

AGEING TESTS ON GOLD LAYERS AND BONDED CONTACTS

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Studies have been made of the adhesion of gold thin films on NiCr and Mo layers deposited on ceramic substrates. It is shown that the ageing behaviour and the related long-term adhesion losses are complex processes and the various contributing mechanisms are identified.

1. INTRODUCTION

Searching for physical reasons for the degradation of integrated circuits, we find that the weak links are often the thin-film metallizations, the bonded or soldered contacts, and the hybrid metallizations. The metal layers mainly limit the lifetime of the microelectronic devices during their application.

Several attempts have been made to undertake an analysis of the degradation kinetics of gold thin and thick films which is closely related to the material structure.^{1,2} This is in contrast to the analysis of statistical reliability, where usually a black box analysis of the electronic behaviour of a device leads to probability data.³

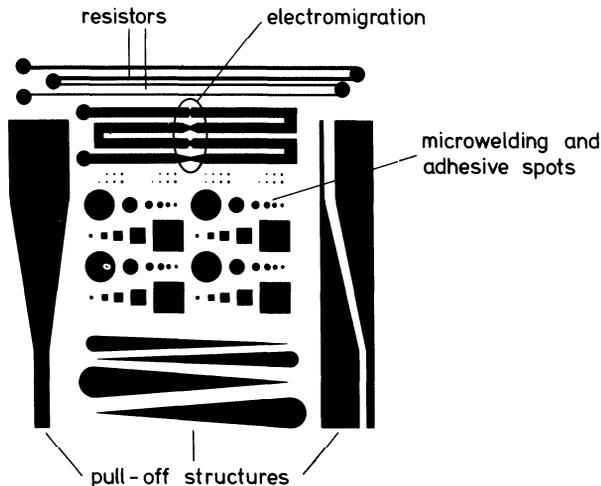


FIGURE 1 The test pattern, used for ageing measurements by DC resistivity and adhesion tests

All material failure is dynamic. It advances by rate processes which have threshold conditions and characteristic growth kinetics.⁴ In particular, the physical properties of Au thin films change during their life time under the influence of various factors,

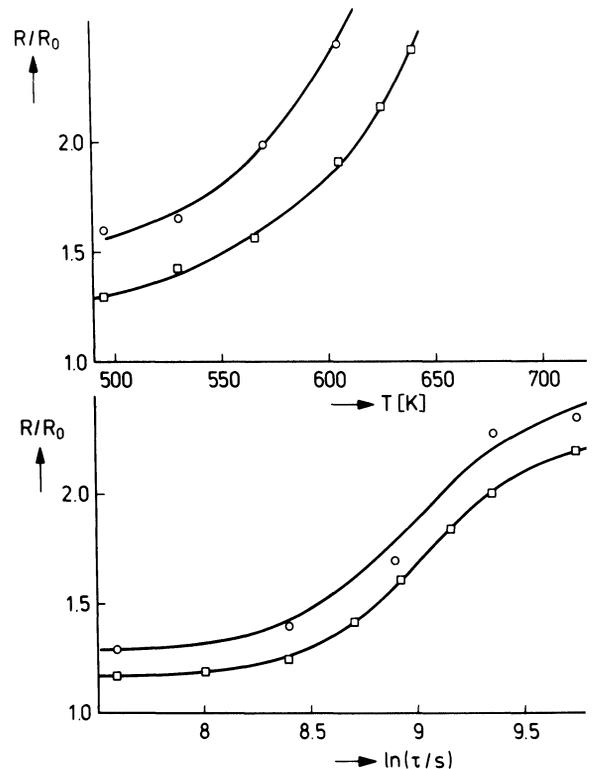


FIGURE 2 Resistivity changes of a sputtered $0.5 \mu\text{m}$ Au layer on a 50 nm NiCr adhesion layer. \square = heat treatment without electromigration; \circ = heat treatment with electromigration ($1.1 \times 10^7 \text{ A cm}^{-2}$ DC current density)

such as the grain size, the lateral homogeneity of the grain structure, the chemical composition, the defect accumulation at the free surface and the interfaces, and finally the mechanical stresses.

