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PHASE ANALYSIS OF EXPLOSIVE WELDED Ti-Cr/Ni STEEL IN AS-RECEIVED STATE AND AFTER HEAT TREATMENT USING SYNCHROTRON

ANALIZA FAZOWA POŁĄCZENIA Ti-Cr/STALI Ni WYTWORZONEGO METODĄ ZGRZEWANIA WYBUCHOWEGO W STANIE WYJŚCIOWYM ORAZ PO OBRÓBCE TERMICZNEJ Z WYKORZYSTANIEM PROMIENIOWANIA SYNCHROTONOWEGO

Surface coatings protection is one of the most important processes ensuring efficient and economic use of basic materials, mostly of lower-quality. At interface of clad and basic material intermetallic phases are formed, representing quite different matrix with dissimilar properties unlike the welded materials. One type of surface coating is explosive bonding which belongs to group of pressure welding. The work is focused on interface shape line, inhomogeneities in vicinity of the wave joint both in basic material and in vicinity of weld line of the Ti and Cr/Ni stainless steel (SS) matrix. Investigated weld was both in as-received state and after heat treatment carried out at 600°C/90 minutes/air. Presented phases have been identified using X-ray diffraction performed by synchrotron. The Tiα, Fe-fcc, Fe-bcc and intermetallic phases Fe_2Ti were detected at interface area.

Keywords: Ti-Cr/Ni, explosive bonding, interface area, heat treatment, hardness, phase analysis

Jednym z najważniejszych procesów zapewniających skuteczne i ekonomiczne uwarunkowane stosowanie materiałów konstrukcyjnych jest pokrywaniepowierzchni warstwą ochronną. W strefie połączenia następuje formowanie się faz międzymetalicznych charakteryzujących się całkowicie odmiennymi od materiałów łączonych własnościami. Jednym ze sposobów pokrycia powierzchni materiałów jest zgrzewanie wybuchowe, które należy do metod ciśnieniowych. Praca została zogniskowana na charakterystyce kształtu linii połączenia, niejednorodnościach w okolicy rozchodzenia się fali wybuchu obydwu materiałów, jak i w okolicach linii samego połączenia Ti oraz Cr/Ni stali niskowęglowej (SS). Badania fazy przetopionej w obydwu materiałach prowadzono po przeprowadzeniu obróbki termicznej, którą przeprowadzono w temperaturze 600°C przez 90 minut, bez atmosfery ochronnej. Pojawiające się fazy zostały przebadane z wykorzystaniem dyfrakcji promieni rentgenowskich w synchrotronie. Ujawniono fazy Tiα, Fe-fcc, Fe-bcc oraz fazę międzymetaliczną Fe_2Ti w obszarze połączenia.

1. Introduction

The interface surface layers of the bonded metals contain non-metallic films such as oxide films that are detrimental for any metallurgical bond, are swept away by plasma jet during explosion. The joint is wave in nature and is usually stronger than the weaker parent metal Fig. 1.

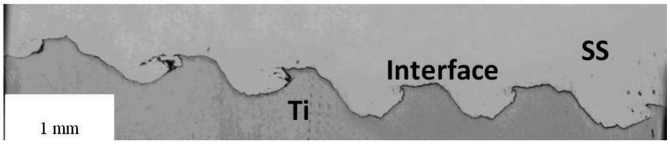

Fig. 1. Typical wavy bonding zone Titanium with SS steel

As formerly Cowan and Holtzman proposed [1], the formation of bonded zone waves in explosion clading is analogous to fluid flowing around an obstacle in which a regular formation of eddies was observed. Similar view expressed Klein [2], Burkhardt et al. [3] observed interface transition from a smooth to a wavy character in dependence on the critical collision velocity in clading angle. Bergmann [4] and Bahrani et al. [5] presented pictorial description for an asymmetric system in more details. As Fig. 2 shows a model of wave mechanism where : a) cutting and flow shaping, b) forming areas of turbulence and recess, c) ending process.

