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Adhesion of NiCu Films DC Biased Plasma-Sputter-Deposited on MgO (001)

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Abstract: NiCu films about 60 nm thick were deposited on MgO (001) substrates at 230 ℃ by DC plasma-sputtering at 2.7 kV and 8 mA in pure Ar gas using a $Ni_{90}Cu_{10}$ target. A DC bias voltage U_s of 0, −60, −110 or −140 V was applied to the substrate during deposition. The adhesion of the film to the substrate was studied using a scratch test as a function of U_s. The application of U_s is very effective in increasing the adhesion of the film to the substrate. In conclusion, the adhesion increases with cleaning the substrate surface by sputtering off impurity admolecules during the film initial formation due to the energetic Ar ion particle bombardment.

Key words: NiCu film; plasma-sputter-deposition; negative bias voltage; adhesion

The adhesion of a film to a substrate is an important property for further successful applications. However, the adhesion is one of the least understood properties [1]. Mattox [2] reviewed the effect of energetic particle bombardment on the adhesion of film to substrate. The adhesion could be generally enhanced by cleaning the substrate surface and by forming compound and/or transition layer between the film and the substrate [3–6]. The present work aims to investigate, with the use of a scratch test, the effect of U_s on the adhesion of NiCu films DC biased plasma-sputter-deposited on MgO(001) substrates. Brief discussion will be given about the present result referring to the previous result of cross-sectional TEM observations [7, 8]:

1 Experimental Procedure

The chamber of the DC biased sputtering system was evacuated with a turbo molecular pump. The other arrangements for sputter-deposition were similar to those described previously [9]. NiCu films about 60nm thick were deposited on MgO (001) substrates at 230 ℃ by DC plasma-sputtering at 2.7 kV and 8 mA in pure Ar gas (99.9995% in purity) using the $Ni_{90}Cu_{10}$ target (99.99% in purity) of 50 mm in diameter. A bias voltage U_s of 0, −60, −110 or −140 V was applied to the substrate during deposition. The distance between the target and the substrate was 80 mm. Prior to deposition, the chamber was evacuated to a pressure lower than 1×10^{-4} Pa. After cooling the cylindrical trap surrounding both the target and the substrate with liquid N_2, the pressure in the chamber was elevated to about 5 Pa by supplying Ar gas and then the sputtering was started. The adhesion of the film to the substrate was measured using a scratch test equipment (RHESCA Co.). As the sample was horizontally moved with the stage at 10.0 μm/s, the film was scratched by the diamond needle having 15 μm pole radius which was vertically loaded at 21.58 mN/mm. The pattern of the scratched region was observed by an optical microscope (OM). Now, the adhesion of film to substrate F can be given by [10]

$$F=[W/(\pi R^2 H_B - W)]^{1/2}\times H_B \qquad (1)$$

where W is the force to peel the film from the substrate, R the pole radius of the diamond needle head and H_B the Brinell hardness of the substrate.

2 Results

Figure 1 shows the OM photographs of the scratched regions of the films. The scratched position where the film prepared at $U_s=0$ V begins to be completely peeled is shown by an arrow in figure 1 (a). Besides the positions with the same scratch load as for $U_s=0$ V are marked by arrows for $U_s=-60$ V (figure 1 (b)), −110 V (figure 1(c)), −140 V (figure 1(d)), respectively. Thus, as can be seen from figure 1, only the film prepared at $U_s=0$ V could be completely peeled from the substrate while the other films could not be peeled by applying the same needle force. **Figure 2** shows the scratch curves corresponding to the OM images in figure 1. The abscissa expresses the scratch data, *i.e.*, the force loaded to the needle which was horizontally moved at 10.0 μm/s and loaded at 21.58 mN/mm. The ordinate expresses the vertical position of the diamond needle in an arbitrary unit but in the common

