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## THE EFFECT OF HEAT TREATMENT ON THE PROPERTIES OF ZIRCONIUM – CARBON STEEL BIMETAL PRODUCED BY EXPLOSION WELDING

### WPLYW OBRÓBKİ CIEPLNEJ NA WŁASNOŚCI ORAZ STRUKTURĘ BIMETALU CYRKON/(STAL WĘGLOWA) WYKONANEGO TECHNOLOGIĄ ZGRZEWANIA WYBUCHOWEGO

This paper assesses the effect of the heat treatment on the mechanical properties and the structure of the bond zone in zirconium (Zr 700) – steel (P355NL2) bimetal. The investigations were performed for welds with varied characteristics of the bond zone. Experimental results were presented concerning the strength tests (the values of peeling –  $R_o$ , shear –  $R_s$  and tensile  $R_m$  strength), measurement of hardness, and structural analyses carried out in micro- and macro- scale.

Microhardness measurements shows that the increase in the detonation velocity during explosion welding causes the increased share of the melted area in the bond, the increase of the deformed area and consequently hardening at the interface. Applying heat treatment lowers the hardening in the bond zone, which is more pronounced in the base material, i.e. steel. This effect results from the full recrystallization of the grains in the plastic deformation area. The selection of temperature is paramount as the higher the temperature, the lower tensile resistance of the bond zone. Applying the same settings for materials with different characteristics showed the treatment did not considerably influence the shear strength, but significantly affects tensile and peeling strength.

*Keywords:* explosive welding, Zr(carbon steel) clad, strain hardening, melted zone, intermetallic phases

W pracy poddano ocenie wpływ obróbki cieplnej na własności mechaniczne oraz strukturę strefy połączenia bimetalu cyrkon (Zr 700) – stal węglowa (P355NL2). Badania prowadzono dla bimetalu wytworzonych przy zróżnicowanej prędkości wybuchu determinującej zróżnicowaną charakterystykę strefy połączenia. Przedstawiono wyniki badań wytrzymałościowych, pomiaru twardości oraz badań strukturalnych strefy złącza.

Pomiary mikrotwardości pokazują, że wzrost prędkości detonacji podczas procesu spajania powoduje zwiększony udział obszarów przetopionych w złączu, wzrost umocnienia oraz rozrost strefy odkształcenia plastycznego w pobliżu granicy połączenia. Zastosowanie obróbki cieplnej redukuje wielkość umocnienia w strefie połączenia. Zjawisko to, szczególnie silnie obserwowane w materiale podstawowym (stali), jest efektem pełnej rekrytalizacji ziaren w strefie odkształcenia plastycznego łączonych metali. Podczas wyżarzania próbek o akceptowalnym udziale stref przetopień w złączu wraz ze wzrostem temperatury wyżarzania zmniejsza się wytrzymałość plateru na rozciąganie i odrywanie. Zmiany parametrów wyżarzania nie mają jednak większego wpływu na wytrzymałość na ścinanie.

## 1. Introduction

The construction of a long life chemical and process apparatus that would be highly resistant to corrosion but also durable, and characterized by a relatively low manufacturing cost, requires the application of reactive metals and their alloys deposited on other structural materials. Zirconium and its alloys are one of those metals, currently used in chemical industry and nuclear power industry. The application of zirconium is, however, limited in modern industry due to its high cost. One feasible solution is using multilayer structures, wherein the base material that meets the construction requirements is a proper grade of steel, whereas zirconium used as

the cladding material acts as a material with special design (e.g. corrosion resistance, radiation resistance, etc.). Joining base and cladding material poses a problem in large-sized elements of reactors and apparatuses, like walls or perforated bottoms. In cases like this explosion welding technology may be applied.

Technological aspects of explosion welding have been described in works by many authors [1-6]. Suffice it to say then that a durable joint of requisite mechanical properties is obtained as a result of the collision of a flyer plate and a base plate with appropriate speed. The flyer plate is propelled by the impact of gasses – detonation products from the explosive charge. The collision energy is determined, among others, by

