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JOINT HYDROGEN SUSCEPTIBILITY OF 304 SS WELDED WITH TITANIUM

PODATNOŚĆ POŁĄCZENIA NA WODOROWANIE W UKŁADZIE PLATERU STAL 304 SS ZGRZEWANYM WYBUCHOWO Z TYTANEM

Welds of thick plates (304 SS) clad with Ti of commercial purity in as-received state and also after subsequent heat treatment and/or after hydrogen charging were investigated. Fatigue tests were carried out at amplitude of 20 Hz and in case of bimetal without hydrogen charging also at amplitude of 40 Hz. After heat treatment, charged welds showed higher threshold level than the welds without heat treatment. Energy dispersed analyses (EDA) of fracture surfaces showed that failure predominantly occurred in joint. Hydrogen induced cracking (HIC) response of bimetal samples demonstrated favourable results both after welding and after subsequent heat treatment. Short and thin cracks were observed, exclusively located in mixed zone, where approx. 16-20 at. % of Ti using EDX (energy dispersed analyser) was revealed. By application of monochromatic synchrotron radiation Ti-α, Fe-fcc, Fe-bcc and intermetallic phase Fe_2Ti were detected.

Keywords: 304 SS-Ti weld, fatigue, hydrogen response, phase analysis

Badano połączenia grubych blach platerowanych (304 ss) z Ti o czystości technicznej po zgrzaniu, jak i po częściowym procesie termicznym i/lub po wodorowaniu. Wykonano testy zmęczeniowe o amplitudzie 20 Hz a w przypadku bimetalu bez wodorowana również o amplitudzie – 40 Hz. po przeprowadzeniu wygrzewania, obciążone połączenie ujawniło wyższy poziom progu niż połączenie, w których nie wykonano obróbki termicznej. Analiza EDA powierzchni łączenia udokumentowała silny udział pęknięć na powierzchni łączenia. Wodorowanie silnie wpływa na inicjację pękanie (HIC), w bimetalowych próbkach w stanie po platerowaniu, jak i po obróbce termicznej. Zaobserwowano, krótkie i cienkie pęknięcia, które zlokalizowane były w strefie połączenia, gdzie zaobserwowano (analiza SEM/EDX) fazy zawierające 16-20 wt.% Ti. Przez zastosowanie monochromatycznego promieniowania synchrotonowego wykryto fazy Ti-α, Fe-fcc, Fe-bcc oraz fazę międzymetaliczną typu Fe_2Ti.

1. Introduction

Explosive cladding enables to weld various materials which cannot be joined by any other conventional welding or bonding techniques. The upper clad material generally shows better properties, while the basic material can be of worse quality [1-3]. Numerous papers were focused on welding conditions [1, 4, 5], metallographic parameters including wave length, amplitude and adiabatic bands at the bonded zone and nearby areas [6-10], on hardness level across the weld [7, 8] and on intermetallic phase analyses [4, 5, 8, 11]. Results partially differed as well as the methods of analyses. Lublińska et al. [12] reported that hydrogen after cathodic charging caused decrease of shear strength of 410S and C-Mn steel bimetal. Mudali [13] presented acceptable corrosive resistance of dissimilar Ti and 304 SS bimetal in HNO_3. Mazancová et al. [14] published the first HIC (hydrogen induced cracking) results of stainless steel clad with Ti, which revealed promising hydrogen response. Regarding fatigue tests and hydrogen susceptibility of bonded materials and bimetal welds particularly, there is information sporadic. Namely, the bonding line and hard, brittle intermetallic phases, formed in wave curls during explosive welding process, represent potential positions for hydrogen trapping, even though it is known that both mentioned materials show high hydrogen resistance [15]. Regarding fatigue of the bimetal interface, there is also practical none information. Situation has been complicated by thin layer of clad material. By use of conventional techniques, an exact quantification of formed intermetallic phases during explosion in area of interface is difficult, as well. Relatively wide beam of the EDX (energy dispersed analyser) penetrates to micro-meter depth of analysed material unlike the monochromatic synchrotron radiation (electrons or positrons) going through the studied sample and enabling to win a set of hundreds of X-ray diffraction (XRD) records on bonding line and its nearby. Even when bimetal interface has a wave character in nature, it does not form regular sinusoid and twists itself in different depth positions. It is why the problem demands more complex analy-

