Selection of the Proper Diffusion Welding Parameters for the Heterogeneous Joint Ti Grade 2/AISI 316L

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The creation of the heterogeneous joints at materials with the different physical and mechanical properties is always problematic. As one of methods by which can be achieved very good results is there a diffusion welding. The aim of paper is to show the possibilities of diffusion welding utilization at creation the heterogeneous joints between Titan grade 2 and high-alloyed austenitic steel AISI 316L. The fundamental theory of diffusion and also scheme and realization of experimentally created diffusion welds in the thermal-mechanical simulator Gleeble® 3500 is described in the article. Furthermore, procedure of technological parameters selection when optimization of heterogeneous joint strength properties including metallographic evaluation are taken into account, are also presented.

Keywords: diffusion welding, Gleeble® 3500 and 3800, 316L steel, Ti Grade 2, processing parameters

1 Introduction

Welding represents one of the widespread technologies used for creating permanent joints. However, during the application of welding methods there can occur defects whose magnitude depends on the base materials to be welded and process parameters and has a significant influence on the weld mechanical properties. Particularly, when welding dissimilar materials, many difficulties may arise from differences in physical, thermal and chemical properties (i.e. difference in melting points, thermal expansion or formation of brittle intermetallic phases) and may lead to the formation of defects within the joint and poor corresponding mechanical properties. These difficulties can be intensively decreased or overcome by using special welding methods, such as diffusion welding. In that case, since solid state bonding is accomplished by diffusion of the materials species across the interface, the area of structural heterogeneity is restricted to approximately tens of micrometers and problems such as segregation or cracking generally encountered in liquid phase welding techniques are avoided.

The use of appropriate intermediate metal (Ni [5,6,9,10] or Cu [8]) has been reported to improve the mechanical properties of the diffusion bonded joints between titanium and stainless steel. However, some studies have shown that direct diffusion bonding between titanium and stainless steel is possible [11,12,13,14] and despite the formation of brittle intermetallic compounds in the diffusion zone, maximal strength of 76% of that of titanium is reported in the bonded joints [15].

This article presents the experimental procedure of diffusion welding for titanium Grade 2 and AISI 316L austenitic steel. The work focused on achieving the highest tensile strength of the dissimilar weld without using any intermediate layer.

2 Diffusion theory

Diffusion in the solid matters can be (as any transport effect) viewed from the two points of view – in light of phenomenology and microscopy. Phenomenological diffusion theory deals with the overall balance of transport process in solid substance undertaken at the given conditions. Microscopic perspective is considered in the atomic diffusion theory. The basic explanation of atomic diffusion theory is introduced for example in books [1] or [3].

General phenomenological diffusion concept is described as thermodynamic non-returnable events given by a flow of thermodynamic quantities. This is defined by their amount which passes through the surface unit by time unit from one stage to the other or to a system. According to Onsanger, the flow Ji of thermodynamic value i is linearly dependent on each thermodynamic motion power Xi which depends on specific system according to the equation (1), [1]

$$Ji = \sum_i Lij \cdot Xi, \text{[depends on variable Xi]} \qquad (1)$$

Where:

Lij - Osanger's kinetic coefficients , [depends on variable Xi]

Xi - thermodynamic motion powers (e.g. self-diffusion by vacant mechanism when the only motive powers are gradients of primary element chemical potential) [depends on chosen thermodynamic variable].

According to Fick, diffusion occurs due to the gradient of concentration. It is a special case of general understanding of diffusion which is only valid when simplified assumptions are taken into account and when it is impossible to describe complicated transferable effects. In spite of that, Fick's explanation is technically important due to its simplicity and high number of gathered values of the overall diffusion coefficient D in different thermodynamic systems.

