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Ceramic Coatings on Package Lids for Radiation Protection

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Ceramic Coatings on Package Lids for Radiation Protection

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Abstract

A study was conducted to determine the feasibility of coating gold plated kovar lids with colloiddally bonded aluminum oxide. Radiation that is incident on a gold plated lid generates a large number of photoelectrons. These electrons can enhance the damage to microelectronic devices and circuits (ICs). The primary purpose of the coating is to stop the electrons emitted from the lid that would otherwise increase the damage

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to the IC. A coating system consisting of ~95 wt% alumina (0.5 μm particles) and ~5 wt% colloidal silica (10 nm particles) was developed. The coating was applied to the lids as an aqueous suspension which was then dried to form a porous coating. Coating processing conditions were optimized so that crack-free, uniform coatings with the required thickness (~80 μm) could be consistently produced. Preliminary data have indicated that the coated lid can be attached to the IC package using current belt furnace sealing procedures. The adhesion and mechanical integrity of the coatings were evaluated by submitting coated lids to centrifuge and shock testing. Selected coatings successfully withstood the shock test and 85% were undamaged after being subjected to an acceleration of 30,000 g's. Several types of radiation tests were performed to determine the effectiveness of the coating to stop electron penetration. Evaluation testing included gamma dose enhancement and X-ray induced photocurrent enhancement. The results for lids with coatings 80 or 150 μm thick were compared with results for uncoated kovar and ceramic lids. Based on these preliminary results, it was concluded that an aluminum oxide coating 80 μm thick can effectively block the radiation induced electrons emitted from gold coated kovar lids that would otherwise increase the damage to the IC.

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Introduction

Integrated circuits (ICs) are assembled in various package configurations prior to incorporation in a device. The package consists of conducting metal pathways embedded in an insulating ceramic or polymer. The primary functions of the package are: 1) to provide efficient electrical interconnection between various terminals on the IC, 2) to distribute power to the IC, 3) to assure environmental protection and 4) to provide connections at the next assembly. Typically, for SNL applications a silicon die is attached to the substrate of a package, wire bonded, and hermetically encapsulated within the package by soldering a package lid onto the top of the package. Figure 1 represents a typical IC and package diagram.

Figure 1. Diagram of a packaged IC with a hermetically sealed lid.

Package lids can be fabricated from either a ceramic, such as aluminum oxide, or a metal such as kovar. The Microelectronics Development Laboratory at SNL primarily uses kovar lids for strength considerations. Kovar lids do not fracture as readily as ceramic lids during normal IC package processing. The kovar lids used at SNL are plated with coatings of first nickel and then gold to improve the solderability of the lids to the package. Au-Sn solder preforms are welded to the outside perimeter of the plated kovar lids. The lids are then soldered hermetically onto the Au-Ni plated package seal rings to protect the ICs inside from moisture. The presence of any water vapor within the package cavity can be extremely detrimental to device reliability.

A potential problem associated with gold plated kovar lids is that when exposed to certain radiation environments, a significant number of low energy electrons are emitted from the kovar and especially from the gold coating. Electron emission occurs due to two different processes: the photoelectric effect (x-ray energies typically below ~500 keV) and the Compton effect (x-ray or gamma ray energies typically above ~500 keV). In an IC package, electrons emitted from the package lid can strike the IC and lead to increased radiation induced photocurrents and to total dose damage.^{1,2}

In order to design a lid coating that would stop low energy emitted electrons so that they would not strike the IC, the electron stopping power of materials must be considered. High energy electrons traveling through a material are slowed down by two processes: inelastic Coulomb collisions with

bound atomic electrons and radiative emission (bremsstrahlung) in the electric field of the atomic electrons and nuclei.³ The magnitude of these effects depends on the atomic structure of the medium. A useful value for comparison of the electron stopping ability of various materials is the CSDA (continuous-slowing-down-approximation) range, which is also a function of the initial electron energy. These values are generally expressed for a given material as the mass per unit area required to stop an electron with a given initial energy. The values can be easily converted to a penetration thickness by dividing by the density of the material. Table II gives the values for the CSDA range of electrons in dense aluminum oxide with energies in the range of interest.³

Table I: Range of electrons in dense aluminum oxide.

Electron Energy (keV)	CSDA Range (g/cm ²)	CSDA Range (μm)
50	5.5x10 ⁻³	14
75	1.1x10 ⁻²	28
100	1.8x10 ⁻²	45
150	3.5x10 ⁻²	88
200	5.6x10 ⁻²	140
250	7.9x10 ⁻²	199

Since the electrons emitted from the lids generally have energies <500 keV, they can be stopped by a relatively thin layer of material. This has been confirmed in previous experiments² using "bilaminate" layered materials in place of the IC package lids. Moreover, it is most important to stop the lower energy electrons since they are more likely to deposit their energy in the active region of an IC. The goal of this work was to develop a coating material for

the inside surface of gold coated kovar lids which would be capable of stopping the radiation induced electrons, especially the low energy ones, and which would be compatible with existing IC package processing.

