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Article

Characterization of ultrasonic soldering of Ti and Ni with Ni/Al reactive multilayer deposition

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Abstract: The joining of Ti and Ni at low temperatures was analysed in this work. For joining pure Ti and Ni coins of 1.5 mm in thickness were used. Reactive multilayer thin films/foils with nanometric period (bilayer thickness), in particular Ni/Al multilayers, have been used to promote joining in two thickness of 28 and 55 nm. The ultrasonic soldering with SnAgTi active solder has been used for "hard-to-solder" material. The structural evaluation of soldered joint was studied by optical microscopy and EDX analysis. The structural analysis was focused to the creation of intermetallic layers in the joint interface. The mechanical properties of solder joints were tested by shear strength.

Keywords: soldering; multilayer; ultrasonic

Introduction

Joining is fundamental for the construction of highly sophisticated devices and components and promotes the development of advanced technologies. In particular, the development of novel methods for producing sound joints in dissimilar and advanced materials is essential. However, there is no universal process that will perform adequately on all materials in all geometries and joining of two different materials. An interlayer or filler material is frequently needed in dissimilar joining [1].

Typically, titanium alloys have high strength to weight ratio, good corrosion resistance (even at high temperatures) and are biocompatible, which makes them attractive for several applications from biomedical to aeronautic field. The joining of these alloys to themselves and to other materials is difficult due to their high reactivity. Conventional fusion welding methods often lead to the formation of brittle intermetallics in the weld zone. This problem was recently overcome by laser welding of Ti-6Al-4V to NiTi using a 75 μm thick copper interlayer to limit diffusion and hinder the formation of brittle intermetallics [2]. Promising alternatives to join Ti alloys could emerge by using reactive multilayers as filler material [3÷6].

The self-propagating nature of some reactive multilayers has potential for joining applications and has been a topic of intense research during the last decades [e.g. 6,7]. Reactive multilayers are composed of tens, hundreds, or thousands of alternating individual layers of reactants having large negative enthalpy of mixing. Nanoscale multilayers have improved diffusivity and reactivity. For certain designs, these multilayers exhibit fast, high temperature reactions which make them attractive to be used as highly localized heat sources. External heating initiates the reaction locally, releasing heat that drives the reaction forward in a self-propagating wave. The heat released by the exothermic reaction in Ni/Al reactive multilayer foils has been used to melt braze alloys promoting joining [e.g. 8,9]. Typically, reactive multilayers thin films are prepared by physical vapour deposition (sputtering or evaporation). Several metal/metal multilayers films/foils have been investigated, in particular Ni/Al, Ti/Al, and Ni/Ti.

The exact dosing of ultrasonic power, parallel to the surface of molten metal, in combination with local heating in melts makes it possible to concentrate the energy within a small volume. In this way, the effect of mechanical action on surfaces is suppressed, melt oxidation is reduced and the durability of soldered joints improved [10]. Ultrasonic activation is one of progressive methods, which rapidly amplifies most of physical processes as wetting, spreadability, solder capillarity and diffusion by aid of mechanical-elastic oscillations with frequencies from 18 to 70 kHz and intensities of 0.1 to 1.0 MW/m^2.

Great emphasis is pay to the versatility of the newly developed soldering alloy. Lead-free solder will be used for soldering of Ti and Ni alloys. Soldering alloys with a working range (260 °C to 450 °C), corresponding to solders for higher application temperatures [11÷13].

Similar and dissimilar joints are processed by fluxless soldering using power ultrasound, taking into account the most recent trends in joining materials through the application of non-toxic and recyclable consumables, namely by using solders free from lead and cadmium. Reactive multilayers thin films are used in order to enhance the soldering process.

Materials and Methods

The substrates of Ti and Ni with the purity of 4 N were used in experiments. Ti and Ni base materials were selected for producing similar and dissimilar ultrasound soldering joints. Cylindrical samples (ϕ=15 mm) 1.5 and 2.5 mm thick, as well as square samples (1x1x2 mm) were cut and prepared by conventional grinding with SiC paper up to 2000 mesh. For the shear tests a cylindrical sample is joined to a square sample.