The knowledge of concentration is summarized by the two Fick's laws. According to the first Fick's law (2), the diffusion flow J_A of element A atoms throughout given time and in the direction of axis x and throughout the surface unit depends on the gradient of element concentration $\frac{\partial C_A}{\partial x}$ and is proportional to diffusion coefficient *D.* Simply stated, the first Fick's law expresses speed and amount of atoms which diffuse through material.

$$J_A = -D\frac{\partial C_A}{\partial x}, \quad [\mathrm{mol \cdot m^{-2} \cdot s^{-1}}] \tag{2}$$

In case of variable diffusion flow, the change of concentration is connected with time. There are termed so-called non-stationary diffusion which is described according to the second Fick's law (3),

$$\frac{\partial C_A}{\partial t} = \frac{\partial}{\partial x}\left(D\frac{\partial C_A}{\partial x}\right), \; [\mathrm{mol \cdot m^{-3} \cdot s^{-1}}] \tag{3}$$

Where:
C_A - concentration of element A [$\mathrm{mol.m^{-3}}$],
x - the direction of diffusion concentration change [m],
t - amount concentration change time [s],
D - the diffusion coefficient [$\mathrm{m^2 \cdot s^{-1}}$].

3 Experiments

The chemical composition of materials used in this study is given in Table 1. Both materials were received in the form of bars of 12mm diameter. Their room temperature mechanical properties are given in Table 2. These data serve as reference data for evaluation of diffusion welded joints strength.

Tab. 1 Measured chemical composition of investigated materials *[wt%]*

316L	***C***	***Mn***	***Si***	***P***	***S***	***Cr***	***Mo***	***Ni***	***Cu***
	0.03	*1.96*	*1.02*	*0.05*	*0.02*	*18.5*	*2.24*	*13.1*	*1.02*
Ti Grade 2	***O***	***N***	***C***	***H***	***Fe***	***Al***	***V***	***Ni***	***Mo***
	0.25	*0.03*	*0.08*	*0.015*	*0.3*	-	-	-	-

***Tab. 2** Measured mechanical properties of investigated materials*

	Yield stress $R_{p0.2}$ [MPa]	***Ultimate stress R_m [MPa]***	***Elongation A_5 [%]***	***Hardness HV10 [HV]***
Ti Grade 2	*319*	*485*	*30.2*	*211*
316L	*361*	*567*	*46.9*	*217*

Diffusion bonding experiments were performed by using the thermo-mechanical simulator Gleeble® 3500 and 3800 where samples are heated by Joule effect. For welding experiments were used cylindrical samples of length l = 50 mm and diameter d = 12 mm. Front contact faces of cylinders were machined [2,4] on the roughness Ra = 1.14 µm for steel 316L and Ra = 0.81µm for Titan Grade 2 and cleaned with the acetone. Welding tests were carried out under vacuum of $1.3 \cdot 10^{-1}$ Pa. Samples were heated to the bonding temperature with a heating rate of 2°C/s. Recommended diffusion bonding temperatures should vary about 0.7x melting temperature, corresponding to 1165°C for Titanium Grade 2 and 1050 °C for 316L steel. However, during preliminary tests, it was observed that mechanical properties of Titanium sharply decreased at temperature exceeding 950°C and compressive forces higher than 0.7 kN (6.2 MPa) already caused significant bulk plastic deformation which is undesirable. So bonding temperature were chosen as below 950°C. Bonding time, temperature and force were changed within the range shown in table 3.

***Tab. 3** Used diffusion welding parameters*

Temperature [°C]	*825 - 950*
Time [min]	*20 – 90*
Compressive force [kN]	*0.3 – 0.7*

Temperature was controlled by using a K-type thermocouple welded 1mm from the interface on the Titanium side as illustrated in Fig. 1. The temperature at the steel side was also measured 1mm from the interface. Temperature distribution in samples for homogeneous as well as heterogeneous combinations is shown in Fig. 2. From this figure it is evident that a non-uniform temperature distribution occurs during welding of heterogeneous joint Ti Grade 2/316L due to different heat conductivity. Maximal temperature was achieved in the Ti sample in the distance approx. 2.5 mm from the interface. It can also be seen that temperature decrease is steeper in steel than in titanium alloy.