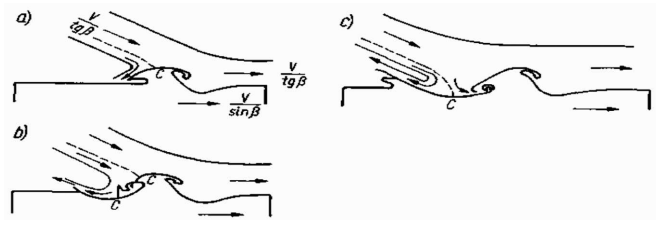

Fig. 2. Model of wave mechanism: a) cutting and flow shaping, b) forming areas of turbulence and recess, c) ending process

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Numerous authors have been orientated on explosion and metallographic parameters in their papers [6, 7]. Some researchers [8-10] have tried to analyse interphase and its close region by use of SEM (EDAX) or conventional X-ray. Those experimental techniques are not too accurate in this case and analysis using synchrotron enables a more precise focus on bonding line. Principle of synchrotron could be shortly described as it follows.

Bunches of charged particles (electrons or positrons) circulate in a storage ring. The ring is designed with straight section, where an insertion device forces the particles to execute small oscillations which produce intense beams of radiation. The radiation then passes through a number of optical elements, such as a monochromator, focusing device, etc., so that a beam of radiation with the desired properties is delivered to the sample. Fig. 3 schematically shows key components of a typical experimental beamline at a third generation source.

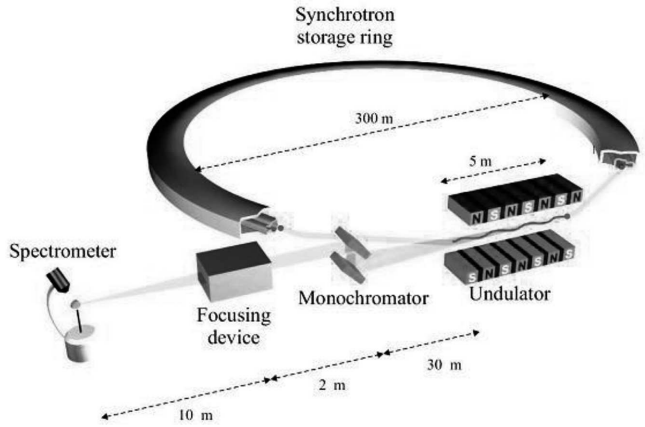

Fig. 3. Scheme of a typical X-ray beamline of the third generation of X-ray source

The electrons do not follow a purely circular orbit in the storage ring, but traverse through straight sections where lattices of magnets, so-called wiggler or undulator insertion devices, force them to execute small amplitude oscillation. At each oscillation X-rays are emitted and, if the amplitude of the oscillations is small, then different contributions from the passage of a single electron add coherently, and a very intense beam of X-rays results.

Undulators replaced other device called wigglers that had the same working procedure but were not able to produce so small angle of the particles oscillation, which is directly responsible for the angular divergence and following interference in the beam. Wigglers are still used for the applications in the lower range of intensity. As for the bunch of particles in the storage ring, the device used to chase the trajectory and bend the path, is called bending magnet. It is the simplest device able to produce X-rays by changing the path of a particle. The second key component is the monochromator, as in many applications it is required to work at a particular average wavelength. It may also be desirable to choose the wavelength band width. Monochromators made from perfect crystals through to multi layers allows use different variations of working parameters. To work with small specimen it may be desirable to focus the monochromatic beam down to as small size as achieve able. This is accomplished by devices such as X-ray mirrors and refractive Fresnel lenses [11]. For those reasons this experimental techniques (synchrotron) are accurate in this case to analysis phases in bonding line area.

2. Experiments and solution technique

The experimental part of this work is focused on the study of material phase composition both in as received state and after followed heat treatment (HT). Applied the HT was annealing at 600°C for 1.5 h in a protective atmosphere followed by air cooling. In both samples phases were studied close to the interface.

The studied material is Cr/Ni SS cladded with titanium of commercial purity. Titanium showed followed chemical composition (wt. %): 0.01C, 0.05Fe, 0.05O, 0.005N, 0.006H and the Cr/Ni SS had as it follows (in wt. %): 0.04C, 0.45Si, 1.96Mn, 18.42Cr, 9.74Ni, 0.065P and 0.011S. Samples of Ti and Cr/Ni SS sandwich were machined by mechanical cutting to avoid any change in microstructure. The thickness of base material Cr/Ni SS was 110 mm and the Ti layer corresponded to 6 mm (in sequence).