The base materials were previously coated with Ni/Al multilayer thin films. Multilayer thin films with nanometric bilayer thicknesses (period) were deposited by magnetron sputtering (Fig. 1) using two targets (Al and Ni). Different periods were obtained by varying the substrates' rotation speed. All the other deposition parameters were kept constant (deposition pressure and time, power applied to each target, substrates' cleaning, etc.). The power applied to each target was adjusted in order to obtain a near equiatomic average chemical composition. Ni/Al multilayers with modulation periods (Λ) of approximately 25 and 50 nm were prepared. The objective is to enhance the soldering process and check if Λ could influence the joints' quality.

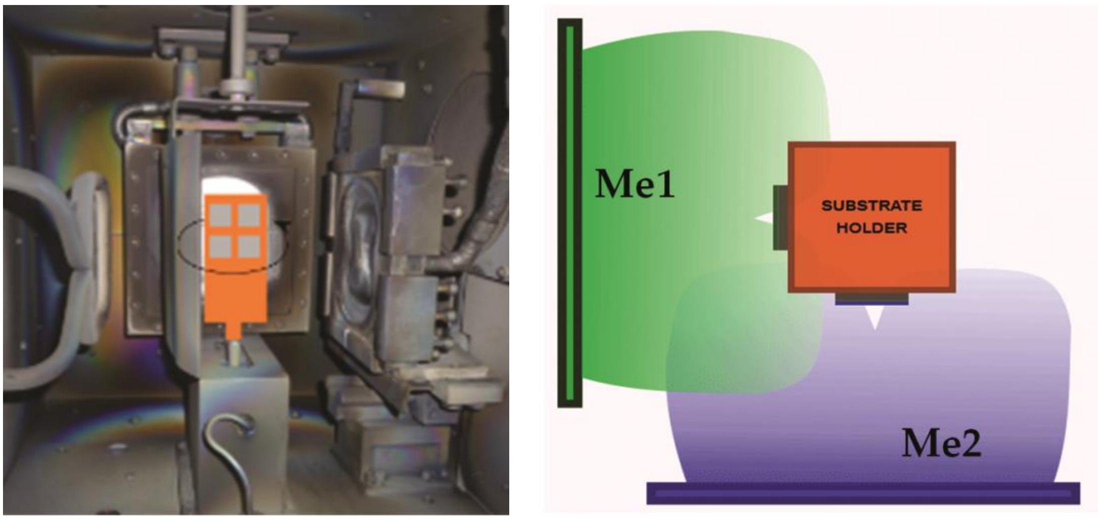

Fig. 1. Ni/Al reactive multilayer thin films deposited by dual cathode magnetron sputtering

The nanolayered structure of the multilayer thin films was confirmed by field-emission gun scanning electron microscopy (FEG-SEM). The image in figure 2 allow the Ni and Al alternated layers to be distinguished, confirming the desired modulation period (close to 25 nm). According to Thornton's model, the Ni/Al thin film exhibits a columnar morphology.

Ti/Ni joints were prepared by ultrasound soldering using a SnAgTi solder. It is a commercial solder with the labeling S-Bond 220. An ultrasonic equipment type Hanuz UT2 was used for soldering with the parameters presented in table I.

The encapsulated ultrasonic transducer consisting of a piezo-electric oscillation system and a titanium sonotrode with the tip diameter of ø3 mm was used for solder activation. The soldering temperature was 50 °C above the solder liquidus temperature. The soldering temperature was checked by a continuous temperature measurement using NiCr/NiSi thermocouple. Soldering was performed in such a manner that the solder was first deposited on the base material heated at soldering temperature and it was then heated at liquidus temperature. The molten solder was activated by a power ultrasound without application of flux and/or shielding atmosphere. This material is quickly laid on the next base material while attaching the surface with the solder layer deposited by ultrasound activation and it is immediately centered. In this way, the desired joint is finally formed after solidification. Schematic representation of this procedure is shown in figure 3.

