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METAL-CERAMIC ENCLOSURE FOR SURFACE MOUNTING

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ABSTRACT

The paper is devoted to the problem of brittle fracture in gold being a component in soldered joints between the leads from a metal-ceramic vibrator enclosure and internal pads in a digital temperature compensated crystal oscillator enclosure when working on creation of a prototype batch of temperature compensated crystal oscillators GK-362 having an intended frequency. In the course of work, the gold concentration in the soldered joint was calculated and the interaction between the eutectic tin-lead solder with the internal pads of the digital thermally compensated crystal oscillators was described, and the data on characteristics of the material of the pads in the digital temperature compensated crystal oscillators were obtained to improve the quality of the soldered joints. Requirements for the material of the pads of the digital temperature compensated crystal oscillator enclosures were formulated to avoid brittle fracture in gold contained in soldered joints with the leads from a metal-ceramic vibrator enclosure. Recommendations for the production of higher quality products are given.

1. INTRODUCTION

Currently, the electronic industry uses the maximum allowable values of gold amount dissolved in the eutectic tin-lead solder, including when creating metal-ceramic enclosures, above which brittle fracture of solder is likely to occur (Altshuller et al, 1984; Achenbach et al, 2000). This threshold of gold content is about 3% by weight. Below it is shown that brittle fracture of solder joints develops also at substantially lower bulk concentrations of gold. Sn-based solders are the most used solders in the electronic industry for surface mount technology and similar applications (Boychuk and Mikayeva, 2018; Altshuller et al., 1984). However, industrial production often requires also joining parts made of different material combinations such as ceramics/metal. In soldering such joints, it is inevitable that the ceramic material is wetted with metallic solder. For decades, metal-porcelain restorations

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have represented the majority of tooth-colored laboratory-fabricated restorations due to their low cost when compared with metal-free restorations, easy cementation technique, and excellent clinical performance. As a result of the high cost of precious alloys and advances in porcelain technology, the use of nickel-chrome (Ni-Cr) and cobalt-chrome (Co-Cr) based alloys as framework materials for fixed dental prostheses (FDPs) has increased considerably. These alloys allow excellent quality treatments due to their satisfactory mechanical properties at less thickness and their hardness, elasticity, and tensile strength compared with precious alloys and all ceramic systems.

Research is at present oriented toward direct bonding of ceramic materials by application of active solders (Kosyhk and Lepetaev, 2003; Boguslavsky, and Litvinov, 2008). This approach reduces the time required for joint fabrication, the hygiene of the working environment is improved, and the economy of production is also enhanced. The paper is devoted to the problem of brittle fracture in gold being a component in soldered joints between the leads from a metal-ceramic vibrator enclosure and internal pads in a digital temperature compensated crystal oscillator enclosure when working on creation of a prototype batch of temperature compensated crystal oscillators GK-362 having an intended frequency.

2. METHODOLOGY

One of the problems encountered in the manufacture of an experimental batch of digital temperature-compensated crystal oscillators (DTCCOs) was brittle fracture in gold contained in the solder joints between the leads of the metal-ceramic vibrator enclosures and the internal areas of the DTCCO enclosures (Boychuk & Mikayeva, 2018; Mikayeva, 2018). With temperature influences on the experimental DTCCO assemblies, frequency signal began to disappear at the output in several assemblies.In the course of the study, it was established that solder joints of metal-ceramic enclosures did not withstand multiple temperature loads in 20% of cases within the temperature range from - 60 to + 85 °C (Patent No, 2003).

In the Russian electronics industry, the eutectic tin-lead alloy which consists of 63% tin (Sn) and 37% lead (Pb), is most often used for soldering compounds (Boguslavsky & Litvinov, 2008). Melting eutectic tin-lead alloy begins at a temperature 183°C. This temperature avoids damage to the polymer base of the printed circuit board and a molding compound of the component. Soldering is usually carried out at a temperature 235°C. Solder retains its viscosity, since the temperature is low enough and surface energy is high enough to ensure wetting of all surfaces with which the solder comes in contact. The calculation of the gold concentration in solder joints is relatively simple. It is carried out if it is necessary to solder over a gold-plated coating. The gold mass concentration percentage is calculated by the following formula:

$$wt\%Au = \frac{wtAu}{wtAu + wtSnPb} \cdot 100 \qquad (1).$$

The mass of the material can be calculated by the formula:

$$wt = V \cdot p, \qquad (2),$$

where V is the volume of the material, and p is the density of the material.