Knowledge about the processes concerning the ageing makes it possible to establish a thermodynamical calculation for simple cases e.g. like the recovery of defects¹ or the diffusion of humidity through encapsulations⁵ the latter of which is thought to govern layer degradation by corrosion. In Section 2 of this paper we shall describe the structural defect dependent ageing processes in pure and sandwich layers of Au.

The term "ageing" is used to describe variation with time, temperature and electrical stress which produces irreversible change in physical and chemical properties during the treatment. In Section 3 the problem of the adhesion between the layers and the substrate will be discussed in terms of the ageing kinetics.

2. THE AGEING KINETICS OF GOLD LAYERS

Pure, fine-grained fcc. metal films with a thickness of the order of 0.5 μm to 3 μm have, in contrast to the respective bulk metals, a high density of atomistic defects. This is expressed in terms of a high internal stress⁶ and of a high dislocation density.⁷ Both show high rates of recovery, due to diffusion and transformation in the crystalline structure, at temperatures below the bulk recrystallization temperature of 300–360°C for pure gold.⁸

For a precise determination of the ageing kinetics using the MEECHAN-BRINKMAN (M-B) method,⁹ the DC resistivity is a useful physical property: it is

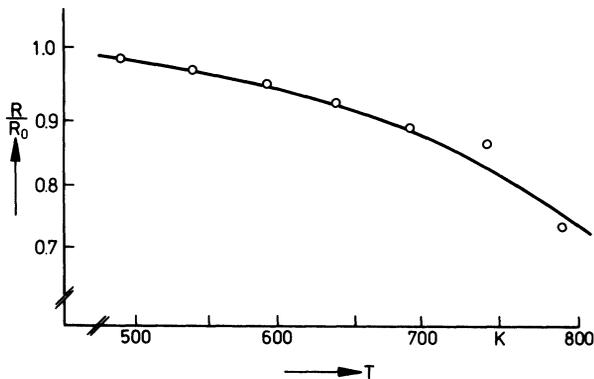


FIGURE 3 Resistivity decrease of a sputtered 0.5 μm Au layer on a 50 nm Mo adhesion layer

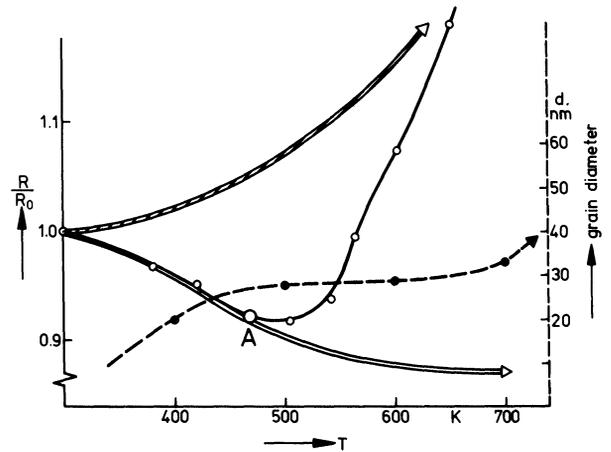


FIGURE 4 Resistivity change of an evaporated 5 μm Au layer
 ○ = measured values
 ● = the grain coarsening of thin Au layers (right scale)
 — = resistivity decrease due to recovery
 — = resistivity increase due to Cr diffusion into the Au

sensitive to the defect concentration and it is measured in a simple way with high accuracy.¹

The applied test pattern, Figure 1, contains strips with different cross sections. The two configurations with wide strips are used for investigating the electromigration kinetics. The narrow strips are used for the DC measurements. The other sections of the test pattern contain square and round spots used for adhesion tests on bonded contacts, and areas for diffusion tests using electron microprobe analysis.