Table I. Soldering parameters

Ultrasound power [W]	400
Working frequency [kHz]	40
Amplitude [μm]	2
Soldering temperature [°C]	310
Time of ultrasonic activation [s]	5

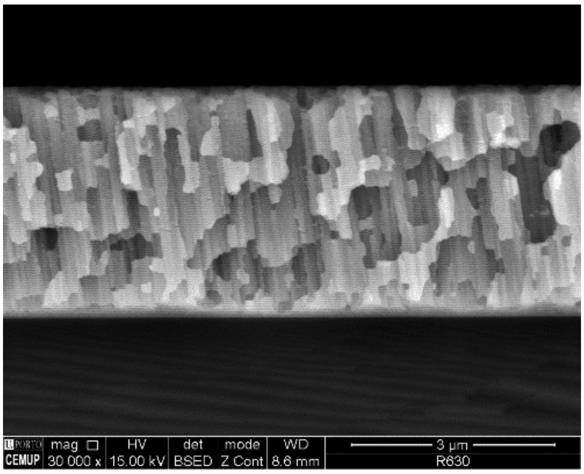

Fig. 2 Cross-section SEM images of a Ni/Al multilayer thin film with a period (Λ) close to 25 nm

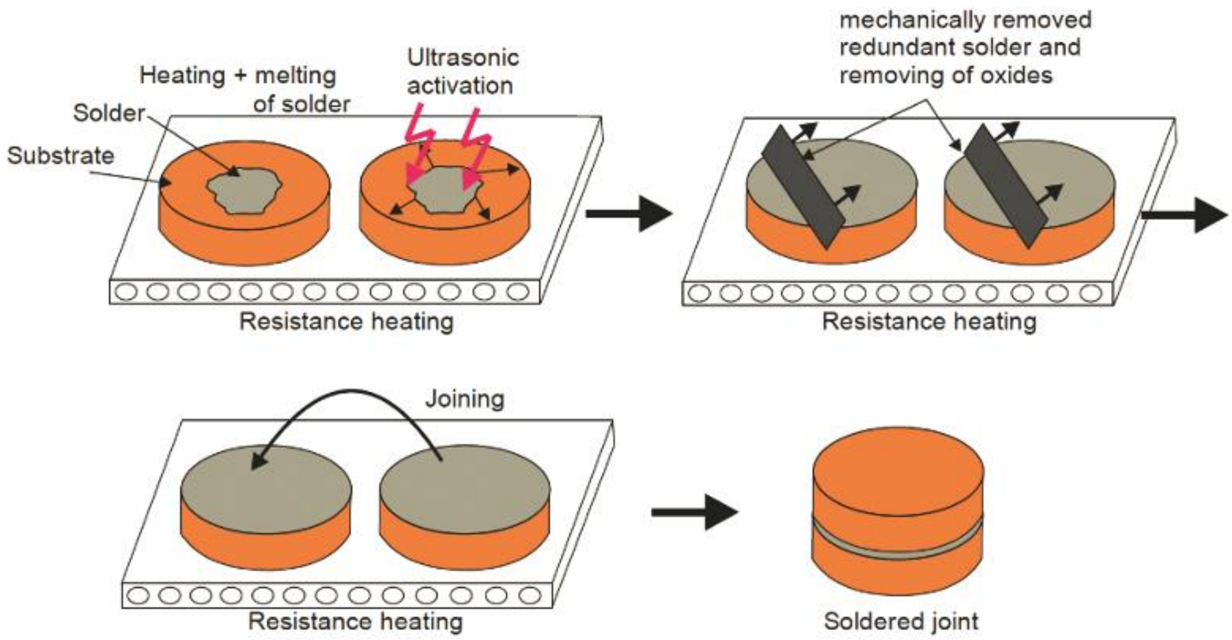

Fig. 3 Schematic representation of soldering process at the presence of ultrasonic power

The joints were observed by SEM and analysed by energy dispersive spectroscopy (EDS) in order to evaluate the chemical composition at the joints' interface. A JEOL Scanning Electron Microscope JSM-IT300 and a Zeiss Merlin FEG-SEM were used. The joints' cross-sections for SEM were prepared using standard metallographic techniques. Mechanical characterization of the joints was carried out by nanoindentation and by shear testing.

Results

The joints were formed by interaction of Ti and Ni base materials, Ni/Al multilayer films and SnAgTi solder. Figure 4 shows a SEM analysis of Ti/Ni soldered joints. The multilayer film with the desired modulation close to 55 nm and close to 28 nm was used for coating of base materials. From the Ni side the mixing of solder and film, the distinguishable alternate layers could be observed. From the Ti side the interface is without cracks. The coated Ti substrate has been wetted by the solder. Inside the solder's matrix several cracks can be observed.

