Soldered joints of Ag electrode to ultra-thin metallic Au layer on ceramic substrate

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Abstract. Among various methods of microjoining applied to electronic equipment soldering technologies are of great importance. In the construction of sensors a technique of a reflow soldering, common for microelectronic devices assembly, is rather not applicable because of the unique design of the sensor. Connecting external leads to thin metal layer of contact pads is a particular challenge. Special requirements are placed on devices and joints operating at cryogenic temperatures. The joints operating in cryo-temperatures should be characterized by good and stable electrical conductivity, tightness and the required mechanical strength. In the paper the new method of soldering of Ag leads to thin Au layer on ceramic substrate is presented. A special feature of the new soldering method is the very short time (2-3 seconds) in which the solder (In) is in the liquid state. The manufactured joints are characterized by a very low resistance as well as a linear and continuous current-voltage characteristic in the temperature range of 15 to 300 K.

1. Introduction

Microjoining is an important technology which is crucial for manufacturing of modern microelectronic devices (e.g. IC), assembly of electronic circuits but also for sensors, batteries and optoelectronics. There are many technologies involved in process of microjoining: solid state bonding, soldering and brazing, wire bonding, fusion microwelding, solid-state diffusion bonding, diffusion soldering and brazing, resistance microwelding, adhesive bonding [1]. The various methods of soldering are most widely used for electronic packaging processes such as surface mount technology (SMT), pin-through-hole (PTH), ball grid array (BGA), and flip-chip (FC) interconnects and also for joining of outer leads in sensors. Soldering technology is used to make the electrical and mechanical connections of electronic components working over a wide temperature range. Special requirements are placed on devices and joints operating at cryogenic temperatures [2-5]. The joint made by soft soldering should be characterized by good and stable electrical conductivity, tightness and the required mechanical strength, which is stable during storage and operation. The use of a standard soldering technology for connections to thin metallic layers is especially difficult. In the case of layers with a thickness of <200 nm it is practically impossible because of permanent damage to the layer. The new method we present in this paper allows to make a permanent soldered joint to a thin metallic layer on a ceramic substrate.
2. Experiments

2.1. Materials and methods
We proposed a method for producing the solder joints between metallic layer on a ceramic substrate and silver outer lead. The considered problem concerns sensors on ceramic substrates used in the temperature range from 300 to 10 K. An ultra-thin Au layer of a thickness of 400 nm was sputter on Al₂O₃ ceramic substrate. Ag foil strip 35 µm thick and 2 mm wide was used as an outer lead of a sensor. Indium was used like the solder material in presented method. The indium played a key role, because ensures flexibility and strength of the solder joint. Properties of joints soldered using a new method were characterized by microscopic investigations. Electrical properties of joints were determined in wide range of temperature from 10 to 300 K.

2.2. New soldering method
The most important feature of the presented method of joining is ability of operation at cryogenic temperature. The use of indium allows for joining metals with different coefficients of thermal expansion. The method consists in indirect heating and melting of the indium on a ceramic substrate coated with Au, and then introducing the Ag strip together with the flux to facilitated the joint of both surfaces. Basically the whole process has three main steps (Figure 1).

![Figure 1. Stages of the new soldering process of an Ag foil to a thin Au layer on a ceramic substrate.](image)

The first step in the new soldering process (Figure 1a) is to place indium on a ceramic substrate coated with an Au layer. Indium is used in solid form, preferably as a small ball. Heating and melting of the indium takes place indirectly, without the use of fluxes, by heating the surface of the ceramic substrate with a concentrated stream of hot air. The absence of flux at this stage of the process is crucial because prevents indium from spreading over the gold surface. The Au layer remains continuous and undamaged. The heating process is volumetric, which facilitates the subsequent spread and penetration
of the solder. The second step (Figure 1b) in the soldering process involves placement the Ag foil with the flux. The Ag foil with the flux deposited on it is introduced after the indium has melted. The flux in the form of a gel was applied to the joined fragment of the foil. This is the key stage of the new soldering. The introduced flux changes the surface tension of the solder and it lead to a instant wetting of the surfaces to be joined. As a result of the strong adhesion, indium is trapped between the joined surfaces. The solder spreads very quickly and evenly, which does not damage the Au layer and causes the formation of inter-metallic compounds. After joining the soldered surfaces, the cooling process begins. This is the third and final step in the soldering process (Figure 1c). The permanent and durable joint between the Ag foil and the Au layer is the result of soldering process.

3. Results and discussion
The produced joints were subjected to microscopic observations at low magnifications of 5-10 x. The image of a typical joint of Ag strip soldered with indium to thin Au layer on the Al₂O₃ substrate is shown in Figure 2a. In all tested joints, almost no flow of indium over the edge of the attached Ag strip was observed. The joint area of joint was limited to the size (width) of the Ag strip. This advantageous feature results from the mechanism of creating a soldered joint according to the proposed method.

