Properties of High-SpeedGoldElectrodeposits AppliedToCeramicElectronicPackages

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In this study, the Ni diffusion barrier characteristics of a relatively high-speed (> 8 mA/cm²) gold deposit electroplated upon electrodeposited Ni in an in-line, conveyorized system were correlated with the Au layer's microstructural characteristics, including surface morphology, texture (preferred orientation), crystallite size, and microstrain. Depositing the Au upon a Ni surface which had been recrystallized by heat-treatment (sintering) achieved, in the as-plated condition, the microstructural features important to good diffusion barrier performance (large grain/crystallite size, lower defect density). If deposited on heat-treated Ni, the Au deposit contained relatively large grains and had a slightly preferred <111> orientation. It was a better Ni diffusion barrier than the finergrained Au deposited on unsintered Ni. The latter Au deposit had a slightly preferred <100> orientation. In addition to larger grain size, the Au deposited on sintered Ni contained larger crystallites or sub-grains and a lower degree of nonuniform microstrain. The lower density of grain boundaries and defects limited the extent of Ni diffusion to, and oxidation on, the gold surface during heat testing in air at 450 °C for 5 min. Annealing the Au deposited on an unsintered Ni surface at 335 °C slightly improved the diffusion barrier performance by increasing crystallite size and apparently reducing defect density. Further improvement was obtained using a 450 °C anneal. Diffusion that occurred during annealing, however, limited the extent to which annealing improved the performance.

Single and multilayer alumina ceramic packages commonly use refractory metallization (Mo, W) and Ni/Au plated finishes (Fig. 1). After nickel plating, a diffusion bond is established between the refractory metallization and the Ni layers, usually by a high-temperature heat-treatment or sintering step. The sintered Ni is then over-plated with another Ni layer, then a Au layer or with Au directly to provide and preserve die and wire bondability, lid sealability and solderability.

For ease of handling, the manufacture of ceramic packages, such as the single-layer leadless chip carriers shown in Fig. 2, is carried out on rectangular arrays of many individual package sites. If the Ni/Au layers are applied by electroplating, individual package sites on an array are interconnected so that plating current can be supplied to all areas of the array.

Conventional plating methods for patterned arrays of this type involve rack plating. For economy of scale, racks are often designed for the simultaneous plating of large numbers of arrays in high-volume tanks. These methods can introduce variations in the array-to-array plating quality because each array experiences a somewhat different plating environment. Anode/cathode geometry, solution agitation, and local chemistry may vary widely over a large rack, resulting in differences in functional properties, microstructure and plating thickness. One way to reduce array-to-array quality variations is to process the arrays using small volume cells in an inline continuous, conveyorized plating system. In a plating line of this type, each array will see the same physical, chemical, and electrical environment.

To achieve acceptable manufacturing rates in small volume cells, gold electrodeposition must be carried out at current densities somewhat greater than those used in conventional systems. In the nearly neutral solutions designed for conventional rack plating, direct-current plating is usually carried out at less than 5 mA/cm².¹ Excessively high current densities lead to degradation of quality, such as discoloration, roughness, thickness non-uniformity, and increased porosity.² To achieve the required rates in small volume cells, high-metal and electrolyte concentrations, rapid solution movement through the cell, and vigorous agitation are required.

One of the most important qualities of the Au finish is its ability to inhibit the diffusion of Ni through the Au layer during heating. With exposure to the heat and oxidizing



Fig. 1—Typical metallization/plating structure on co-fired ceramic IC packages.



Fig. 2—Single-layer leadless ceramic chip carriers showing: (A) die bond and wire bond pads; (B) solder pads.