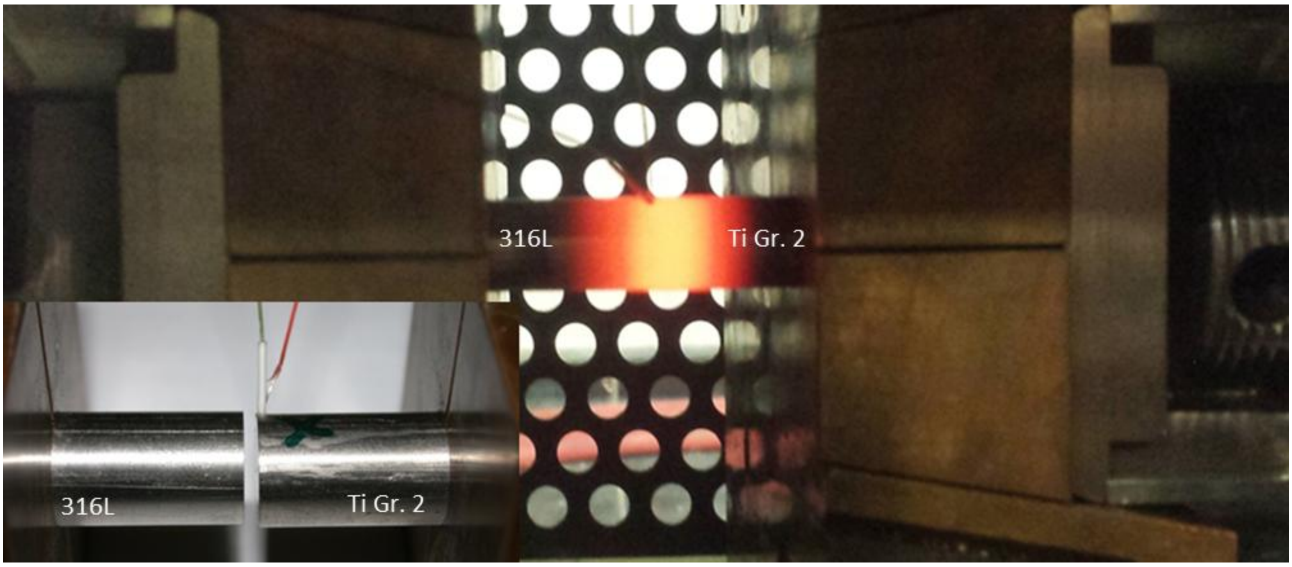

Fig. 1 *Samples of 316L steel and Ti Gr 2 clamped in the high-temperature jaws of Gleeble®*