The DC measurements were carried out at 78 K (liquid nitrogen) after each isochronal (increasing temperature and constant time interval) and isothermal (increasing time, constant temperature) heat treatment.¹ The classification of the measured

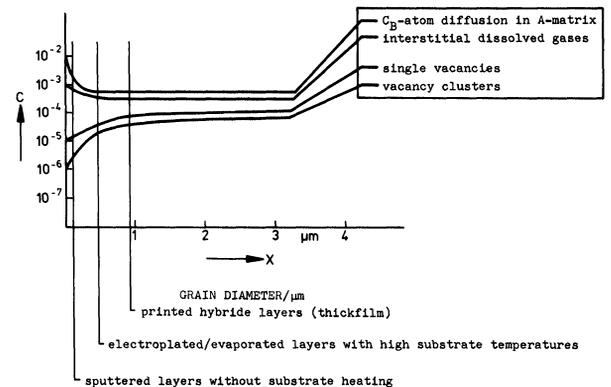


FIGURE 5 Structure defect concentrations beneath high angle grain boundaries^{1 5}

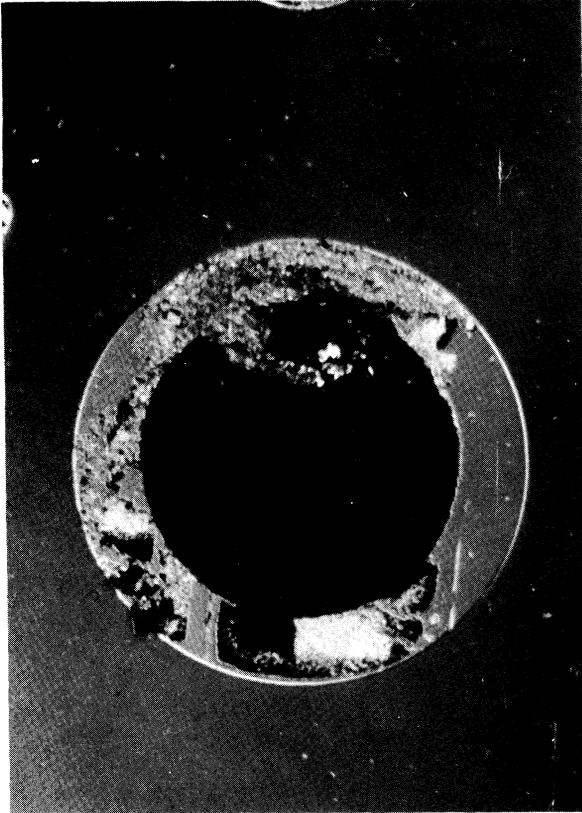


FIGURE 6 The corrosion of an adherence layer of 50 nm Ti under a 0.1 μm sputtered Au layer (spot diameter 1 mm)

resistivity variations leads to the following types of behaviour:

- The diffusion sensitive systems, e.g. Au thin films with NiCr adherence layers show a remarkable increase in the DC resistivity during the isochronal heat treatment, Figure 2. This is explained by the diffusion of adherence layer components into the pure gold layer. It should be noted that an additional DC current during the heat treatments increases the residual resistivity. In this case, additional defects near grain boundaries are introduced, the diffusion of the Ni and Cr into the Au is not markedly disturbed.
- The non-diffusion sensitive systems show a continuous decrease of the DC resistivity within the limits of a few percent. Figure 3 shows the behaviour of a 0.5 μm Au thin film on a 50 nm Mo adherence layer. In the bulk we do not find any solid state miscibility. The recent observations of the diffusion of Mo through Au layers¹⁰ must be interpreted by grain boundary diffusion.

Thus we find two basic kinds of behaviour which govern, in a more or less complex manner, the ageing characteristics of the measured systems, Au layer/adhesion layer. If the diffusion and the recovery both contribute to the change in the DC resistivity, the resulting plots can be separated to show the contributions. Figure 4 shows the ageing kinetics of a 5 μm thick Au film on a very thin Cr adherence layer in the temperature range up to 350°C. At lower temperature, the recovery of Au vacancies near the grain boundaries takes place.¹ Above the point A ($T \cong 200^\circ\text{C}$), the mobility of the Cr atoms predominates and leads to increasing DC resistivity (the diffusion coefficient of the low-concentrated Cr in the Au matrix is

$$D = 1.5 \times \exp -(0.4 \pm 0.06)/kT) \text{ cm}^2 \text{ s}^{-1}$$

The diffusion of Cr (and of Ni) from the NiCr adherence layers into the Au thin films can be examined by electron microprobe analysis of the thin Au films. The mass center of the adherence layer elements shifts upwards from the original position into the volume of the Au layer. For example, in a 0.1 μm thick Au layer on a 10 nm NiCr adherence layer, the original ratios between the characteristic X-ray intensities are:

$$\text{Au}(M\alpha_1) : \text{Cr}(K\alpha) : \text{Ni}(K\alpha) = 1:0.105:0.044.$$