Table 1 Ni/Au Finishing Sequences

Seguence 1	Sequence 2
Ni Strike	Ni Strike
Ni Plate (1-2 µm)	Ni Plate (~2 µm)
Ni Sinter	Ni Sinter
Ni Strike	Au Strike
Ni Plate (2-5 µm)	Au Plate (O.8-1.5 μm)
Au Strike	Au Plate (0.8-1.5 µm)

conditions of package assembly and interconnection, Ni diffuses to the top surface of the gold where it is subsequently oxidized. Nickel oxide on the Au surface can interfere with die-bonding, wire-bonding, lid sealing and soldering. Because the rate at which Ni arrives at and NiO forms on the Au surface is relatively rapid compared to that predicted from bulk diffusion rates, Hall and Morabito³ proposed a grainboundary diffusion model for this transport of Ni through the deposited Au layer. Porosity in the Au deposit has also been cited as a cause for accumulation of Ni on the Au surface.⁴ Analytical studies have confirmed that the Ni transport rate through deposited Au during heating in air is consistent with the grain-boundary diffusion model.^{5,6} Our earlier work established that there is a relationship between gold microstructure and the extent and distribution of NiO accumulation on the Au surface during heating.^{7,8} In general, deposits characterized by large, faceted grains demonstrated the best performance. This is believed to result from the lower density of grain boundaries in these large-grained deposits.9

Experimental Procedure

In this study, pure, electronic-grade gold deposits were electroplated under d-c conditions in an in-line, conveyorized system at relatively high speed (>8 mA/cm²). Two different Ni/Au finishing sequences were examined (Table 1) using the processes described in Table 2.

The sintering of Ni was carried out at a temperature of 850 to 920 °C for 10 min. in a reducing atmosphere (N_2/H_2). Samples generated by Sequence 1 were also evaluated after



 $Fig. \ 3-Nickel \ concentration \ on \ Au \ surfaces \ after \ 450 \ ^\circ C/5 \ min/air \ heat \ test$ and 30-sec sputter.

Table 2 Finishing Processes

Ni Strike - Sulfamate/Chloride

Ni conc. (g/L)	
рН	
Temp (°C)	
Current density (mA/cm ²)	

Ni Plate^b - Sulfamate

Ni conc.(g/L)	75
Boric acid conc. (g/L)	
Ni bromide (g/L)	
рН	3.5-4.5
- Temp (°C)	
Current density (mA/cm ²)	

Au Strike^c - Buffered KAu(CN),

Au conc. (g/L)	
рН	3.8-4.2
Temp (°C)	54
Current density (mA/cm ²)	

Au Plate^d - Buffered KAu(CN),

Au conc.(g/L)	
рН	5.8-6.4
Brightener	Pb (1-2 ppm)
Temp (°C)	
Current density (mA/cm ²)	8 - 12

^b Techni-Nickel Sulfamate (FFP), Technic, Inc., Cranston, RI.

Orostrike C, Technic, Inc., Cranston, RI.
MIL-G-45204C, Type III, Grade A, ACR 434HS, Technic, Inc., Cranston, RI.

e PHI Model 660, Physical Electronics, Eden Prairie, MN

^f PAD V Goniometer, Scintag, Inc., Cupertino, CA.

post-plate annealing. Two different annealing processes were carried out in a reducing atmosphere (H_2/N_2) . In the first, the temperature was 400 to 450 °C for 12 min. In the second process, the temperature was 300 to 335 °C for 8 min. The former will be referred to as the 450 °C anneal, the latter as the 335 °C anneal.

After the finishing process, Au thicknesses were determined by X-ray fluorescence. Auger electron spectroscopy(AES) was used to determine the concentration of NiO on the Au surfaces after they were subjected to a heattest to promote Ni diffusion and oxidation. This procedure involved placing the parts in an air oven operating at 450 °C for 5 min. Data were obtained using a scanning Auger microprobee in the survey mode. Prior to analysis, the specimens were sputtered for 30 sec. using a 3.5 KeV Ar⁺ beam. Under these conditions, the sputter rate of electroplated Au in the absence of surface films was determined to be about 400 Å/min. The data obtained after 30 sec of sputtering time were used as an indicator of both the concentration and depth of the NiO. As in our previous work, it was used as a "figure of merit" to compare finishes.⁸ The following peaks were used for quantitative analysis: O -503 eV, Ni -848 eV, and Au -85 eV. Unless otherwise noted, the measurements were performed on the die pads of the package samples. The estimated range (max.- min.) of measurements on or near the same measurement area was 5 atomic percent with a detection limit of 2 atomic percent.