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the stand-off distance and detonation velocity (explosion energy). However, the collision creates additional technological issues resulting from the increase of stresses and large strain deformation in the bond zone. This phenomenon causes a substantial strain hardening of regions adjacent to the bond interface. Moreover, exceeding the process settings, especially the detonation velocity, promotes the formation of brittle and hard fusion zones in the joint which tend to crack. This process was vastly described in earlier works, e.g. [5-6, 10-11]. Hardening of the bond zone significantly affects the shaping process of explosion welded materials which consist of straightening, bending, punching, welding etc. Therefore, the thermal treatment is an important issue in fabricating clads. The point, or even the necessity, of performing proper heat treatment procedures is documented in works [7-10]. The most important issue in the case of heat treatment used for layered materials with highly diversified properties is the selection of process parameters. It depends mostly on the chemical composition of welded materials and their dimensions because of their capability to deform during heating and cooling cycles. On the one hand, we deal with the base material usually made of structural carbon or low-alloy steels, requiring sufficiently high temperature of annealing and long heating times. On the other hand, a permanently deposited cladding material, often originating from reactive metals, is characterized by a high sensitivity to the high temperature oxidation or the deterioration of its properties due to the dissolution of atmospheric gasses such as hydrogen and nitrogen.

The present paper undertakes to analyze the effect of heat treatment on the hardening changes in explosively welded bimetals of zirconium (flyer/moving plate) and carbon steel (base plate). The paper analyses three cases of bimetal clads, obtained as a result of the application of varied detonation velocities, which allowed receiving a bimetal of a diversified hardness and increasing share of the fusion in the bond. The share of the fusion zone in the analyzed cases was representative for three cases: minimal, acceptable and unacceptable from the point of view of the quality of the weld. The effect of heat treatment on the mechanical properties and the structural changes of the bond zone of zirconium/carbon steel bimetal was analyzed.

**2. Material and research methodology**

The material for this study was produced by means explosion welding in the form of testing plates with dimensions 300x500 mm<sup>2</sup>. The base plate was a sheet made of grade P355NJ2 carbon steel designed for operation in elevated temperatures, whereas the flyer plate was a technical grade Zr700 zirconium. The chemical composition according to the manufacturer's certificate was given in Table 1.

Blasting works were carried out at a blasting site by „EXPLOMET” (EXPLOMET High-Energy Techniques Works), Opole. The welding process was performed with parallel set-up at variable detonation velocity ( $v_1 < v_2 < v_3$ ) for respective clads, and a constant stand-off distance between the plates ( $h = 6 \text{ mm}$ ). The designations of plates with the values of settings for respective clads were presented in Table 2.

TABLE 1

The chemical composition of joined metal sheets, as per the supplier's certificate

Basic material	Chemical composition [wt%]						
	C	Mn	FeCr	H	O	Zr+Hf	N
Zr 700	<0.002	–	0.05	<0.0003	0.05	>99.2	<0.002
P 355NL2	C	Mn	Si	P	S	Cr	
	0.17	1.13	0.35	0.008	0.011	0.15	
	Cu	Ni	Mo	Al	Nb	Fe	
	0.17	0.285	0.035	0.045	0.019	rest	

TABLE 2

Designation of the produced plates and the relations between process parameters

Designation plate	Detonation velocity $v_D$ [m/s]	Stand off distance $h$ [mm]
1.0 $v_D$	2200	6
1.3 $v_D$	2800	
1.6 $v_D$	3500	

The detonation velocity ( $v_D$ ) during explosive welding tests was monitored in all cases by means of a system of three optical fibers with an 'Explomet' measuring instrument. Varied detonation velocity was obtained as a result of the application explosive charges from the ammonite group with a diverse composition (varied explosion energy).

After cladding, the entire surfaces of all materials underwent ultrasonic tests in order to determine the continuity of the bond. The material for further analyses (in as-welded state and prior to heat treatment) was sampled from regions without visible traces of discontinuity, at a section perpendicular to sheet surface and parallel to the detonation front motion. Heat treatment (annealing) was performed in order to remove, or at least minimize, undesired effects of hardening in the bond zone associated with explosion welding. Heating was performed at 600°C during 90 minutes, based on the literature data, e.g. [8-10]. The scheme of respective operations of heat treatment was presented in Fig. 1. The samples were heated together with the furnace up to 300°C for 35 minutes. Then the material was heated up to 600°C at 100°C/h. After reaching the temperature of treatment, the material was annealed