A heat treatment by continuous temperature increase from 155°C to 325°C in vacuum in 1.6 h, leads to an increase of the relative Cr and Ni X-ray signals:

$$\text{Au}(M\alpha_1) : \text{Cr}(K\alpha) : \text{Ni}(K\alpha) = 1.0:0.126:0.060.$$

This corresponds to diffusion coefficients of $5 \times 10^{-11} \text{ cm}^2 \text{ s}^{-1}$ and $\geq 10^{-9} \text{ cm}^2 \text{ s}^{-1}$ for Cr and Ni respectively at $T = 200^\circ\text{C}$. These values are in agreement with observations of the surface concentration of Cr in Au by other physical methods.^{10,11}

The kinetics of the observed ageing process in Au thin films which react with the adherence layers and change their own defect distributions become still more complex if we take into consideration the equilibrium defect distribution near free surfaces and grain boundaries (Figure 5.) The ageing analysis using the M-B method gives values of the activation energies of the predominant reaction of the lattice defects.

3. ADHESION LOSSES OF BONDED CONTACTS

If the adherence layers react by diffusion with the Au layers, the residual gold-rich interface to the oxide

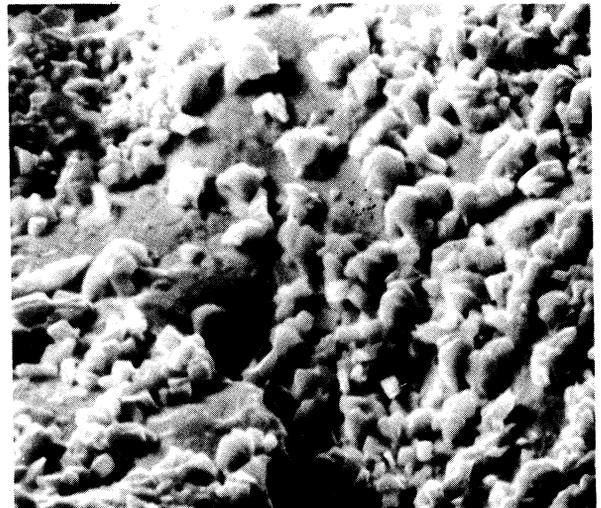
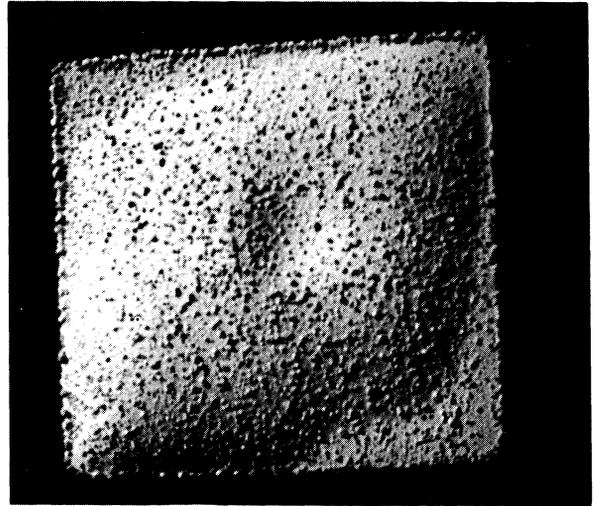


FIGURE 7 Crystallization effects in a $0.5 \mu\text{m}$ Au nm $\text{Ti/SiO}_2\text{-Si}$ substrate system

- (a) chemical reaction products of *Ti*-silicate (length of bottom edge = $34 \mu\text{m}$)
- (b) vaulting of a structured layer system (length of side of square = 0.75 mm)
- (c) crystal growth through the Au layer (length of bottom edge = $17 \mu\text{m}$)

substrate does not have sufficient adhesion. Consequently the "adhesion" must be related to the time and temperature dependent longterm ageing kinetics.^{1,2} The term "adhesion" is to be understood as the mechanical resistance which a multilayer structure consisting of a thin film and a substrate has to "pull-off," blistering and other irreversible kinds of destruction.