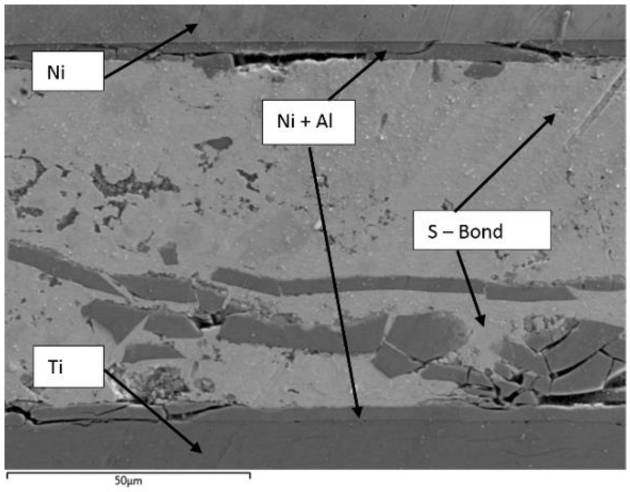

Fig. 4. SEM analysis of Ti/Ni soldered joints

Microstructure of SnAgTi solder (Fig. 5) indicates in the three crystalline phases, β-Sn, Ag_3Sn (gray contrast) and α-Ti_6Sn_5 (dark gray contrast) were identified. The Ti element was dissolved in the Sn–Ag solution at high temperature and then precipitated out of solution and formed α-Ti_6Sn_5 during the cooling process. The distribution and dispersion of α-Ti_6Sn_5 crystals in the Sn–3.5Ag based solders also showed that the titanium had been melted well.

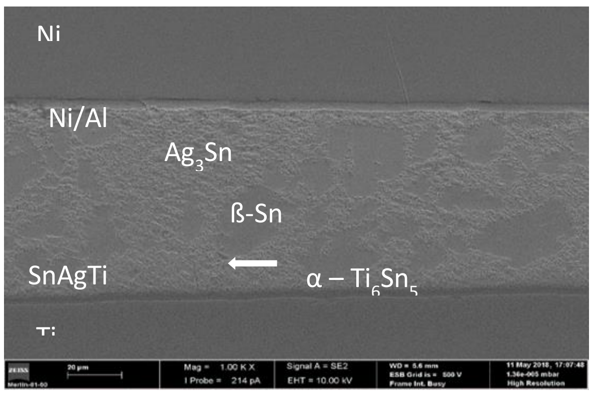

Fig. 5 Microstructure of SnAgTi solder

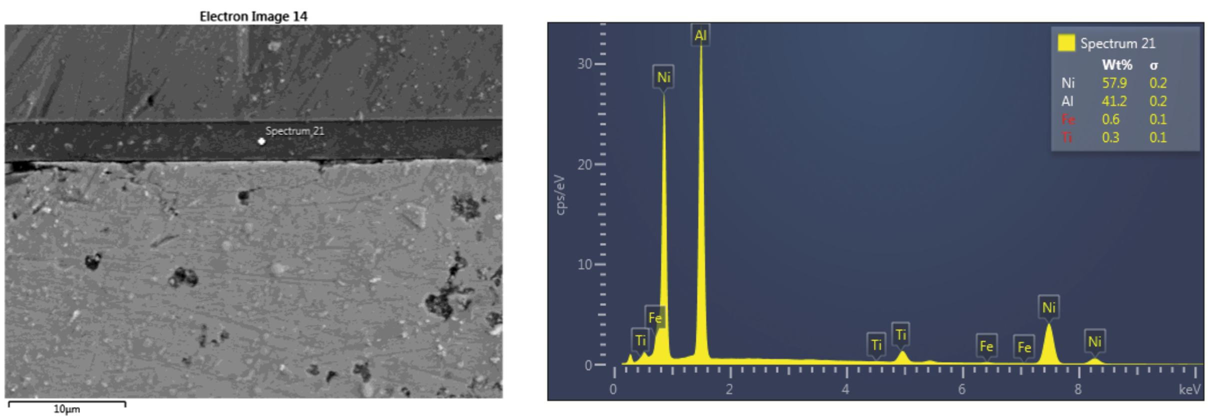

Fig. 6 SEM analysis on Ni/Al multilayer

The Figure 6 shows the distinguishable alternate layers but in the interface of film and solder several cracks can be observed on both sides of the base materials. Also no adhesion between film and solder can be observed in the interface. The detailed SEM analysis confirmed the presence of Ni/Al in the substrate and solder interface.

