STRUCTURE OF BIMETALS INVESTIGATED BY SYNCHROTRON RADIATION

SAKSL Karel[1], OSTROUSHKO Dmytro[1], MAZANCOVÁ Eva[2], SZULC Zygmunt[3], MILKOVIČ Ondrej[4], ĎURIŠIN Martin[1], BALGA Dušan[1], ĎURIŠIN Juraj[1]

[1]*Institute of Materials Research, Slovak Academy of Sciences, Košice, Slovak Republic, EU*

[2]*Faculty of Metallurgy and Materials Engineering, VŠB – Technical University of Ostrava, Ostrava, Czech Republic, EU*

[3]*Explomet – Opole (High-Energy Techniques Works), Opole, Poland, EU*

[4]*Faculty of Metallurgy, Technical University of Košice, Košice, Slovak Republic, EU*

Abstract

Bimetals targeted for industrial applications are usually designed to combine the properties of two dissimilar metals, e. g. high strength, high hardness and low cost of steels with chemically resistive titanium. In this work the structure of one particular, but for industries very interesting, bimetallic system – Cr/Ni stainless steel clad with titanium has been studied. The material was prepared by explosion welding, a method capable of joining a wide variety of similar or dissimilar materials. Our analysis is based on X-ray micro-diffraction experimentation utilizing hard monochromatic X rays focused down to micrometer size. In this way the bimetal in bulk form was analysed and microstructural differences between the joined materials and their interface were determined.

Keywords: Explosive welding, intermetallics, X-ray diffraction, elastic strains

1. INTRODUCTION

Explosive welding is method applied for joining of wide variety of similar or fundamentally dissimilar materials that cannot be joined by any other welding or bonding technique. Under conditions of controlled explosion, two or more materials can be joined by pressure, generated by detonation of explosives placed on the top of a welded material. Impact on plates at their contact surfaces is governed by the laws of ideal fluid where shock wave of amplitude ranging from 10 to 100 GPa propagates through materials causing strong deformation pronounced mainly at the bonded zone areas. Intensity of the impact substantially exceeds yield strength of basic materials forming wavy interface often seen with spikes or jets of one material indented to another one [1]. As has been formerly proposed by Cowan et al. [2], formation of the wavy bonded zone in explosive cladding is analogous to the formation of an oscillating wave and vortex street in fluid flow passing an obstacle. In case of explosive welding of Ti/stainless steel Manikandan et al. [3] demonstrated that at high kinetic energy conditions, dissipated heat causes melting of the mixture, leading to molten zones, where FeTi and Fe_2Ti intermetallic were identified. Additionally energetic conditions of joined interlayer of different thickness and showed that an increase in the interlayer thickness resulted in an increase in intermetallic layer as a consequence of high kinetic energy loss [4]. It is important to notice that non-metallic films (typically oxides) existing naturally on metal surfaces are swept away by explosion generated plasma leaving metallic surfaces clean for instantaneous compressed by very high pressure. In this work we present structural observation on one particular, but for the industry very interesting bimetallic system - Cr/Ni stainless steel cladded by titanium (Ti). Stainless steels are commonly used materials in industries such as energy conversion, chemical, petroleum and many others. Their applicability in these industries is however influenced by chemical resistivity of the material against transporting/containing media determining product lifetime against particular transporting/containing medium/environment. Titanium on the other hand, is one of the most important non-ferrous metals and finds extensive application in aerospace industry, because of its light weight, excellent corrosion resistance, high strength level, attractive fracture behaviour and high melting point. The cost providing a component whose full thickness is completely comprised of titanium is however for many applications strictly cost prohibitive. Hard, strong and relatively cheap stainless steel cladded by corrosion resistive titanium combines advantages of both materials gaining rapid acceptance and use by industry. The study presented here is unique, because the material was investigated in bulk form – as it is, in a breadth of several millimetres. The data were recorded in transmission by a micro-diffraction experiment, utilizing high-energy X-rays focused down to micrometer size area. The space resolved X-ray micro-diffraction experiment brought several new and very interesting findings related to the material system. It can be believed, that the knowledge gained by this work will contribute to a deeper structural understanding of the Ti / stainless steel bimetal, but also in structure predictions of explosively welded materials in general.

