

THE STUDY INTO THE TRANSITION AREAS OF A SOLDERED JOINT OF ITO CERAMICS

Martin PROVAZNÍK, Roman KOLEŇÁK, Monika KOLEŇÁKOVÁ

Abstract

The paper deals with solderability analysis of ITO ceramics ($\text{In}_2\text{O}_3 / \text{SnO}_2$) by metals. SnInAgTi soft active solder was used for experiments. Solder was activated by power ultrasound in air without flux. The analysis of the interface of phases between solder and ceramics was carried out to determine ultrasonic impact on active metal and identify creation mechanism of joint on the ceramic side.

Key words

ultrasonic activation, active solder, ITO (indium-tin oxide) ceramics

Introduction

The current technical practice is characterized by the increasing use of ceramic materials. This especially concerns electrotechnical area. There is enormous demand for the conductive joining of ceramics with metals. Soldering by active solders is a trend in this area at present. Such solders contain an active element which reacts with the surface of the ceramic material. This enables its wetting and formation of the reaction layer. The solders have a very low wetting angle, they enable soldering at low temperatures, without flux and additional protection.

The most frequently used active metal is titanium. Reactive product transforms the surface energy of ceramics and enables the wetting of the solder. The active element moves from the whole solder volume to both soldered materials. In interface of the soldered joint, a reaction layer with thickness of several μm that containing reaction products of active elements and substrate is formed [1].

Activation of the solder is done mechanically or by using a very high temperature effecting the active element. Mechanical application is done by scraping, spreading by metal

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brush (soldering of: Cu, Al, CrNi steel), vibrations and ultrasound over 20 kHz (soldering of: ceramics, non-metallic materials). Working cycle of mechanical application is approximately 10 times shorter than high temperature activation and does not need application of vacuum or protective atmosphere.

Experimental

A sample was prepared by the machinery displayed in Fig. 1. The soldering process comprised the heating of soldered materials by a hot plate to the soldering temperature in the range of 150-160 °C. The maximum temperature of soldering was limited by the temperature of 160 °C, when surface oxidation of ITO ceramics increases. Heating by steps was chosen to reach steady heating of both materials. A strap shaped solder made by fast cooling technology was placed on a heated substratum as shown in Fig. 2. The solder was activated by a titanium peak of the ultrasonic equipment after melting-down. The activation time was chosen in interval 1 to 5 seconds in one contact point. Tab.1 shows the parameters of the US equipment. The solder was applied on the second substratum in the same way. Both prepared parts are joined and pushed softly. The presence of active solder provides high plasticity of the phase interface.

Soldering samples were fixed after splitting and prepared by a standard metallographic methods. Consequently, they were analyzed by light microscope and scanning electron microscope. For further documentation of the mechanism of joint formation, we made X-ray diffraction analysis of concentration profiles of separate elements.

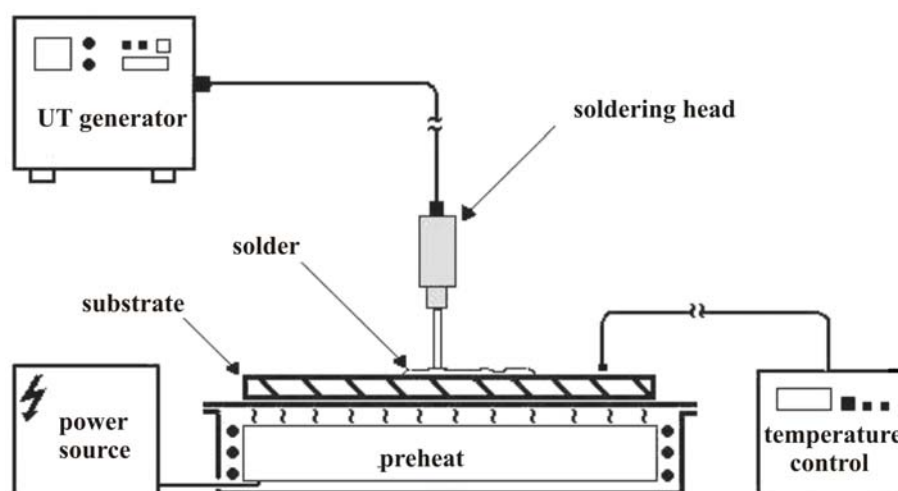


Fig. 1 Diagram of equipment for soldering by ultrasound

PARAMETRES US EQUIPMENT

Table 1

Output power - intermittent service	[W]	max. 400
Operative frequency	[kHz]	40
Input power	[W]	max. 600
Time adjustment range	[s]	0.1 - 9.9 for 0.01