For phase analyse synchrotron was used. The samples of dimension 40 x 20 x 10 mm were cut from the bulk. Subsequently, individual pieces were embedded to poly-fast, then grinded (P300, P600, P800, P1200, P2500), polished in PHOENIX 4000, Buehler (grinding paste of 0.006 mm graininess with distilled water) and finally etched in KROLL (100ml H_2O + 6ml HNO_3 + 3ml HF) [12] for about 3 second.

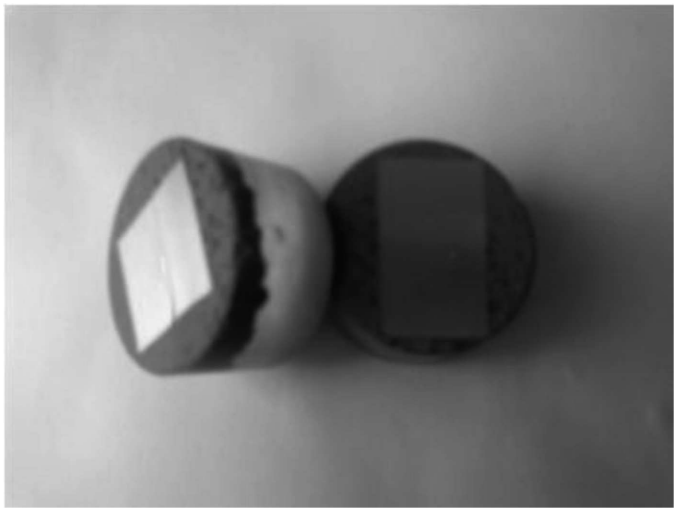

Fig. 4. Samples for phase analysis

The first XRD measurements were carried out using the BW5 experimental station [13] located at the DORIS III positron storage ring (energy 4.45 GeV, current 140-100 mA) at HASYLAB/DESY, Hamburg, Germany. The energy of the incident beam was 100 keV, lambda corresponded to 0.124 Å. The specimen was scanned shot-by-shot along a path of total length of 40 mm with step width of 1 mm. During each step, the sample was illuminated by highly intensive hard X-rays for 2 seconds. The resulting 2D XRD patterns were recorded using a Perkin Elmer 1621 detector. The collected data were then integrated into 2 Theta space by using the FIT2D software [14]. The sample-detector distance, detector orthogonal-

ity with respect to the incoming radiation, as well as precise radiation energy was determined by fitting a standard reference LaB6 sample [15].

To determine the structure orientation and possible phase transformations in the interface a hard X-ray micro-diffraction experiment was performed at beamline P07 [16] at PETRA III (positron storage ring operating at energy 6GeV with beam current 100mA) [17]. During the experiment, monochromatic synchrotron radiation of energy 80.09keV (λ = 0.01548nm) was used. The beam of photons was focused by compound refractive lenses down to a spot size of 2.2 μm ×34 μm. The specimen was scanned shot-by-shot along a straight path of total length 0,4 mm with step width of 1 μm. During each step, the sample was illuminated by highly intensive hard X-rays for 0.5 seconds. The resulting 2D XRD patterns were recorded using a Perkin Elmer 1621 detector [18]. The intensity was integrated to 2 Theta space by using the Fit2D software [19].

3. Results and their analysis

Results of wavy interface region, as shown in Fig. 5, analyzed in cross section were studied in interface geometry. As the sample was analysed in transmission, it had to be ensured that the interface region was straight in the direction of the beam. Otherwise, a detailed interface analyses would not have been possible. The sample investigated at beamline BW-5 and P07 has afterwards been metallographically analysed in the three dimensions. To do so, small amounts of the sample were removed by grinding and polishing and six cross sections have been analysed, see Fig. 4. The same technology was used for sample preparation in work [20]. The part of interface that has been analysed by hard X-ray microdiffraction was regular and the segments were sufficiently straight. This can be explained by the use of an orthogonal cutting operation.

