both sides and were oriented so that the surface normal was within 4° of the [0001]. Except for ultrasonic cleaning in tepid water with detergent, no further substrate preparation was used.

The films studied were 5 to 7 μm thick. The crystallinity of the films was determined using X-ray diffraction Laue back-reflection photographs using a 1 mm diameter beam of unfiltered Cu radiation. Fig. 2 gives representative Laue photographs of copper films for various substrate temperatures. The films vary from a single-crystal character through a preferred orientation texture to complete polycrystallinity with some superimposed preferred orientation. Below 240° C Laue patterns indicated increasing disorientation or randomness with decreasing temperature. At 228° C the films exhibited a high degree of preferred orientation and were completely polycrystalline at 108° C. In each of the three photographs of copper on sapphire, the weaker set of reflections is due to the sapphire substrate. The pseudo-sixfold symmetry is due to the twinning of the copper film. The completely polycrystalline copper film on glass was obtained during the same 350° C run, as the single-crystal film.

2.2. Ion-Etching

The ion-etching study was made on the apparatus described by Tsui [5] as shown in fig. 3. The two parallel plate aluminium electrodes, $3\frac{1}{2}$ in. in diameter, are spaced about 2 cm apart. A fused silica plate, $\frac{1}{16}$ in. thick, is placed on the cathode which is water-cooled from the back side. The copper-coated sapphire wafer to be etched is positioned at the centre of the quartz substrate. High vacuum silicone grease is used to improve thermal contact between the wafer and dielectric substrate. The base pressure of the vacuum system is about 5×10^{-8} torr. Ultra high purity argon gas is bled into the system at a rate of about 0.3 l min^{-1} and a pressure of about 5 to 12×10^{-3} torr is maintained in order to obtain

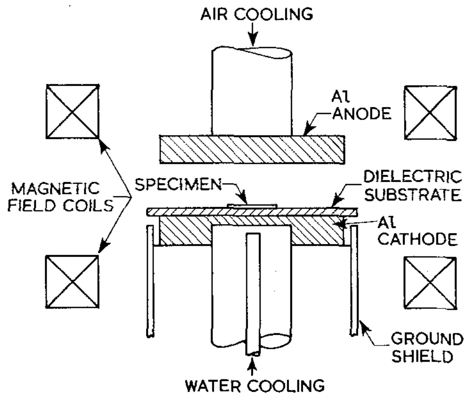

Figure 3 Schematic diagram of the ion-etching apparatus (after R. T. C. Tsui).

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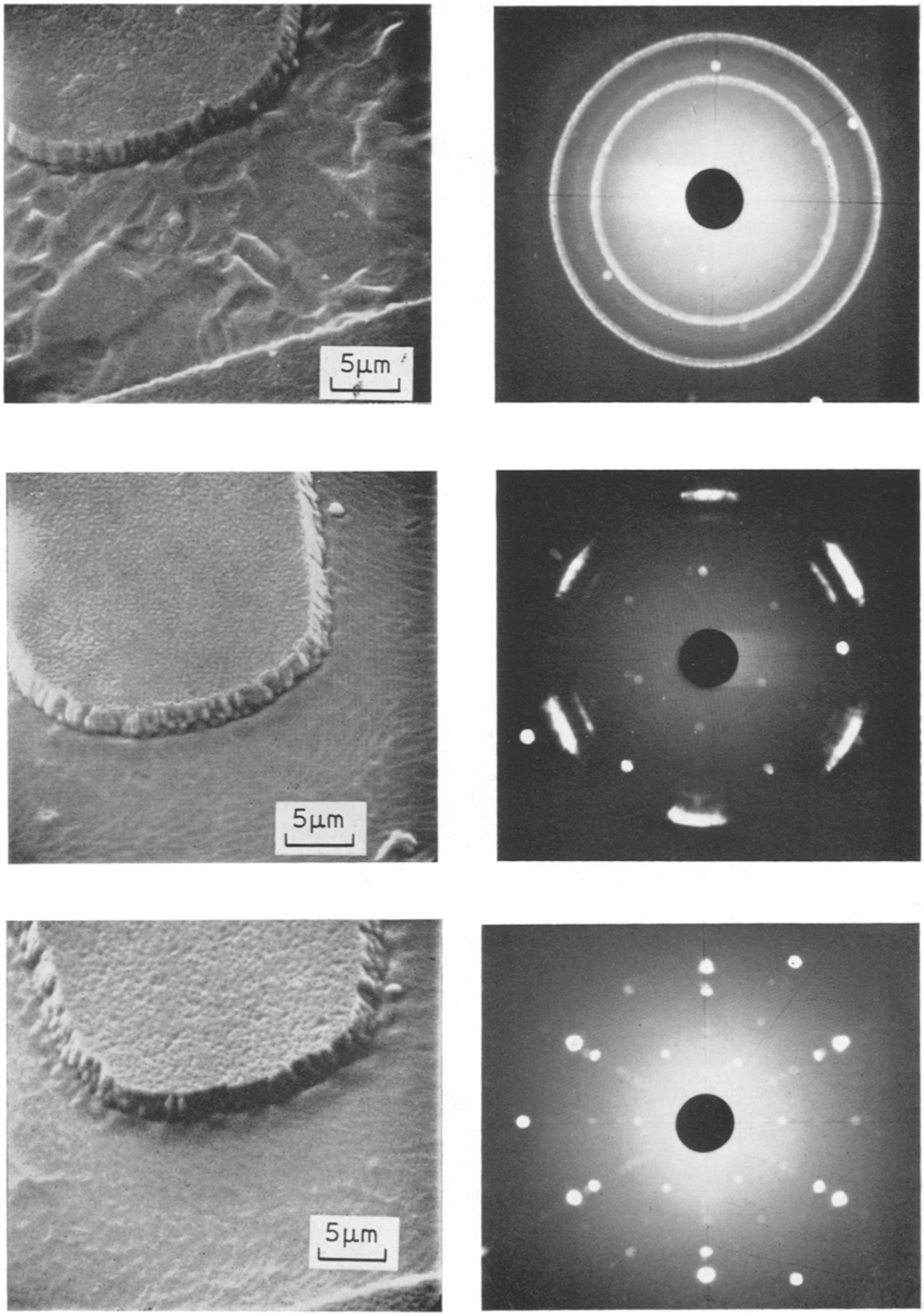