In this section we do not consider the zero-hour adhesion which can be related closely to fracture mechanics. The long term adhesion is based on the creep fracture of composite structures involving the influence of corrosion at the crack tip. The adhesion layer also reacts chemically with the oxide substrate. Figure 6 shows the corrosion in the flaw between the substrate and the Au layer. Figures 7a, b show the formation of crystals out of the plane of the adhesion layer. These chemical reaction products are growing through the Au layer (Figure 7c). It should be noted that these reactions of crystalline transformation are not stress dependent as in the case of the whisker growth through a hard cover layer.^{1,3} (The driving

force originates from the loss in the free enthalpy by means of the large crystal formation.)

The effect of an external tensile force e.g. attached to a microwelded beam lead is shown diagrammatically in Figure 8.^{1,2} The different geometrical relationships of the arrangement consisting of the substrate/thin film/beam lead system lead to different kinds of tensile stress distributions. The sharper the crack tip the higher will be the local maximum of the normal stress in the thin film. Table I shows the lifetimes of diffusion sensitive adhesion layer/Au layer systems which were contacted by thermocompressive beam lead bonds and subjected to steady-state creep tests.

The diffusion governed process of the adhesion loss by sharp crack propagation is explained by the

TABLE I
The limits of lifetime of beam lead bonded Au thin films with diffusion sensitive adhesion layers on Si-SiO₂ substrates

Au thinfilm, thickness [μm]	Adhesion layer, thickness [nm]	Mechanical stress [MPa]	Temperature range [°C]	Lifetime limits [h]
0.50	NiCr, 50	2	250 to 300	9 ± 2.5
		2	30 to 100	100 ± 30
		10	250 to 300	4 ± 1.5
		10	30 to 100	12 to 20
0.10	NiCr 10	2	120 to 200	2.2 ± 0.5
		2	30 to 80	10 ± 2
		6	30 to 80	≤ 6
0.10	Ti 10	2	200 to 300	6 to 60
		6	100 to 200	5 to 50

model shown in Figure 9. The creep stability of the fine grained thin film is governed by the grain boundary creep rate. This is accompanied by creep induced diffusion of the adhesion layer elements through the grain boundaries in the gold. The kinetics is determined by the vacancy migration shown in Figure 9. This leads to a steady-state creep rate $\dot{\epsilon}$ given by

$$\dot{\epsilon} = \frac{2\delta\sigma\Omega}{kTR^3} \frac{D_1D_2}{n_2D_1 + n_1D_2} :$$

where δ = width of the grain boundary, σ = shear stress, Ω = average atomic volume, R = grain radius, D_1 = diffusivity of Au vacancy, D_2 = diffusivity of the

adhesion layer element in Au, n_1, n_2 = initial concentration of the components.

Using the simplification that no chemical bonds are ruptured in the cracking interface, we get, for the example of Au grains (with $R \cong 100$ nm, $d \cong 0.3$ nm, $\Omega = 0.007$ nm³, $\sigma = 10$ MPa, $T = 300-400$ K,) and Cr as the tracer diffusor, (the grain boundary self diffusivity of Au D_1 as in Gupta reference,² the Cr diffusivity in Au D_2 as in Holloway reference:^{1,4}

$$\dot{\epsilon} = 3 \times 10^{-10} \text{ s}^{-1} - 3 \times 10^{-7} \text{ s}^{-1},$$

if we assume grain boundary occupation densities of between 0 and 10%.