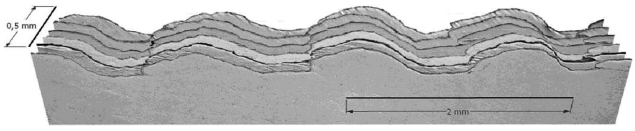

Fig. 5. Resulting of wavy interface region

Figure 5 shows 3D plot of obtained XRD patterns taken shot-by-shot going from the Cr/Ni SS to Ti. The picture clearly indicates abrupt change of the XRD pattern (roughly in the middle) corresponding to interface between this two materials.

The XRD pattern from the interface region is shown together with patterns taken approximately 10 mm away from the interface in both directions (in Ti and to Cr/Ni SS), where black line represents sample without the HT and the red line sample with the HT. Titanium consists of Single phase hcp-Ti alpha phase (S.G.: P63/mmc, a=2.95 Å, c=4.6846 Å) and austenitic steel consists of fcc-Fe phase (S.G. Fm-3m, a=3.599 Å). Interesting position is the interface region where mixing of this two phases is clearly visible. However, detail inspection of the pattern inclines to conclusion that there are two low intensive peaks (marked by the "**o**" sign – see Fig. 7) in addition coming from the third phase. All existing phases between Fe and Ti listed in ICDD PDF2 database [21] has been checked, but position of the peaks match up well with pure bcc-Fe phase (S.G. Im-3m, a=2.866 Å). Consequently, it can be stated the interface region probed in area 1x1 mm consists of the hcp-Ti, fcc-Fe solid solution and bcc-Fe like phases.

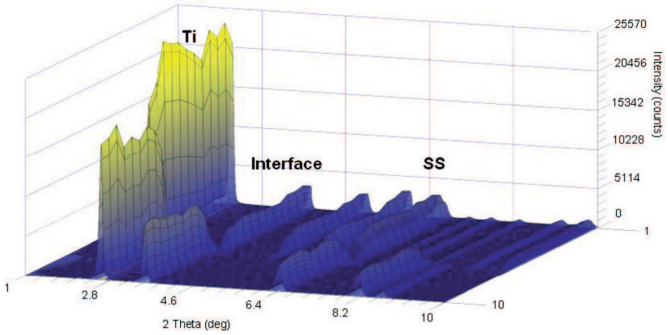

Fig. 6. 3D plot of obtained XRD patterns of Ti-Cr/Ni SS bimetal

In samples after the HT, similarly as in samples without the HT, 10 mm from the joint towards to the austenitic steel the fcc-Fe phase was detected. In close vicinity of the bonding line mixture of alpha Ti phase and fcc-Fe, bcc-Fe ferrite phase was observed (Fig. 7). There are differences in the proportion of the fcc-Fe between the sample without the HT and after the HT. Sample after the HT shows significantly lower level (approximately by 60%, see diffraction maxima for images with "o" in Fig. 7). From that it can be concluded, deformation-induced austenite to ferrite transformation caused by explosion shock can be reverted by appropriate the HT. No other intermetallic phases have been detected by this method. By going down from the interface to steel, the volume amount of the bcc-phase is rapidly decreasing, but 2 mm away from the border it can be still detected. Using presented method none intermetallic phases were revealed.

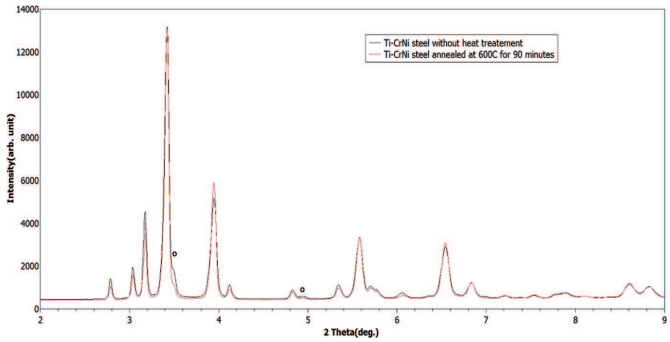

Fig. 7. XRD pattern from the interface area of Ti-Cr/Ni SS by synchrotron (BW5) – compared materials without the HT and after the HT