2. EXPERIMENTAL

2.1. Material and sample preparation

The Cr/Ni stainless steel of chemical composition (in wt. %): Fe–69.314, Cr–18.42, Ni–9.74, Mn–1.96, Si–0.45, P–0.065, C–0.04 and S–0.011 was clad by commercial pure titanium (in wt. %): Ti–99.879, Fe–0.05, O–0.05, C–0.01, H–0.006 and N–0.005. The thickness of Cr/Ni steel was 110 mm and of the Ti clad 6 mm. Sizes of the welded sheets were 2600 × 2600 mm. Both materials were explosively welded by the EXPLOMET-Opole company under mild conditions. Conditions under which the materials were joined is the know-how of the company; but the curvy shape of the interface and the number of vortexes in the joining region (Fig. 1 in this paper) is very similar to the material E5 (Fig. 7a in Manikandan et al. work [3]) prepared under the conditions of horizontal collision point velocity Vc 2350 $m \times s^{-1}$ and kinetic energy loss ΔKE 2.44 $MJ \times m^{-2}$. The bimetal was further annealed at 600 °C for 1.5 hour in air atmosphere. This temperature and dwell time was chosen in order to homogenize the grain size and chemical composition of the titanium and to relax internal stresses of the bimetal. The thermal treatment was chosen on the basis of previous works, after which mechanical properties including hydrogen response of the material show the most favourable results. From a material cube of 5 mm an edge length was cut off so that Ti/steel interface was roughly in the middle parts of four faces. The two parallel faces of the cube subjected to the micro-diffraction experiment were finely grinded, polished and etched in order to remove residual stresses introduced by the cutting process.

2.2. Instrument used for the microdiffraction experiment

To determine phase composition and microstructure parameters of the interface and surrounding regions, a hard X-ray micro-diffraction experiment was performed at beamline P07 at PETRA III (electron storage ring operating at energy 6 GeV with beam current 100 mA). During the experiment, monochromatic synchrotron radiation of energy 99.16 keV (λ = 0.0125031 nm) was used. The beam of photons was focused by compound refractive lenses down to a spot the size of 2.2 µm x 34 µm. The sample was positioned perpendicularly to the direct beam by adjusting the tilt of supporting cradle with precision ±0.25 degree adjusted by the steepest transition (measured by absorption) between the Ti and the steel. The materials interface was scanned shot-by-shot along a straight path (orange full line marked in Fig. 1) of length 0.4 mm with step width of 1 µm. The scan was continued further down to the steel by coarser 0.1 mm step up to the total distance 5.4 mm (dashed orange line marked in Fig. 1). 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. The intensity was then integrated to 2θ by using the Fit2D software.

