Electrocomponent Science and Technology, 1980, Vol. 7, pp. 159–162
0305–3091/80/0701–0159$06.50/0

PROPERTIES OF NICKEL FILMS PREPARED BY R.F. SPUTTERING AND INTERDIFFUSION ANALYSIS OF Ta_2N–Ni FILMS

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(Received March 13, 1980)

Ni film deposition by r.f. sputtering and etching conditions have been investigated. The contact resistance and adhesion of this Ni layer deposited directly onto Ta_2N films have also been studied in air and at elevated temperatures. In this combination, the Ta_2N/Ni interdiffusion analysis was carried out by the Secondary Ion Mass Spectroscopy (SIMS).

1. INTRODUCTION

A mechanically strong Nickel layer with a low contact resistance could be deposited directly on to Tantalum Nitride (Ta_2N) presputtered film.

The classical heat treatment of the generated Ta_2N film resistors can be done at 250°C for 16 hours without significant diffusion of Tantalum in the Nickel conductor layer. On the other hand the diffusion of Nickel in Tantalum decreases the contact resistance and improves the adherence between the two layers.

A nickel oxide layer less than 150 Å is formed during the heat treatment. This layer was removed in the soldering process.

2. FILM PREPARATION

The nickel films were deposited by r.f. sputtering techniques[1] using a Randex (model 3400) module.

Three types of substrates were used; corning glass, alumina (MRC) hybrid quality and optically polished alumina for SIMS analysis.

The glass substrates (58 x 70 mm) were used in the study of the deposition rate and the resistivity of the Ni films as a function of the argon pressure and the input r.f. power. The choice of the glass substrates for these experiments was due to their good surface. The substrate-to-target distance was maintained at 5 cm and the back ground pressure was 6×10^{-7} Torr. The target was a disc of 8 in diameter and sputtering was carried out at an r.f. input power of 1.5 kW.

Figure 1a represents the resistivity (ρ) and deposition rate (D) of the sputtered Ni films as a function of various argon pressures. The resistivity was determined by four point probe techniques. Deposition rate was determined by weight measurements before and after sputtering.

From Figure 1a, we see that the optimum condition to obtain low resistivity values necessitates an argon pressure in the range of 1.5 to 2.2×10^{-2} Torr. At 3×10^{-2} Torr, the deposition rate decreases with increasing pressure. This is due to the excessive collisions of the ejected atoms which prevented them from reaching the anode.

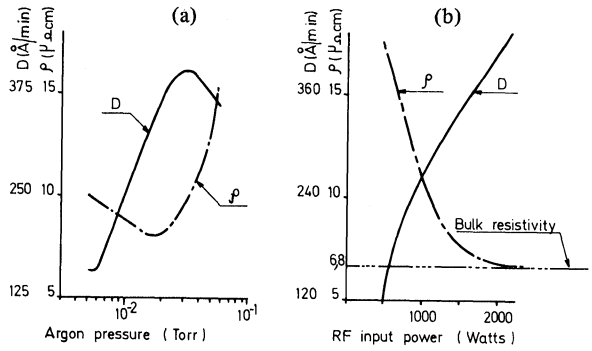

FIGURE 1 (a) Resistivity and deposition rate vs. argon pressure. (b) The dependance of resistivity and deposition rate on the r.f. input power.

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In Figure 1.b, the deposition rate and the resistivity are plotted against the r.f. input power for argon pressure of 1.7×10^{-2} Torr and sputtering time of 20 minutes. At input power of 2.5 kW, the resistivity reaches its theoretical bulk value of 6.84 $\mu\Omega$cm.

2.1 *Contact Resistance and Adhesion Measurements*

In the present study, the Ni layers were deposited on alumina ceramic substrates (50 x 50 mm), which were precoated with reactively sputtered Ta_2N (500 Å, $\rho = 250\ \mu\Omega$cm.)[2] The effect of accelerated ageing conditions on both contact resistance and adhesion to Ta_2N have been investigated.

A special test mask was designed with intermittent resistor pattern and conductor pads (see Figure 2a). To obtain the required thin film pattern, a selective etching method was used. It was found that the best etching solution for Ni films is cerium nitride at 35°C, with etching rate of 350 Å/min. The other etching solutions such as iron chloride left a veil of nickel oxide.

The pattern used for the contact resistance measurements had four intermittent Ta_2N resistors each of one third square in series with five squares Ni coated resistors. We have used the following equation to deduce the values of the contact resistance (Rc).

$$Rc = \frac{Rt + 3Rs - \sum_{4}^{1} Ri}{6}$$

where Rt, Rs and Ri are the total resistance of the intermittent pattern, the sheet resistance of conductor coated Ta_2N and the Ta_2N intermittent resistances respectively.

The initial contact resistance between the Ta_2N and Ni layers measured from as prepared intermittent resistors ranged between 60 mΩ/contact and 100 mΩ contact. After ageing in accelerated conditions in air at 250°C for 16 hours and then at 150°C for 2000 hours, the contact resistances were always stable and showed no change in their initial values.

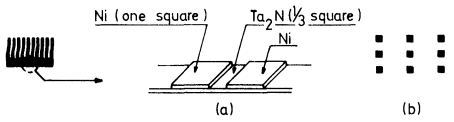

FIGURE 2 Parts of the test pattern used in (a) contact resistance measurements (b) pads for adhesion test.

2.2 *Adhesion Test*

For the adhesion test, we used the wire peel test "Du Pont Test"[3] and each alumina substrate had nine test pads of uniform size, 2.0 x 2.0 mm (see Figure 2b). In this method, a 0.6 mm tin-plated copper wire is bent at one end to enable it to be soldered onto the conductor pad. The Ta_2N/Ni films are heat treated firstly at 250°C for 16 hours and then the soldering process was done by dipping in a bath of molten solder held at a constant temperature at 220°C for 5 seconds. The measurements were made on a tensile testing machine Istron.