The synchrotron (BW5) using is not too accurate in this case and enables a more precise focus through bonding line. For this reason microdiffraction experiment has been performed at the beamline (P07). Phase analysis from the interfacial region proved existence of the hexagonal close packed Fe_2Ti intermetallic phase together with main matrix components fcc-Fe (austenite) and bcc-Fe (ferrite), see Fig. 8 and its insert, where black line represents sample without the HT and the red line sample with the HT. Comparison between explosion welded (as-prepared) and the sample after heat treatment testify effective reduction of intermetallic compound by the

annealing, visible in the mentioned Fig. 8 as reduction of corresponding Bragg's peaks intensities. In order to quantify volume amount of intermetallic phase in the samples we performed Rietveld refinement of the XRD patterns. Volume percentage Fe_2Ti in the as-prepared sample is about 17.5% while after the HT is approx. by 70% less (5.25 vol. %).

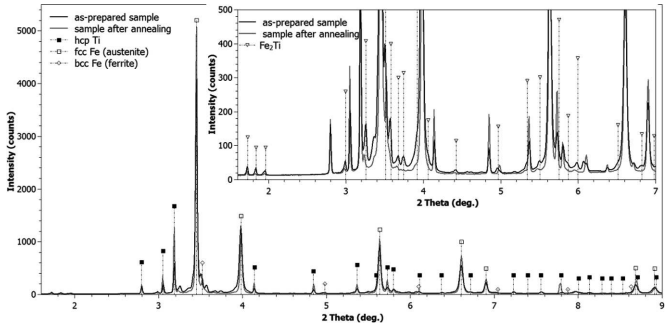

Fig. 8. XRD pattern from the interface area of Ti-Cr/Ni SS analyzed using synchrotron (P07) – compared materials without the HT and after the HT

In addition, we quantify the amount of intermetallic phase measuring from centre of the interface to both materials. The results are shown in Fig. 9 where black line represents sample without the HT and the red line sample with the HT. The phase is visible in region wide approx. 204 μm and for the annealed sample is significantly lower compared to the as-received state. The measurement proved that the HT is effective procedure to dissolute unwanted intermetallic compound and to improve the whole material lifetime.

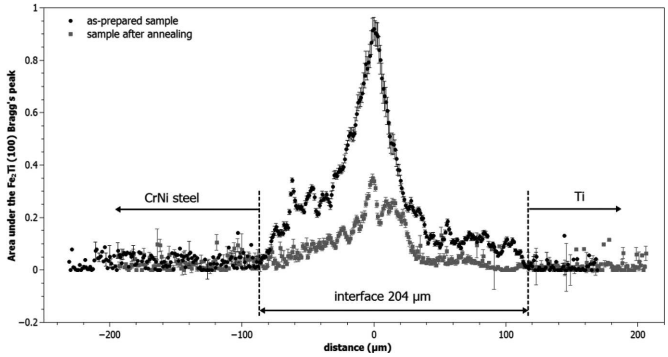

Fig. 9. XRD pattern from the interface area of Ti-Cr/Ni SS analyzed using synchrotron (P07) – compared materials without the HT and after the HT measuring from center of the interface for both materials

4. Summary

The results could be summarised as follows: the XRD pattern by synchrotron (BW5) showed that the interface region of both the samples probed in area 1×1 mm consist of the hcp-Ti, fcc-Fe solid solution and bcc-Fe like phases. The sample after annealing at 600°C/90 min./air shows significantly lower amount of the bcc-Fe phase. Other intermetallic phases were not detected by synchrotron (BW5).

Phase analysis from the interfacial region synchrotron (P07) proved existence of the hexagonal close packed Fe_2Ti intermetallic phase together with main matrix components fcc Fe (austenite) and bcc-Fe (ferrite). In order to quantify volume amount of intermetallic phase in the samples we performed Rietveld refinement of the XRD patterns. Volume percentage of Fe_2Ti phase in the as-prepared sample is about 17.5% while after the HT is ~70% less (5.25 vol.%). The measurement proved that the HT is effective procedure to dissolution unwanted intermetallic compound causing improvement of the whole material lifetime.

In future works it would be useful presented results to verify using other explosively welded samples.

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