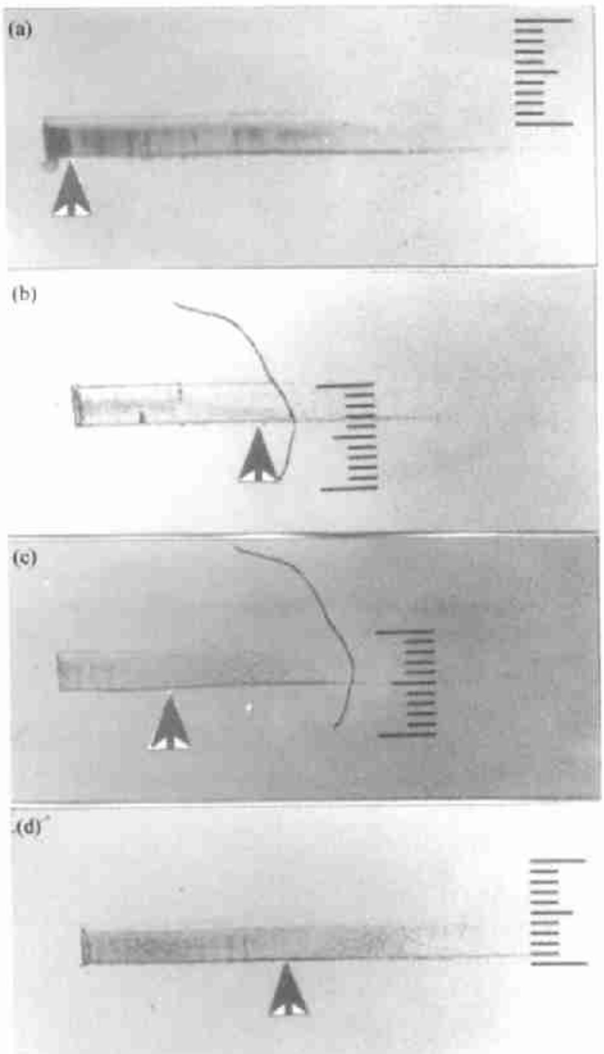

Figure 1 Optical microscope (OM) photographs of scratches of the NiCu films prepared at various U_s, (a) 0 V; (b) −60 V; (c) −110 V; (d) −140 V. The minimun unit on each photograph represents a common scale of 0.01 mm. Arrows mark the force position equivalent to that where the film prepared $U_s = 0$ V is completely peeled from the substrate.

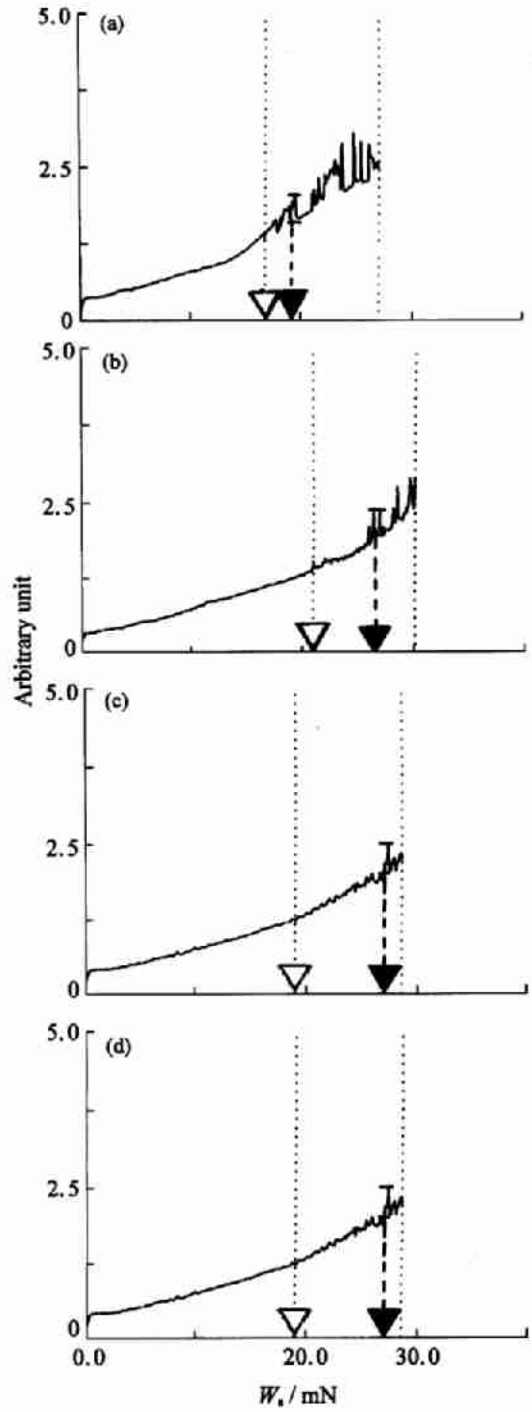

Figure 2 Scratch curves of the NiCu films prepared at various U_s, (a) 0 V; (b) −60 V; (c) −110 V; (d) −140 V. Solid arrows indicate the force position W_s. Open arrows indicate the force position where the film begins to be partially peeled by the needle.