Microstructural characteristics of the deposits were obtained using SEM analysis to determine surface morphology and X-ray diffraction analysis to obtain crystallographic orientation, crystallite size estimates, and estimates of the microstrain. Phase identification was performed using a twoaxis (Ω , 2 θ) goniometer^f operating in the θ - 2 θ mode. Copper K α_1 , radiation (λ = 1.54060 Å) originated from a sealed tube operating at 45 kV and 40 mA. The samples were stepscanned from 38 to 138 °2 θ in steps of 0.03 °2 θ with a count time of 1 sec/step. A variable slit was employed to illuminate a 1-mm width of the sample. The peak positions, intensities, and line-widths were analyzed.

Results

Auger Analysis

In Fig. 3, the Ni concentrations on the Au surfaces after the heat-test and 30 sec of sputtering are shown. The as-plated Au thicknesses on these samples are also represented. Although no upper limit has been determined for this test, experience has shown that deposits on finished product which, as a result of the heat-test, develop Ni (as NiO) concentrations in the range of 30 to 40 at pct are of marginal quality with respect to final assembly requirements. Successful assembly depends, however, on the assembly methods and conditions used. Packages assembled using glass die attachment and Au-Sn lid sealing have even more stringent requirements.⁸

On unannealed samples finished using Sequence 1, 20 to 40 at pct Ni was determined to be present. When Sequence 2 was used, the pct Ni was significantly decreased. In this case, the amount of Ni found was near or below the limits of detection for the Auger analysis method used. Depositing the Au layer directly on a sintered Ni surface (Sequence 2), rather than an as-plated Ni surface (Sequence 1), resulted in a major improvement in the Au layer's ability to inhibit Ni diffusion.

The impact of post-plate annealing on the performance of the Au as a Ni diffusion barrier is seen in Fig. 3. The 335 °C anneal may have brought about a small improvement in Ni diffusion inhibition, although the strong dependence of this property on Au thickness remained. After 450 °C annealing, significant improvement was seen, especially for the thinner deposits. The amount of Ni detected was nearly independent of Au thickness over the 0.8 to 1.2 μ m range. Neither annealing process, however, resulted in an improvement as large as that which occurred when Sequence 2 was used with no anneal.

SEM Analysis of Surface Morphology

For Au layers on samples processed by both Sequence 1 and Sequence 2, the surface morphology (Fig. 4) primarily reflected the features of the W/Ni underlayer. Slight differences in the Au surface morphology were observed, however. The deposit obtained using Sequence 2 included a greater number of larger Au grains with recognizable facets. Figure 5 shows that sintering extensively reordered the Ni underlayer. When this is done just prior to Au plating, it evidently facilitates the development of larger, faceted Au grains.

When the "as-plated" Au deposit obtained using Sequence 1 was annealed at 335 $^{\circ}$ C, a slight modification of the Au surface morphology was observed (Fig. 6). The 450 $^{\circ}$ C anneal brought about a more profound alteration of the structure, resulting in significant recrystallization and a reduction of small-scale roughness.

X-RayDiffraction

Preferred Orientation

The Au surface morphology differences observed in Fig. 4 indicate that there are microstructural differences between the as-plated, or unannealed, Au deposits obtained using Sequences 1 and 2. To further characterize these differences, samples of both were subjected to room temperature x-ray diffraction analysis. The preferred orientation was determined by analysis of the diffraction intensities in the manner recently described by Lee *et al.*¹⁰ The data, referenced to NBS powder data,¹¹ were used to determine a "texture fraction," TF, for various reflections.

$$\Gamma F\{hkl\} = \frac{I\{hkl\}/I_0\{hkl\}}{\Sigma[I\{hkl\}/I_0\{hkl\}]}$$

annealed Au layers.



Fig. 4—SEM images of Au surface morphology comparing samples finished using Sequences 1 and 2.

Fig. 5—SEM images of Ni surface morphology just before Au plating.