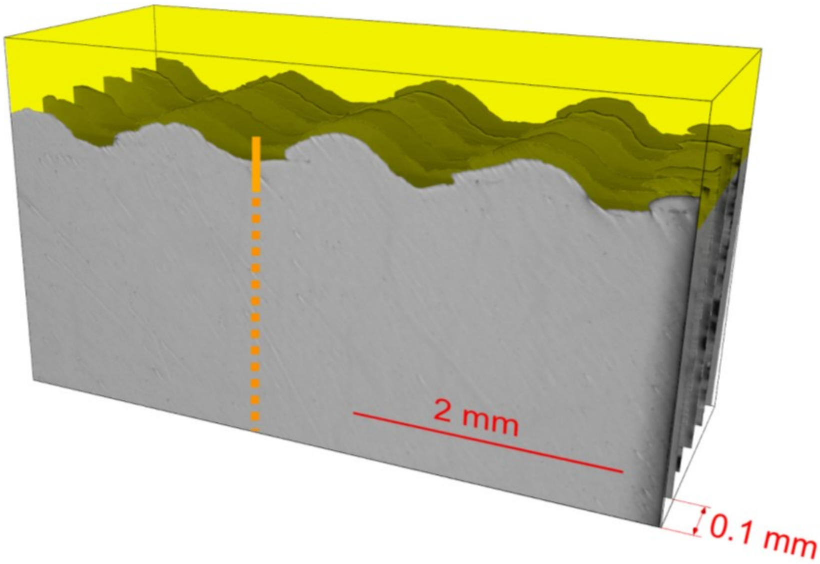

Fig. 1 Three-dimensional view (6 cross sections) of the Cr/Ni stainless steel/Ti bimetal investigated by micro-diffraction. Steel (gray areas at the bottom) titanium (transparent yellow color). The orange full line represents shot-by-shot scan along a straight path of length 0.4 mm with a step of width 1 μm. The scan was continued further down to steel side with a coarser step 0.1 mm (dashed part of the line).

3. RESULTS

3.1. Phase composition

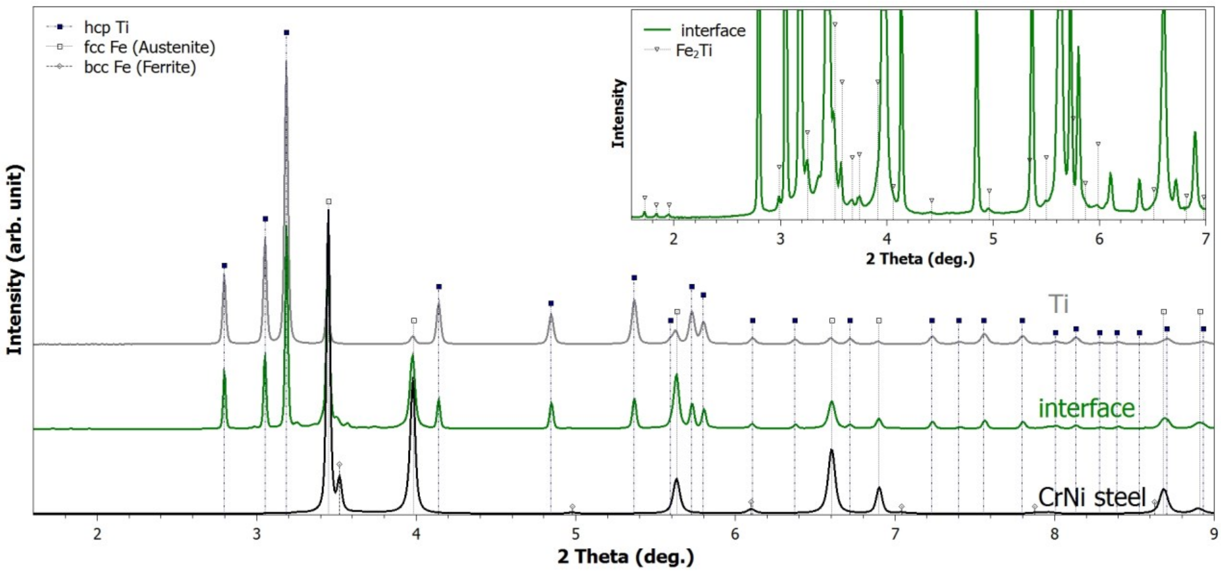

Fig. 2 XRD patterns obtained from the investigated bimetal.

Fig. 2 shows XRD patterns from different parts of the bimetal: steel side measured 230 μm far from the center of the joint (black curve at the bottom), titanium 170 μm from the centre, but in the opposite direction (gray curve on the top of the picture) and centre of the joint itself (represented by green curve in between and enlarged in the figure insert). Steel measured close to the joint consists of two phases: fcc-Fe (austenite) and bcc-Fe (ferrite) occupying the volume fractions ~94 % and ~6 %, respectively - percentages determined by the Rietveld refinement method. The place on the titanium side is composed of hcp-Ti (α Ti phase) 94 vol. % and residual austenite 6 vol. %. Material in the centre of the joint consists of the following phases mentioned here along with corresponding space groups, refined lattice parameters and estimated volume percentages: fcc-Fe (austenite; S.G.: Fm–3m; a = 0.3599 nm, 75.7 vol. %), hcp-Ti (alpha phase; S. G.: P63/mmc; a = 0.2959 nm, c = 0.4694 nm; 14.5 vol.%), bcc-Fe (ferrite; S.G.: Im–3m; a = 0.2879 nm, 0.8 vol. %) and hexagonal intermetallic phase Fe_2Ti (S.G.: P63/mmc; a = 0.4804 nm, c = 0.7822 nm; 8.9 vol. %).

