SOLDERING OF CERAMIC MATERIALS

Roman KOLEŇÁK

Author: ***Assoc. Prof. Roman Koleňák, PhD.***

Workplace: ***Department of Welding, Institute of Production Technologies, Faculty of Material Science and Technology in Trnava, Slovak University of Technology***

Address: ***J. Bottu 23, 917 24 Trnava, Slovak Republic***

Phone: ***+421 (33) 5521 002 kl. 90***

E-mail: ***roman.kolenak@stuba.sk***

Abstract

Active lead-free solders for soldering metallic and ceramics materials. High temperature activation in vacuum. Mechanical activation by ultrasonic energy. Parameters of ultrasonic soldering. Wettability of ceramics (Al_2O_3, ITO, SiO_2, TiO_2) and non-metallic materials (Si, Ge, graphite). Microstructure of combined joints ceramics/metal. Quantitative and qualitative microanalysis of joint interface.

Key words

active solders, non-metallic materials, mechanical activation, ultrasonic energy

Introduction

The ceramic materials that are not provided with metallic solderable coatings can be soldered only with active solders. The active solders contain an active element – usually Ti which during soldering process reacts with the surface of hard solderable material at formation of a reactive layer – Fig. 1, which then allows wetting of ceramic material and thus also joint formation. Active solders, similarly as commercial brazing/soldering alloys are classified according to melting point into soldering, brazing alloys and high-temperature brazing alloys.

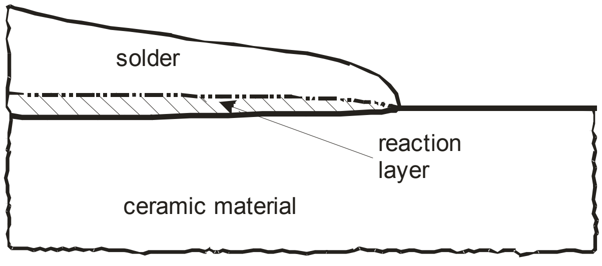

Fig. 1. *Scheme of formation of reaction layer on ceramic material*

Classification of active solders

Active solder can be, by the principle of activation of active element in molten solder classified into the solders destined for:

- ✓ high-temperature activation,
- ✓ mechanical activation.

The active solders on the basis of Sn and Pb for high-temperature activation were produced by Degussa company with designation CS 1 and with composition Sn90AgTi2 and CS 2 with composition Pb94InTi2. Soldering temperature of these solders is 850 to 950 °C. This solder are at present produced on order by the company BrazeTec, GmbH, Germany.

The solder for mechanical activation with composition SnAg6Ti4Ce and commercial designation S-Bond 220 is supplied by Euromat, GmbH, Germany. The recommended soldering temperature is 250 to 280 °C [2].

The SnAg6Ti4Ce solder can be used also at high-temperature activation: however, only small wetting angles are achieved. An example is shown in Fig. 2, where the solder at temperature 860 °C in vacuum of 10^{-2} Pa wetted the Al_2O_3 ceramics with wetting angle of 62°. The experiment was performed in cooperation with the Institute of Materials and Machine Mechanics of SAS Bratislava.

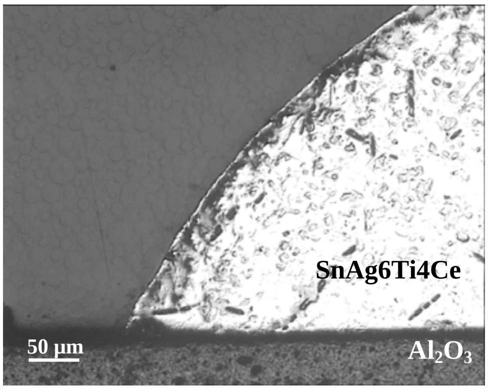

Fig. 2. *A droplet of SnAg6Ti4Ce solder on Al_2O_3 substrate with wetting angle of 62°*

High-temperature activation

Is a process which takes place at high-temperature (850 to 950 °C), mostly in a vacuum furnace, or in the furnace with shielding atmosphere of argon of helium – Fig. 3.

It was proved experimentally that the lowest temperature at which a good wetting of Al_2O_3 ceramics was attained is 780 °C at application of Ti as active element. However, wet-ting additionally depended also on the content of Ti active element in the solder [3].