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I{hkl} and I₀{hkl} are the integrated intensities of the sample and powder reference respectively for a particular {hkl} set of reflection planes. Preferred orientation or texture is present when TF for a particular family of reflection planes, {hkl}, exceeds the mean TF for the reflections analyzed. When this is true, more of the deposit's crystals have their <hkl> directions perpendicular to the plated surface than would be the case in a randomly oriented sample. The larger the quantity [TF{hkl} - TF(Mean)], for a particular {hkl}, the greater the degree of preferred orientation. If all crystals were oriented in the <hkl> direction, TF{hkl} would equal 1. In this study, seven sets of reflection planes were analyzed and TF(Mean) = 0.143. If the Au deposit was completely random, TF{hkl} would equal TF(Mean) or 0.143 for each {hkl}.

The results of this analysis applied to the samples obtained in this study are shown in Fig. 7 and Table 3. These samples were not subjected to the 450 °C / 5-min / air heat test prior to XRD analysis. When plated on an unsintered Ni surface (Sequence 1), the Au is deposited with a weakly preferred <100> orientation. For the Au deposit on sintered Ni (Sequence 2), the most preferred orientation is in the <111> direction. The preference is, however, not strong in either case. Results for annealed samples obtained by Sequence 1 are also listed in Table 3. The texture of the sample annealed at 335 °C is similar to that of the unannealed deposit; that is, it has a slight <100> preferred orientation. Annealing at 450 °C shifted the preferred orientation to a slightly preferred <111> orientation similar to the texture of the unannealed deposit produced by Sequence 2.

ParticleSize/MicrostrainAnalysis

X-ray diffraction lines are broadened by both sub-grain particle (crystallite) size and the presence of nonuniform lattice microstrains.¹² Peak line widths and positions for the 111 and 222 lines, representing the {111} set of planes, and for the 200 and 400 lines, representing the {100} planes, are listed in Tables 4 and 5. The K α_2 contribution to the line widths and peak positions was removed by profile fitting, but the line widths were not corrected for instrumental broadening. Although the instrumental broadening was not removed, the line width data for a particular diffraction line can be used to compare the samples qualitatively.



Fig. 7—Preferred orientation of crystals of as-plated gold deposits expressed as the texture fraction for various crystal directions.

Annealing the deposit at 335 °C brought about a reduction in line widths. This implies that the crystallite size was increased and/or the nonuniform microstrain reduced by this anneal. As a result of the 450 °C anneal, however, the lines were broadened somewhat. In addition, the diffraction peaks were significantly shifted to new 2θ positions (Table 5). Figure 8 shows the position of the {111} peak of the 450 °C annealed sample to be shifted to larger 2θ compared to this peak's position for the unannealed and 335 °C annealed samples. The peaks for the other planes are also shifted to greater 2θ by the 450 °C anneal, as Table 5 shows. This shift to larger 2θ indicates, by Bragg's Law, that the interplanar spacing, d, has been reduced for at least a portion of the material analyzed. The bulk diffusion of Ni into the Au crystal lattice from the Ni/Au interface and from grain boundaries where Ni has accumulated by grain boundary diffusion would explain the shift of the diffraction peaks to larger 2θ , because Ni has a smaller atomic radius.^{13,14} The line broadening is a result of the inhomogeneous chemical composition of the Au layer resulting from the existence of Ni concentration gradients within it. The smaller shifts of 2θ and the line width reductions brought about by the 335 °C anneal may indicate that less of this bulk Ni diffusion has occurred during this operation and that the major microstructural effect is crystallite size increase and/or microstrain reduction. Microstructural changes of this nature brought about by the 450 °C anneal could be masked in the x-ray data by the effect of bulk Ni diffusion.

If more than one order of reflection is observed for the same set of planes, the extent of peak broadening can be used to estimate the crystallite size and microstrain perpendicular to the surface for that set of planes. Weil¹³ has recently reviewed this method, which is based upon the following relationship:

$$B_{hkl}\cos\theta = \lambda/D_{hkl} + 4\varepsilon\sin\theta,$$

where B_{hkl} is the corrected peak width (radians) at half the maximum intensity, θ is the Bragg angle (radians), λ is the radiation wavelength, D_{hkl} is the crystallite size, and ε is the



Fig. 8—{111} Au diffraction peak for annealed and unannealed Au (Sequence 1).

root mean square (rms) strain or microstrain. When $B_{hkl} \cos \theta$ is plotted vs. $4 \sin \theta$, the slope is equal to ϵ and the intercept is inversely proportional to D_{hkl} . In Fig. 9, the results of the application of the above relationship to the sets of planes in the <111> and <100> directions are represented for the asplated deposits.

