Electrical Flexibility of Cu Thin Films for Applications in Flexible Electronics

A thesis submitted

in partial fulfilment of the requirement for

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in

Physics

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CERTIFICATE

This is to certify that the thesis titled "Electrical Flexibility of Cu Thin Films for Applications in Flexible Electronics" submitted by **Deepti garg, Roll no. 301604009** in partial fulfilment of requirements for the award of the degree **M.Sc.** in **Physics** from the School of Physics and Materials Science, Thapar Institute of Engineering and Technology, Patiala is a record of candidate's own work carried out by her under my supervision and guidance. The matter embodied in this report has not been submitted in part or full to any other institute for award of any degree.

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Declaration

I hereby declare that the work been presented in this thesis report entitled “**Electrical flexibility of cu thin films for applications in flexible electronics**” by me in partial fulfilment of the requirements for the award of degree of **Masters of Science** in **Physics** Thapar Institute of Engineering and Technology, Patiala is an authentic award record on my work carried out under the supervision of **Dr. Bhaskar Chandra Mohanty**, Associate Professor, School of Physics and Materials Science, Thapar Institute of Engineering and Technology, Patiala. The matter presented in this report has not been submitted in any other university/institute for award of master and science or any other degree.

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Abstract

Great demands for flexible conducting electrodes are promoted considering the rapid development of flexible electronics. Conventional thin-film FCE, however, has limits of low conductivity, poor mechanical reliability and requires complicated fabrication process due to the use of nanowires and nanoparticles. In this work, Al-Cu bilayer thin films were grown on plastic flexible substrate by a DC magnetron sputtering at 75W power. Cu and Al target were used as source for the deposition Al-Cu bilayer. The deposition time for the Cu was 30 min and for Al it was varied (30s, 60s, 120s). The evolution of properties and stability of electrical resistance of Al-Cu bilayer on plastic flexible substrates was evaluated. The properties of resulting thin films were characterized by X-ray diffraction (XRD), and bending test. It was observed that the resistance of the Cu films significantly increased to as much as 40 times of the initial value after 400 bending cycles. The results indicate fast mechanical deterioration of the films due to initiation of cracks and their propagation, which impairs electrical conduction across the films. Compared to a single Cu layer, excellent electrical stability due to bending cycles was observed when a sputter-deposited thinner Al underlayer was used. The Al underlayers of deposition times of 30 and 60 s, shows that resistance remained the same up to 400 bending cycles. This is attributed to the enhanced mechanical integrity and better adhesion with the substrate. However, when a thicker Al underlayer was used, the resistance increased with bending cycles. The results suggest that there exist an optimum thickness of the underlayer which coupes well both with the substrate and the Cu over layer. The obtained results are quite relevant for flexible electronics in that an appropriate underlayer helps in electrical stability of Cu films, which might be used as bottom contact in flexible devices.

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Chapter 1

Introduction

Thin solid films are typically defined as a layer of material having thickness in the range of few millimeters to nanometers. Owing to a large surface to volume ratio, the properties of thin films are very different from their bulk counterpart [1]. Furthermore, it is relatively easier to manipulate the properties of the thin films by process parameters including thickness, deposition rate, deposition temperature, etc. and also by the substrates. Hence, it has been possible to find thin films of same material having a large variation in properties, which has been capitulated to design various devices. Over the years, thin films have been increasingly used in a variety of applications such as micro-electronics, energy generation and storage, solid state lighting, hard and protective coatings, health care systems, automobiles, sensors, etc. [2-4].

1.1 Flexible electronics

Flexible electronics is a technology for building electronic circuits by mounting electronic components and devices on flexible plastic substrates. This technology has drawn considerable research interest in the last decade because of the growing popularity of the flexible devices such as roll-able solar cells, electronic skins, paper like electronic displays etc. [5-7]. Figure 1 shows examples of various flexible thin film devices.

Figure 1.1: Examples of flexible thin film based devices [8].