Production principle of foil active solder

1. Molten alloy is extruded by fine pressure of inert gas (argon, helium) through the rectangular slot of the jet placed near the cooling of copper disk.
2. The rotating cooling disk gradually touches the molten solder. The disk creates a thin layer of solid alloy which the roller takes away.

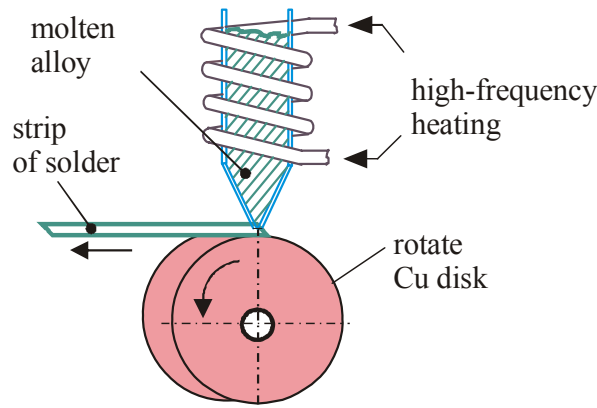


Fig. 2 Solder production by fast cooling technology [2]

The solder in foil shape with parameters 0.15 mm x 7 mm, was made for experimental purposes by the Physical Institute of the Slovak Academy of Science in Bratislava. The solder was made of foundry alloy ready casting into ingot-mould.

Experiment results

On the sample of active solder, a differential thermal analysis was made. Process of the analysis is recorded in Fig. 3. From DTA graphs, we can specify thermal areas with phase metamorphoses. Temperature 116 °C characterised the start of Sn-In molten eutectic. Temperature 156 °C was equivalent to molten temperature of pure indium.

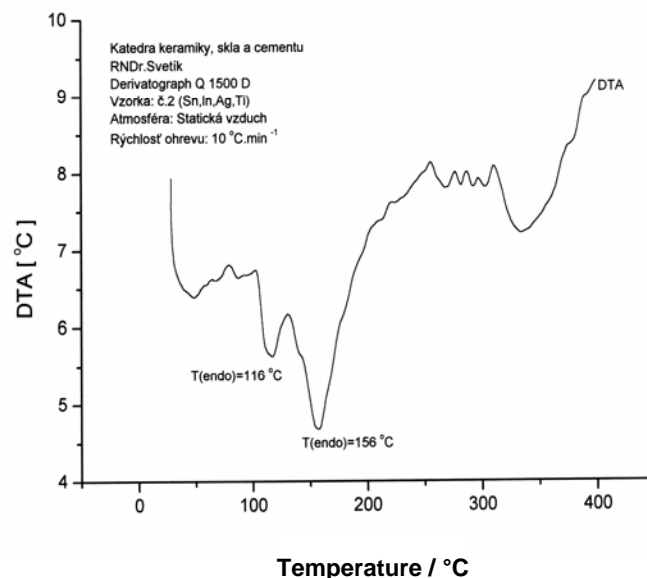
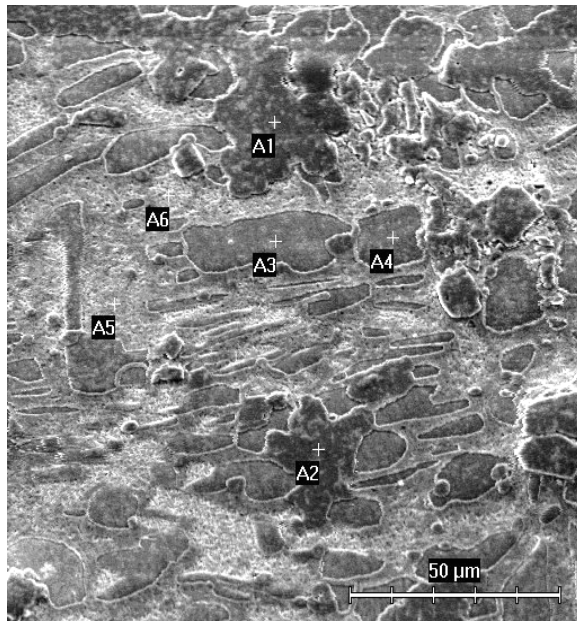