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sis in 3D form what is part of following-up research. Moreover, the first study by application of monochromatic synchrotron radiation indicated dissimilar phases at bimetal interface and its vicinity [16] unlike results found by conventional EDX [4, 5, 11]. Information about hydrogen response and about limit states of various mechanical properties of 304 SS clad with Ti is useful, because given bimetal is applied in heavy chemistry, special technique, shipbuilding, in energetic and in the latest time it has been considering for geothermal energetic, where sour environment degrades bimetal material.

That is also way the presented work is aimed at hydrogen induced cracking and hydrogen susceptibility of mentioned bimetal under fatigue tests. Both conventional technique and monochromatic synchrotron radiation were applied for chemical analysis of bimetal weld area.

2. Experimental procedure

For study explosively welded bimetal of stainless steel 304 SS with Ti of commercial purity was used. Chemical composition of Ti was followed (wt. %): 0.01C, 0.05Fe, 0.05O, 0.005N, 0.006H. The 304 SS showed this chemical composition (wt. %): 0.04C, 0.45Si, 1.96Mn, 18.42Cr, 9.74Ni, 0.006P and 0.011S. In EXPLOMET-Opole both materials were explosively welded and subsequently the clad plate was subjected to ultrasonic testing. Cladding process has been know-how of above mentioned firm. The thickness of the 304 SS and Ti layer corresponded to 110 mm and to 6 mm. A half of studied material was heat treated (HT) at 600°C/1.5h with air cooling. With respect to thickness of clad Ti, 10 mm round bars of 100 mm length for fatigue tests were machined. In the joint area the bars were reduced to 7.8 mm in diameter. Figure 1 shows schema and real appearance of machined fatigue tensile bar. The evaluation of bimetal service life was tested under alternating load tension-pressure at amplitude of 20 Hz in the range from 160 MPa to 40 MPa according to the ČSN EN ISO 6892-1 Standard by multifunction LSV100kN machine. Amplitude of 40 Hz was used only for samples after the HT. For fatigue tests of bimetal at ambient temperature, by hydrogen charged specimens of weld (electrolytically in 0.1 M solution of H_2SO_4 for 8 hours at current density 15 mA.cm^{-2}) in as-clad state and also after the HT were prepared.

Fig. 1. Appearance of used fatigue tensile bar

Susceptibility of given bimetal to HIC (hydrogen induced cracking) was the second part of investigation. Bimetal samples of dimensions 20×14.5×100 mm were manufactured and then exposed in corrosive solution A (NaCl, CH_3COOH saturated with H_2S) for 96 hours, according the NACE Standard TM 0284-2011, item No. 21215 [17]. At the beginning of the test the pH corresponded to 2.66 and the final pH was 3.96. The test temperature reached 25±3°C. Critical parameters of the HIC the CLR (crack length ratio), the CTR (crack thickness ratio) and the CSR (total crack sensitivity) were mathematically evaluated according to the NACE Standard TM 0284-2011, item No. 21215 [17]. The corrosive test and own evaluation were in accord with the above mentioned Standard and the results were confronted with ANSI/API specification 5L generally used for oil country tubular goods. According to the mentioned specification the parameters CLR, CTR and CSR should be equal and/or lower than 15%, 5% and 2% (introduced in sequence). Each of three exposed samples (20×14.5×100 mm) was divided into four same perpendicular sections. Consequently, nine samples were prepared for the metallographic evaluation, again in accord with above mentioned NACE Standard [17]. Welded materials were etched in mixture of nitric acid and hydrofluoric acid and in water solution of hydrochloride and nitric acid. Presented material was investigated both in as-clad state and also after the HT. Metallographic and micro-fractographic evaluation was carried out by light microscope Carl Zeiss Axio Observer A1m and the electron microscope SEM JEOL LSM-6490 equipped with EDX OXFORD INCA ENERGY 350.