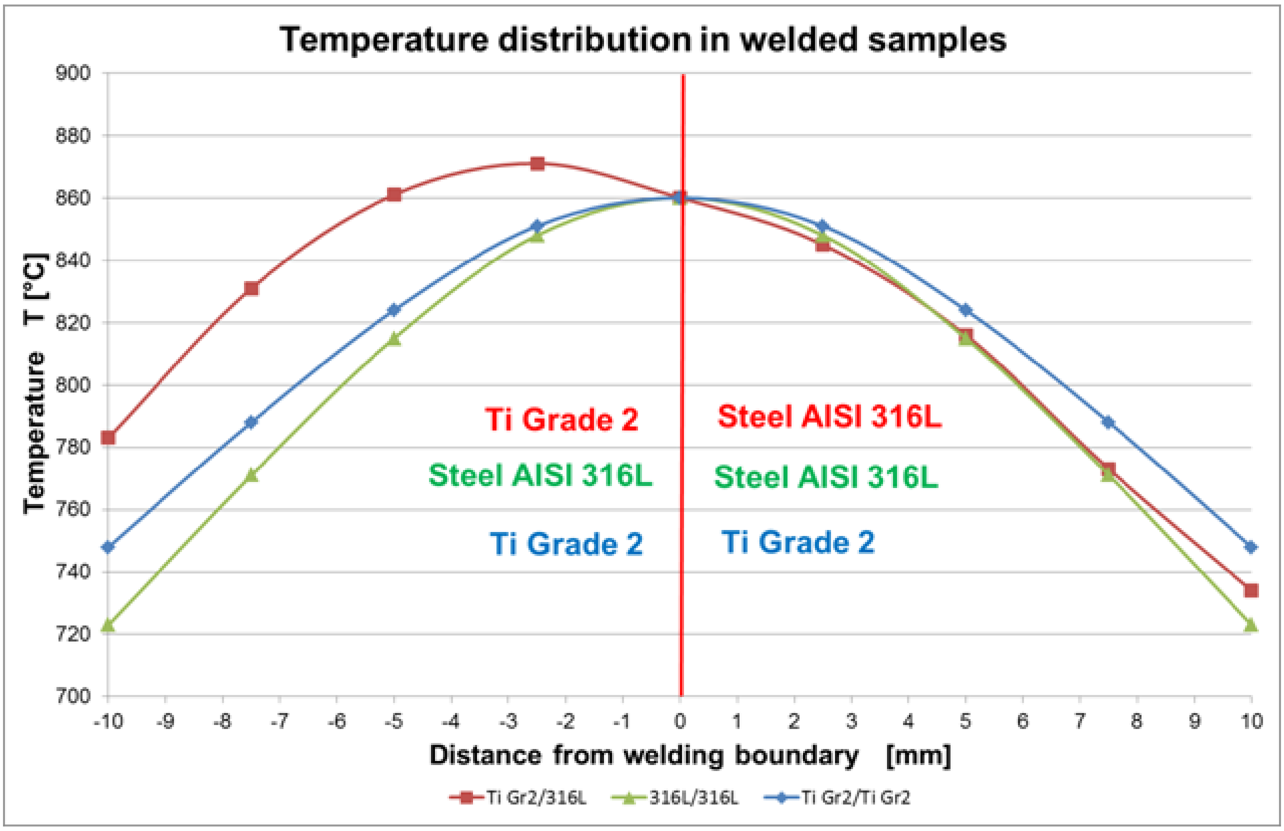

Fig. 2 *Temperature distribution in Ti Gr2 and 316L steel samples during homogeneous and heterogeneous diffusion welding at 860°C*

For metallographic examinations, samples were cut longitudinally through the bond and placed in a resin for grinding and polishing. Then they were examined using both light and scanning electron microscopy including EDX-measurements. Microhardness (HV0.02 and HV0.03) was measured through the bond. Tensile strength tests of bonded samples were evaluated at room temperature with the bonded interface at the centre of the gauge length.

The bonding quality was assessed based on the plastic deformation of metals during bonding (which should be minimal), diffusion zones on both sides of the interface as well as on the ultimate tensile strength values (which should be maximal).

4 Results and discussion

The tensile strengths of diffusion bonded samples using different parameters are shown in table 4. All samples fractured in the elastic regime and did not reach the yield strength. The best strength results were achieved by using the following combination of processing parameters: temperature T = 860 °C, compression force F = 0.5 kN and exposition time t = 40 min. With this parameters combination, a joint tensile strength of 254 MPa was measured, which corresponds to strengths achieved for the similar combinations of materials at diffusion welding [7]. However, in this case was this magnitude achieved with the very small local deformations. Evaluation of the diffusion weld quality can be also performed by means of chosen NDT methods as is e.g. ultrasonic Time-of-flight diffraction technique [8].

Tab. 4 Results of the static tensile tests for diffusion welded joints Ti Gr. 2/316L

Surface preparation	*Atmosphere [-]*	*Temperature [°C]*	*Force [kN]*	*Time [min]*	*Fracture stress [MPa]*
machined	*vacuum*	830	-1.1	40	233
machined	*vacuum*	845	-1.1	20	217
machined	*vacuum*	860	-0.5	40	254
machined	*vacuum*	860	-0.8	40	207
machined	*vacuum*	860	-0.6	90	222
machined	*vacuum*	860	-0.8	90	230

The microstructure of diffusion welded joints is shown in fig. 3. The interface is quite regular and does not show many defects. Starting from the interface, two different zones can be observed in the Ti side: a uniform zone, zone 1 and a lamella zone, zone 2 - as is illustrated in fig. 3b. EDX measurements shown in fig. 4a reveal that Ti diffuses much less in the steel while Fe and Cr diffuse on significant distances into the Ti part. It can be seen that the zone 1 is enriched with Fe and Cr and zone 2 is enriched with Fe. Further measurements in the latter zone show an alternating structure of lamella enrich in Fe and lamella rich in Ti, see Fig 4b. The zone 2 was observed only for bonding temperatures higher than 850°C. And the width of both zones increases with increasing bonding temperatures (for a constant bonding time). At T = 825 °C the width of zone 1 is x=28±2 μm, at 860°C x= 31±2 μm, at 900°C x= 35±3 μm and at 950°C x =63±3 μm under the time of exposition 40 min. Hardness measurements show a strong increase in hardness up to 430 HV0.03 in zone 1 and intermediate hardness values around 350 HV0.03 in zone 2 as displayed in Fig. 5.