The detailed SEM analysis (Fig. 7) shows the interaction of substrate with Al element in the Ni/Al multilayer. From Ti side and also from Ni side. These demonstrate that cavitation effect has been created in the soldered seam during the fluxless ultrasound assisted soldering process, which induces the wetting of SnAgTi solder on the multilayer film.

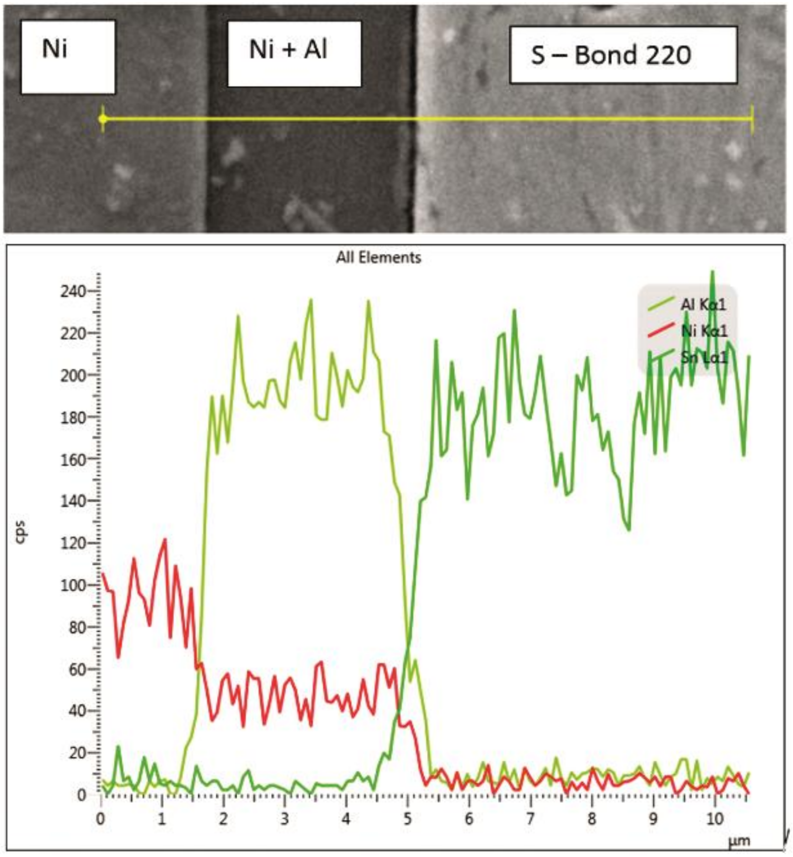

Fig. 7. SEM analysis of substrate's interaction with Al element in the Ni/Al multilayer

Hardness and Young's modulus were determined by nanoindentation using the Oliver and Pharr analysis method. A Micro Materials – Nano hardness apparatus equipped with a Berkovich diamond indenter was used. Before the indentation experiments, the tip area function was calibrated using fused silica as standard. The nanoindentation experiments were carried out for joints where suitable regions were observed by SEM. In these cases, loading/unloading experiments were run up to a maximum load of 3 mN with a dwell period of 30 s at 0.3 mN during unloading for thermal drift correction. Several indentation matrixes with 100 measurements each were selected in order to test joint's interface (solder and film), as well as both base materials. A summary for hardness of are in the table II.

Table II. Hardness of Ti/Ni soldered joints using the Ni/Al multilayer

Hardness [GPa]	Ni	Film Ni side	Solder	Film Ti side	Ti
Ti/Ni [Λ≈28 nm]	3.6±0.38	23.5±5.3	0.27±0.095	10.8±2.0	2.8±0.25
Ti/Ni [Λ≈55 nm]	4.6±0.54	30.4±9.3	0.48±0.22	Not possible	3.6±0.2

The highest hardness shows the multilayer film and the lowest the solder. The sample of Ti/Ni (Λ ≈ 55 nm) indicates the nonuniformity of hardness level. The hardness of substrates shows the comparable results. From these results it is not possible to have the conclusion about the influence of the multilayer films thickness to the hardness.

Shear tests were carried out to determine the shear strength of the soldered joints. The shear strength was determined on a versatile tearing equipment type LabTest 5.250SP1-VM. A new-developed shearing jig was used for directional change of axial tearing force (see Fig. 8). This shearing jig ensures a uniform loading of the specimen by shear in the plane of base material/solder interface. The tests were performed on 3 specimens of each base materials' pair. The values of shear strength of created joints are shown in table III.