Microscopic energy dispersive microanalysis (hereinafter referred to as EDM) with the use of a scanning electron microscope (hereinafter referred to as SEM) is one of the methods for estimating the gold concentration in soldered joints. The analysis of the zones of jointing was carried out with special metallographic polished sections of the soldered joint (Fry, 2007). The X-ray peaks intensity in the EDM spectrum is proportional to the concentration of the element under study in the sample. These peak intensities can be corrected for the number of atoms, the absorption degree and the fluorescence effects. Such an approach is considered as “semi-quantitative”; nevertheless, it is very useful for estimating the concentration, which is necessary when analyzing brittle fractures of soldered joints of metal-ceramic enclosures (Kosyhk & Lepetaev, 2003). The gold concentrations in already destroyed solder joints determined according to EDM / SEM data were within the range from about 5 to 6% by mass of Au, what qualitatively confirms the threshold value of 3% by mass of Au in the compound.

3. RESULTS

Molten eutectic tin-lead solder is divided during solidification into two solid fractions: a phase with a high lead content (Pb phase) and a phase with a high tin content (Sn phase).The microstructure of tin-lead solder after solidification is shown in Figure. 1.

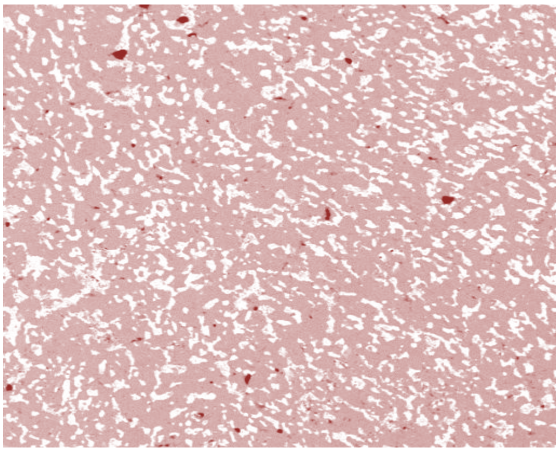

Figure 1 - Microstructure of eutectic tin-lead solder (image obtained with the use of electron backscattering method and a scanning electron microscope with a magnification of 1700X) at a moderate to high cooling rate. Bright areas correspond to Pb phase, and darker to Sn phase.

Additional phases are formed when gold is dissolved in the eutectic tin-lead solder. The most important are the phases with the formation of intermetallic compounds (IC) AuSn4 and AuSn2. In the system under consideration, several more intermetallic compounds can be formed, including AuSn, Au5Sn, Au2Pb, AuPb2, and AuPb3. Figure 2 shows the microstructure of a solder joint strongly damaged by brittle fracture. Brittle fracture in the joints of metal-ceramic enclosures arises as a result of a large volumetric fracture of rigid Au-Sn IC wafers in the viscous Sn-Pb matrix.

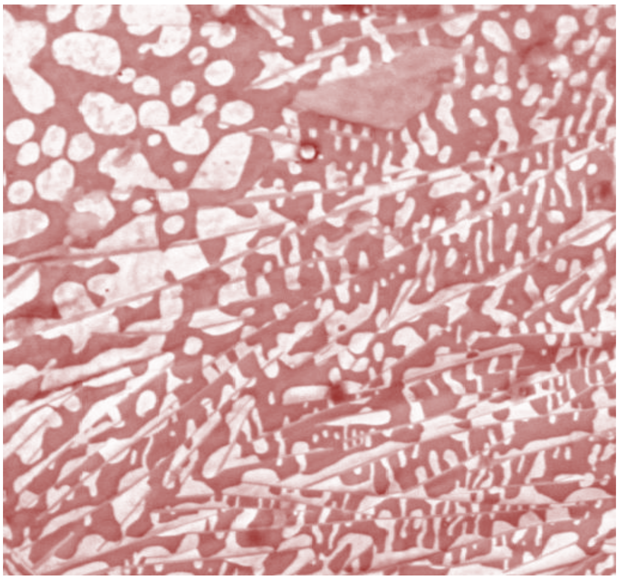

Figure 2 - Microstructure of eutectic tin-lead solder with brittle fracturein gold (image obtained with electron backscattering on a scanning electron microscope with a magnification of 3400X). The bright areas are Pb phase, the darker areas are Sn phase, the intermediate contrast areas are Au-Sn IC (first of all, such ICs like AuSn4 and AuSn2).