Fig. 3 DTA of SnInAgTi solder

Fig. 4 describes heterogeneous microstructure of SnInAgTi soft active solder in the molten state.



	Ti [at.%]	Ag [at.%]	In [at.%]	Sn [at.%]
A1	3.1	27.6	55.1	14.3
A2	1.9	29.3	53	15.8
A3	0	0.3	11.3	88.5
A4	0	0	12.1	87.9
A5	2.7	0	64.7	32.7
A6	0	0	71.4	28.7

Fig. 4 Microstructure of SnInAgTi active solder [3]

Quantitative chemical analysis of solder is shown in Fig. 4. Dark grains, denoted as 1 and A2 are expressively enriched by Ti and Ag. Other pallid grains (areas A3, A4) and matrix (position A5, A6) are explicitly composed by the elements of In and Sn.

To identify the phase composition of solder after moulding, a radiographic diffraction analysis was carried out by the Physical Institute of the Slovak Academy of Science in Bratislava, which identified the following phases: In_3Sn , InSn_4 , Ti_6Sn_5 , Ag_3Sn , AgIn_2 and Ti_3Ag [3]. We can therefore identify dark areas of a solder as a Ti_3Ag phase, which demonstrates the high affinity of titanium to silver.

Fig. 5 shows the phase interface of solder and ITO ceramics. The concentration profiles of individual elements across the interface suggest that Ti is responsible for the joint formation, though the big effect of Indium is also supposed.

Provable effect of power ultrasound is, that the solder is able to fill the narrow spaces among the ceramics grains, which confirms a high degree of wetting the ceramics by solder.