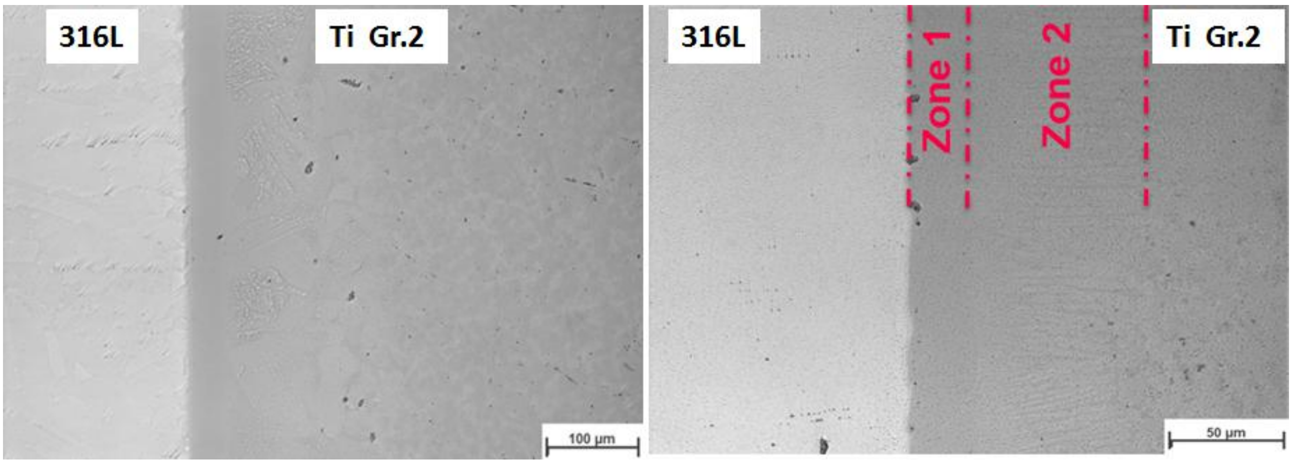

Fig. 3 *Light optical microscope image of the diffusion welded sample with a) 875°C, 90', 0.7 kN and b) 900°C, 40', -0.5 kN*

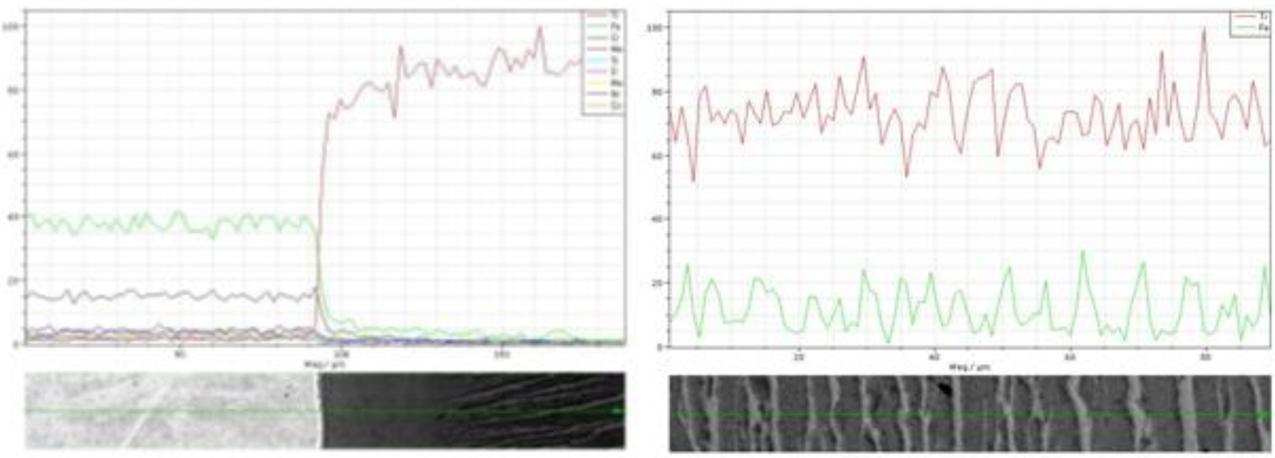

Fig. 4 *EDX measurements through the diffusion welded joint (a) and in the zone 2 (b)*

