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CHARACTERIZATION OF WELDED DUPLEX STAINLESS STEEL AFTER ANNEALING

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Abstract

In this work microstructure analysis and microhardness testing was carried out on duplex stainless steel X2CrNiMoN22-5-3 after welding and annealing. Investigated stainless steel was welded using two welding procedures. The root of weld join was performed with TIG process and for filling SMAW process was used. Analysis of microstructure and microhardness was carried out before and after annealing. Annealing of welded joint was performed at temperature of 850 °C in duration for 60 minutes followed by cooling in air. After annealing, the samples were metallographic prepared. Optical microscopy and scanning electron microscopy with energy dispersive spectrometer were used for characterization of microstructure. Results shows that before annealing microstructure was consisted of ferrite and austenite, and after annealing presence of sigma phase was determined in weld metal. Microhardness values of samples before and after annealing was determined by Vickers method. A significant increase in microhardness values was observed in weld metal after annealing (from 294.9 HV1 to 357.0 HV1).

Keywords: *duplex stainless steel, microstructure, microhardness, welding, annealing*

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INTRODUCTION

Stainless steel is steel which has a resistance to staining, rusting, and pitting in the air. Generally, stainless steels define a chromium content higher from 11 % but less than 30 % [1]. They can be produced, with specific restriction in certain types, in conventional ways, and used in the as-cast condition. All the stainless steels types can be classified into five major classes: ferritic, austenitic, duplex, martensitic, and precipitation hardened.

The structure of duplex stainless steels is a structure which contain of ferrite and austenite, that is roughly half ferrite and half austenite [2]. The ratio of ferrite and austenite has an important effect on properties of these steels.

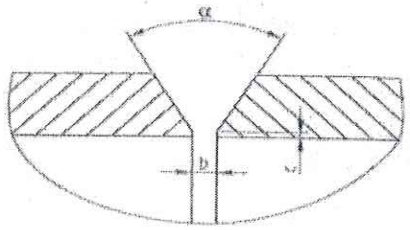
At annealing from 1000 °C to 1150 °C only austenite and ferrite are present in microstructure of duplex stainless steels. If temperature of annealing decrease below 1000 °C the duplex stainless steels are not more stable and other phases are formed, like carbides, chromium rich phases etc. The sigma phase is chromium rich intermetallic phase which most often occurs, and its compositions is the most Fe-Cr. Intermetallic sigma phase is hard and brittle phase which significantly affected on properties i.e. reduce impact energy, ductility and corrosion resistance of stainless steels [3,4].

Duplex stainless steels can be welded, and weld metal also contains a mixture of austenite and ferrite [5-8]. The solidification of weld metal occurs as ferrite and no austenite forms at the end of solidification process. The ferrite is stable in the solid state at elevated temperatures, and transformation to austenite carried out below the ferrite solvus. During transformation the austenite first formed along grain boundaries of ferrite [2]. Also, Folkhard [9] mentioned that the precipitation of sigma phase starts in weld metal after about 10-15 minutes after exposure to elevated temperatures 650 – 950 °C.

The aim of this study is microstructural characterization of a welded joint made from duplex stainless steel of grade X2CrNiMoN22-5-3 as well as analysis of annealing effect on microstructure and microhardness.

MATERIALS AND METHODS

Welding of duplex stainless steel (AISI/ASTM 2205, W.Nr. 1.4462, EN X2CrNiMoN22-5-3) was performed by TIG process for root of welded joint, and for filling by SMAW process on a 16 mm thick of plates, Fig. 1. The chemical composition of investigated steel was determined on an MA-ARL 8660 optical emission quantometer and presented in Table 1. The BOHLER CN 22/9 N-IG electrodes was used as filler material for TIG process while BOHLER FOX CN22/9 N-B electrodes was used for SMAW process, Table 2.



b = 2 mm, c = 2mm, α= 60°

Figure 1. Shematic illustration of joint preparation

Table 1. Chemical composition of investigated duplex stainless steel plates (determined by optical emission quantometer), wt.%

C	Mn	Si	Cu	V	Mo	Al	Cr	Ni	N
0.03	1.52	0.36	0.26	0.02	3.15	0.005	22.21	5.27	0.171

Table 2. Chemical composition of BOHLER CN 22/9 N-IG and BOHLER FOX CN22/9 N-B electrodes, wt.% [10]