In weld and its nearby different phases were found by a hard X-ray micro-diffraction synchrotron radiation (focused to 2.2×34 μm). Experiment was performed at beam-line P07 at PETRA III (electron storage ring operating at energy 6 GeV with beam current 100 mA) [18]. Monochromatic synchrotron radiation of energy 99 keV was used. The raster step size corresponded to 1 μm in total length of 401 μm. Consequently, 401 XRD records were won for next integration and numerical processing. Other information concerning the analyses found by use of synchrotron is accessible in paper [16].

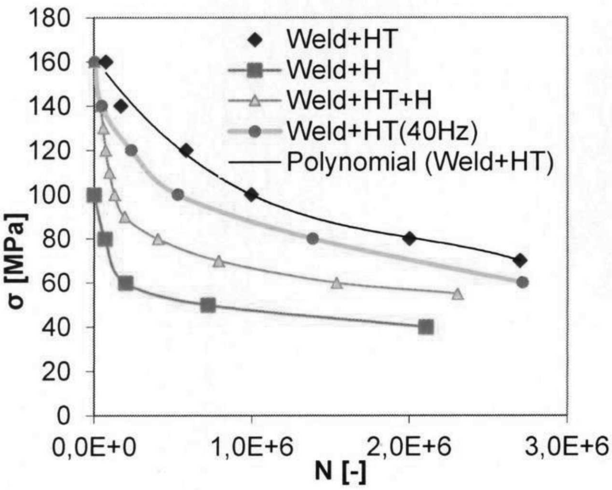

Fig. 2. Dependence of loading on number of cycles to rupture

3. Results and discussion

3.1. Fatigue and micro-fractographic study

Results of detected fatigue response are summarised in Fig. 2 and piece of information was presented recently [8]. As Fig. 2 demonstrates, bars after the HT without hydrogen showed the best threshold level at the highest load of 80 MPa while threshold level of the HT samples after hydrogen charging was shifted to 55 MPa and it corresponds to 68.8% life time of the samples without hydrogen charging. Threshold level of life time of samples without the HT and exposed by hydrogen was lying on level of 40 MPa, representing 50% life time of uncharged samples. It demonstrates an important harmful hydrogen influence on embrittlement of bimetal bonding line, especially being in as-clad state, without the HT. The applied HT represents optimised HT mode being able to make the Ti matrix more homogeneous and to decrease internal stress formed during the explosive welding [13]. Life time of testing bars after the HT and hydrogen charging corresponded to 17% of the tensile strength of samples in as-clad state [8]. Of course, initiation mode of fatigue crack depends on rate of plastic deformation, hence on level of effective tension [19]. In studied case the cycling rate was constant. Beside structure homogeneity, grain sizes play also a role in initiatory stage of failure. Internal stresses formed during welding were decreased after the HT and Ti matrix was simultaneously homogenized and grain size slightly refined as it was presented recently [8]. The bonding line of any bimetal shows typical wavy character, according to the types of joined materials, velocity of clad material, angle of the running material and the explosive type [1, 7, 10]. All fatigue bars were broken in the weld or in curls of the welded area. All fracture surfaces of testing bars did show more or less wavy character as it can be seen in Fig. 3, in given case for hydrogen uncharged sample. At 40 Hz of cycling threshold level of the HT bimetal life time was by 10 MPa lower than at 20 Hz, as it Fig. 2 shows.