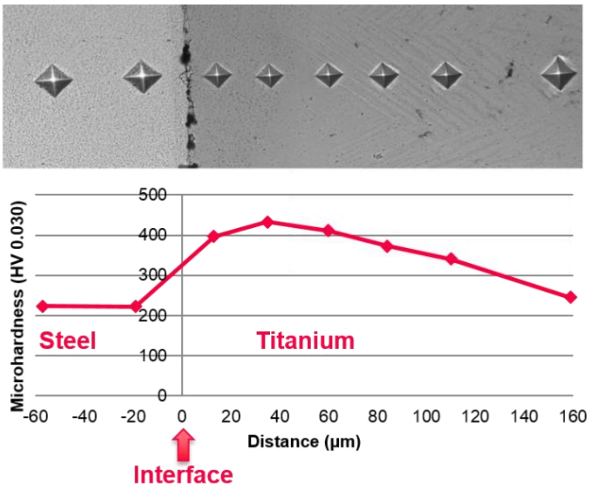

Fig. 5 *Hardness profile through the diffusion welded joint (950°C, 40', -0.5 kN)*

Beside the bonding temperature, time and pressure, other parameters such as contact surface roughness, environment in which the diffusion takes place (vacuum, shielding atmospheres and so on), contact areas cleanness have also influence on the welded joint properties. Because the possibilities to change processing parameters (temperature, pressure and time) were greatly used in the first phase of experiments, the second phase of these experiments concerned mostly about the influence of the sample surface roughness and environment in which the diffusion process takes place. For this second phase of experiments, the parameters permitting to obtain the highest tensile strength were used: temperature 860 °C, compression force 0.5 kN and time t = 40 min.

Firstly, samples having grinded contact areas (Ti Grade 2 - Ra = 0,48 µm; 316L - Ra = 0,39 µm) and subsequently samples with polished contact areas (Ti Grade 2 - Ra = 0,27 µm; 316L - Ra = 0,14 µm) were bonded in the vacuum of $3 \cdot 10^{-1}$ Pa. Afterwards, the same tests were performed by using either Ar shielding atmosphere of purity 99.9993% or mixture of gases Ar/He at ratio 50/50 %. Tab. 6 summarizes results from the static tensile tests performed on the diffusion joints prepared as mentioned before.

Tab. 6 Results of the static tensile tests for optimized diffusion welded joints Ti Gr. 2/316L

Surface preparation	*Atm. [-]*	*Temp. [°C]*	*Force [kN]*	*Time [min]*	*Yield str. $R_{p0.2}$ [MPa]*	*Fracture stress [MPa]*	*Fracture elongation [%]*
grinding	*vacuum*	*860*	*0.5*	*40*	*-*	*281*	*-*
polishing	*vacuum*	*860*	*0.5*	*40*	*-*	*287*	*-*
grinding	*Ar*	*860*	*0.5*	*40*	*328*	*336*	*0.2*
polishing	*Ar*	*860*	*0.5*	*40*	*326*	*341*	*0.3*
grinding	*Ar/He*	*860*	*0.5*	*40*	*-*	*321*	*-*
polishing	*Ar/He*	*860*	*0.5*	*40*	*326*	*329*	*0.1*

The diffusion welded heterogenous joint Ti grade 2/AISI 316L with the polished contact areas and performed in the Ar shielding atmosphere as well as the tensile sample machined from this workpiece are shown in Fig. 6.