Values of D and ε , determined graphically from the line broadening and peak position data in Tables 4 and 5 are collected in Table 6. It can be seen that the Au crystallites produced by Sequence 2 are larger than the crystallites developed by Sequence 1. A comparison of the slopes reveals that, for the Au applied to a sintered Ni surface (Sequence 2), the slopes are lower, indicating that the rms strain is less than in the Sequence 1 deposit. In the case of both Sequences 1 and 2, D was larger and ε , somewhat less for the {111} set of planes. It should be pointed out that estimations of particle size from line-broadening data are not considered accurate for D > ~0.1 μ m.¹² In addition, the line widths were not corrected for instrumental broadening. Accordingly, the graphically determined values of D and ε in Table 6 should be regarded only as indicating trends.

Discussion

In this study, pure Au deposited from a Pb-brightened, buffered KAu(CN), solution at relatively high current density (DC) was found to have a weakly preferred <111> or <100> crystal orientation. This result is in agreement with previous studies of pure Au electrodeposits from similar electrolytes.¹⁶⁻¹⁸ When deposited on an unsintered electrodeposited Ni surface, the Au layer was found to be slightly <100> preferred, but was slightly <111> preferred when deposited on a sintered Ni surface. This shift to <111> preference is accompanied by an improvement in Ni diffusion inhibition during heating in air. The improvement in this property that comes about if the Ni is sintered at 600 °C or greater just prior to Au deposition has been noted by Wakabayashi, et al.,19 and reported in a previous study.8 It is not clear whether the improved Ni diffusion performance is the result of the shift to slightly <111> preferred orientation or is the result of other structural modifications that were



Fig. 9—Graphical estimation of Au crystallite sizes ans microstrain for sets of planes in the <111> and <100> directions (as-plated deposits).

Texture	Fraction of	Table As-plat	e 3 ed & Anne	aled Au	Deposits
Texture Fraction, TF					F
Process	Au anneal	<111>	<100>	<110>	<311>
Seq. 1	None	0.2	0.22	0.18	0.15
	335 °C	0.21	0.22	0.19	0.17
	450 °C	0.24	0.2	0.19	0.14
Seq. 2	None	0.24	0.2	0.19	0.14

Table 4	
Diffraction Line Widths of As-plate	d
& Annealed Au Deposits	

		Line Width, B(°)			
Process	Au Anneal	111	222	200	400
Seq. 1	None	0.196	0.365	0.313	0.707
	335 °C	0.126	0.223	0.207	0.516
	450 °C	0.215	0.502	0.276	0.749
Seq. 2	None	0.118	0.202	0.196	0.427

Table 5
Diffraction Peak Positions of As-plated and Annealed
Au Deposits Compared To Powder Reference

		Peak Position, $2\theta(^{\circ})$			
Process	Au Anneal	111	222	200	400
Seq. 1	None	38.33	81.83	44.55	98.31
	335 °C	38.35	81.90	44.61	98.45
	450 °C	38.45	82.13	44.71	98.68
Seq. 2	None	38.37	81.86	44.57	98.26
Powder ¹⁰		38.18	81.72	44.39	98.13

Table 6

			ε	D (µ	lm)
Process	Au Anneal	<111>	<100>	<111>	<100>
Seq. 1	None	0.069	0.11	0.092	0.074
	335 °C	0.038	0.096	0.12	0.19
Seq. 2	None	0.032	0.065	0.12	0.1

observed to accompany this change. Although it may contribute, this modest shift in preferred orientation is unlikely to be the major reason for the large improvement in diffusion performance.