For all purposes, the flexible electronic systems are expected to have low weight, increased yield strength, low costs, ruggedness, increased barrier properties and larger fracture strain. Consequently, the various functional layers used to fabricate the flexible electronic devices are required to satisfy the above requirements simultaneously. For instance, in designing a flexible solar cell, one should ensure that all components (for example, bottom electrode, top electrode, p-layer, n-layer, and buffer layers in inorganic thin film solar cells) need to be equally flexible. Electrical/mechanical failure of any single layer will result in complete failure of the device.

Thin metallic films have been routinely used in electronic devices, including the flexible ones. Among various choices available, metallic films have been the first choice as bottom or top electrode in electronic devices because of their high conductivity, excellent adhesion between substrate and the film and inertness. In the case of flexible devices, a strong adhesion between the film and substrate is required so that electrical properties during repeated bending are sustained. For designing fully flexible devices, thin metallic films are generally deposited on the flexible substrates as the high ductility of metallic films can ensure the excellent adhesion and stability of the system. Although Cu, Ag, Al, etc. have been successfully used in many cases [9], there are reports of development cracks in the films due to the tensile stress and mechanical cycling when the well bonded metallic thin films to the substrate is bended up to longer strain. Figure 1.2 shows cracks in metallic films [10] as a consequence of repeated bending of the films up to a large bending strain. To overcome this problem, traditionally a buffer layer is used.

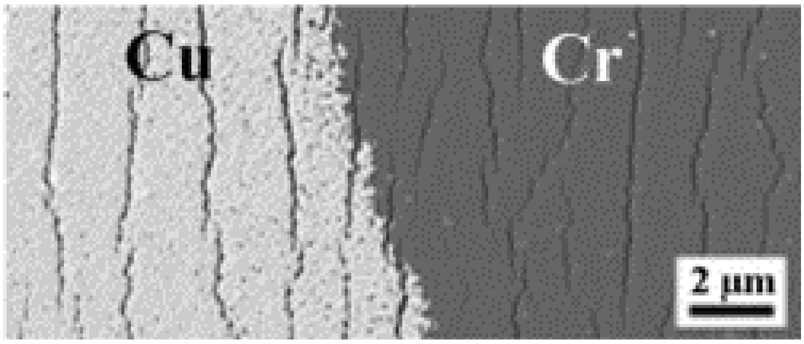

Figure 1.2: Cracks in metallic thin films due to bending of the films [11]

1.2 Flexibility/electrical properties of Cu films for bottom electrode applications

Cao *et al.* [12] deposited Cu thin films on polymer substrate by dc magnetron sputtering at room temperature. The dc sputtering power was 80W. It revealed that the average crack spacing of Cu thin films decreases with higher strain rate, which exhibits a remarkable strain rate sensitivity. The R/Ro also increases with the increment of strain rate which results the increase in crack density.

Xiang *et al.* [13] deposited Cu thin films on polymer substrate by dc magnetron sputtering with no Ar plasma but with a 20 nm carbon – release layer. The dc sputtering power was 200W. It was found that ductility of thin film depends on the interface between the film and the substrate and intergranular fracture. The strong adhesion between the film and the substrate can sustain the film to larger strain i.e. up to 30% without causing any microcracks in the film as compared to weak adhesion which can sustain the strains up to 2% only.

Kim *et al.* [14] deposited Cu thin films on polymer substrate by thermal evaporation at room temperature with deposition rate of 25 nm/min. It was concluded that when a tensile load was applied, nucleation of cracks started and as a result, there was increase in the electrical resistance. This further reduced the number of bending cycles as there was increase in the fatigue stress. Hence, the bending is measured by the change in the resistance and the abasement in the electrical properties was due to the fatigue damage.

Zhang *et al.* [15] deposited Cu thin films on polyimide substrate by dc magnetron sputtering at room temperature with deposition rate of 0.6 nm/sec. The thickness of Cu thin film was 500 nm. The Cr interlayer was deposited by the same above conditions but with deposition rate of 0.55nm/sec. It was found that with the decrease in thickness of Cu layer, the twin density as well as number of twins per grain decreases which follows the linear law formation.