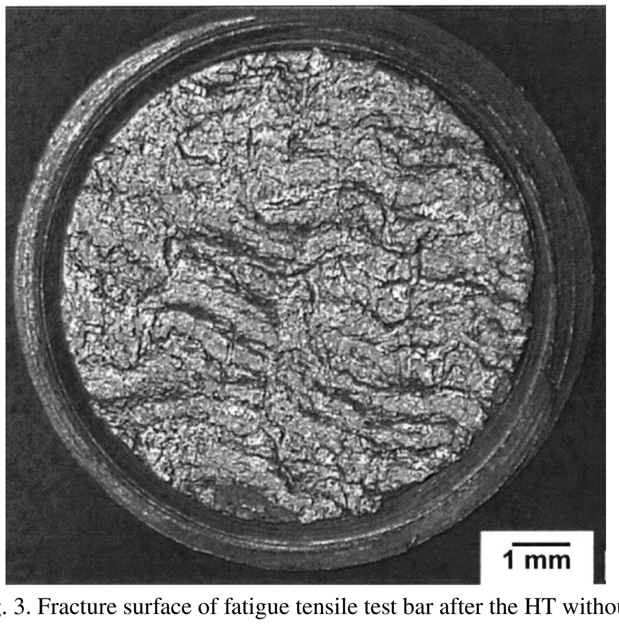

Fig. 3. Fracture surface of fatigue tensile test bar after the HT without H-charging

By use of EDX the carried out EDA (energy dispersed analysis) proofed the fracture was preferentially realized in mixture matrix with high Ti content lying in the range from 66 to 97 at. % showing smooth dark surface areas (2a, 2b in Fig. 4) and/or secondarily in lighter grey surface marked as 3a, 3b (with approx. 43 at. % of Ti) in Fig. 4. The brightest smooth cleavage facets (areas 1a-1c in Fig. 4) were observed in minority, showed 20 at. % of Ti and 48 at. % of Fe and could indicate FeTi or Fe_2Ti presence. In curls of weld Manicandan [20] detected slightly higher portion of both elements, also by EDX. Revealed Ti and Fe portions lie in eutectic region of phase diagram for the intermetallic compounds FeTi and Fe_2Ti. The SEM beam penetrates through sample surface to micro-meter depth and so the analysis is not so precise. Sporadic appearance of pure Ti was also analysed (see black spot 4 and 4b in Fig. 4). The presented results confirm that the cracks initiation and their propagation were predominantly realized in the region with the highest Ti portion as it was also observed in works [1, 8]. Analysis in section across the bimetal interface proved that chemical composition with 65-97 at. % of Ti corresponded to bonding line, whereas the approximately 40 and/or 20 at. % of Ti represented mixed phases situated in curls of the waves. At the interface of the curls and Ti matrix occurrence of practically pure Ti was detected. Consequently, the fatigue bars rupture predominately occurred just at bimetal interface and secondarily in area of mixed phases of curls. Neither un-welded spots nor cavities were detected at the 304 SS-Ti interface and/or close neighbourhood, which could negatively influence the fracture.

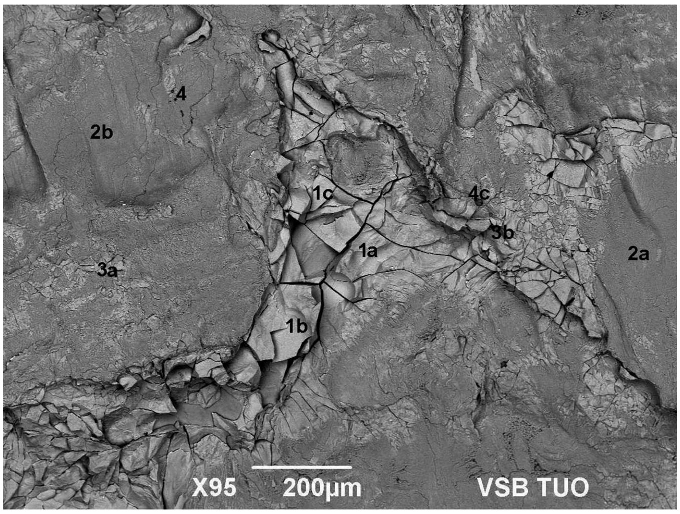

Fig. 4. Fracture surface micrograph showing cleavage fracture and micro-cracks propagation in fatigue bar after hydrogen charging without the HT. Titanium contents (EDA) in regions (1a, 1b, 1c) = 20 at. %, (2a, 2b) = 66 – 97 at. %, (3a, 3b) = 43 at. % and (4, 4c) = 100 at. %