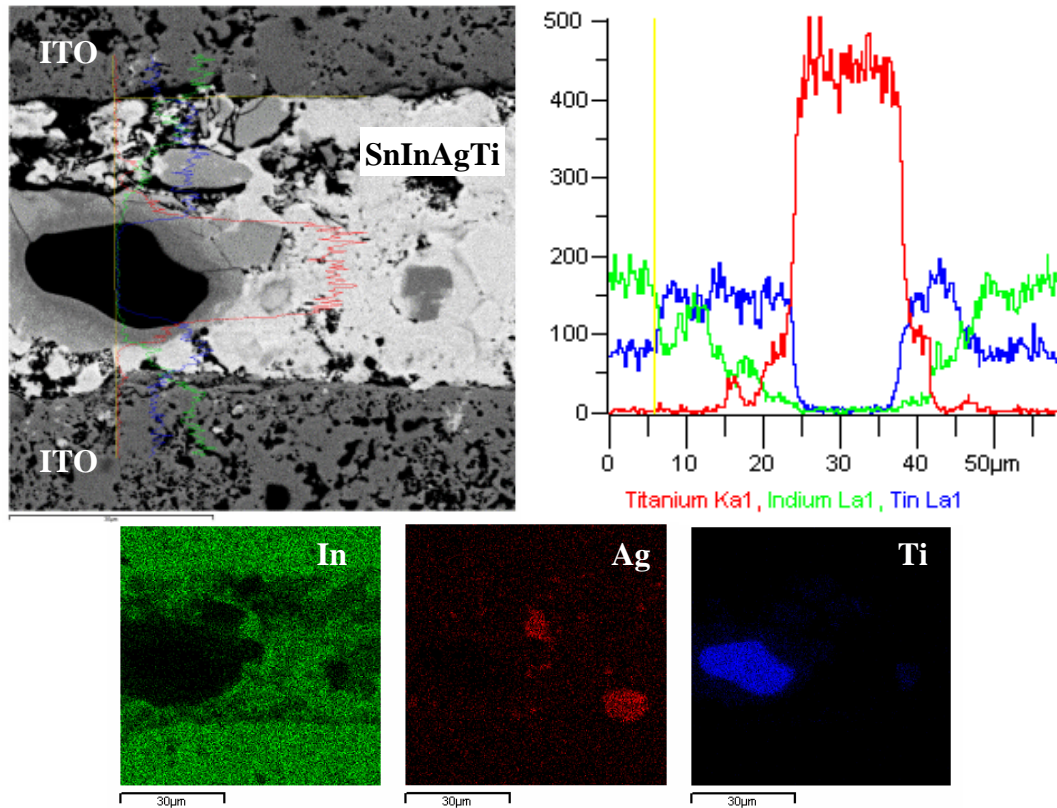


Fig. 5 *SnInAgTi - ITO interface and concentration profiles of individual elements over the interface*

Indium markedly supports joint formation in the monitored interface. The experiments show clearly that the mission of an active element in solder does not always have to appear. In this case, Ti is indifferent (does not form any provable phases), it remains fixed in the solder (particularly in Ti_3Ag phase) and it does not markedly participate in the joint formation.

Since indium has a high affinity to oxygen, we can assume that it combines in the soldering process with atmospheric oxygen with complex oxide genesis of indium, which provides an input into the reactions with the surface of the ITO ceramics by the simplified model shown in Fig. 6.

A contribution to the subject

Our contribution to the subject could be summarised as follows:

a soft active SnInAgTi solder has been designed and manufactured by the methods of fast cooling,

- our analyses of the phase structure of the solder identified the following phases: In_3Sn , InSn_4 , Ti_6Sn_5 , Ag_3Sn , AgIn_2 , Ti_3Ag ,
- the SnInAgTi solder drenched ITO ceramics with the use of power ultrasound,
- studies into the interfaces showed that when a joint with ITO ceramics is formed, indium from the solder participates preferentially.

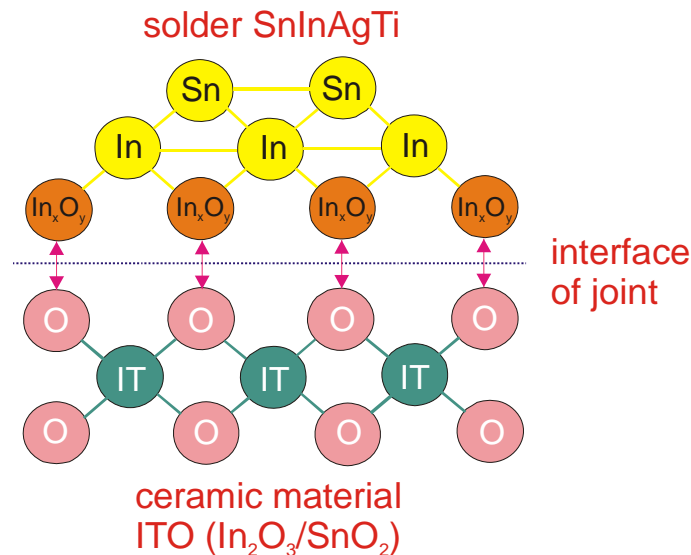


Fig. 6 A simplified model formation of a joint between ITO ceramics and SnInAgTi solder [4]

Conclusion

The result of experiments verified the possibility of making a soldered joint between metal and ITO ceramics. The soldered joint was formed by using mechanical activation power of ultrasound on air, without flux. It was proved that SnInAgTi solder reacts with surface layers of the connected metals while forming reactive products of various thicknesses and qualities. This proves that the solid substrates dissolve in the solder to form diffusion joints. There is no evidence of any diffusion area on the interface between the solder and ITO ceramics. The solder is able to leak into the spaces between the grains thus forming a mechanical joint. Indium plays a major role in producing the joint. Indium provides oxide inputs that react with the ceramics and create a chemical bond.

Acknowledgments

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