Figure 2 was processed to estimate the fracture area of the IC. As a result, an estimate of the fracture area of 21.1 % was obtained. This is a severe brittle damage to the solder joint. It can be assumed that the degree of brittle damage should depend on the morphology of the solid intermetallic Au-Sn phases in a softer Sn-Pb matrix. Au-Sn IC wafers shown in Fig. 2 are very thin; their thickness is about 0.25 microns. At a fixed gold concentration, thinner wafers have a larger total area of the contact surface (interface) with the surrounding matrix. Therefore, they make a more powerful contribution to the overall mechanical behavior of the solder joint (Buck & Hoff, 2002; Eltsov et al, 2011). A preliminary conclusion can be made that such general rule as the brittle fracture threshold with a gold mass of 3% should be applied with caution.

Image analysis was used to study the fracture zone in Au – Sn ICs. In this case, images of metallographic sections or open soldered surfaces obtained by the method of recording backscattered electrons using a scanning electron microscope were investigated.

The image of metallographic sections is obtained by selecting the specified level of image "saturation" represented in a monochrome scale divided into 256 gradations, corresponding to the monochrome range of values inherent to ICs. This color coding is convenient, since Sn and Pb phases in the above images fall approximately in the middle of the scale. The IC fracture zone is calculated by counting pixels of a given saturation, which is then divided by the total number of pixels in the image. For example, Figure 2 shows the image with a fracture which occupies 21.1% of the area.

In this case, in order to establish that brittle fracture in gold is available, sections of finished soldered joints of metal-ceramic enclosures were used (Murasov, 2011). The gold content in the main volume of the welded joint was measured using EDM. Based on the measurements, it was assumed that the gold content in the solder was equal to 10% of the soldered joint mass. The fracture area of the Au-Sn IC was measured by an analysis method (Fig. 3) and turned out to be ~28%. This means that the gold content in the tested solder joint was about three times the allowable limit for brittle fracture in gold. The thickness of the gold coating on unsoldered leads of metal-ceramic enclosures was also measured and was equal to ~ 288 micro inches. This is too thick for electrodeposited gold coating.

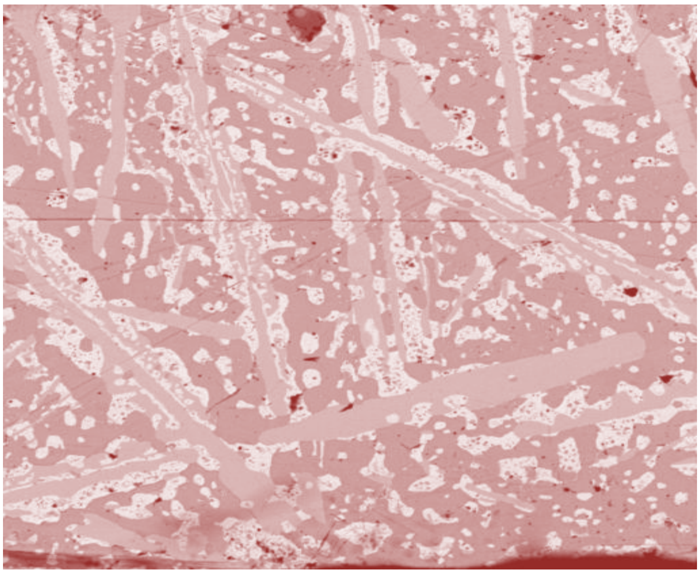

Figure 3: Microstructure of the solder joints between metal-ceramic enclosures. The distribution of Sn phase (dark scale) and Pb phase (light scale), as well as the Au-Sn IM (intermediate scale) are shown.

The authors could not find data on the molar volume of AuSn4in literature. The theoretical molar volume value can be determined by such AuSn4parameters as the unit cell and the lattice. AuSn4 has a rhombic crystal lattice with the following parameters: a = 6.51241 Å, b = 6.51621 Å, c = 11.70651 Å. Each unit cell of a crystal consists of four Au atoms and 16 Sn atoms. The molar volume will be equal to 75.0 cm^3 / (AuSn4moles), accordingly. In other words, one Au layer micron turns into a 7.4 microns ofAuSn4 layer. Using data on the AuSn2 crystal lattice, the volume expansion ratio during transition from Au toAuSn2 can be estimated as 4.2. This is a significant value. The data obtained explain why AuSn4 and AuSn2 lead to the formation of such large fractures in the microstructure when Au concentration amounts only to 3% of the solder joint mass.