Shaoxiong *et al.* [16] deposited Cu thin films of thickness 520nm on aluminium nitride substrate (AlN) by dc magnetron sputtering at room temperature with deposition rate of 25sccm. The titanium of thickness 30nmn interlayer was deposited to improve the adhesion and electrical conductivity between Cu film and substrate due to mismatch of thermal coefficient. The lowest electrical resistivity was achieved 2.75µΩcm for Cu/Ti bilayer film as compared to Cu thin film. As a result there is decrease in the cracks and advancement in thin film density. Even after 50 thermal cycles, the resistance still remain the same.

Nanshu *et al.* [17] deposited Cu thin film on polymer substrate by dc magnetron sputtering at room temperature. The deposition power was 24w. When subjected to tensile loading resistance(R) increased with the length and the microcracks was obtained at elongation of 40%. But when the Cr layer was under layered, then the microcracks was obtained at elongation of 50%.

Nanshu *et al.* [18] deposited Cu thin film on polymer substrate by dc magnetron sputtering at room temperature with deposition rate of 0.39nm/s. The deposition power was 200w. The thickness varying from 50nm to 1µm and is controlled by increasing the deposition time. With the increase in thickness of Cu thin films, there is decrease in yield strength which further increases the failure strain in the (111) textured films the fraction of (100) grains increases as we increase the thickness beyond 500nm. As result, at lower yield strength, larger grain size are obtained.

Yin *et al.* [19] deposited Cu thin films on polyimide substrate by thermal evaporation at room temperature with pressure of 7.0×10^{-5}Pa. The Al buffer layer was introduced to improve the interface between the film and substrate, to upgrade crystallinity of Cu and to turn down internal thermal stress which results in enhancement of the electrical conductivity of Cu thin film electrode. As result the lowest electrical resistivity was achieved i.e. 3.05 μΩ·cm for Al-Cu layer as compared to bulk material. It exhibited excellent adhesion and flexibility under bending radius of larger than 6mm over 1000 cycles. R/R0 less than 1% was exhibited in thermal shock tests with 500 cycles.

1.3 Motivation and objective

The brief literature review presented above shows that introducing a buffer layer between the metallic coating and substrate helps to increase the stability of electrical conductivity of the device in the repeated bending conditions. In view of this, the main objective of this work was to evaluate the electrical flexibility of Cu films deposited by DC magnetron sputtering in different bending conditions. We have found that electrical properties of the films were significantly deteriorated. However, insertion of a very thin Al buffer layer dramatically improves the electrical stability of the layer.

Chapter 2

Experimental Techniques

In this chapter a brief description of growth of thin films and their characterization is presented. The strategy used for growth of thin films is presented in section 2.1 and the techniques used for analyses of thin films is described in section 2.2.

2.1 Sputtering deposition technique

When the surface layer of material is bombarded with highly energy particles, then ejection of atoms occurs. This ejected species are made to deposit on a substrate. This phenomenon is key to the sputtering deposition of thin films [20]. It involves many parameters that can affect the deposition process such as pressure of the deposition ambient, substrate temperature, sputter power, etc.

Sputtering is carried out in a high vacuum chamber as shown in **Figure 2.1**. The chamber is containing inert gas mostly Ar which is known as **sputtering gas** and is only used since it has moderate mass so that the momentum can be easily transfer from the target to bombard the atoms. The target is that material which is to be deposited on the substrate and is connected to negative voltage supply. The substrate holder in the chamber which is facing towards the target is maintained at ground potential [21]. The high voltage leads to the ionization of atoms which is exhibited by dense plasma in the chamber as shown in **Figure 2.2** which is maintained between target and substrate throughout the deposition time. These ions bombard the surface of target and ejection of atoms takes place which are made to condense on a substrate to form film. This process is carried out only when the kinetic energy of the ejected atoms is too much high as compared to the thermal energies which leads to the uniformity of film. The ratio of sputter atoms to the bombarding atoms is known as sputter yield which depends on the characteristics of the incident ion, material composition, experimental geometry and binding energy. At very high energies, sputtering yield decreases due to increasing penetration depth. Sputtering involves the energy of few eV and as result execellent adhesion is achieved in sputtering.