scale from figure 2 (a) to (d). The vertical dotted lines marked by open arrows indicate the positions where the needle may begin to partially peel or penetrate the film. The vertical position of the needle begins to oscillate as it peels or penetrates the film. The amplitude of this oscillation is proportional to the average depth to which the needle penetrates into the film. **Table 1** shows the force W_s loaded to the needle where the amplitude reaches a common constant value as marked by solid arrows in figure 2 (a) to (d). The substitution of W_s into W in equation (1) gives an apparent adhesion F_s where H_B

Table 1 Values of the force W_s with the same amplitude in each scrath data curve shown in figure 2

U_s / V	0	−60	−110	−140
W_s / mN	19.2	26.7	26.9	26.9

was estimated to be 788 by measuring the microhardness of MgO substrate. The variation of F_s with U_s has to correspond the U_s dependence of the true adhesion F though F_s must be smaller than F. The variation of F_s with U_s is plotted in **figure 3**. The result shown in figure 3 suggests that the adhesion of the NiCu film to the MgO(001) substrate appreciably increases with an application of U_s from −60 V to −140 V.

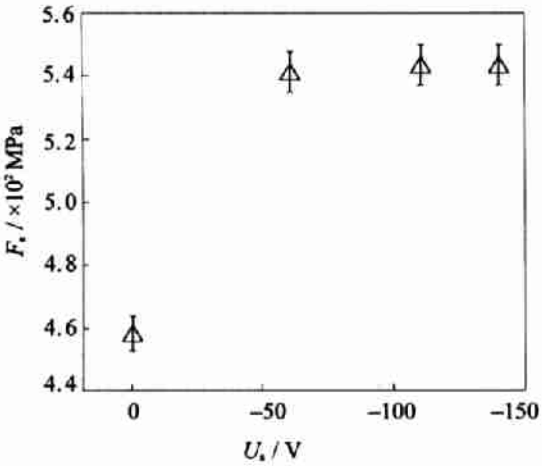

Figure 3 A variation of the apparent adhesion F_a corresponding to W_c estimated from figure 2 with U_s.

3 Discussion

In general, the cleaning of the substrate surface, the formation of compound and/or transition layer between the film and the substrate can cause an enhancement in the adhesion [2–6]. **Figure 4** shows the cross-sectional TEM microphotographs of the NiCu films prepared at various U_s. As can be seen from it, the interface between the film and the substrate is very sharp, suggesting that no transition layer between them is formed at any U_s. Besides, the cross-sectional electron diffraction patterns indicate no compound formation in the films at any U_s [7]. Furthermore, the high resolution cross-sectional TEM observation also confirms no formation of compound and transition layer between the film and the substrate [8]. Therefore, it may be said for the present case that the adhesion increases with an application of U_s principally because residual impurity admolecules are sputtered off from the substrate surface during the film initial formation by the energetic Ar ion particles accelerated by U_s towards the substrate. Now, not only the kinetic energy of the incoming Ar ion particles but also the ratio of the number of incoming Ar ion particles to the number of incoming atoms to be deposited have to contributed to sputter off the residual impurity admolecules. Both factors are controlled by U_s. The average kinetic energy E_a of the incoming Ar ion particles and the ratio of the incident Ar ion flux J_{Ar} to the incident atom flux J_{NiCu} are listed as a function of U_s in **table 2** [11, 12]. E_a was approximately calculated in terms of the Davis and Vanderslice theory [13] and J_{Ar}/J_{NiCu} was

Table 2 Values of average energy E_a of Ar ion particles striking the substrate and ratio J_{Ar} / J_{NiCu} of Ar ion flux to adatom flux at any U_s