3.2. HIC study

After exposition of the bimetal samples in corrosive solution A [17] with bubbled H_2S the metallographic investigation revealed numerous thick and shorter cracks in wave curls, where mixed phases were detected. Cracks length was lying in range from 0.01 to 0.08 mm the thickness was minimally 0.01 mm and maximally 0.22 mm. Maximal values of the cracks dimensions were detected in sporadic cases only. The total number of all revealed cracks was 84 (samples without the HT) and 56 (samples after the HT). Cracks predominantly showed 45° -90° orientation towards the bonding line and none ran to any basic material. None cracks were observed outside the wave curls. No cracks and cavities were also revealed at bimetal interface or in its neighbourhood, as well as inclusions, which could significantly influence the hydrogen response. On the basis of detected cracks and their dimensions the Table 1 summarises results of calculated HIC parameters. Samples 1-9 represent sectioned three originally exposed samples (in accord with [17]).

TABLE 1

Hydrogen induced cracking parameters without the HT/after the HT [%]
(CLR = Crack length ratio, CTR = Crack thickness ratio, CSR = Total crack sensitivity)

Sample	CLR	CTR	CSR	CLR	CTR	CSR
	Individual values			Mean values		
1	1.04/ 2.19	2.47/ 0.14	0.003/0.001	1.44/2.21	2.47/0.30	0.01/0.002
2	1.25/ 1.37	2.25/ 0.13	0.01/0.002			
3	2.04/ 3.08	1.99/ 0.62	0.01/0.003			
4	3.07/ 3.68	2.17/ 0.70	0.01/0.002	3.55/1.52	2.95/0.30	0.01/0.002
5	3.42/ 0.50	2.93/ 0.04	0.01/0.001			
6	4.15/ 2.37	3.75/ 0.16	0.01/0.002			
7	2.25/1.80	2.30/3.66	0.01/0.012	2.39/1.67	2.24/3.00	0.01/0.010
8	1.92/1.60	1.90/2.80	0.01/0.005			
9	3.02/1.60	2.51/2.62	0.01/0.013			

As it from Table 1 follows, all parameters are satisfactory and comply with the requirements of the ANSI/API specification 5L. The worst individual CLR parameter showed 72%, resp. 75% reserve (sample 6/4-without the HT/after the HT). Regarding the CTR parameter the minimal reserve corresponded to 25% or 27% (sample 6/7-without the HT/after the HT). The total CSR parameters are approximately on the same level and the worst results showed 99.5% reserve.

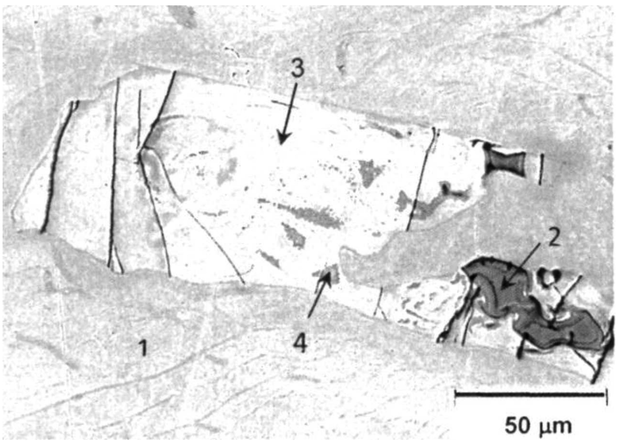

Fig. 5. Micrograph of section after the HIC exposition in NACE corrosive solution [17] with detected crack. Distinct grey regions represent different Ti content of bimetal wave curls (EDA). Mark 1 represents 304 SS, mark 2=90-100 at. %, mark 3=16 at. % and mark 4=20 at. % of Ti

Figure 5 shows SEM micrograph of the welded 304 SS steel with Ti after exposition in corrosive solution. Sample was slightly etched so that grey nuances as well as cracks would be better distinguished. The sinusoidal interface with curls orientated in direction of explosion is obvious. The cracks were detected only in area of mixed phases, mostly located just in curls. Propagation of cracks into basic 304 SS or into Ti was not revealed as it from Fig. 5 and/or from Fig. 6a, 6b follows. By use of EDX different grey regions were analysed. In Fig. 5 the number 1 corresponds to the basic 304 SS, number 2 practically represents Ti matrix (90-100 at. %), and number 3 showed mixture of (in at. %) 16 Ti, 16 Cr, 57 Fe, 8 Ni and Al, Si and/or Mn balanced was revealed, while the area marked by number 4 consisted of (in at. %) 20 Ti, 11 Cr, 61 Fe, 6 Ni and Al or Si and/or Mn. The last two chemical compositions are similar to ones of areas 1a-1c in Fig. 4.