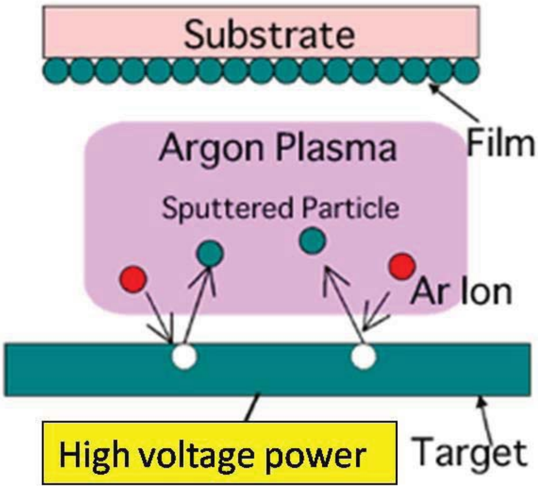

Figure 2.1: Sputtering process in high vacuum chamber

Figure 2.2: Photograph of dense plasma in the machine

Sputtering processes are divided into different categories: RF (radio frequency), DC (direct current) sputtering which are explained as follows:

2.1.1 DC (direct current) Magnetron Sputtering

DC sputtering is most suitable technique used to deposit metallic materials with the help of magnets. In this process, positively charged Ar+ can smoothly accelerated towards the target and from there ejected atoms are deposited on substrate to form film as shown in **figure 2.3**. The power used in this process is DC supply and also it is cheap on proceedings with large substrate sizes [22].

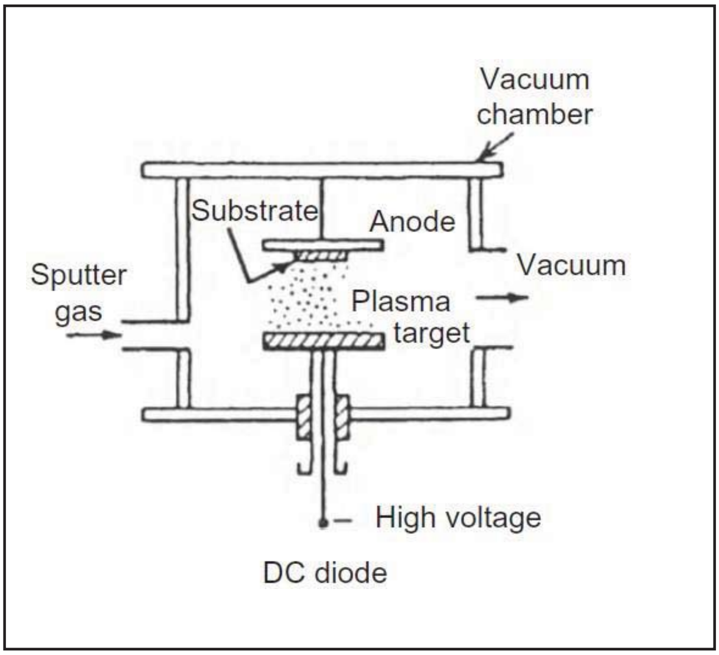

Figure 2.3 DC Magnetron Sputtering process

2.2 Fabrication of Al-Cu bilayer thin films on plastic flexible substrate

In this work, the Al- Cu bilayer thin films were fabricated by the sputtering on plastic substrate i.e. transparent sheet of thickness 100µm with dimensions of 2.5×3.5cm as shown in **figure 2.4**. The substrate was cleaned by ultra -sonication for 5 minutes in DI water. The Al-Cu bilayer: Cu films were firstly deposited on transparent flexible substrates at room temperature via dc magnetron sputtering in high vacuum chamber with base pressure of 4.8×10^{-6} mbar. The Ar gas was allowed to flow in the chamber through mass flow controller at 34 SCCM and working pressure was achieved 2.6×10^{-3} mbar. The sputtering power was 75W and the deposition time is set to 30 minutes.