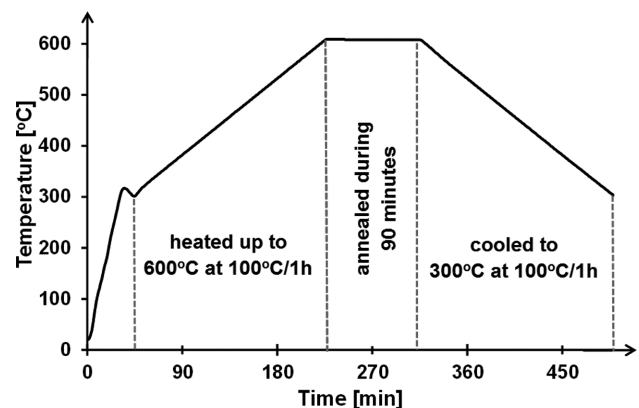


Fig. 1. Heat treatments of (carbon steel)/Zr clad

during 90 minutes and afterwards cooled at 100°C/1h down to 300°C. The final stage was cooling the sample in air.

Tests of mechanical properties were carried out for as-welded samples and samples after heat treatment according to EN13445-2 standard. Shearing, peeling and tensile tests were carried out as required for explosion welded clads for the purpose of determining the values of  $R_S$ ,  $R_O$  and  $R_m$ , respectively. Mechanical properties were tested on fatigue machines WPM ZD20 and Instron 6025.

The evaluation of the hardening of the bimetal was carried out based on the systematic measurements (in micro-scale) by Vickers method, using LECO MHT Series 200 microhardness tester. The tests were performed for the respective layers adjacent to the joint and in the longitudinal section of the bimetal. The measurements were recorded in as-welded samples along the line perpendicular to the bond interface (3 series) in places without melted zone. After recording hardness measurements, the surface of specimens was polished and subsequently thermally treated and measurements were repeated in the same spots. The measurements were done in accordance to ISO 6507-3:1996 (metal hardness measurement by Vickers' method below HV 0.2) under 50G load.

Material for tests was sampled from as-welded clads and those which were thermally treated. The metallographic specimens were made on the section perpendicular to the surface of the sheet and parallel to the direction of detonation front movement through mechanical grinding and polishing (abrasive papers and diamond pastes with descending grade), and then polishing and etching with LectroPol 5 polisher using Struers TM A3 electrolyte.

Initial metallographic analyses were conducted on a non-treated material by means of an optical microscope Olympus IX 70, and an image analyzer LECO IA 32. The aim of this analysis was determining the characteristic of the joint and the quantitative volume of the fusion zone in respective specimens. The above mentioned analysis was performed based on the measurement of basic parameters of the bond interface - the length of the bond line  $L$ , height ( $H$ ) and wave length ( $n$ ) as well as the area of the fusion surface ( $P$ ) (Fig. 2).

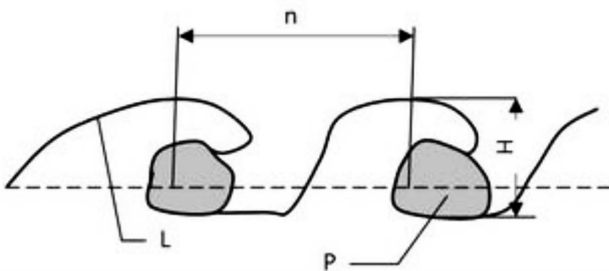


Fig. 2. Basic bond parameters:  $H$  – wave height,  $L$  – length of the bond line,  $n$  – wave length,  $P$  – summary area of 'fusion' surface

The measurements for each sample were taken in random regions on the average measurement length of over 10 000  $\mu\text{m}$ . Based on average values of the above parameters Equation 1 was used to determine the fusion depth equivalent coefficient ( $RGP$ ), describing the volume of the fusion zone in the bond:

$$RGP = \frac{S}{L} \quad [\mu\text{m}] \quad (1)$$

where:  $S$  – the sum of  $P_i$  surface of fusion regions in  $\mu\text{m}^2$   
 $L$  – the length of the bond line  $\mu\text{m}$ .

### 3. Results and discussion

#### 3.1. Structural observations

The materials used were characterized by a fully recrystallized state. The microstructure of the zirconium plate (flyer plate) consists of  $\alpha$  phase grains with dimensions between 70 to 170  $\mu\text{m}$ . The microstructure of carbon steel (base plate) was characterized by a ferrite (sized 10-20  $\mu\text{m}$  on average) and pearlite (4-20  $\mu\text{m}$ ) grains. Applying various detonation velocities allowed obtaining bimetals with different bond characteristic, however, in all instances the joint interface was of wavy nature (Fig. 3a-c). It was observed that increasing the detonation velocity from  $1.0v_D$  to  $1.3v_D$  and even more pronouncedly to  $1.6v_D$  promoted the formation of melted inclusions in the bond zone (Fig. 3b and c).

