Rohs requirements for electronic applications: measurements of characteristics of silver epoxy adhesives solder joints

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Abstract – It is emerged the necessity to modify the soldering process of all the devices under the RoHS European Directive [1], in order to eliminate the presence of lead. So our research is oriented to study the use of new materials for connections in electronic devices and to measure their characteristics and reliability performances. In this paper it is shown the preliminary characterization of a new study of the use of conductive materials without lead for the adhesion of the plate, which has piezoelectric properties, with the fingers' comb. Found a possible variability of the samples production process, it appeared opportune to apply a Design of Experiments method (D.o.E.) [2], to understand the degrees of correlation between the interested variables. After having chosen the output control characteristics of the process, (measures of electrical resistance of every single element of transducer's array) the variables, which are potentially responsible of the uncertainty on the aforesaid measures, have been characterized. The samples and the measurement system are implemented and the results are discussed in this paper.

I. Introduction

With the assumption of Directive 2002/95/CE concerning the Restriction of certain Hazardous Substances (RoHS) [1], it is needed the experimentation of various lead-free substances for the realization of solder joints in RoHS compliance electric and electronic equipments.

The application of the RoHS Directive is critical where the use of new substances will imply the variation of the production processes, like in the event of the Sn-Pb solder joints dismissal.

However today, the wettability of the majority of lead free solder does not appear as good as the Sn-Pb one and the quality control will be a critical factor. The reliability of the lead free solders will be carefully study and up to the present the thermomechanical performances of the conductive adhesives or alloys, that will have to replace the Sn-Pb joints, are not known [3].

The aim of this research is to carry out experimental and comparative studies on soldering made up new epoxy adhesives, epoxy glues characterized from the presence of a percentage of metal "drowned" in the matrix, in the event under investigation silver. The epoxide glue that for its nature it is insulator must be rendered conductive adding to a considerable amount of conductive material, in the event under investigation silver, that must create a continuous bridge from part to part for being able to lead current. This procedure induce to considerable values of viscosity of 2.000 ÷ 25,000 cPs, that it is one of the main characteristics of the conductive adhesive. A first phase regarded the design and, then, the implementation of a soldering process with conductive glue of a piezoelectric transducer (ceramic) with a comb of fingers, micro-connections between the substrate of piezoelectric material and the printed circuit. Considering that the break of a single electrical connection could compromise the functionality of the whole apparatus, it is necessary to guarantee extremely resistant and reliable joints, so as to be able to tolerate the electrical and mechanical stresses that they take place during all the operating life.

To compute the effect of the factors, which influence the solder joint's process, an interesting statistical method is D.o.E. (Design of Experiments). The implementation of D.o.E. allows to comprise the influence of not usually in ranges collected factors, that they are never used; to this aim it is necessary, therefore, to estimate the effects of every single factor on the process and quantify the possible interaction of them, that is the difference of effect that a variable could have in reliance on another.

It will be necessary to compare the obsolete Sn-Pb solder joints with conductive adhesives ones, specifying the different behaviours in order, for example, to the temperature and the realization times of the joint, the wettability, the residue of the flux, the thermal expansion, the voids generation and the electrical conductivity.

II. Proposed Approach

In order to reduction of development's time of the processes, to become more efficient the use of the resources, to optimize product and to reduce costs for non conformity, we implement a D.o.E. methodology [2] to produce the sample under test. Through this methodology it was possible to identify the meaningful variables and their correlation (phase of screening) and to identify the optimal answer (phase of optimization).

Initially the research is concentrated on the problematic of connection between comb's fingers and piezoelectric; we studied different conductive glues with a content of silver and lacking in lead in observance to the recent directive [1], that it limits the use of dangerous substances.

To compare the reliability and functional performances among the produced samples, we applied D.o.E. with the purpose to know the variables effect of process, therefore to realize a punctual quality control. The factors, taken into account in the planning of D.o.E. for the samples realization (piezoelectric layer - silver conductive adhesive-fingers), were the characteristics of conductive glues (in terms of chemical elements quantity), the thickness of conductive glues and the polymerization temperature of the glues [4].

In a two levels factorial plan the factors come regulated on two different values. Many experimenters support that the limits must be as far away as possible from the normal operative values [5], so we choose:

FACTOR	LOW LEVEL	HIGH LEVEL
Temperature of Polymerization	80°C	120°C
Thickness Conductive Glue	7 µm	94 µm
Type of Glue	A	B

Tab. 1: Planning of levels of the 3 factors without interactions

A plan of two levels with three variables has 2^3 possible combinations of factors: the eight tests to carry out are indicated for the measure of the electrical resistance, the output of experiment [3].