In addition to being capable of stopping the electrons, the coating must meet several other critical requirements. Due to outgassing and degradation concerns, the coating must not contain any polymeric or organic material. The coating has to withstand the temperature at which the lid is soldered onto the package, 270°C. Ideally, the coating should not require processing above this temperature since the solder preform is pre-attached to the lids by their manufacturer. The absolute maximum processing temperature limitation is 450°C, since nickel from the nickel coating diffuses through gold coating above this temperature. The thermal expansion mismatch between the coating material and kovar cannot be so great that the stress produced during heating and cooling will damage the coating. The coating has to adhere well enough to the lid and have enough strength to be able to withstand standard IC package acceptance testing. Finally, the level of naturally radioactive impurities in the coating has to be extremely low (<10 ppb) since emission of alpha particles during decay can also produce IC errors. The final coating requirement was a concern because commercial aluminum oxide powders generally contain thorium and/or uranium impurities.⁴

In this evaluation the feasibility of aluminum oxide as an acceptable coating material was studied. Alumina was chosen for several reasons. Since it consists of low atomic weight elements, it would not emit a significant number of electrons when irradiated.^{1,2} Alumina is also thermally and chemically stable. Although Th and U impurities are a consideration, low α -emitting

alumina powders are now commercially available. Thermal expansion mismatch should not be a problem since the thermal expansion coefficients of kovar and alumina are relatively well matched in the temperature range of interest. Finally, a coating of aluminum oxide particles can be processed at low temperature by drying a stable aqueous suspension. However, it was not known whether coatings of this type would have adequate strength, adhesion and uniformity.

Since alumina powders do not sinter to any extent below 900°C, even if the particles are less than 0.5 μm in size, any coating made from alumina particles will tend to be porous and have a relatively low strength. Porosity is not detrimental to the ultimate function of the coating and its presence just increases the thickness of the coating required to stop the electrons. However, low strength and poor adhesion can be major problems. To try to improve these mechanical properties of the coating, colloidal bonding was used.

The technique of colloidal bonding has been used in the past to increase the green strength of ceramics.⁵ In this technique, a small volume fraction of colloidal size particles (5 to 50 nm) of a second material, usually silica, is added to an aqueous suspension of particles of the major phase. When this mixture is dried, the colloidal particles bind the larger particles together by chemical bonding (condensation of surface hydroxyl groups). Reprecipitation of material dissolved from the colloidal particles may also occur which further increases the strength. For the present application, the presence of a small fraction of high purity colloidal silica in the coating should not have any detrimental effects since it is composed of low atomic number elements, has high chemical and thermal stability and low U and Th contents.

Several techniques for application of the coating were considered. Electrophoresis was not attempted because it does not work well in aqueous systems and does not produce strong coatings in the absence of polymers. In addition, masking would be required on the portion of the lid that is not coated. Spraying the aqueous suspension onto a heated lid was considered. This technique would also require some degree of masking. Unfortunately, a possibility existed that the dry coating on the substrate could be damaged by the wet spray. It was found through experimentation that the simplest way to form the coating is to first coat the desired area of the lid with the suspension and then allow it to dry by heating. This particular technique was chosen because of its simplicity and because it did not appear to have any major drawbacks.

There are, however, several concerns with this coating technique. By adding colloidal particles to a suspension, the maximum coating thickness that can be dried without cracking is decreased. This occurs because the small colloidal particles will produce small pores which generate a high capillary stress when partially filled with a liquid. Therefore, it is possible that a coating with enough colloidal silica to provide adequate dry strength may have a maximum crack-free thickness less than the thickness required to stop the photoelectrons. Producing a coating with a uniform thickness by this technique is difficult. Since drying normally commences from the edges of the coating and proceeds inward, the thickness of the coating increases towards the center. A "dimple" which is much thinner than the surrounding area is generally formed at the very last region of the coating to dry. Drying conditions had to be

carefully controlled to assure that the coating thickness was sufficient to inhibit electron penetration.

The goal of this work was to study the feasibility of using colloidally bonded alumina coatings on Au-Ni plated kovar lids to stop the Compton and photoelectrons emitted by the lids. Coating processing techniques were studied to determine the optimum particle sizes for the alumina and silica, composition, and drying conditions. Coatings were then evaluated to determine adhesion and mechanical integrity, compatibility with current processing, and effectiveness at stopping electrons.