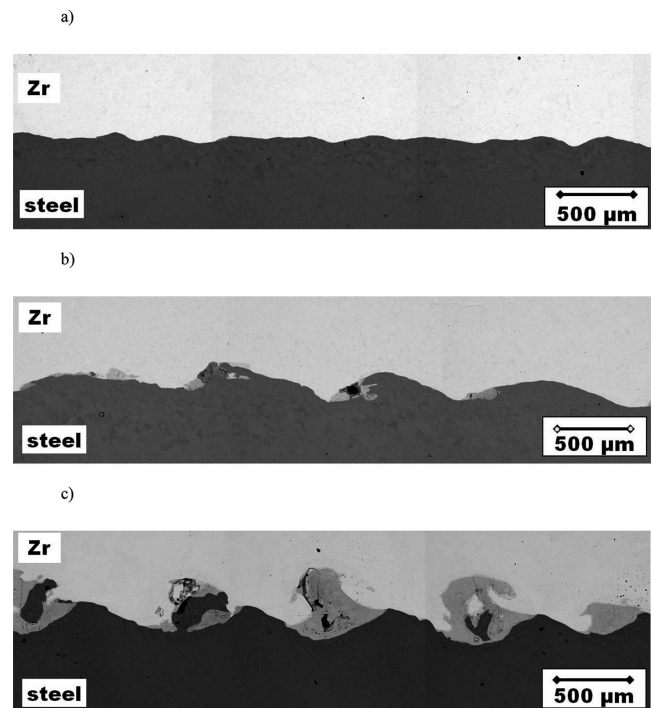


Fig. 3. Characteristics of near-the-interface zone of Zr700/P355NL2 clad (in the 'as-bonded state') for detonation velocities of: (a)  $1.0v_D$ , (b)  $1.3v_D$  and (c)  $1.6v_D$

Structural observations of the bond zone in as-welded clad showed a strongly hardened layers and material displacement towards the direction of the detonation front. Strongly strain hardened structure was particularly clearly seen in the region of ferrite grains (Figs. 4a and 4b). Moreover, in the regions adjacent to the bond interface, in steel and zirconium, thin layers of very fine grains were identified. The width of the strongly deformed structure in steel was three times smaller than the thickness of the analogous region in zirconium. Heat treatment caused considerable changes in the structure of base and cladding material. Equiaxed grains can be identified near the joint interface in the analyzed area presented in Fig. 4c which confirms the full recrystallization of the strain hardened structure.