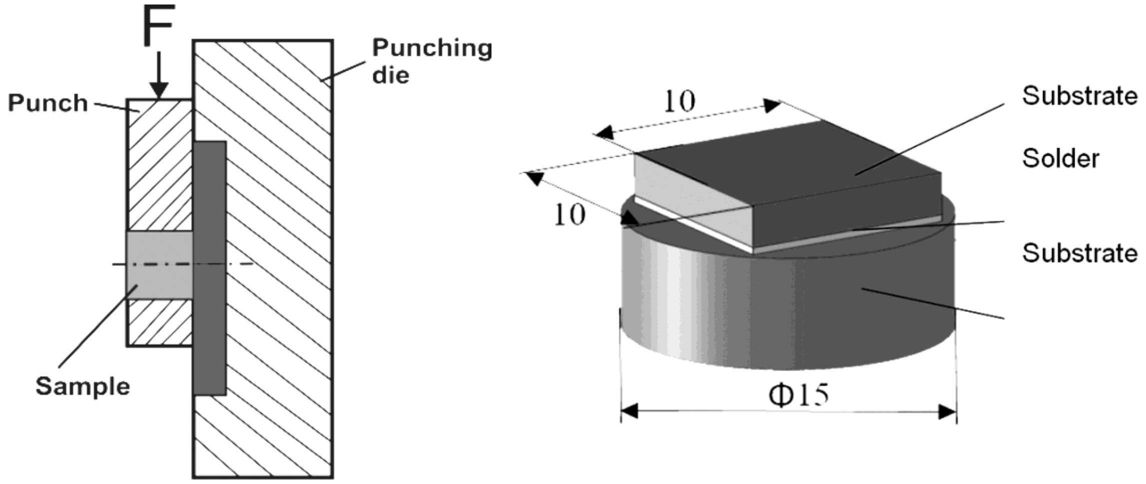

Fig. 8. Scheme of shear strength measurement

Table III. Values of shear strength of created joints

Soldered joints	Shear strength (MPa)
Ti/Ni (Λ≈28 nm)	36
Ti/Ni (Λ≈55 nm)	45

The soldered joint Ti/Ni (Λ ≈ 55 nm) has the highest shear strength of aproximatelly 45 MPa, which is 9 MPa higher than that of soldered joint Ti/Ni (Λ ≈ 28 nm), respectively. The obtained results of strength values are comparable with experimental results of different authors. From these facts it may be concluded the difference of shear strength values for different films thickness.

Conclusions

The Ni and Al alternated layers to be distinguished, confirming the desired modulation period (close to 25 nm). The Ni/Al thin film exhibits a columnar morphology. The cross-sections of Ti/Ni joint (Λ ≈ 55 nm) shows that from the Ni side the mixing of solder and film could be observed. From the Ti side the interface is without cracks. The SEM analysis observation of Ti/Ni joint (Λ ≈ 28 nm) shows the distinguishable alternate layers but in the interface of film and solder several cracks can be observed on both sides of the base materials. Also, no adhesion between film and solder can be observed in the interface.

Microstructure of SnAgTi solder indicates in the three crystalline phases, β-Sn, Ag_3Sn and α-Ti_6Sn_5 were identified. The detailed SEM analysis shows the presence of reaction layer between the Al/Ni thin films and solder. These demonstrate that cavitation effect is created in the soldered seam during the fluxless ultrasound assisted soldering process, which induces the wetting of SnAgTi solder on the multilayer film.

Nanoindentation experiments across the joints - the highest hardness shows the multilayer film and the lowest the solder. The sample of Ti/Ni (Λ ≈ 55 nm) indicates the nonuniformity of hardness level. The hardness of substrates show the comparable results. From these results it is not possible to have the conclusion about the influence of the multilayer films thickness to the hardness. Shear strength - The soldered joint Ti/Ni (Λ ≈ 55 nm) has the highest shear strength of aproximatelly 45 MPa, which is 9 MPa higher than that of soldered joint Ti/Ni (Λ ≈ 28 nm), respectively. The obtained results of strength values are comparable with experimental results of different authors. For samples from pure disimilar metal joints, the difference of shear strength values for different films thickness can be seen.

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Conflicts of Interest: “The authors declare no conflict of interest.” “The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results”.

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