Every single sample has been realized cutting piezoelectric with a step of 0,34 mm longitudinally, in order to obtain 64 elements, which contain one finger on the left side and one on the right side.

Electrical resistance measure is acquired with an automatic measurement system (figure 1) constituted by a LCR bridge "AGILENT 4284A", a two micro-needles tracking system (figure 2), a webcam "Q-tec" (300K), by an optical microscope 10x and a computer running a LabView® program, which was dedicated to the automatic store of the resistance values in a frequency range *1kHz ÷ 100 kHz;* as an example in figure 3 we can see the front panel of automatic measurement during a reference measure of the resistance of gold layer deposited on the ceramic sample. This range is sufficient to find a possible damage or breakdown of the electrical connections: inductive effects would have been added beyond *100 kHz*. The eight obtained outputs at *1kHz*, with ±3% of uncertainty, for every realized sample are brought back in table 2.

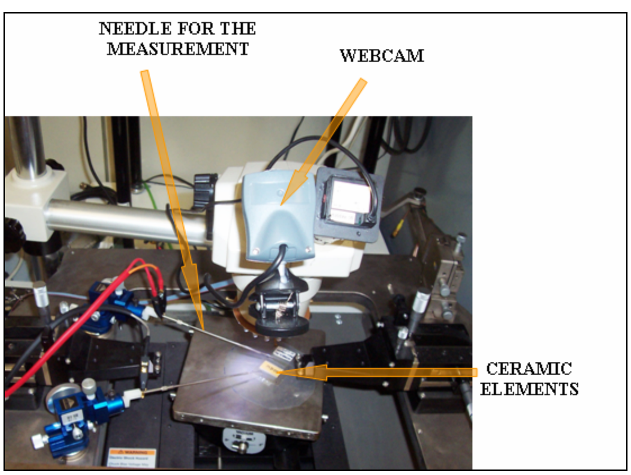

Figure 1: The implemented measurement system for the electric resistance measure

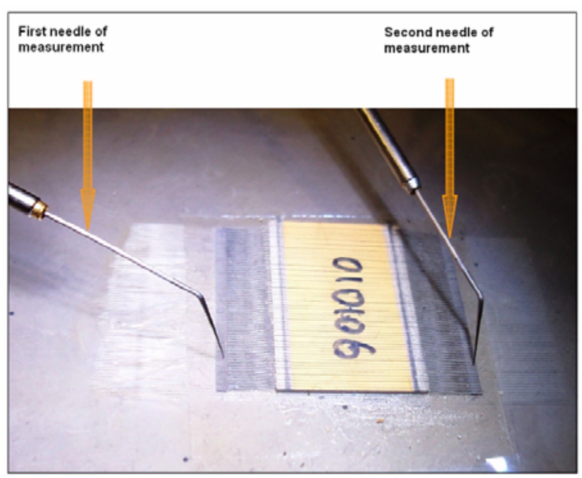

Figure 2: Detail of the system for the electric resistance measure

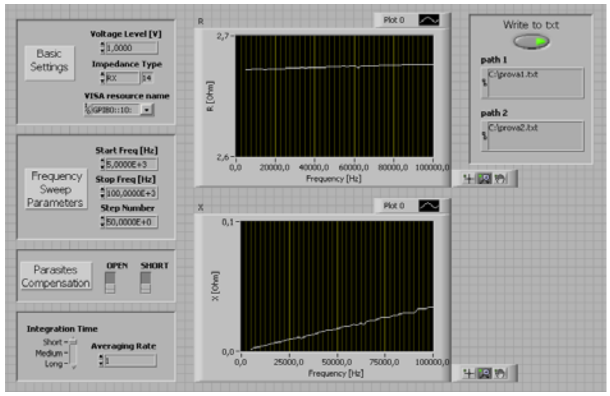

Figure 3: Front panel of automated measures

Thickness of conductive glue (F_2)	Solder material (F_3)			
	A		B	
	- Polymerization Temperature + (F_1)		- Temperature of polymerization + (F_1)	
-	5,07±0,11	5,15±0,41	5,40±0,04	5,43±0,07
+	4,74±0,25	4,82±0,34	5,03±0,14	5,14±0,24

Table 2: electrical resistance measures for the 8 samples of Ag conductive glue

Parallel we realized two transducers with Sn-Pb welding with the scope to do comparison between the two type of soldering. In table 3 we show the values of electrical resistance found at f =1kHz for Sn-Pb joint. It can be observed that at the moment of the realization the values of contact resistance for solder joints realized with silver conductive adhesive and metallic alloys Sn-Pb appear similar.