Figure 2.4 Inside and outside photograph of machine used for fabrication Al-Cu bilayer

Hence in order to improve the adhesion between the Cu film and flexible transparent substrate, Al buffer layer was introduced in between Cu thin film and substrate. The Al film were deposited on the transparent flexible substrate at room temperature via dc magnetron sputtering with base pressure of 5×10^{-6} mbar. The working pressure achieved was 3.5×10^{-3} mbar at rate of 30 SCCM. The DC sputtering power was kept at 75W and the thickness was controlled by varying the deposition time. The two set of samples with different deposition time i.e. 30 sec and 60sec were prepared. In the prepared samples only the thickness of Al layer was changed and the Cu thickness was kept same in each case. The schematic and photographs of Al-Cu bilayer is shown in **figure2.5**.

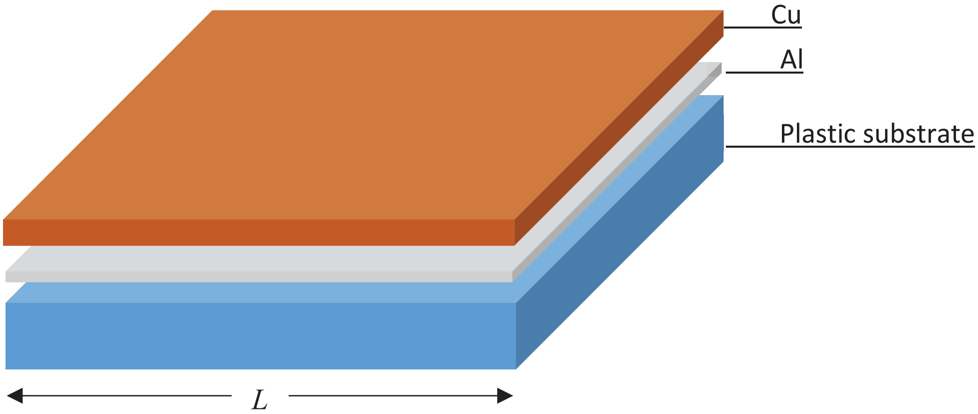

Figure 2.5 Schematic of Al–Cu bilayer

2.3 Characterization Techniques

Crystal structure and phase purity of the films were assessed from the X-ray diffraction (XRD) patterns of the films. The XRD measurements were carried out using a Bruker D-8 advance diffractometer using Cu K_α (λ = 1.54 Å). Typical current and voltage during the measurements was about 40 mA and 45 kV.

The bending test was carried out using an in-house designed set-up, shown in **Fig. 2.6**. The schematic diagram of the bending experiment is presented in **Fig. 2.7**. The sample is held between two parallel holders of length (*L*) 25 mm, of which one holder is kept fixed and other holder is allowed to move free and is attached to a micrometer. By moving the micrometer, the sample is bent into a concave shape, thus inducing strain in the samples. Depending upon the distance to which the micrometer is moved (i.e. by varying ΔL), the amount of strain is varied. This design has been often used in literature to induce a bending strain at the central region of the film [23].