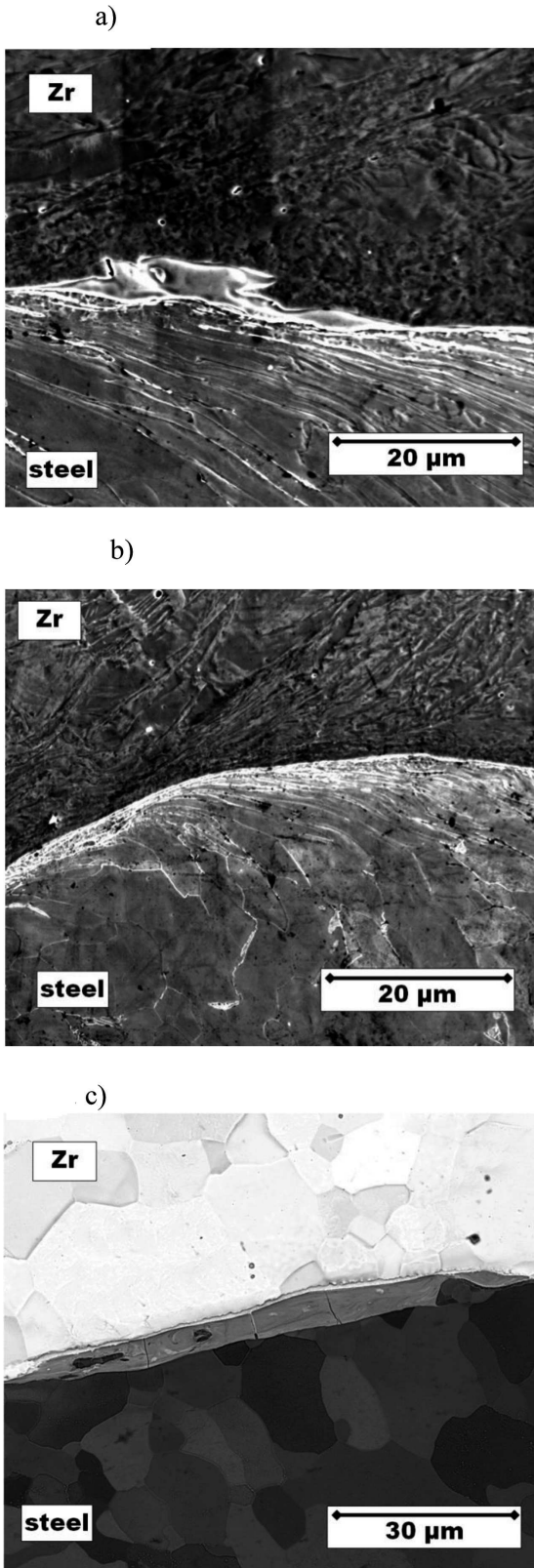


Fig. 4. Microstructure observed at optical microscopy scale showing strong deformation of near-the-interface layers of bonded sheets: (a) bottom and (b) crest of the wave, (c) microstructure of the clad after heat treatment

Detailed analyses based on the measurement of the length of the bond line  $L$ , height ( $H$ ) and wave length ( $n$ ) as well as the area of the fusion surface ( $P$ ) are presented in Table 3 and Fig. 5.

TABLE 3

Parameters describing wave shape and the quantity of the melted zone

Designation plate	Length of the bond line $L$ [ $\mu\text{m}$ ]	Wave height $H$ [ $\mu\text{m}$ ]	Wave length $n$ [ $\mu\text{m}$ ]	Melt surface area $P$ [ $\mu\text{m}^2$ ]	Melt depth equivalent RGP [ $\mu\text{m}$ ]
$1.0v_D$	12 685	58	454	5 596	0.5
$1.3v_D$	13 648	148	1014	137 484	10.0
$1.6v_D$	16 823	240	940	912 943	54.0

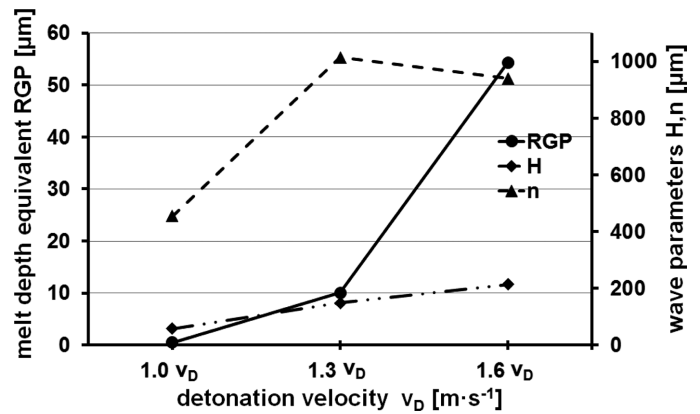


Fig. 5. Parameters of the interface

Applying the detonation velocity ( $1.0v_D$ ) caused the formation of the bond interface characterized by a regular wave with the smallest height, among the studied cases, and the wave length of  $454 \mu\text{m}$ . At  $1.0v_D$  only a small amount of the fusion zone was observed ( $RGP \sim 0.5 \mu\text{m}$ ). The rise in the detonation velocity by 30% and 60% caused a marked increase of the joint parameters. At the detonation velocity of  $1.3v_D$  the wave length increased twofold (up to  $1014 \mu\text{m}$ ) whereas the height 2.5 – fold (up to  $148 \mu\text{m}$ ). For this set of technological parameters of welding the volume of the fusion layer in the bond increased up to  $10 \mu\text{m}$ , which is considered a threshold value from the point of view of the quality of the bond ('good bond'). Increasing the detonation velocity up to  $1.6v_D$  did not impact the wave length ( $n$ ) whose value was  $940 \mu\text{m}$ , however, a fourfold rise in the height of wave ( $240 \mu\text{m}$ ) was noticed compared to the wave height generated using the smallest detonation velocity. Using the greatest detonation speed ( $1.6v_D$ ) caused the RGP coefficient in the interface area to grow to an unacceptable level of  $54 \mu\text{m}$ . Optical microscopy observations of the interface zone of bimetals after heat treatment did not demonstrate any changes in the bond interface parameters or the volume of the fusion layer.