Experimental Procedure

Two high purity alumina powders with different average particle sizes were initially evaluated: Sumitomo AKP-HP (Sumitomo Chemical, Tokyo, Japan; average particle size $\approx 0.5 \mu\text{m}$) and Criceram A6 (Criceram, Paris, France; average particle size $\approx 10 \mu\text{m}$). Colloidal silica sols from two sources, Nyacol (Nyacol Products Inc., Ashland, MA) and Cabot (Cabot Corp., Tuscola, IL), were obtained. The sols evaluated and some of their properties are given in Table II. To determine the level of U and Th impurities, the following materials were analyzed using neutron activation analysis: Sumitomo AKP-HP alumina, Cab-o-sperse 3295 silica (dried sol) and Nyacol 9950 silica (dried sol).

Aqueous suspension of the alumina powders (~ 20 vol % alumina) were prepared using Darvan C (R. T. Vanderbilt) as a dispersant (~ 0.5 wt% Darvan C). The pH of these suspensions was about 9, which is similar to that of the colloidal silica sols. Therefore, the silica sols could be added directly to the

suspensions without causing coagulation. Initially, a number of combinations of aluminas and silicas were qualitatively evaluated for their cracking behavior during drying and their strength and adhesion after drying. Based on these results, the combination of Sumitomo alumina and Cab-o-sperse S 3295 silica was used.

Table II: Properties of some colloidal silica sols.

Manufacturer	Grade	Wt. % Solids	pH	Particle Size (nm)
Cabot	Cab-o-sperse S 3295	15	9.5 - 10	8
Cabot	Cab-o-sperse S 2095	17	9.5 - 10	14
Nyacol	215	15	11	3 - 4
Nyacol	830	30	10.5	8
Nyacol	2040	40	10	20
Nyacol	9950	50	9	100

Based on the data in Table I, two nominal coating thicknesses were chosen to initially evaluate the mechanical integrity and effectiveness in stopping the electrons: 80 and 150 μm . Coatings processed with enough silica to impart reasonable strength cracked during drying when the coating thickness was greater than ~ 100 μm . A polymeric binder, polyvinyl alcohol (PVA, MW = 15,000) was added to the suspension to prevent the cracking phenomenon. The composition of the coating suspension was 95 wt% alumina, 4.5 wt% silica, and 0.5 wt% PVA. Since polymeric material is not desired in the final coating, the pyrolysis behavior of the PVA in the coating was studied using thermogravimetric analysis.

The coatings were applied by pipetting the desired volume onto the inside surface of standard 1 cm square Au-Ni plated kovar lids and then spreading the material using the pipette tip. Since the dry coating uniformity was very sensitive to the manner in which it was dried, a variety of drying techniques

were evaluated. Techniques ranged from simply placing the uncovered coated lid on a level hot plate to controlling the drying direction by gradually removing a glass slide above the coating and/or moving the lid gradually onto the hotplate. The drying of the coatings which were evaluated further was done by gradually sliding the lid from a surface at room temperature, where the coating was covered by a glass slide ~1 mm above it, onto a hotplate at 100°C, where the coating was not covered. Drying took about 1 minute to complete. The microstructure of the coating was examined using the scanning electron microscope. The coated lids, 8 of each of the 2 chosen thicknesses, were then evaluated without further heat treatment.

The coated lids were then subjected to centrifuge testing. Lids were subjected to a constant acceleration to a 30,000 g force for 1 minute. A complete visual examination was performed on the lids prior to and immediately following the test. A mechanical shock test, involving 800 g shocks and a 20 g vibration at 120 Hz for 4 seconds, was then performed on the lids, followed by another visual inspection. Several experiments were then performed on the lids to evaluate the ability of the coatings to stop radiation-induced electrons. The relative x-ray induced photocurrent enhancement for endpoint energies of 350 and 900 keV was measured for uncoated standard kovar lids, for standard kovar lids with 80 or 150 μm thick coatings and for ceramic (alumina) lids, using the SNL pelletron source.¹ The relative gamma dose enhancement was also determined for the same sets of lids using a ^{60}Co gammacell source by comparing threshold voltage shifts as a function of dose for doses between 2 and 100 krad(Si) at a rate of 1 Mrad/hr.⁶

Based on the results of the previous experiments, a second group of lids, a total of 17, were coated with a suspension that did not contain any PVA such that the dry thickness was 100 μm . The composition of these coatings was 95 wt% Sumitomo alumina and 5 wt% silica from Cab-o-sperse S 3295. The coated lids were heated for 30 minutes on a hotplate at 250°C prior to clamping onto ceramic packages. They were then sealed in the standard belt furnace at 270°C, tested for hermeticity, given a centrifuge test at 30,000 g's, and then retested for hermeticity. A particle impact noise detection (PIND) test was performed after the hermeticity retest to determine whether the coatings remained intact.