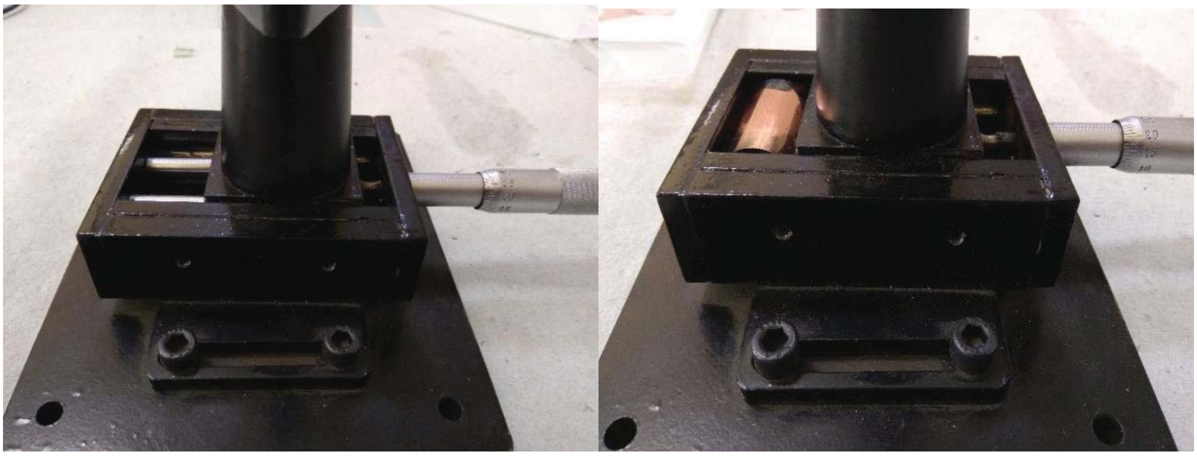

Figure 2.6: Photographs of the bending test set up (a) without (b) with the sample.

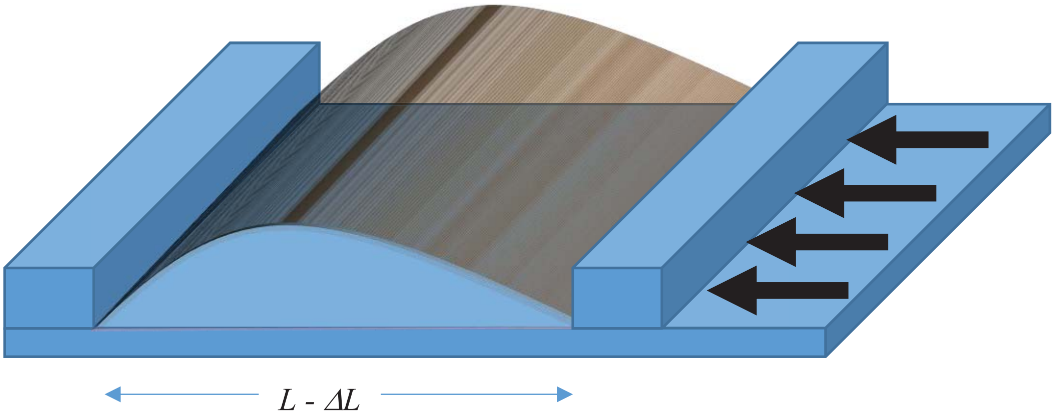

Figure 2.7: Schematic diagram of the bending experiment

The Cu films with and without Al under layer were subjected to the bending test in different conditions. In the first set, samples were bent for different values of ΔL ranging from 0 to 15 mm and their electrical resistance with a two probe multimeter was determined. In the second set, for each value of ΔL, samples were bent and released for a number of cycles up to 400. The resistance was measured after each cycle to study the electrical stability of the films.

Chapter 3

Results and Discussion

This chapter presents the results of the XRD measurements and the analyses of the bending test results to assess the electrical stability in repeated bending conditions.

3.1 XRD results

XRD patterns of the Cu films revealed to be of FCC structure characterized by three peaks at 43.4°, 50.4°, and 74.2° corresponding to (111), (200) and (220) planes, respectively. The typical patterns with an Al under layer with different thicknesses is shown in Fig. 3.1. The sample with the thinnest Al under layer (i.e., of deposition time of 30 s) revealed the similar pattern as of the sample without any underlayer. Owing to very small thickness of the Al layer, no peak corresponding to Al, was observed. Note that the dominant peak at about 25.83° corresponds to the PET substrate [24]. For all thicknesses of the underlayer, the nature of the patterns remains the same. However, the intensity of the Cu peaks significantly decreased with respect to the substrate peaks indicating reduced crystallinity with increase in thickness of the Al underlayer. This suggests that the growth mechanism of Cu films through sputtering is different for growth on plastic substrate than that on the Al-coated plastic substrate. Nevertheless, phase purity of all samples is established from these patterns.