The brittle fracture in gold phenomenon in Indium 6.4R solder (62Sn-36Pb-2Ag, T m = 179 °C) is very similar to the brittle fracture effect in eutectic tin-lead solder (63Sn-37Pb, Tm = 183 °C), since the alloys themselves have a number of similarities. When Ag concentration is 2%, the eutectic compound of Ag3Sn is formed in the intermetallic phase. Since three silver atoms are necessary for the formation of an intermetallic bond, the intermetallic phase concentration of Ag3Sn in the solder microstructure is very small.

A system of equations was compiled to determine the volume percentage of the intermetallic component AuSn4 relative to the mass percentage of gold in the Indium 6.4R solder. It should be noted that the intermetallic phase of Ag 3 Sn was not taken into account in this system to simplify the calculations.

Using the EDM data on the mass percentage of gold in the Indium 6.4R solder, it is necessary to calculate the percentage volume of $AuSn_4$ in IC.

pSn= 7.3 g / cm3; pPb= 11.34 g / cm3; pAuSn4= 8.985 g / cm3; AWSn= 118.71 g / mol; AWPb= 207.2 g / mol; AWAu= 196.97 g / mol;

$V\%Sn + V\%Pb + V\%AuSn4 = 1;$

$V_{sn} + V_{Pb} + V_{AuSn4} = V_{total};\ V_{pb} = M_{Pb} / p_{Pb};\ Vsn = M^*Sn / pSn$ (3),

where M^*_{Sn} is the mass of Sn in the primary phase.

$V_{AuSn4} = M_{AuSn4} / p_{AuSn4}$ (4)

$MSn + MPb + MAu = 1$ (5)

As well as for the results of energy-dispersive microanalysis.

$MSn + M_{Pb} + M_{AuSn4} = 1$ (6)

$MSn = M^*_{Sn} + M^{**}_{Sn},$ (7)

where M^{**}_{Sn} is Sn mass in the phase of $AuSn_4$formation.

$MAuSn4 = MAu + 4 \cdot M^{**}_{Sn}$ (8)

$M^{**}_{Sn} = (M_{AuSn4} - M_{Au}) / 4$ (9)

$M^{**}_{Sn} = MAuSn4 \cdot 4 \cdot AWSn / (AWAu + 4 \cdot AWSn)$ (10)

$MAu = MAuSn4 \cdot AWAu / (AWAu + 4 \cdot AWSn)$ (11)

$4 \cdot MAu / Awau = M^{**}_{Sn} / AWSn$ (12)

$M^{**}_{Sn} = 4 \cdot MAu \cdot AWSn / Awau$ (13)

For Indium 6.4R solder

$MPb = 0.58\ MSn,$ (14)

Where *MAu*, *MSn*, and *MPb* are the values obtained as a result of energy-dispersive microanalysis.

$V_{AuSn4} = M_{AuSn4} / p_{AuSn4}$ (15)

$MAuSn4 = 1 - M^*_{Sn} - MPb$ (16)

$MAuSn4 = 1 - (MSn - M^{**}_{Sn}) – MPb$ (17)

$MAuSn4 = 1 - (MSn - 4 \cdot MAu \cdot AWSn / AWAu) – MPb$ (18)

The resulting data were substituted into the expression (1).

$$V_{AuSn4} = (1 - MPb - (MSn - 4 \cdot MAu \cdot AWSn / AWAu)) / pAuSn4 \quad (19)$$

$$V\%_{AuSn4} = 100 \cdot V_{AuSn4} / (V_{AuSn4} + V_{Pb} + V_{Sn}) \quad (20)$$

4. CONCLUSION

The calculations performed suggest that the volumetric percent of the intermetallic component AuSn4 is approximately 3.33 times the percentage mass of gold in a joint.

Thus, we can conclude that brittle fracture in gold greatly affects the reliability of soldered electronic components. Careful study of the design of soldered joints is required to ensure reliable operation of the product. It is necessary to calculate the potential gold content in the solder joint between the metal-ceramic enclosures. This is an important parameter that needs to be fixed at the design stage. Analysis of EDM / SEM and processing the images obtained by electron backscattering on a raster electron microscope is a useful tool for assessing the degree of brittle fracture in gold contained in actual solder joints between metal-ceramic enclosures. Brittle fracture in gold can be prevented by carefully designing the joints and analyzing the corresponding hardware.

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