Solder joint	**Electrical Resistance**	**Thickness**
63Sn-37Pb	5,34± 0,25Ω	37±7μm
63Sn-37Pb	5,29± 0,28 Ω	36±8μm

Table 3: electrical resistance measures for the 2 samples of Sn-Pb solder joint

The electrical resistance measures is used to realize a model in function of the results and to generate some plots to estimate the effects of the involved variables. The purpose is to chose factors that have more important effects for the soldering process: the effects diagrams and the interactions diagram can be plotted to this aim [4].
As results we obtained that the "thickness of conductive glue" and "curing temperature" have a rather elevated effect on the final value of the resistance. Although the two factors seem to have important effects, it's necessary to observe their interaction because this can exalt or cancel a main effect [4].

III. Analysis of samples realised with conductive adhesive solder joints

For the experimental study on new conductive adhesive soldering was initially demanded a deepened analysis of these new materials: the environmental scanning electron microscope (E.S.E.M) was a valid support in order to obtain or morphologic or chemistries information, so as to identify and to estimate the advantages and disadvantages of the realization of the new soldering, according to the form and dimension of metallic fragments drowned in the conductive adhesive [5].
To understand the macroscopic characteristics of the glue, we took a vetronite board of the same dimensions of the ceramics, that it is used for the realization of the transducer, and we drew up of the conductive glue.
We used a vetronite board of 14 x 25 mm on which it has been carried out the drawing up of the conductive glue and the relative phase of spin-coating, then we glued the vetronite on a aluminium layer (for a greater ductility); we realised the sample for the E.S.E.M. analysis dipping it in a bath of transparent resin, to which is followed a phase of milling and polishing of the cross-sectional surface to analyze.

With the phase of centrifugation of the glue A, we can obtain a layer much homogenous and uniform or in the gel phase or after the phase of polymerization (figures 4).
This allows to obtain a soldering much efficient from the adhesive point of view, since the finger “will be wetted” on all the surface from the conductive glue. At the end of soldering process between a finger and the other one the glue layer appears uniform and covers perfectly the “naked” part of the ceramics.
Instead the B type analyzed glue does not introduce the same uniformity, in fact after the spin-coating we can see some poor zones of conductive glue, their maximum width is 250 µm (figures 5); all this induces us to think to a soldering with minor mechanical resistance regarding to the A type glue.

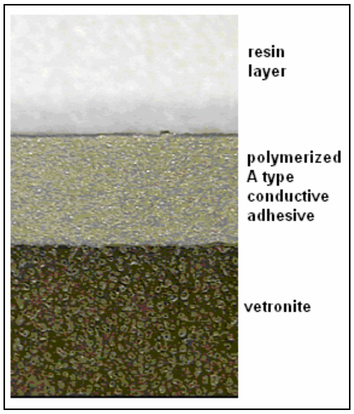

Figure 4: layer of polymerized (@80°C) A type conductive adhesive of 50 µm

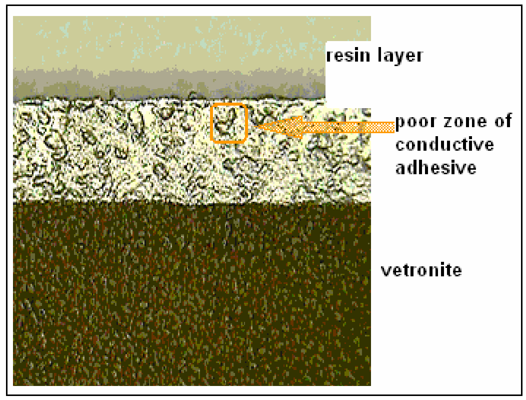

Figure 5: layer of polymerized (@80°C) B type conductive adhesive of 50 µm, localization of poor zone.