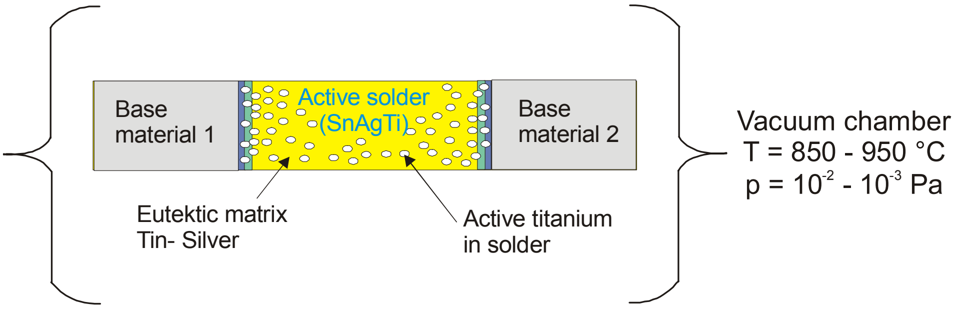

Fig. 3. *Scheme of soldering with high-temperature activation in vacuum [4]*

With increasing soldering temperature (activation temperature) at high-temperature activation also wetting of ceramic material is improved, however on contrary degradation of base metal by erosion may occur, if a ceramic material is soldered in combination with a metallic one. An example of working cycle of soldering in a vacuum furnace type of PZ 810 is shown in Fig. 4.

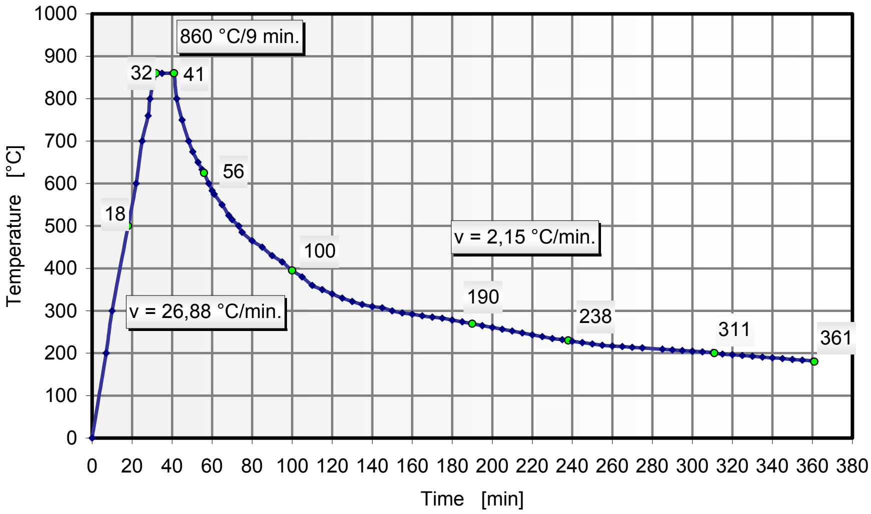

Fig. 4. *Working cycle of soldering in vacuum furnace type of PZ 810*

The working cycle (Fig. 4) consists of a rapid heating at soldering temperature, 9 min. holding time and a long slow cooling down (for about 320 min), which assures the reduction of residual stresses in the joint. During small cooling down, the diffusion processes continue, what is negatively exerted in growth of diffusion zone in BM and solubility of steel in the solder. The example is shown in Fig. 5. The figure shows the diffusion zone in width ~ 70 μm and the solubility zone of austenitic CrNi steel in SnTi3 solder, soldered at temperature 860 °C/9 min. in vacuum with a slow cooling rate (v = 2,15 °C/min.).