### 3.2. Strength tests

The strength tests performed for materials in 'as-welded' state allowed evaluating the quality of the received joint. For all three cases, tensile tests, shearing tests and peeling tests were performed in order to evaluate of  $R_m$ ,  $R_s$  and  $R_o$ , respectively; the average values of these parameters are shown in Table 4 and Fig. 6. After the analysis of the shearing and peeling tests it can be claimed that the increase of the detona-

tion velocity and the volume of the fusion layer in the bond at the same time caused a deterioration of the strength properties. This effect is less pronounced in the shearing test which gave the result of 351 MPa for  $1.0v_D$  specimen and 281 MPa for  $1.6v_D$  specimen. It points to a 20% loss of strength. For the peeling and tensile tests the highest values of strength were also recorded for the samples with the smallest detonation velocity of  $1.0v_D$ ,  $R_o = 499$ MPa and  $R_m = 544$ MPa, respectively. In the case of the highest detonation velocity  $1.6v_D$  both trials gave similar values around 180 MPa, which is a 40% loss of strength against peeling and 35% against shearing. The specimen with the acceptable threshold volume of the melt near the interface ( $1.3v_D$ ) displayed high, comparable to  $1.0v_D$  specimen, values of tensile strength and low resistance to stripping comparable to the specimen made at the highest detonation velocity ( $1.6v_D$ ).

TABLE 4  
Metallic radii of rare earth metals and magnesium [12]

plate	without heat treatment			after heat treatment		
	$R_s$ [MPa]	$R_o$ [MPa]	$R_m$ [MPa]	$R_s$ [MPa]	$R_o$ [MPa]	$R_m$ [MPa]
$1.0v_D$	351	449	544	351	390	396
$1.3v_D$	321	144	562	281	53	387
$1.6v_D$	281	159	180	293	53	159

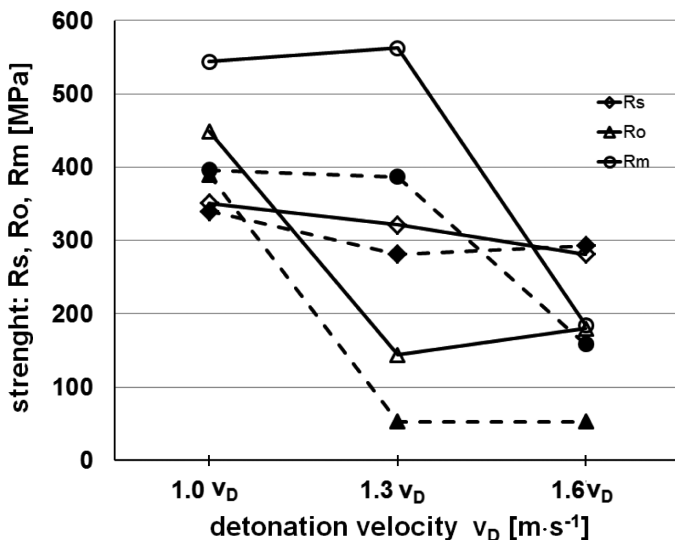


Fig. 6. Mechanical properties (shear strength, peel-off strength, ultimate strength) of the clad in the 'as-bonded' state (continuous line) and after heat treatment (dotted line)

In the case of the peeling test performed on the specimens cut out of clad formed at  $1.3v_D$  and  $1.6v_D$  it was observed that the joint metals were peeled at the joint. Whereas in the case of specimen fabricated at  $1.0v_D$ , i.e. without the fusion zone – it happened at the cladding material, i.e. Zr. Analyzing the shearing results it was determined that specimens with a low but acceptable RGP coefficient ( $0.5 \mu\text{m}$  and  $10 \mu\text{m}$ ) were sheared in the cladding material, whereas for the specimen with the value of RGP equal to  $54 \mu\text{m}$ , the shearing occurred in the bond.