3.2. Space resolved studies

With a narrow (μm sized) and hard X-ray beam we were able to scan material along a well-defined trajectory and obtain crystallographic information about the material from a very tiny volume (0.0022 × 0.034 × 5 = 0.00037 mm^3). The microdiffraction experiment performed allows us to determine structure of the investigated material as a function of space (space resolved studies). Fig. 3 shows normalized areas under the hexagonal Fe_2Ti (100) peaks as a function of distance. The position of the Fe_2Ti (100) peak maximum we set at zero. Positive numbers mean direction to the titanium side while negative numbers to the steel side. The green curve represents distribution of the hcp Fe_2Ti phase in as-prepared material (after explosion welding and annealing at 600 °C for 1.5 hour) while the red curve represents amount of the phase after second annealing (post heat treatment) at 600 °C for 1 hour. As it can be seen the second annealing significantly reduces (by more than 66 %) the amount of the intermetallic phase. Hard and brittle intermetallics (micro-hardness of the phase is 1420 HV0.01) are typically unwanted in explosion welded materials since under static or dynamic stress it can play a role of crack initiator, causing material decohesion at the joint. Therefore, application of the second annealing is advised to increase ductility and fatigue resistance of the material enhancing lifetime and/or durability of this bimetal in many applications.

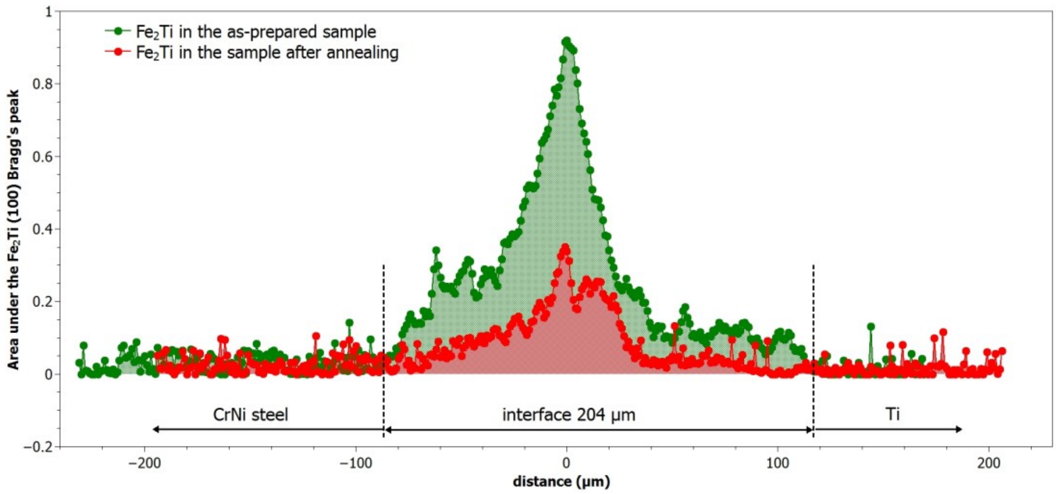

Fig. 3 Amount of the hcp-Fe_2Ti phase in as-prepared sample and after the second annealing at 600 °C for 1 hour.

Further we analysed internal strains of the austenitic phase induced by explosion and compare it for two material stages: in as-prepared stage and after the second annealing. Our approach is based on calculation of distances between (200) crystallographic planes measured azimuthally along the (200) Debye-Scherrer rings. Fig. 4 a. and b. shows 2D XRD patterns of the two samples taken from the centre of their interfaces. Radius of the (200) Debye-Scherrer ring is inversely proportional to distances between the (200) crystallographic planes "d" while dashed arc shown in Fig. 4a indicate azymuthal angle, along which the d-values has been calculated. Comparison between d-values distributions obtained from as-prepared and secondly annealed samples is shown in the Fig. 4c in polar plot representation. From this comparison is obvious that the second annealing (represented by red curve) narrowing and homogenize the d-values distribution (is less noisy and more circular) what is direct consequence of relieve of internal stresses in the material.