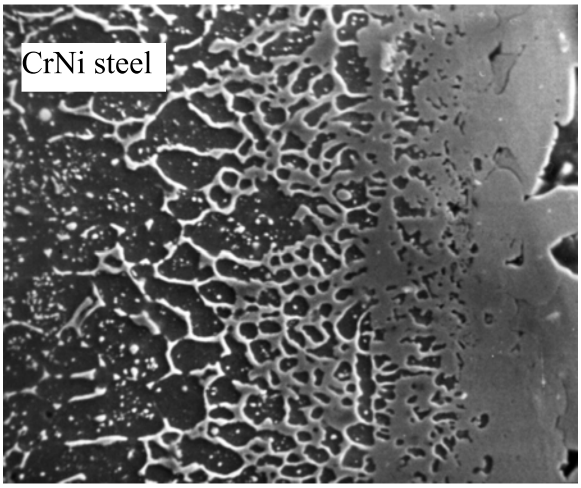

Fig. 5. *Diffusion zone and solubility zone of CrNi steel in Sn solder*

Mechanical activation

Advantage of this approach is that on contrary to solders activated by high temperature, which need considerably higher soldering temperature and can be used only in vacuum or shielding atmosphere, the mechanically activated solder can wet the metallic and non-metallic materials in the interval of temperatures from 240 to 280 °C. This unique property is enabled by the alloying elements: cerium, praseodymium, neodymium (rare earth elements), which occur in small amounts in solder matrix and which form the protective barrier for active metal Ti – Fig. 6 [5].

Mechanical activation can be performed by:

- ✓ scratching (for example with stainless wire brush, steel spatula etc.)
- ✓ vibrations (50 – 60 Hz),
- ✓ ultrasound (20 – 60 kHz).

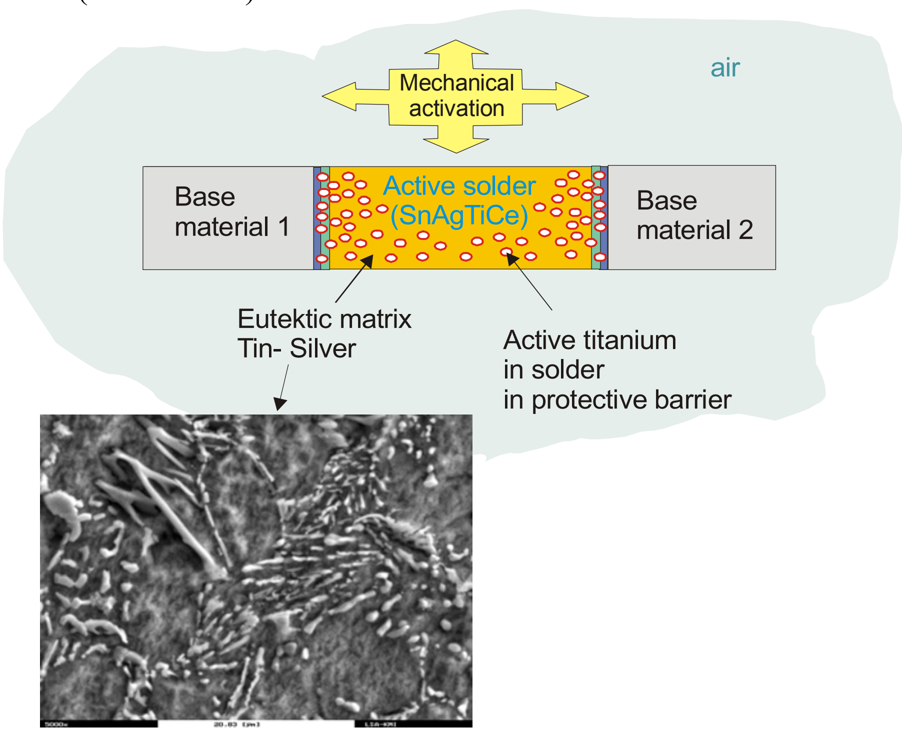

Fig. 6. *Scheme of fluxless mechanical activation in the air (S-Bond process [6])*

Mechanical activation (especially ultrasonic) enables wetting of metallic and ceramic material with SnAg6Ti4Ce solder with very small wetting angle, below 20°. Since the flux in not used in soldering and the solder is spread only due to effect of mechanical activation, the SnAg6Ti4Ce solder cannot be used directly for capillary soldering.

Experiments have shown, that with scratching and vibrations only metallic materials, for example CrNi steel, aluminium and its alloys, copper and its alloys etc. can be used. An example of microstructure of the joint of high-purity aluminium (99,99 %) fabricated with SnAg6Ti4Ce solder and obtained by scratching without flux is shown in Fig. 7. The joint was formed by interaction of the silver component of solder with the surface of Al parent metal at formation of Ag_3Al intermetallic phase.

In soldering of ceramic, non metallic (Si, Ge etc.) and hard to solder metallic materials as tungsten, tantalum, hafnium etc. it is necessary to use ultrasonic activation with the frequency over 20 kHz.