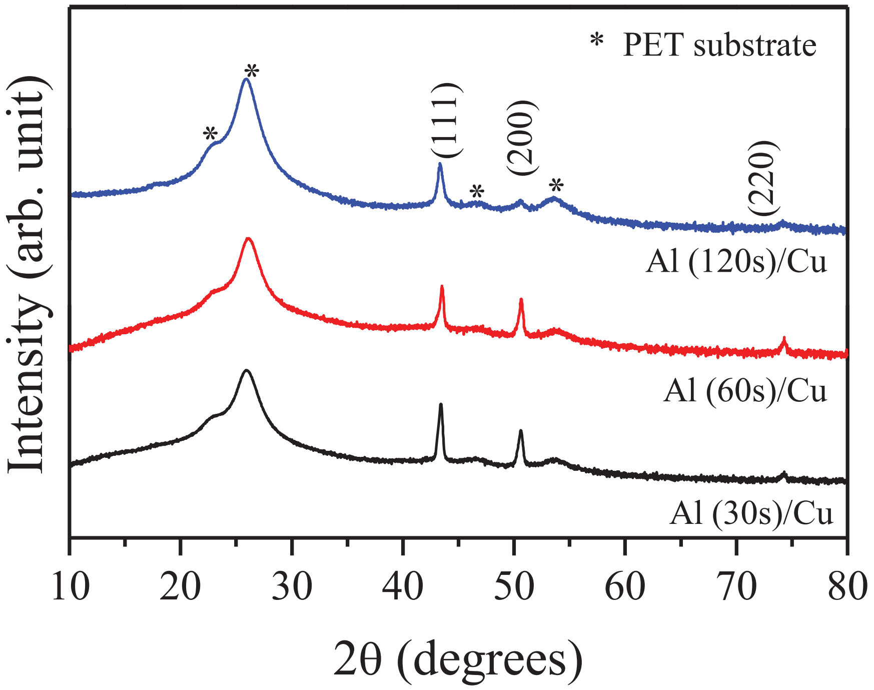

Figure 3.1: Typical XRD patterns of Cu films with an Al underlayer

3.2 Bending test results

The electrical stability of the Cu films with and without Al underlayer was studied from the results of the bending tests. Figure 3.2 shows the sheet resistance change (R/R_0) with the number of bending cycles for ΔL = 10 mm, where R_o is the initial resistance and R is the resistance which is measured after each bending cycle. As observed from the figure, for the Cu film without any underlayer the resistance of the films progressively increased up to 150 bending cycle. For higher bending cycles, the resistance sharply increased (for instance, by 40 times of the initial value after 400 bending cycles) indicating fast mechanical deterioration of the films. It might be possible due to initiation of cracks and their propagation, which can impair electrical conduction across the films.