Results and Discussion

The neutron activation analysis was not able to detect any U or Th in any of the alumina or silica samples analyzed, indicating that their concentrations were less than the detectability limit of 50 ppb by mass. These results indicate that all of the materials analyzed had acceptably low levels of Th and U.

Coatings initially made from the two alumina powders indicated that those from the Criceram powder could be made thicker than those from the Sumitomo powder without cracking during drying. However, when a given amount of silica sol was added to either alumina powder suspension, coatings containing the Criceram alumina were considerably weaker when dry. Both of these effects were most likely due to the difference in particle size between the two alumina powders: larger particles produce larger pores and thus lower capillary stress during drying so they can be made thicker without cracking, but they also

have fewer interparticle contacts per unit volume which makes them relatively weaker. The relative density of the Sumitomo alumina coatings was estimated to be about 50%.

As stated above, the maximum thickness coating that could be dried without cracking decreased considerably when any of the silica sols were added to the alumina suspension. This occurred because the small size of the silica particles created small pores and thus a larger capillary stress during drying.

The Cab-o-sperse S 3295 silica sol produced the highest strength coatings for a given vol% silica, probably because of their small size, but the other silicas also gave coatings with reasonable strengths. Crack-free coatings with 95 wt% Sumitomo alumina and 5 wt% Cab-o-sperse S 3295 silica ~100 μm thick could be produced without PVA and over 150 μm thick with 0.5 wt% PVA. The combination of the Sumitomo alumina and the Cab-o-sperse S 3295 silica was therefore chosen for the rest of this work since coatings with the desired range of thicknesses and reasonable strength could be produced.

Since the coating suspension wet the surface of the gold coated lids well, the suspension could be spread into a thin film. On some lids, the suspension accidentally came into contact with the solder rim on the edge of the lid while it was being spread out with the pipet. Some of the suspension was then drawn into the gap between the rim and the lid and left some ceramic material there after drying. The effect of this material on the sealing of the lids is discussed below.

As previously mentioned, the uniformity of the coating was very dependent on the way that it was dried. Since the last region of the coating to dry forms a "dimple" that consists of a central thin region surrounded by a thicker

region, it is necessary that drying be carried out in such a way that the dimple is not in the central part of the coating which is directly above the IC. This was accomplished by making the coating dry starting from only one edge and then allowing the drying front to move across to the opposite edge. This was achieved by a combination of two techniques.

In the first method, a glass slide was placed ~1 mm above the coating, parallel to the lid, such that all of the coating was initially covered by the slide. The slide suppresses evaporation of water from the coating by trapping water vapor near the coating surface, thus increasing the local partial pressure of water vapor. The slide was then slowly moved relative to the lid to uncover the coating from one edge, thereby allowing more rapid evaporation to take place starting from one edge. The speed at which the slide was moved was such that the drying front was just able to keep up with it.

The second technique used to establish unidirectional drying consisted of sliding a coated lid along a horizontal surface from a surface at room temperature onto a hotplate at ~100°C so that one edge of the coating was parallel to the boundary between the room temperature and hot surfaces. This produced a steep temperature gradient in the lid and caused drying to occur from the edge heated first across to the opposite edge, when the speed of the lid was such that the drying front could just keep up with it. The best results were obtained when the slide cover was removed while the lid was slid onto the hotplate. When this was done, the dimple formed within a few millimeters of the edge opposite the edge where drying started.

One problem with unidirectional drying was that it produced a gradient in coating thickness. The coating was thinnest at the first edge to dry and

thickness increased across in the direction of drying. The magnitude of the gradient was $\sim 50 \mu\text{m}/\text{cm}$. To minimize the thickness gradient, the surface on which the lid rested was tilted slightly from level in such a way that the first edge to dry was lower than the last. This decreased the gradient to $<10 \mu\text{m}/\text{cm}$.

Scanning electron micrographs of the microstructure of the Sumitomo alumina/Cab-o-sperse S 3295 silica coatings are shown in Figure 2. No pinholes, cracks or large pores were observed. However, some heterogeneity, in the form of silica-rich regions, was present. These regions probably formed during drying after the alumina particles came into contact with each other and the much smaller silica particles retained in the pore liquid were concentrated where the last water in an area evaporated.

Figure 2. Scanning electron micrographs of the top surface of an alumina/silica coating.