The strength test results for bimetals after heat treatment show that this process strongly affects values of tensile and peeling strength, whereas it only slightly influences shearing test results. The graph presented in Fig. 6 (dashed line) of the strength properties changes for specimens treated thermally is comparable to the progression of specimens prior treatment. In the case of tensile strength ( $R_m$ ) the heat treatment caused about 30% decrease in the strength of specimens with the lowest ( $0.5 \mu\text{m}$ ) and the highest acceptable ( $10 \mu\text{m}$ ) volume of the fusion layer. In the case of  $1.6v_D$  specimen the tensile strength ( $R_m$ ) after heat treatment dropped by 10% compared with the as-welded specimen. A reverse trend was observed in the peeling test ( $R_o$ ). The strength of the specimen where the fusion layer occurred in the bond zone ( $1.3v_D$ ,  $1.6v_D$ ) decreased by about 30%. Whereas in the specimen free of melted zones the decrease reached 15% in comparison to bimetals prior to heat treatment. In the case of shearing ( $R_s$ ) a slight strength decrease ( $\sim 10\%$ ) was recorded in the region. In all the analyzed cases, thermally treated specimens were destructed at the joint.

### 3.3. Microhardness measurements

The analysis of hardening was performed based on systematic measurements of microhardness under the load of 50G. The measurement was taken for as-welded and thermally treated materials throughout its length and in direct vicinity of the bond zone (up to 0,5 mm from the joint interface). The analysis of the hardness distribution across the entire section of the bimetal prior to heat treatment demonstrates that the increase in the detonation velocity affects hardening especially in the base material. In either of the analyzed materials an increase of hardness was noticed in comparison to hardness before explosion welding. A detailed analysis of hardening across the entire section of a clad was presented in paper [12]. The present study focuses on the analysis of the effect of thermal treatment on the most strongly hardened regions of the bond zone, i.e. in the distance of 0.5 mm from the bond interface.

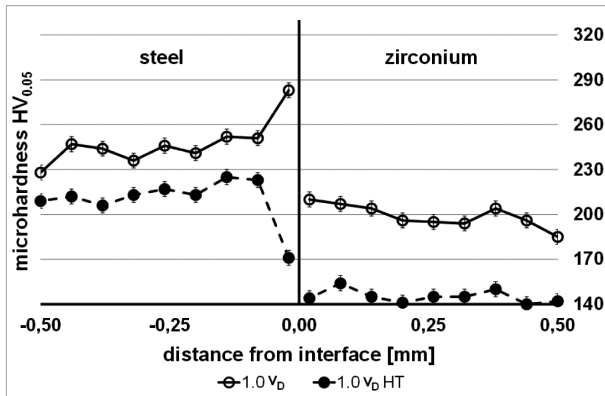
It was observed that the highest hardness for the base material in all cases occurred at about  $20 \mu\text{m}$  from the joint interface (Fig. 7a-c). For the plate fabricated with the highest detonation velocity ( $1.6v_D$ ) a hardness of  $346 \text{HV}_{0.05}$  was recorded and it is about 16% higher than the hardness of the plate made with  $1.3v_D$ . At the detonation velocity of  $1.0v_D$ , microhardness reached  $265 \text{HV}_{0.05}$ , which means that it decreased by 13%.

For cladding material, the hardness was about  $230 \text{HV}_{0.05}$  in the direct vicinity of the joint interface regardless of the detonation velocity. As the distance from the bond interface increases, the hardening decreases and for the clad without the fusion zone it stabilizes at the distance of around  $80 \mu\text{m}$  (Fig. 7a) and for  $1.3v_D$  at  $140 \mu\text{m}$  (Fig. 7b). In the case of the highest volume of fusion in the bond it did not reach a steady level across the whole test area (Fig. 7c).

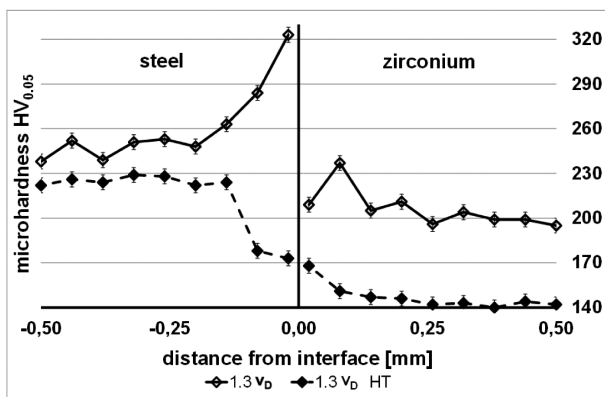
After the analysis of hardening changes resulting from the heat treatment, it can be claimed that the greatest decrease of hardness was observed directly near the bond interface. The test results show that for all the analyzed cases the hardness is similar to the hardness of steel prior to cladding