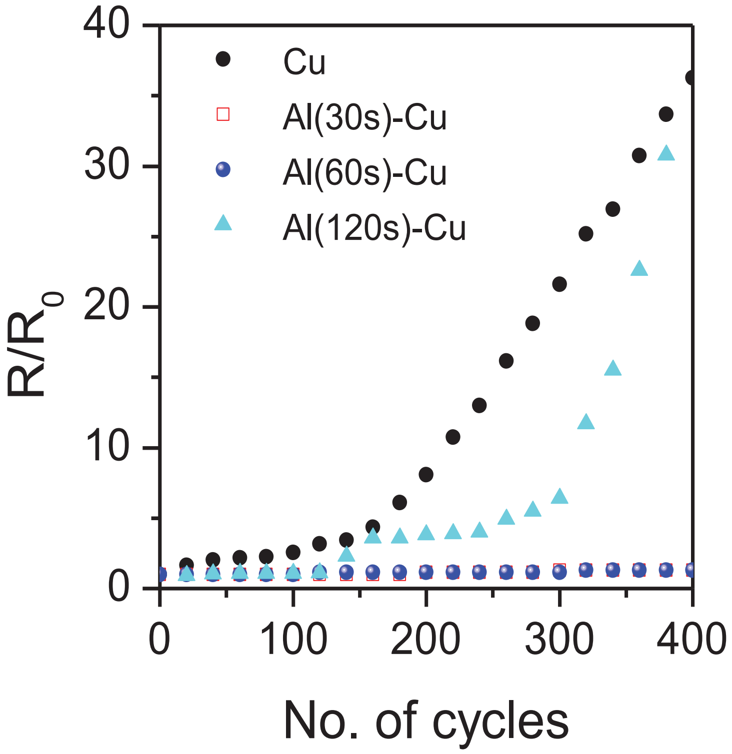

Figure 3.2: Variation of R/Ro with number of bending cycles

However, significantly different behavior was observed when an Al underlayer was introduced. For Al underlayer of smaller thicknesses (i.e., films of deposition time of 30 s and 60 s), the normalized resistance (R/R_0) remained almost constant at unity value up to 400 bending cycles. The results suggest excellent electrical stability of the samples. The improved results can be thought to arise due to enhanced mechanical integrity and better adhesion with the substrate. However, the performance degraded, as observed from the figure, when the thickness of the underlayer was increased. For the Al layer of deposition time of 120 s, the

R/R_0 ratio remained better than the Cu layer and almost comparable to the Cu layer with thinner Al underlayer up to 140 bending cycles. However, beyond this the R/R_0 ratio increased rapidly compared to the Cu films with thinner Al underlayer, though it remained less than that of the single Cu layer. It was observed that due to repeated bending of the samples, the Cu film peeled off, resulting in increased resistance of the samples. The photographs of the sample (Cu layer with Al layer of 120 s) before and after the bending test are presented in Fig. 3.3 that clearly shows damaged surface after 400bending cycles.

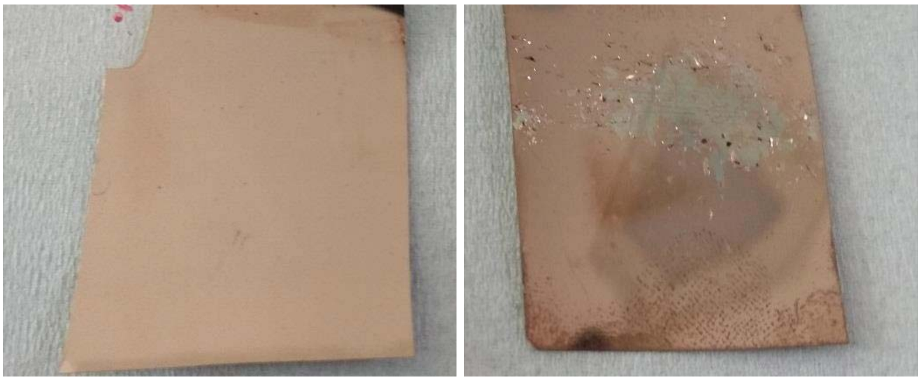
(a) (b)

Figure 3.3 Photographs of Al-Cu bilayer samples (a) before bending (b) after bending

Conclusion

We have evaluated stability of electrical resistance of thin Cu films grown by DC magnetron sputtering on flexible transparency sheets in multiple bending conditions. It was observed that the resistance of the films significantly increased to as much as 40 times of the initial value after 400 bending cycles. The results indicate fast mechanical deterioration of the films due to initiation of cracks and their propagation, which impairs electrical conduction across the films. Compared to a single Cu layer, excellent electrical stability due to bending cycles was observed when a sputter-deposited thinner Al underlayer was used. The Al underlayers of deposition times of 30 and 60 s, shows that resistance remained the same up to 400 bending cycles. This is attributed to the enhanced mechanical integrity and better adhesion with the substrate. However, when a thicker Al underlayer was used, the resistance increased with bending cycles. The results suggest that there exist an optimum thickness of the underlayer which coupes well both with the substrate and the Cu over layer. The obtained results are quite relevant for flexible electronics in that an appropriate underlayer helps in electrical stability of Cu films, which might be used as bottom contact in flexible devices.

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