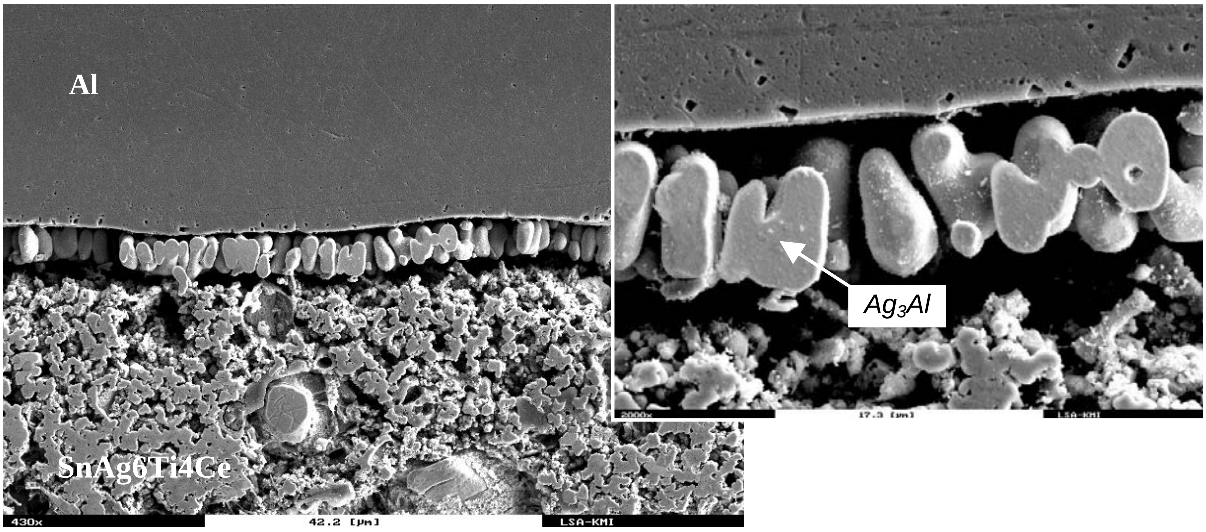

Fig. 7. *Microstructure of joint of high purity Al with SnAg6Ti4Ce solder after deep etching of matrix*

Ultrasonic activation

Experimental equipment destined for soldering ceramic and metallic materials with use of high-power ultrasound is shown in Fig. 8. The equipment consists of ultrasonic generator and ultrasonic head with 400 W power. The ultrasonic head is composed of piezoelectric transducer, concentrating extension and replaceable working tool, made of titanium.

For heating the joints at soldering temperature the hot plate technique was applied. The soldering parameters used at experiments used at experiments are given in Table 1. The scheme of soldering process with ultrasonic head terminated with titanium tool is shown in Fig. 9.

Fig. 8. Ultrasonic generator and ultrasonic head of experimental equipment for ultrasonic activation

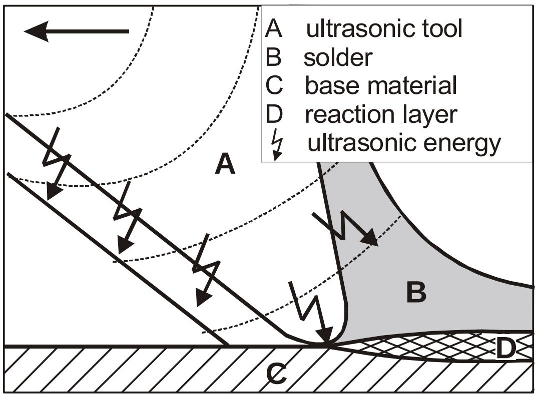

Fig. 9. *Schematic representation of activation of surface of soldered material with US tool [7]*

SOLDERING PARAMETERS Table 1

Frequency	40 kHz
Soldering temperature	270 °C
Time of ultrasound acting	5 s

In experiment with use of ultrasonic activation the Al_2O_3, SiO_2, TiO_2, ITO ceramics, non metallic materials as Si, Ge and graphite, all with purity over 99, 99 % were soldered. These materials were joined mutually or in combination with metals.

It was found out the SnAg6Ti4Ce solder wetted the studied materials with a very small wetting angle. Poor results were attained only in case of soldering pure graphite which was not wetted at all with this solder and thus it was impossible to obtain the joint with graphite by use of SnAg6Ti4Ce solder.