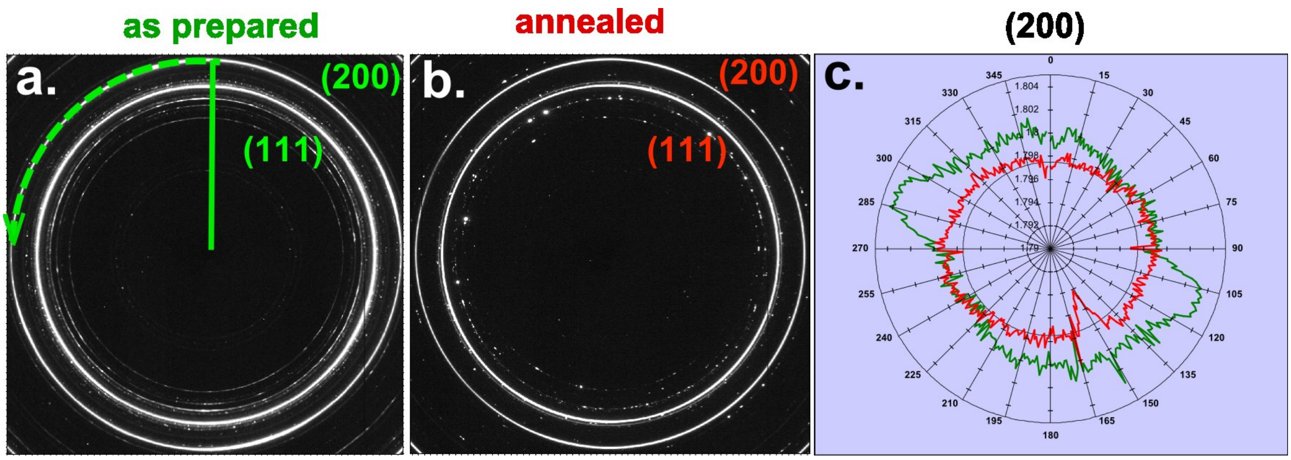

Fig. 4 2D XRD patterns taken from center of CrNi stainless steel/Ti bimetals in as-prepared stage **a.** and after the second annealing **b.** On the **a.** is marked radius of the (200) Debye-Scherrer ring and direction along which the d-values distribution has been calculated. **c.** compares the angular distributions of (200) interplanar distances calculated from the (200) Debye-Scerrer rings shown on figures **a.** and **b.**

Distributions of the austenite internal strains have been evaluated at interface as well as in close vicinity to the joint. Results of the analysis are shown in the Fig. 5. On vertical axis 0 represents "reference" (200) interplanar distance 0.1779 nm obtained from well relaxed steel, in our case measured far (~6 mm) from the centre of the joint, negative values mean compressive elastic strains and positive values tensile type of elastic strains. It's important to notice that the second annealing reversing tensile strains seen at interface of the as-prepared material to more favourable compressive. Tensile stresses in material can add to external forces and can cause cracks to develop; compressive stresses on the other hand can prevent them for starting.