The results of the thermogravimetric analysis, shown in Figure 3, indicated that the PVA is completely pyrolyzed by $\sim 375^\circ\text{C}$. This means that the use of the

binder is compatible with the maximum temperature of the lid itself, but not with the solder rim which melts at 260°C. Therefore, the second set of films that were evaluated were prepared without any PVA.

a.

b.

Figure 3. Thermogravimetric analysis of the dry coating material containing 0.5 wt % PVA. a.) Heated at 5°C/min to 800°C and the cooled at 5°C/min. b.) Heated at 5°C/min to 375°C and held for 2 hr.

All of the PVA containing coatings of both thicknesses, 80 and 150 μm , passed the shock and centrifuge tests without sustaining any damage. The second group of coated lids, those without PVA, were expected to be weaker since they did not contain any organic binder and were therefore heated to 250°C after drying to try to increase their strength and adhesion. These coatings also passed the shock test but only 88% (15/17) passed the centrifuge test, slightly worse than the first group.

The measured x-ray induced photocurrent enhancement for gold coated kovar lids with and without ceramic coatings relative to that of an all ceramic lid is given in Figure 4a for an endpoint energy of 350 keV and in Figure 4b for an endpoint energy of 900 keV. These measurements were performed on standard Sandia diodes. As these figures indicate, the presence of the ceramic coatings decreases the photocurrent enhancement of the kovar lids to less than or equal

a.

b.

Figure 4. The relative x-ray induced photocurrent enhancement for ceramic, Au/kovar and ceramic coated (for two coating thicknesses: 80 and 150 μm) Au/kovar lids at endpoint densities of a) 350 keV and b) 900 keV.

to that of the all ceramic lids, with the thicker coating being only marginally better than the thinner. The presence of the coatings lowered the threshold voltage shift produced by a dose of gamma rays to nearly the same value produced by the all ceramic lids, as shown in Figure 5. From these values of the threshold voltage shift, relative gamma dose enhancements were determined. As shown in Figure 6, the coatings lowered the relative gamma dose enhancement of the kovar lids from 1.5 to 1.1, slightly higher than the value of 1.0 for the all ceramic lid. In this case, no dependence on coating thickness was observed.

These results can be explained by considering the CSDA values for alumina in Table 1. According to the table, the 80 μm thick coating should be able to

stop electrons with energies below ~100 keV, whereas the 150 μm thick coating should be able to stop electrons below ~150 keV. (Note that the coatings are

Figure 5. The measured threshold voltage shift as a function of dose for ceramic, Au/kovar and ceramic coated lids.

Figure 6. The relative gamma dose enhancement for ceramic, Au/kovar, and ceramic coated lids determined from the threshold voltage shift data.

about 50% porous.) The fact that coatings of either thickness can reduce damage almost completely is consistent with the fact that damage is caused primarily by low energy (< 100 keV) photoelectrons, which are stopped even by the thin coating.

The second group of coated lids, those not containing PVA, were sealed to packages using the standard belt furnace. These sealed packages were then tested for hermeticity. Only 47% (8/17) passed the fine leak test. After the centrifuge test, 29% (5/17) passed the fine leak test and 23% (4/17) passed the coarse.

The suspected origin of these poor results is the presence of coating material between the solder rim and the lid which was caused by manual coating the small lids. This problem could therefore be eliminated by more careful coating either by leaving a larger space between the coating and the solder or by applying the coating with a machine.

Summary

Colloidally bonded alumina coatings have been shown to be effective at stopping radiation induced electrons emitted from kovar lids. In fact, the behavior of lids with only an $80\text{ }\mu\text{m}$ thick coating is comparable to that of ceramic lids in terms of x-ray induced photocurrent enhancement and gamma dose enhancement. Optimum coatings were obtained when 95 wt. % Sumitomo AKP-HP alumina and 5 wt. % silica from Cab-o-sperse S 3295 colloidal silica sol were used. The coatings are inexpensive, simple to prepare, compatible with present package processing and meet a number of other coating requirements. However,

there were two problems with the coatings. One problem was the non-hermeticity of a large percentage of the lids after sealing. This was most likely due to careless application of the coating and can therefore be corrected. The more serious problem was the inability of all the coatings without an organic binder to pass the 30,000 g's centrifuge test. There are several possible ways to solve this problem including making the coatings slightly thinner, which will lower the force at the interface produced by the acceleration, or making them slightly stronger by, for example, using a slightly smaller size of alumina powder or by heating for a longer time at 250°C. If these techniques fail, it may be possible to apply the coating prior to attachment of the PbSn solder preform so that it can be heated to higher temperature where strength will increase.

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