An example of microstructure of soldered joint of Al_2O_3 ceramics fabricated with SnAg6Ti4Ce solder in polished condition is shown in Fig. 10. A ronounced reaction layer containing 35 weight % of Ti was formed between the solder and ceramic material.

By a similar mechanism also the joint between the Si parent metal and SnAg6Ti4Ce solder was attained – Fig. 11. From the course of Ti concentration profile it is evident (Fig. 11) that on the Si – solder boundary there is an increased volume of titanium from the solder, which forms the reaction layer and thus supports the joint formation.

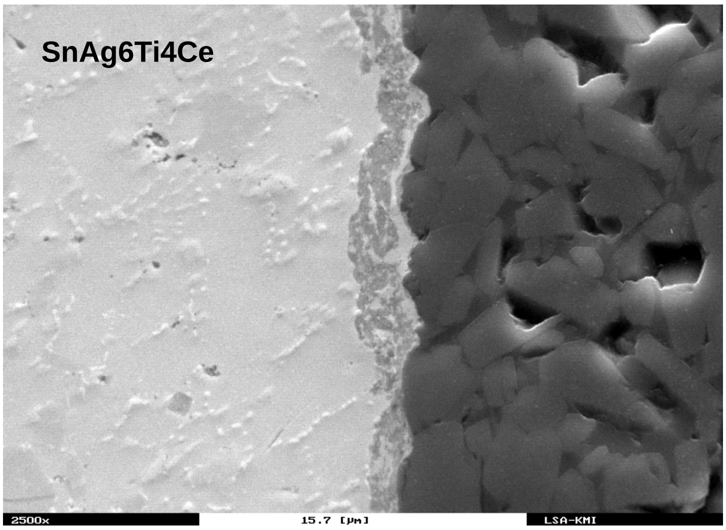

Fig. 10. *Microstructure of Al_2O_3 joint with SnAg6Ti4Ce solder in polished condition*

On the combined soldered joints of Al_2O_3 ceramic - metal, the shear strength tests were performed. The attained values of shear stress of the combined soldered joints of Al_2O_3 ceramics – metal are given in Table. 2. The achieved shear strength of ceramics – metal corresponds to the shear strength achieved by use of common solders.

VALUES OF SHEAR STRENGTH OF CERAMICS – METAL JOINTS

Table 2

Joint type	τ [MPa]
Al_2O_3 – CrNi steel	30
Al_2O_3 – Cu	26
Al_2O_3 – AlSi5	17

Conclusions

Soldering with active solders with application of ultrasonic activation makes possible to solder actually all types of ceramic materials and brittle non-metallic material mutually or in combination with metals without application of high-temperature activation in vacuum furnace. However, at ceramic and non-metallic materials it is necessary to apply the activation by use of high-power ultrasound.

Based on the results of experiments it can be stated that at soldering ceramic materials by use of ultrasonic energy the following advantages, compared to classical method of high-temperature activation in vacuum furnace can be attained:

- ✓ low soldering temperature of about 270 °C, at cooling down from this temperature the formation of residual stresses is eliminated,
- ✓ soldering time is very short (in seconds),
- ✓ cooling time is short, what prevents the formation of undesirable transition zones and erosion of parent metal,
- ✓ vacuum is not needed, soldering is performed in the air without flux.