U_s / V	0	−60	−110	−140
E_a / eV	0	12.8	20.7	24.0
J_{Ar} / J_{NiCu}	0	1.9	1.9	1.7

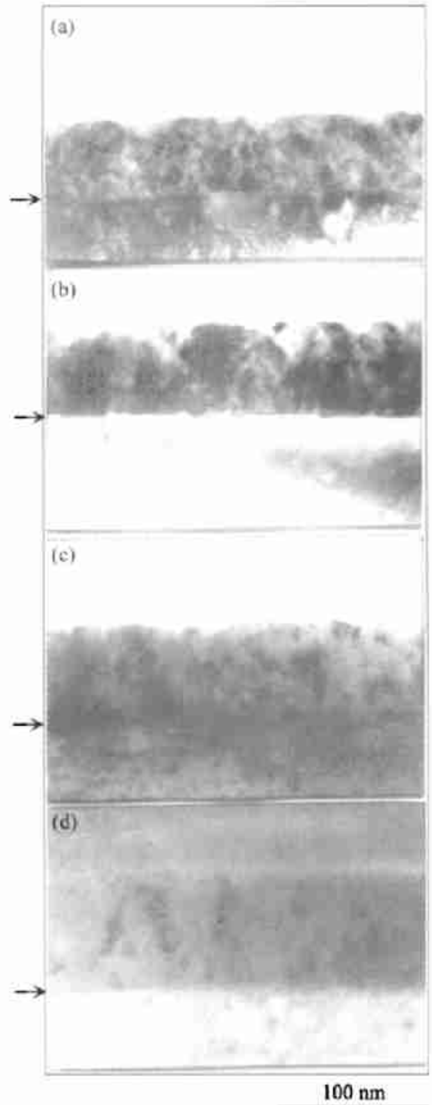

Figure 4 XTEM microphotographs of NiCu films prepared on MgO(001) at various U_s, (a) 0 V; (b) −60 V; (c) −110 V; (d) −140 V.

calculated with the use of the substrate current and the deposition rate with the assumption that the contribution of the secondary electrons from the substrate can be neglected [11]. Table 2 suggests that E_a = 12.8 eV and J_{Ar}/J_{NiCu} = 1.9 are effective enough in increasing the adhesion.

4 Conclusions

The adhesions of the NiCu films DC biased plasma-sputter-deposited on MgO (001) were characterized using a scratch test. The application of the negative bias voltage U_s to the substrate enhances the adhesion of the film to the substrate because the energetic Ar ion particles accelerated by U_s sputter off the residual impurity admolecules from the substrate surface.

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References

[1] M. Ohring: *The Materials Science of Thin Films*, Academic Press, New York, 1992.
[2] D. M. Mattox: *J. Vac. Sci. Technol.*, A7 (1989), p. 1105.
[3] M. J. Mirtich: *J. Vac. Sci. Technol.*, 18 (1981), p. 186.
[4] C. Weaver: *J. Vac. Sci. Technol.*, 12 (1975), p. 18.
[5] L. E. Collins, J. G. Perkins, P. T. Stroud: *Thin Solid Films*, 4 (1969), p. 41.
[6] J. E. Greene, M. Pestes: *Thin Solid Films*, 37 (1976), p. 373.
[7] H. Qiu, M. Hashimoto, A. Barna, *et al.*: *Thin Solid Films*, 288 (1996), p. 171.
[8] H. Maruyama, H. Qiu, M. Hashimoto, *et al.*: *Thin Solid Films*, 299 (1997), p. 59.
[9] H. Qiu, G. Safran, B. Pecz, *et al.*: *Thin Solid Films*, 229 (1993), p. 107.
[10] A. Kinbara, H. Fujiwara: *Thin Films* (in Japanese), Syokabo, Tokyo, 1993.
[11] H. Qiu: *Structural and Physical Properties of Nickel Films Biased DC Sputtering-Deposited on Si(001), SiO_2 and MgO (001)*: [*Ph. D. thesis*]. Japan: The University of Electro-Communications, 1994.
[12] H. Qiu, H. Nakai, M. Hashimoto, *et al.*: *J. Vac. Sci. Technol.*, A12 (1994), p. 2855.
[13] W. D. Davis, T. A. Vanderslice: *Phys. Rev.*, 131 (1963), p. 219.