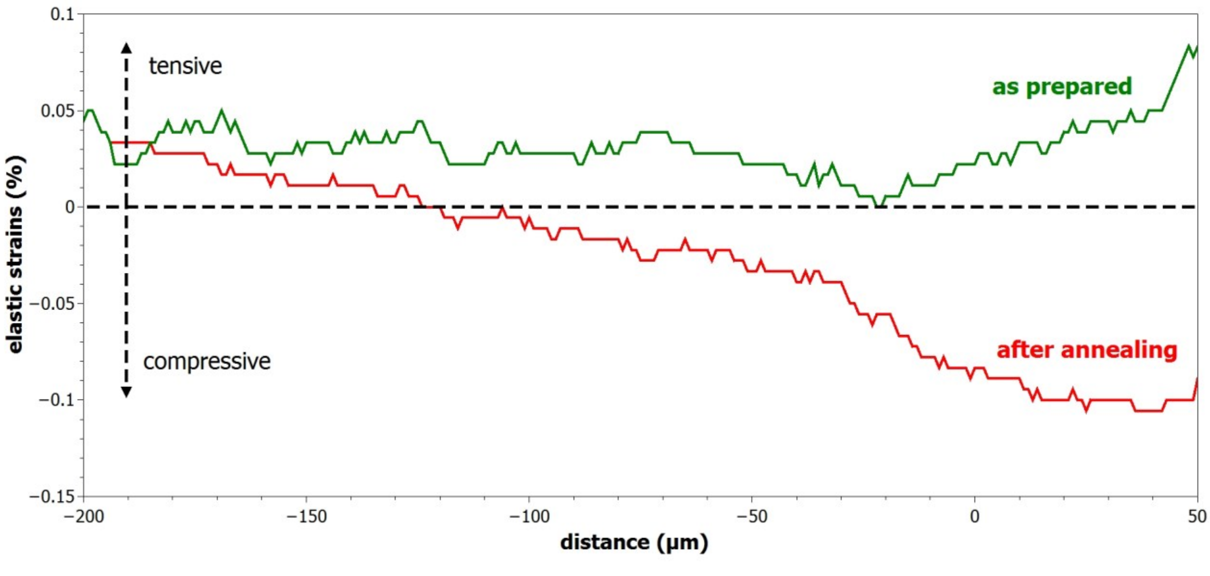

Fig. 5 Space distributions of the austenite internal strains obtained from (200) crystallographic planes

CONCLUSION

Microstructure of the CrNi steel/Ti bimetal was characterized by micro-X-ray diffraction technique applying a hard (99.16 keV) monochromatic X-ray beam focused down to size 2.2 µm × 34 µm. The aim of our work was to provide detailed information about a phase composition and strains of interface of explosively welded titanium on Cr/Ni stainless steel. The bimetal has been evaluated in two stages: as in as-prepared (after explosion welding and annealing at 600°C for 1.5 hour) and after second annealing (post heat treatment) at 600°C for 1 hour. In the weld (the place where materials are joined together) microstructure of both the samples consists of austenite, hcp-Ti, bcc-Fe and newly formed hexagonal intermetallic phase Fe_2Ti. Application of the second annealing significantly reduces (by more than 66 %) amount of the unwanted intermetallic phase and induces favourable compressive strains at the weld region. Both the factors increase ductility and fatigue resistance of the material what consequently enhance lifetime and/or durability of this bimetal for many applications.

. ACKNOWLEDGEMENTS

K. Saksl., M. Ďurišin, D. Balga and J. Ďurišin are grateful to the Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic and the Slovak Academy of Sciences (VEGA Project No. 2/0128/13) and to the M-ERA.NET project "ExploGuard" for the financial support of this work. The work was also realized in frame of project No. LO1203 "Regional Materials Science and Technology Centre – Fasibility Program" and project 7AMB14SK023 funded by Ministery of Education, Youth and Sports of Czech republic.

REFERENCES

[1] BERGMANN, O.R., COWAN, G.R., HOLTZMANN, A.H. Supply Chain Simulation: Experimental Evidence of Jet Formation During Explosion Cladding. *Trans. Met. Soc. AIME*, 1966, vol. 236, pp. 646-653.

[2] COWAN, G.R., BERGMANN, O.R., HOLTZMAN, A.H. Mechanism of bond zone wave formation in explosion-clad metals. *Metall. Mater. Trans. B.*, 1971, vol. 2, No. 11, pp. 3145-3155.

[3] MANIKANDAN, P., HOKAMOTO, K., DERIBAS, A.A., RGHUKANDAN, K., TOMOSHIGE, R., Explosive welding of Titanium/Stainless Steel by Controlling Energetic Conditions. *Mater. Trans.,* 2006, vol. 47, No. 8, pp. 2049-2055.

[4] MANIKANDAN, P., HOKAMOTO, K., FUJITA, M., RAGHUKANDAN, K., TOMOSHIGE, R. Control of energetic conditions by employing interlayer of different thickness for explosive welding of titanium/304 stainless steel. *J. Mater. Process. Technol.*, 2008, vol. 195, pp. 232–240.