By application of monochromatic synchrotron radiation, phase analysis confirmed at bimetal interface and/or in wave curls Fe_2Ti phase (both in as-received state and after the HT), beside presence of Fe-fcc, Fe-bcc and Ti-hcp, as it the first results indicated [16] and as it newly Fig. 7 demonstrates [21], unlike the results presented by Mousavi et al. [22, 23] who detected FeTi intermetallic phase simultaneously with Fe_2Ti, Fe_4TiO_2, NiTi and Cr_2Ti under different explosive loads (R in range from 2.1 to 3.93), while Manikandan et al. [20] revealed in similar bimetal FeTi and Fe_2Ti only. They used EDX. Explanation of those differences can be in technology of own welding, which has been know-how of EXPLOMET-Opole. Their explosion conditions could be marked as medium. The Fe-bcc detection may be ascribed to strong plastic deformation during explosion developed by kinetic energy loss at the interface region, friction and shear of two welded materials followed by enormous cooling rate [24]. This leads also to partial melting of the wave crest regions and to a formation of intermetallic particles [4]. Presence of Fe-bcc could, in superposition with some other negatives, influence quality of the bimetal weld, e.g. decrease interface strength and degrade hydrogen response. Even when portion of that phase was sporadic next research pays deeper attention to this founding.

No cracks and cavities were observed in the investigated bimetal before exposition in corrosive solution, which could be good potential areas for hydrogen trapping. In such free spaces a lot of hydrogen could be lost without negative influence on the welded materials. In detail, the analysis of the bonding line and/or of curls area was already presented [8]. On the contrary, a lot of deformation bands and ultrafine grains of both micro-structures in the vicinity of the interface, as a consequence of strong deformation caused by explosive welding, represent numerous potential traps for more uniform hydrogen redistribution and so better conditions for the more favourable hydrogen resistance [7, 8, 13]. Oxides were also not detected at

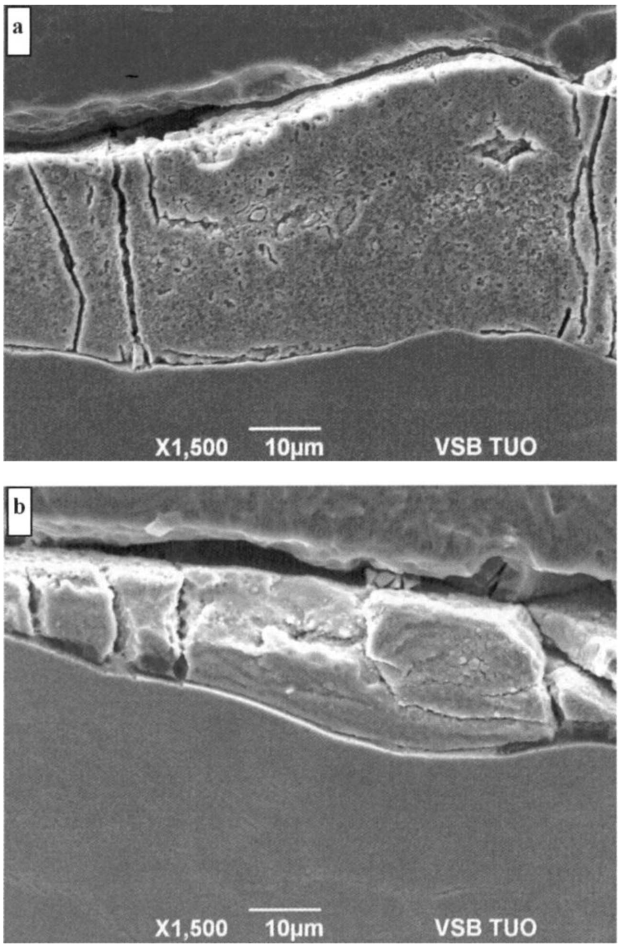

Fig. 6. a) Bimetal interface after HIC exposition without the HT – sample 3; b) Bimetal interface after HIC exposition with the HT – sample 3