The most significant microstructural change is the inclusion of more large-faceted grains in the Au structure (Fig. 4). Our previous studies demonstrated a relationship between the observable presence of large-faceted Au grains and the extent/distribution of NiO on the Au surface after heat testing in air.⁸ In addition to the inclusion of a greater number of large-faceted grains, the Au deposited on a sintered Ni surface contains larger sub-grain crystallites (Table 6). Thus it has a lower concentration of both grain and crystallite boundaries. The relationship between improved grain boundary diffusion inhibition and a reduced concentration of grain boundaries has been demonstrated in a series of studies by Bitler and coworkers.²⁰⁻²⁶

Less non-uniform microstrain was observed in the deposit on sintered Ni. This may imply that the structure is more ordered and has a lower density of dislocations. Dini²⁷ has pointed out that diffusion occurs more rapidly when a metal has been cold worked, in part because dislocation densities are increased, and that many electrodeposits appear comparable to cold-worked metals. In addition, the grain and crystallite boundaries in the deposit on sintered Ni may be of better quality with fewer defects and less misorientation. One of the variables influencing diffusion along a grain boundary is the misorientation between adjoining grains.²⁸ Indeed, the quality of the grain boundary structure may be as important as grain size in producing an Au layer that will attenuate Ni diffusion and maintain good functional performance of the package.

Annealing the Seq. 1 Au deposit at 335 °C does not alter the preferred orientation or surface morphology significantly, but brings about a reduction in the microstrain and an increase in crystallite size. When this annealed deposit was subjected to the heat test, a modest improvement in Ni diffusion performance was observed. Grain boundary diffusion of Ni stimulated by the anneal itself may offset some of the benefits gained by the changes in microstructure that annealing brings about. Katz *et al.*, have pointed out the competition between diffusion and recrystallization or grain growth that accompanies annealing of electrodeposits.²²

Annealing at 450 °C shifts the structure of the Seq. 1 deposit to slightly <111> preferred, much like that found for the as-plated Seq. 2 Au deposit. This is accompanied by changes in surface morphology indicating significant recrystallization. Although this anneal resulted in improved heat test performance, the improvement did not match that of the as-plated Au over sintered Ni. This difference is believed to result from significant amounts of both bulk and grain boundary diffusion of Ni into the Au during annealing at this temperature according to the model of Marx et al.25 Through the microstructural changes it brings about, annealing can improve the Au layer's heat test performance, as this study demonstrates. The improvement, however, is limited by the Ni diffusion that accompanies it. Achieving an improved Au microstructure in the as-plated condition by prior heat treatment of the plated Ni underlayer avoids this complication.

The microstructural properties which this study has associated with improved performance in the heat test are larger grain size, larger sub-grain particle size, and lower microstrain, an indication of lower defect density. These characteristics reduce the amount of Ni that diffuses through grain boundaries to the surface and oxidizes. The result is improved ability to preserve the Au layer's functional properties (bondability etc.) during package assembly.

Summary

In this study, the Ni diffusion barrier characteristics of a relatively high-speed (>8 mA/cm²) gold deposit electroplated upon electrodeposited Ni in an in-line, conveyorized system were characterized by Auger surface analysis. These results were correlated with the Au layer's microstructural characteristics, including surface morphology, texture (preferred orientation), crystallite size, and microstrain. When the Au was deposited on a Ni surface recrystallized by sintering, a larger-grained, more highly faceted microstructure resulted that was a much better diffusion barrier than the finer grained Au deposited upon unsintered Ni. In addition to containing larger grains, the Au deposited on sintered Ni contained larger sub-grain crystallites and a lower degree of nonuniform microstrain. The lower density of grain boundaries and dislocations inferred from these results evidently account for the reduced extent of Ni diffusion through grain boundaries and oxidation on the gold surface during heat testing in air at 450 °C for 5 min. Annealing the Au deposited on unsintered Ni at 335 °C slightly improved the diffusion barrier performance by modifying the Au microstructure. Annealing increased the Au crystallite size and probably reduced defect density. Further improvement was obtained using a 450 °C anneal. Diffusion that occurred during annealing, however, limited the extent to which annealing improved the performance in the heat test. Depositing the Au upon a sintered Ni surface achieved, in the as-plated condition, the Au microstructural features important to diffusion barrier performance (large grain/crystallite size, lower defect density).

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