	C	Si	Mn	Cr	Ni	Mo	N
BOHLER CN 22/9 N-IG	<0.015	0.4	1.7	22.5	8.8	3.2	0.15
BOHLER FOX CN 22/9 N-B	<0.03	0.5	1.1	22.6	8.8	3.1	0.16

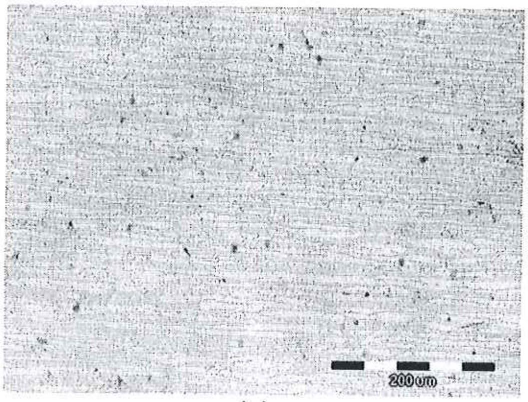
Annealing of welded duplex stainless steel X2CrNiMoN22-5-3 was performed in the electric resistance chamber furnace. The welded joint was annealed at 850 ° C for 60 minutes and then cooled in air.

Metallographic preparation for microstructural characterization was performed by grinding and polishing. The grinding was performed by a Phoenix Beta Buehler device with constant water cooling (papers 240, 400, 600 and 800), and polishing was performed with water solution 0.3 μm alumina (Al_2O_3). The samples were electrolytically etched with an etching solution consisting of 60 mL HNO_3 and 40 mL H_2O (1V for 20 seconds) to detect austenite. A solution consisting of 56 g KOH and 100 mL H_2O (2V for 10 seconds) was used to detect the sigma phase. Optical microscopy was performed on the Olympus GX51 at different magnification. Scanning electron microscopy (SEM) was performed on Tescan Vega 5136MM device equipped with an energy dispersive X-ray spectrometer (EDS).

The microhardness of the welded duplex stainless steel samples was measured by the Vickers method on Leica VHMT device, and the injection force was 9.804 N over a period of 10 seconds.

RESULTS AND DISCUSSION

Microstructural analysis of the welded duplex stainless steel was performed by optical and scanning electron microscopy. Optical microscopy of the base material (BM), weld metal (WM) and heat affected zone (HAZ) were investigated in detail, Figs. 2 and 3. By analysis of the optical micrographs can be observed the existence of two-phase duplex microstructure i.e. the presence of ferrite and austenite in microstructure. Also, it can be obtained that the ferrite is elongated in the rolling direction during the production of the plates of investigated stainless steel. After annealing at 850 °C, the microstructure is still mostly two-phase (ferrite and austenite), but sporadically some precipitates are observed in weld metal. The composition of these precipitates therefore needed to be determined by SEM and EDS analysis.



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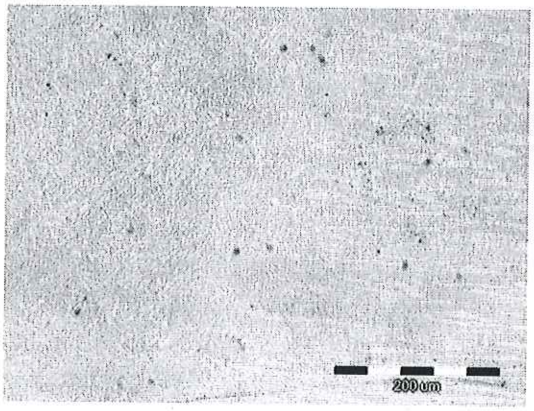

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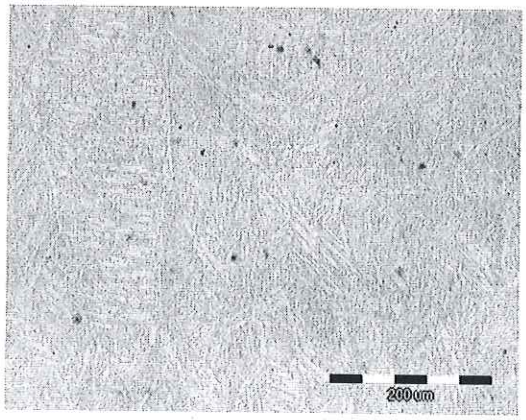