Figure 4 Scanning electron micrographs and Laue back-reflection photographs: (a) polycrystalline, (b) preferred orientation and (c) single-crystal films.

stable discharge conditions. The sputtering conditions used in this study were:

sample surface temperature	– approx. 200° C
power density	– 1.6 W cm^{-2}
argon pressure	– 10×10^{-3} torr
rf voltage across metal electrode (peak to peak)	– 1700 V at 13.56 MHz
dielectric substrate	– α-Al_2O_3 on top of fused silica plate $\frac{1}{16}$ in. thick

3. Ion-Etching Results and Discussion

The results of the ion-etching study of a set of three copper films on sapphire substrates are shown in fig. 4. The photographs were taken in a Cambridge Instrument Co. "Stereoscan" scanning electron microscope. The photographs represent SEM's of polycrystalline copper, copper with a preferred orientation texture and single-crystal copper films. Associated with each SEM is a Laue photograph describing the degree of crystallinity. All three samples were etched simultaneously under the same conditions. Each SEM represents the portion of an ion-etched transmission line showing the unetched upper surface and the etched groove between lines. The etching was achieved using argon ions and sputtered Al_2O_3 as the masking material. Openings were etched in the Al_2O_3 using H_3PO_4 at 75° C. Subsequent to the ion-etching, all the Al_2O_3 was similarly removed with no apparent harm to the copper films.

All the copper films were nominally 6 μm thick and were etched to a depth of 2.3 μm for the polycrystalline sample and 2.8 μm for the single-crystal film. Thus the etched surfaces are copper as well. The nature of the etched surface of the polycrystalline copper is most apparent. Due to the random crystallographic orientation of the individual grains, the etched surface is far from homogeneous, as evidenced by the many cavities of varying depth. One can estimate the grain size to be approximately 5 μm. The texture of the etched surface is considerably improved in the other two samples with the single-crystal sample having the smoothest etched surface. Fig. 5 is a SEM of another ion-etched single-crystal sample showing a very straight vertical profile of a transmission line.

Due to the uncertainty of the overall etch rate in the polycrystalline sample, the etching process would have to be sufficiently long to ensure the removal of all the copper between adjacent transmission lines. In the single-crystal sample the etch rate is very uniform and the etch time could be minimised. This study has shown that the characteristics produced by ion-etching-crystal copper films are superior to those produced from polycrystalline films.

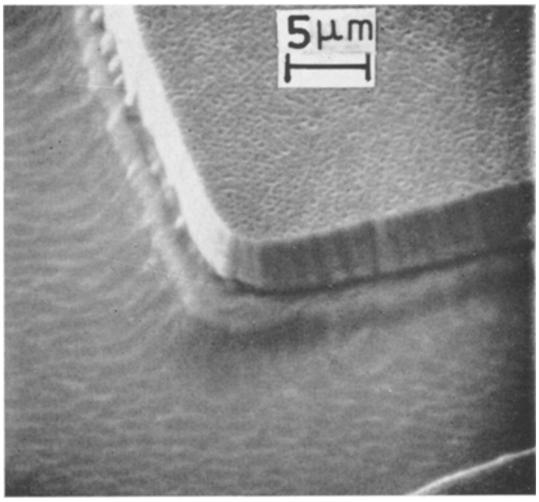

Figure 5 An example of the optimum vertical profile obtainable in ion-etching a single-crystal copper film.

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