The whole system consisting of the beam lead with

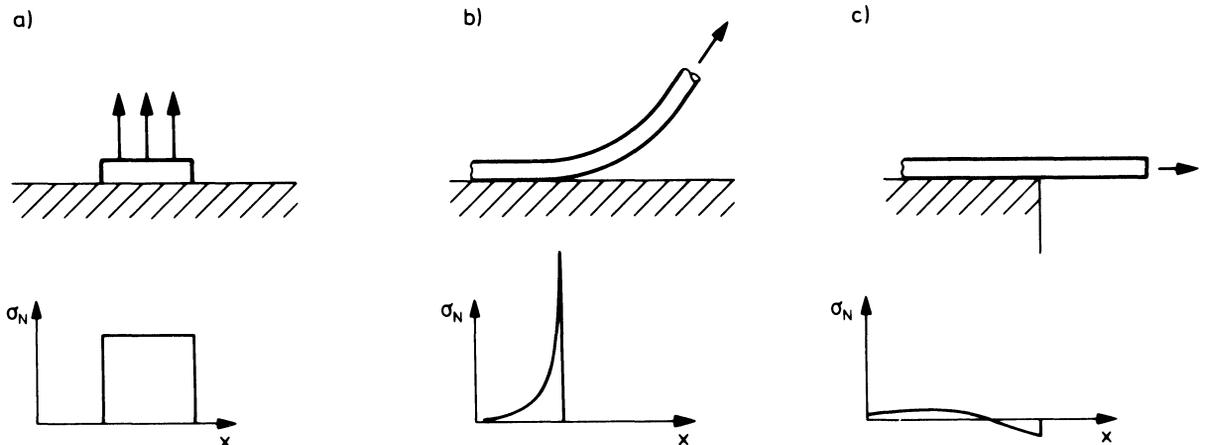


FIGURE 8 Stress distributions for different directions of force introduction

- a) pure normal stress
- b) stress maximum at the crack tip
- c) bending of the beam leads to higher shear and lower normal stress

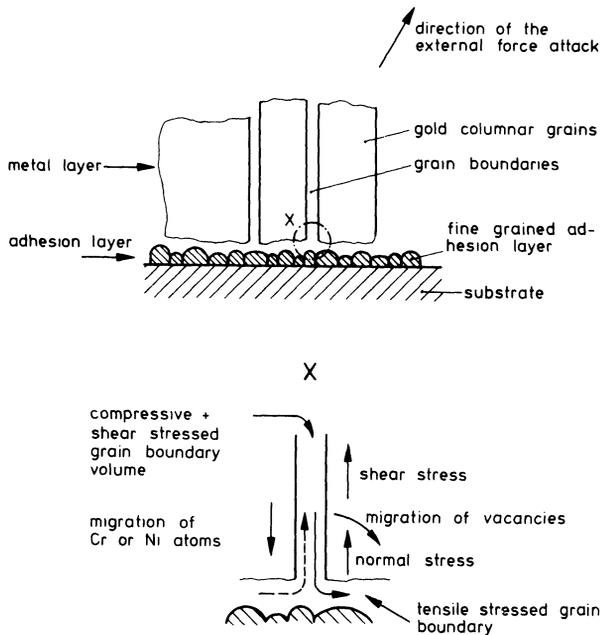


FIGURE 9 The mechanism of grain boundary creep assisted by the segregation of the adhesion layer material in the Au layer grain boundaries.

a curvature radius of ≈ 0.4 mm and a thickness of $0.1 \text{ mm}^{1/2}$ will provide crack velocities between $10^{-9} \text{ mm s}^{-1}$ and $10^{-6} \text{ mm s}^{-1}$. The total crack lengths of 0.2 to 0.35 mm yield lifetimes between 2 h at $T = 400$ K and several days at $T = 300$ K. This is in a good agreement with the experimental results.

4. CONCLUSIONS

It is evident that the ageing kinetics of an Au thin film/adhesion layer composite and the related long term adhesion losses are complicated effects which result from the occurrence of several reactions, namely:—

— the recovery of atomistic defects

- the diffusion of the adhesion layer elements into the gold thin films
- the stress-induced grain boundary creep
- the chemical reaction of the adhesion layer.

The layer thicknesses, the grain and the atomistic defect structure, and the geometry of the introduced external force determine the kinetics of the thin film degradation and of the adhesion losses.

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