Fig. 6 *Diffusion welded sample, T = 860°C; F = 0.5kN; t = 40min performed in the argon shielding gas and testing sample machined from this workpiece*

The microhardness HV 0.02 distribution in the sample mentioned above can be seen in Fig. 7. Each point represents the mean value from 5 measurements, while the maximal standard deviation was 9 HV. It can be seen that a significant increase in hardness values is observed in the Ti side in about 50µm wide zone from the interface where a hardness of 450 HV0.002 is reached while the base material has a hardness of 250 HV0.02.

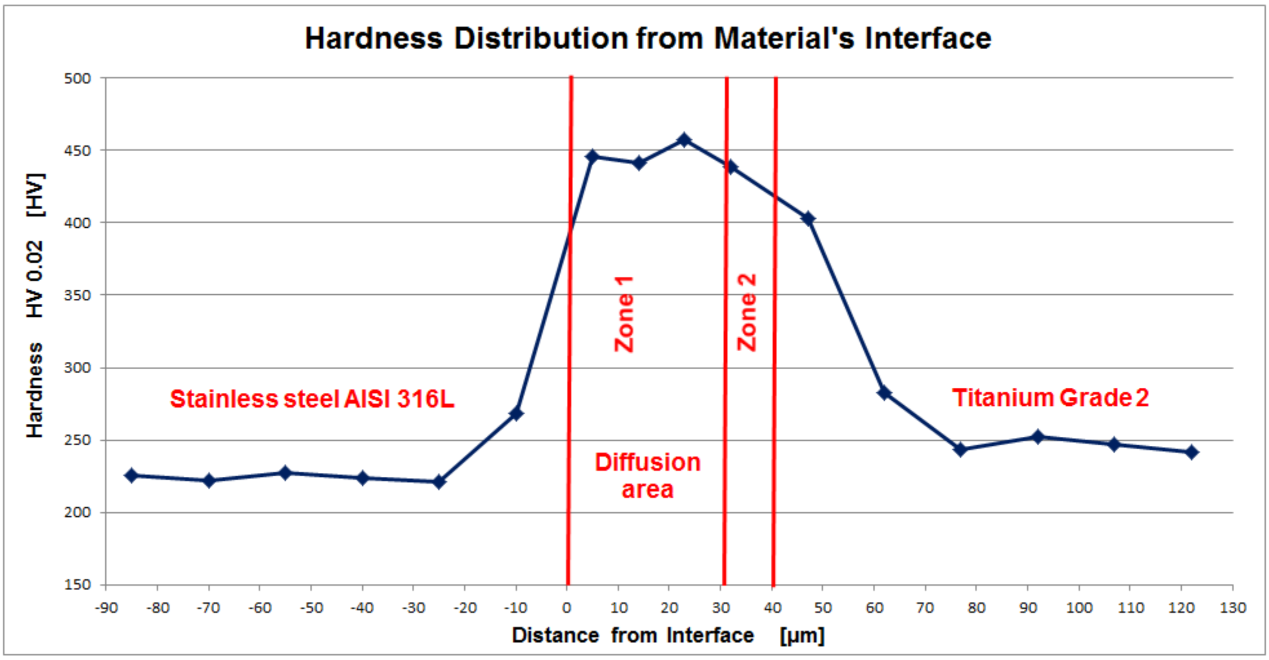

Fig. 7 *Microhardness HV 0.02 distribution over the diffusion welded joint (T = 860°C; F = 0.5kN; t = 40min performed in the argon shielding gas)*

5 Conclusion

Diffusion welding represents a suitable alternative to the fusion welding methods, especially at creating heterogeneous joints. The diffusion rate increases with increasing temperature. Nevertheless, it is also connected to the magnitude of compressive force. The diffusion rate continuously decreases with increasing holding time on the holding temperature until the time when concentrated potentials are equally represented in the joint area is achieved.

The aim of this article was to show how to increase the total strength of the heterogeneous joint from materials Ti Grade 2 and steel AISI 316L by the proper choice of processing and technological parameters. By such processing parameters modification a maximal joint strength of 254 MPa was achieved. Further technological parameters modification led to a joint strength of 341 MPa. However, the total joint strength is still quite low and higher than the yield strength just by few MPa. Further microscopy investigations are required to characterize more precisely the interface (eventual presence of intermetallic compounds) and identify the causes of this low strength.

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