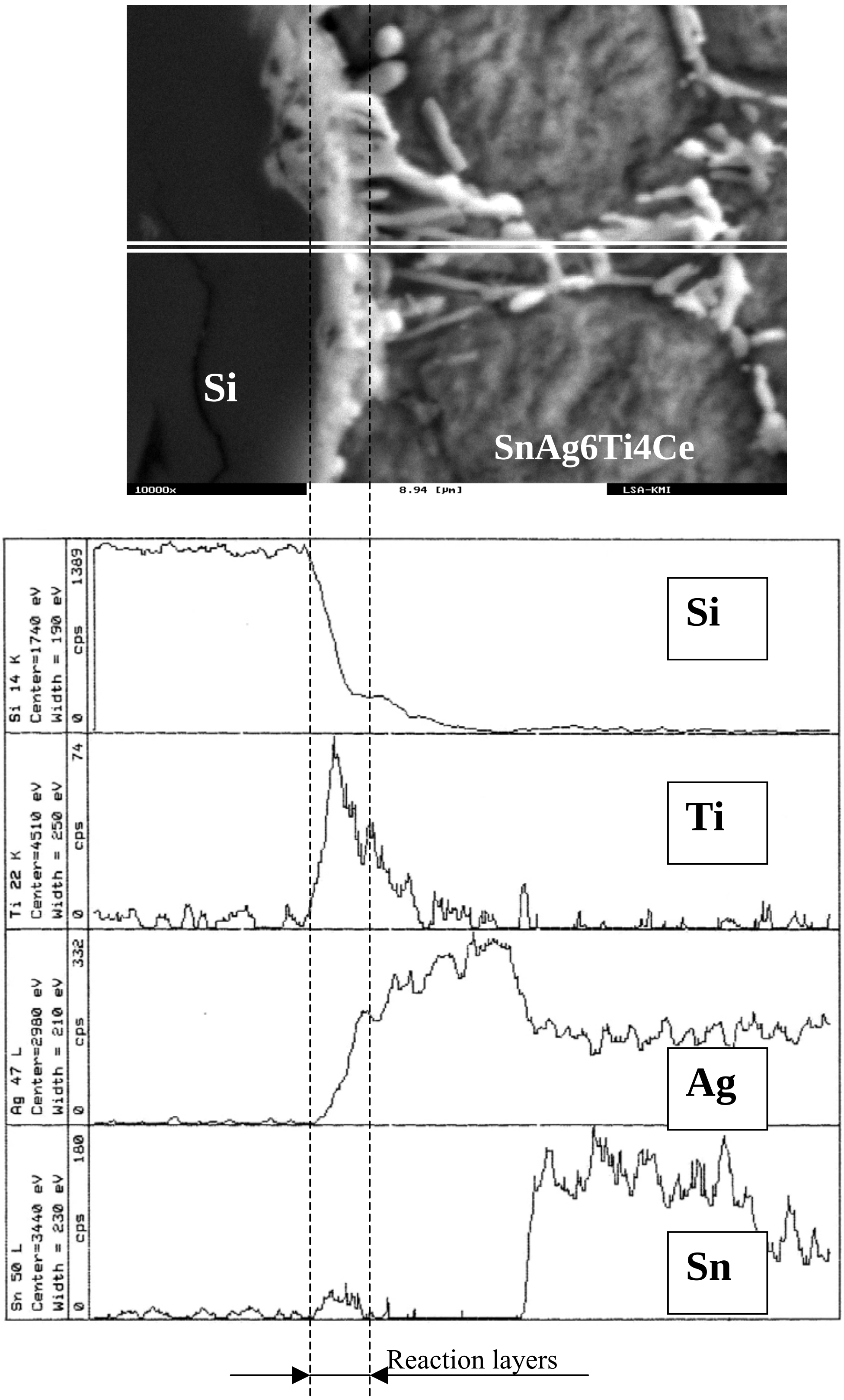

Fig. 11. *Microstructure Si – SnAg6Ti4Ce solder joint and the concentration profiles of elements in this boundary [8]*

This work was supported by projects under the contract No **VEGA 1/0381/08** and **APVT 20-010804**.

References:

[1] KOLEŇÁK, R., TURŇA, M. Soldering of ceramic materials with active solders. In *Zváranie – Svařování*, 2001, 50, č.3 – 4, p. 75 – 78.

[2] www.euromat.de (2009-06-04).

[3] KOLEŇÁK, R. *Solderability of metal and ceramic materials by active solders.* (Scientific monographs). Dresden: Forschungszentrum Dresden, 2008, 72 p. -ISBN 978-3-941405-03-5

[4] S-bondTM Handbook, STB Copyright, USA, 2002.

[5] LUGSCHEIDER, E. Entwicklung von Lotwerkstoffen,http://www.rwth-aachen.de,(2004-3-23).

[6] www.s-bond.com (2009-06-04).

[7] HILLEN, F., RASS, J.I., LUGSCHEIDER, E. Low temperature joining for heat exchangers and other applications. In *ISBC 2000, ASM International.* Albuquerque, New Mexico, 2000.

[8] MAŇKA, L. *Physico-metallurgical aspects of soldering semiconductor materials*. Thesis. Trnava: FMST SUT, 2004.

[9] HODÚLOVÁ, E. Lead-free soldering of coated Al_2O_3 ceramics. In *Engineering Sciences 2 / nadát. 4th international conference of PHD*. Miskolc: University of Miskolc, 2003, pp. 99-102. ISBN 963 661 585 3 ö. - ISBN 963 661 591 8. -