![Microscopic pictures of soldered joints](image)

Figure 2. Microscopic pictures of soldered joints: (a) overall view of typical joint; (b) side view of the connection (visible thin layer of indium solder between the Ag lead and the ceramic substrate); (c) metallographic section of a joint (optical microscope)
The metallographic cross-section of the joint is shown in Figure 2b. The composition of the two-metal alloy depends on the temperature and the kinetics of the solidification process. Ag-In and Au-In systems are involved in diffusion soldering, especially to its low temperature variant - Transient Liquid Phase soldering (TLPS). The TLPS method is used for die attach (flip chip bonding), also in the power module packaging, thermoelectric power module and [6-8]. The duration of these processes depends on the metals used and for Ag-In is 10 min in 210°C [6,7] and 1-10 min in 160-240°C for Au-In [9,10]. It was theoretically and experimentally established that kinetics of intermetallic compounds formation depends on diffusion on interface of Au/In and Ag/In in these processes [10,11].

In the soldering process proposed by the authors, the temperature in the area of the joint did not exceed 200°C. It should however be noted that the soldered joint is dynamically formed within approx. 10 s, during which the indium ball is in a liquid state for 3 s (stage a, Figure 1) and another 2 s after inserting the Ag strip with flux (stage b, Figure 1). Different metallurgical processes take place simultaneously on two interfaces: Ag/In and Au/In. There are two additional factors that contribute to the formation of the joint. The first - the surface tension of the melted indium ball with a diameter of approx. 0.5 mm, much smaller than the joint area (2x2 mm). Surface tension is one of the most important factors in solder alloy formation and has a great value of around 200°C [11]. The second factor is the wetting of Ag and Au surfaces by the melted indium, depending on the surface energy of both layers. As a result, the molten indium flows immediately between Ag strip and Au metallization.

It is clear visible on Figure 2b that a whole Au metallization was converted to intermetallic compounds. Formation of AuIn$_2$ single intermetallic phase can be observed. The same intermetallic was reported at temperature range of 180~300°C when In thickness was larger than that of Au [12]. The intermetallic compound Ag$_2$In grows between Ag and the liquid In. Research [13] have shown that the reaction Ag-In is driven by the diffusion of silver in molten indium and parabolic growth constant is only $6.07 \times 10^{-5}$ cm$^2$/s in 473 K. This indicates that for our soldering process the Ag$_2$In layer has depth of a few micrometers.

The resistance of the joint and Au layer were measured simultaneously in the cooling-heating cycle. The resistance of two sample sections of the same length $L$ (Figure 3) was measured: Au layer ($R_{Au}$) and Au layer together with the joint ($R_J + R_{Au}$). In the second section, the length of the joint is half the length of this section. The presented arrangement of the electrodes allows to the determination of the joint resistance $R_J$ based on the measurement of the resistance of individual sample sections. The measurement is correct assuming that the Au layer is continuous. The 4-probe method was used to measure the resistance of the samples. Resistance measurements were conducted using a Keysight 34420A Micro-Ohm Meter.

![Figure 3. Method of measuring the resistance of samples.](image-url)
Figure 4. Temperature dependence of resistance a) and relative changes of resistance b) of Au layer on ceramic substrate and Ag/In/Au joint.

The results of studies on the effects of temperature on the resistance of Au layer on ceramic substrate and Au/In/Ag joint showed linear dependence of resistance on temperature in wide range (35-300)K (Figure 4). This is a typical ohmic relationship for metals and alloys. The temperature coefficient of resistance of the Au layer sputtered on the ceramic substrate ($\alpha_{Au} = 2.3 \cdot 10^{-3} 1/K$) is lower than the coefficient of bulk Au samples ($\alpha_{Au\_Bulk} = 3.7 \cdot 10^{-3} 1/K$). The vacuum sputtered Au layer is highly defective and inhomogeneous, which is the direct cause of the observed difference. The electrical properties of the joint are determined by the dominant material, silver. The temperature coefficient of resistance of the joint Ag/In/Au ($\alpha_{J} = 3.3 \cdot 10^{-3} 1/K$) is similar to the coefficient of bulk Ag samples ($\alpha_{Ag\_Bulk} = 3.8 \cdot 10^{-3} 1/K$). The observed difference is the result of the formation of inter-metallic compounds at the Ag/In and Au/In boundary in the soldering process.

4. Conclusions
The main feature of the presented soldering method is the possibility of using this type of joint at cryogenic temperature. The usage of indium enables joining metals with different thermal expansion coefficients without concern of damage of the joint due to temperature changes. The temperature characteristics of the joint resistance showed the typical ohmic relationship over the entire temperature range tested. The formation of intermetallic compounds at the Ag/In and Au/In boundaries in the soldering process was observed. The transition layers slightly affect the electrical properties in the presented joint configuration. The results showed that the electrical properties of the joint are determined by the electrode material, silver. Observation of the effect of temperature on the resistance of the joint indicates the potential application of the presented method in the construction of temperature sensors and other cryogenic devices.

5. References
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