( $\sim 175 \text{ HV}_{0.05}$ ). A decrease of hardness ( $\sim 35\%$ ) was noticed for  $1.0v_D$  specimen in comparison to hardness in as-welded state (Fig. 7a), whereas the stronger drop in microhardness reached 45% (Fig. 7b) for the detonation velocity of  $1.3v_D$ . The largest 'softening' of material occurred at the distance of about  $20 \mu\text{m}$  from the bond interface in the bimetal produced at the highest detonation velocity ( $1.6v_D$ ), and it reached 50% as compared to the hardness for as-welded state at the same distance (Fig. 7c).

a)



b)



c)

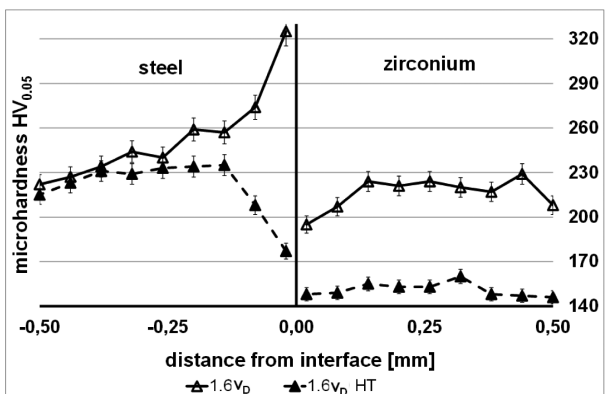


Fig. 7. Microhardness changes near-the-interface for different detonation velocities: a)  $1.0v_D$ , b)  $1.3v_D$  and c)  $1.6v_D$ . Continuous line – clad in the as-bonded state, dotted line – clad after heat treatment. Measurements along 3 lines across the interface. Loading of 50G was applied

Figures 7a-c show that after annealing the size of the softened area in the base material reflects the values of hardening in the base plates and amounts to  $80 \mu\text{m}$  and  $140 \mu\text{m}$  for  $1.0v_D$  and  $1.3v_D$  specimens, respectively. The hardening stabilizes at a farther distance and is around 10% smaller than the hardening directly after welding. For the  $1.6v_D$  specimen after annealing, the hardening stabilizes at the level of  $230 \text{ HV}_{0.05}$  at the distance of  $140 \mu\text{m}$ . In the case of zirconium a softening (reduction of hardening) of about 35-45% occurred in comparison to the as-welded state, practically on the whole analyzed area (up to  $0.5 \text{ mm}$  from the bond interface). The distribution of hardness throughout the entire section of the flyer plate was similar for all cases and fluctuated in the range of  $140\text{-}150 \text{ HV}_{0.05}$ .

#### 4. Summary

The present study analyzes the effect of heat treatment on mechanical properties and structural changes in the bond zone of bimetals made of sheets of steel P355NL2 (base plate) and zirconium (flyer plate) joined by explosion welding. Bimetal were fabricated with variable detonation velocity which determined strongly diversified characteristics of the bond.

- The microscopic analyses showing that the increase of the detonation velocity increases the height and length of the wave and promotes the growth of melted zones in the bond. This phenomenon considerably influences mechanical properties of bimetals. The analysis of strength test results, i.e. tensile test, shearing test and peeling test allows claiming that a small volume of fusion region in the bond considerably improves the strength of the joint, whereas the increase of the *RGP* coefficient above the acceptable value, i.e.  $RGP \gg 10 \mu\text{m}$  causes a drastic decline of strength properties.
- Applying heat treatment causes the decrease of the mechanical properties of clad. However, in the case of bimetals with a low ratio of the melted zone good mechanical properties are maintained. In the case of clads fabricated using high detonation velocity the increase of strengthening (and the depth of its influence) in the interface area is observed, which is confirmed by the microhardness measurements. The effects are particularly well-visible in the base material, where along with the detonation velocity increase the strengthening also increase at the interface area. The analysis of microhardness in Zr700 plates shows the occurrence of hardening in the bond zone, however, regardless of the detonation velocity the hardening remains at a similar level.
- Applying thermal treatment procedures significantly affects the hardening values. The analysis of results of microhardness measured near the interface for the base material confirms a high reduction of hardening down to similar values for all the considered cases. This phenomenon is associated with the reduction of stresses generated by very high pressures and high temperature gradients occurring at the collision point during explosion welding.
- The reduction of the strength properties after heat treatment is undeniably associated with structural changes near the interface.

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