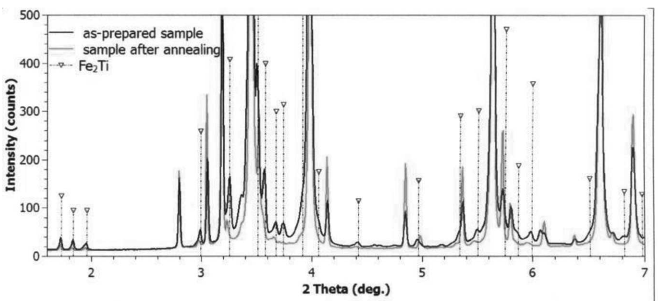

Fig. 7. XRD pattern from the interface area of bimetal 304 SS clad with Ti analyzed using synchrotron (P07) – compared materials without the HT and after the HT

the bimetal interface and in its vicinity, whereas Berdychenko et al. [25] detected TiO, Ti_3O and Ti_2O_3 in bimetal type of C-Mn steel clad with Ti. Lower portion of fine oxides, as well as other particles in matrixes, generally could also influence higher material resistance against hydrogen embrittlement as potential hydrogen traps. On the other side, the mentioned particles could degrade other bimetal properties, especially when those would be found at bimetal interface or in its close neighbourhood in localised form. Any in-homogeneity represents higher danger of hydrogen cracking susceptibility. It can be also state, during the HIC exposition (representing the static test) cracks were solely formed in area of intermetallic phases of curls and not in area of bonding line as it was observed in case of dynamic fatigue test.

4. Conclusions

Explosively welded stainless steel of 304 SS type with Ti was investigated. The attention was paid to tension-pressure fatigue tests (representing dynamic tests) of samples after welding and/or after heat treatment (HT) charged by hydrogen. Hydrogen induced cracking (HIC, representing static tests) in as-weld state and after the HT in accord with the NACE Standard TM 0284-2011, item No. 21215 was investigated.

After the HT, the hydrogen charged bars tested at amplitude of 20 Hz showed safe load boundary at 17% of tensile strength of bimetal weld in as-clad state. The HT samples without hydrogen charging at higher applied amplitude (40 Hz) resulted in life time decrease approx. by 10 MPa in comparison with samples tested at 20 Hz. Fracture surfaces predominantly indicated the rupture in areas where 66-97 at. % of Ti was revealed. This represents bonding line. Area in which 43 at. % of Ti was detected could a presence of FeTi and/or Fe_2Ti (with Cr partially substituting the Fe) indicate.

After the HIC exposition cracks were observed in areas of curls only, where inter-metallic phase of Fe_2Ti type by synchrotron was found. By use of conventional EDX the most frequent mixed phases approximately (in at. %) 20 Ti, 11 Cr, 60 Fe together with balanced portion of Ni, Al, Si and Mn showed. This analysis also corresponds to the areas 1a-1c in Fig. 4 (fatigue tensile test bars). All HIC parameters were satisfactory and complied with the requirements of NACE Standard and ANSI/API specification 5L. After the HT results were more favourable. Minimal reserve of the CLR parameter was 72% (without the HT) and 75 % (after the HT). In case of the CTR parameter it was 25% and 27 % again for not heat treated and for heat treated samples, respectively. After the HIC tests, 84 crack were detected in samples without the HT, unlike 56 cracks observed in samples after the HT. Cracks showed 45°-90° orientation towards the bonding line and none ran to any basic material. Cracks were maximally 0.08 mm long and maximally 0.22 mm thick (two cases only). By application of monochromatic synchrotron radiation sole intermetallic phase Fe_2Ti in wave curls was revealed. Analysis of results is part of work [21].

Presented results of hydrogen response give useful knowledge to producers e.g. of electrolytic cells or components for geothermal energetic, which operate in sour environment.

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