Carrying out the joining at more than 80°C, for example at 120°C, the macroscopic characteristics didn’t vary.
In the A type glue areas with much elevating grain density are alternated to create agglomerates of 5 µm (width) and others in which the concentration is more reduced, but however uniform. Making a blow up of the zone with much elevating grain density of soldering material, it can be noticed like this is formed by metallic grain micro-chains, their form is rather round and the dimension is inferior to 1 µm of diameter.
The silver fragments of the B glue (figure 6) have an irregular form: they are more lengthened regarding grains of the A glue and the dimensions are 1÷2 µm of diameter. The greater dimension of the metallic parts of the B glue demonstrates as it wasn’t possible to obtain an uniform and compact drawing up; however we don’t found inner regions in which there is a complete absence of conductive material.
The successive step was the chemical analysis, from which we can deduce that the relative percentage of the base element (Ag), named filler, for the two glue under test is remarkably greater regarding other atoms. In particular, the A glue introduces a quantity of silver approximately of 53%, while the B glue introduces a percentage of metal more than 60%.

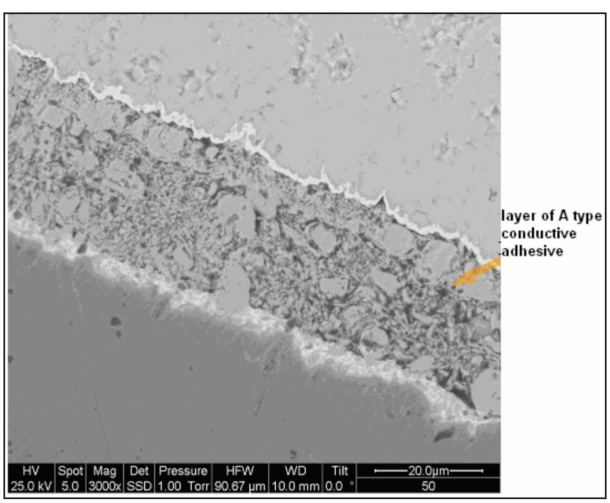

Figure 6: polymerized (@80°C) A type conductive

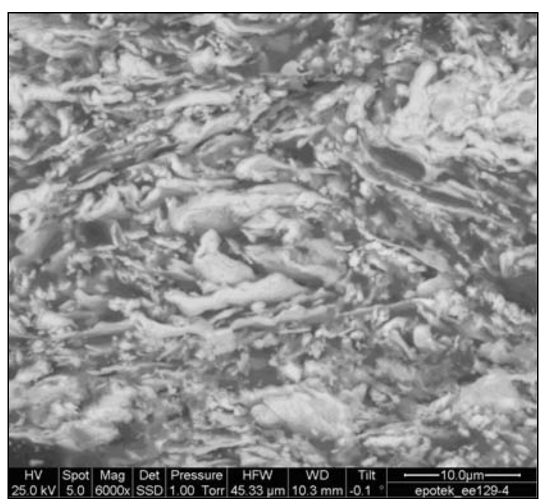

Figure 7: detail of polymerized (@80°C) B type

adhesive (zoom 3.000x) conductive layer adhesive (zoom 6.000x)

IV. Conclusions

The available documentation appeared inadequate in the treatment of silver conductive adhesive in electronic soldering, instead the preliminary study shows the real possibility in the use of these new glues for a reliable and reproduced process of solder joint, in sight of substitution of Sn-Pb solder. We shown a comparison between two different type of silver conductive adhesives, compliance to the new European directive RoHS, with different thickness of solder joint and polymerization temperature lower than the Sn-Pb soldering temperature, so we propose to carry ahead the comparison between conductive adhesive soldering with different characteristics but also with the joints realized with consolidated technology as the Sn-Pb, so as to be able to estimate the reliability of the new technology in absolute, quantifying advantages and disadvantages of the new soldering materials.

Therefore it will be born the requirement to renew the product process of soldering and to research support's materials, to realise an innovative procedure for a correct and reproduced soldering with conductive glues.

In this work the first steps of experimentation for the systematic realization of the samples of transducers, soldered with silver glue has been shown. This experimental approach is fundamental, in future, to subject set of samples to environmental comparative tests (thermal cycles and vibrations [6]), that they will evidence the fundamental factors to control during the phase of adhesion of piezoelectric and, in particular, to be able to compare the reliability of the different solder joints, realised with various conductive glues without lead; the electron microscope analysis will be carry out on realised samples, it will allow to found the location of possible difference in the structure, which will be born after thermal or vibration solicitations.

References

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