Figure 3 shows the breaking strength as a function of the time of heat treatment for different temperatures. In each case the breaking strength was averaged over three substrates (27 pads).

In this test it was noted that:

1) The Ni layer was scratched and left the Ta_2N layer which has a good adhesion to the alumina substrate.

2) Some measurements at 20°C were carried out on the adhesion of Ni layers directly sputtered on alumina and the breaking strength value was found to be approximately that of Ni/Ta_2N layers (7.5 kg/pad).

3) After ageing in either of the accelerated conditions, the Ni layer succeeded the scotch tape test.

From the above remarks, we see the possibility of device application of Ta_2N/Ni system where Ta_2N film was in direct contact with the Ni film.

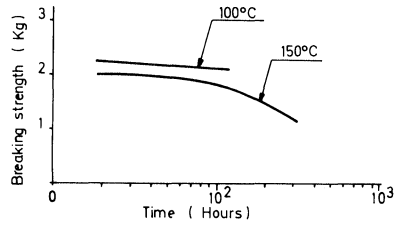

FIGURE 3 Breaking strength vs. time of heat treatment at 100°C and 150°C for pad area of 4 mm².

3. INTERDIFFUSION ANALYSIS

The method used for the study of the diffusion between Ta_2N and Ni layers is the secondary ion mass spectrometry (SIMS). The Ni and Ta profiles in our samples have been determined by a CAMECA

IMS 300 ion analyzer.[4] The measurement of the amplitude of Ni and Ta ion signals was carried out from the heat treated nickel film on Ta_2N as a function of the depth.

The instrument is designed in such a way that only the ions from the centre of the eroded crater (which has a surface area of 7×10^{-2} mm^2) are analyzed, thus eliminating the effect of the redistribution of ions coming from the outer area. The depth of the crater was measured by an optical interference technique. The crater is produced by scanning the ion beam, its bottom is flat and depth profiling can be obtained with a resolution ~150 Å.

Some remarks on interpretation of the SIMS result.

i) The ion amplitudes are presented in relative intensity, while we do not have standard samples for the quantitative analysis.

ii) The ion yields of one element could be varied by many orders of magnitude from one matrix to another (matrix effect), so ion signal linearity does not exist for the same element in the two layers of different nature.

Figure 4 summarizes the Ni and Ta profiles in relative intensity (internal standard $^{27}Al^+$ was used from Al_2O_3) as a result of annealing at 250°C for 16 hours, 450°C and 600°C for one hour. In this figure Ni and Ta profiles of an unannealed sample are also presented.

We can make the following remarks on interdiffusion analysis:

1) In Figure 4a, the existence of a certain quantity of nickel in the unannealed Ta_2N layer is related to the preparation conditions. During r.f. sputtering of Ni, the film temperature was sufficiently raised[5] to cause some Ni diffusion in the Ta_2N film.

2) With increasing annealing temperature there is more Ni diffusion in the Ta_2N film.

3) We observe neither Ni nor Ta diffusion in the alumina substrates up to 450°C, a slight diffusion can be observed at 600°C.

4) Ta diffusion in Ni layer became evident up to 450°C and at 600°C we observed an important diffusion of Ta in Ni.

5) The higher ion intensity was observed at the surface and different interfaces could be attributed to the enhancement of the ion signal due to the presence of chemisorbed oxygen.[6]

6) The ion profile of N_2 as a function of depth was also studied. We found a homogeneous distribution of N_2 in the Ta_2N film and a slight diffusion of N_2 in the Ni film was also noted.

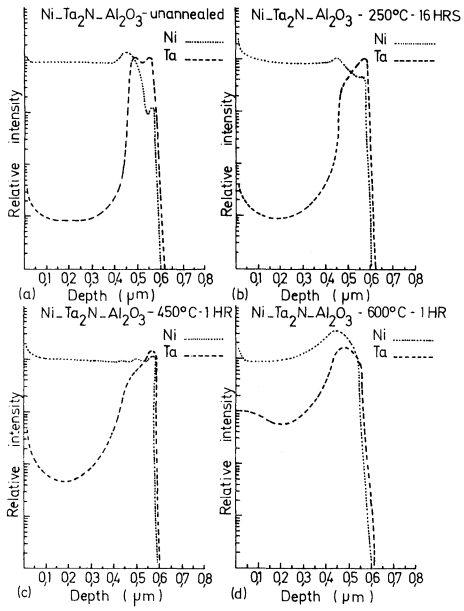

FIGURE 4 Ni and Ta profiles as a function of depth. (a) unannealed; (b) annealed at 250°C, 16 h; (c) 450°C, 1 h; (d) 600°C, 1 h.

4. CONCLUSION

It has been shown in this study that metallizing Ta_2N films directly with Ni conductor films using r.f. sputtering techniques can give satisfying results for contact resistance and adhesion. Moreover, soft solder can be used between the Ni layer and outer connection which simplifies the soldering process.

The contact resistance changes and the breaking strength were examined as a function of heat treatment temperature and time. In these conditions, the contact resistance was stable and the conductor pads succeeded the scotch tape test. Interdiffusion analysis showed a slight diffusion of Ta in Ni film for temperatures of classical heat treatment, this diffusion has no effect on the solderability of Ni.

ACKNOWLEDGEMENTS

The authors wish to thank M. JP. Truffer for his help in film preparation and M. P. Merenda for helpful discussion.

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