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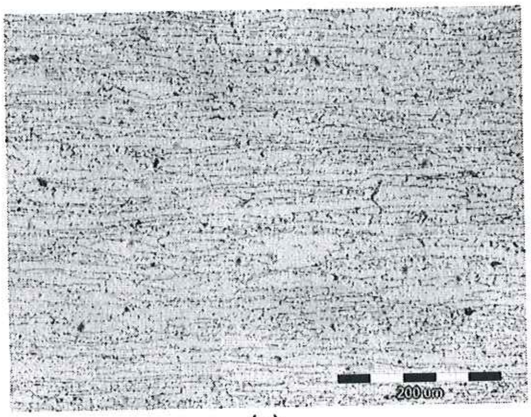
Figure 2. Optical micrographs of welded duplex stainless steel X2CrNiMoN22-5-3
a) base material; b) heat affected zone; c) weld metal; *magnification 200X*



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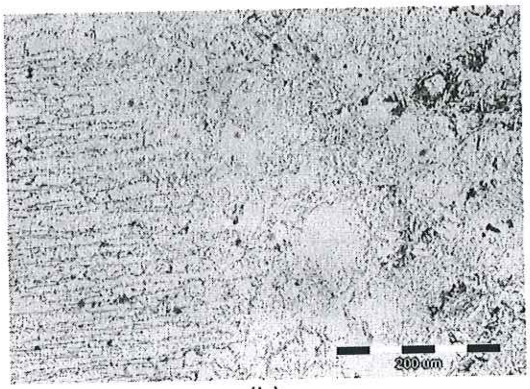

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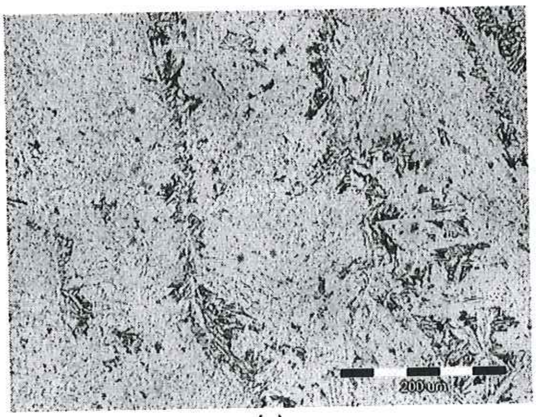

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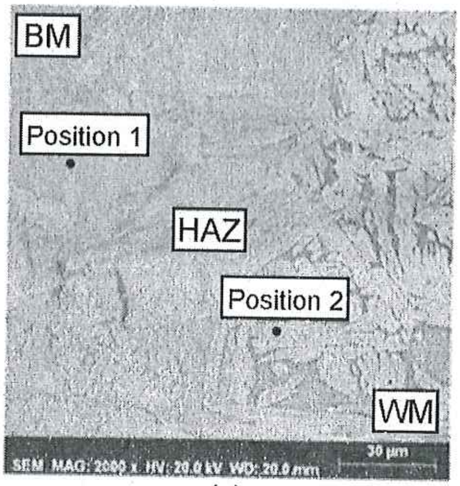
Figure 3. Optical micrographs of welded duplex stainless steel X2CrNiMoN22-5-3 after annealing 850°C/60`/air
a) base material; b) heat affected zone; c) weld metal; *magnification 200X*

Fig. 4a shows a SEM micrograph of the sample before annealing and this image includes the base material, the heat affected zone and the weld metal. On Fig. 4a are marked the positions for chemical composition testing by EDS analysis. Fig. 4b shows the EDS spectrum obtained for position 1. The chemical composition of the analyzed positions is given in Table 3. By analysis of data in Table 3 can be seen that the chemical composition of position 1 was 65.31 % Fe, 22.01 % Cr, 1.69 % C, 4.78 % Ni, 2.81 % Mn, 3.42 % Mo and 0.2% Si (in wt.%). The chemical composition of position 2 was 63.31 % Fe, 21.77 % Cr, 1.72 % C, 7.43 % Ni, 2.56 % Mn, 3.15 % Mo, 0.24 % Si (in wt.%). In SEM micrograph (Fig. 4a) the presence of precipitates was not observed and can be concluded that before annealing the only existed phases in microstructure were austenite and ferrite.



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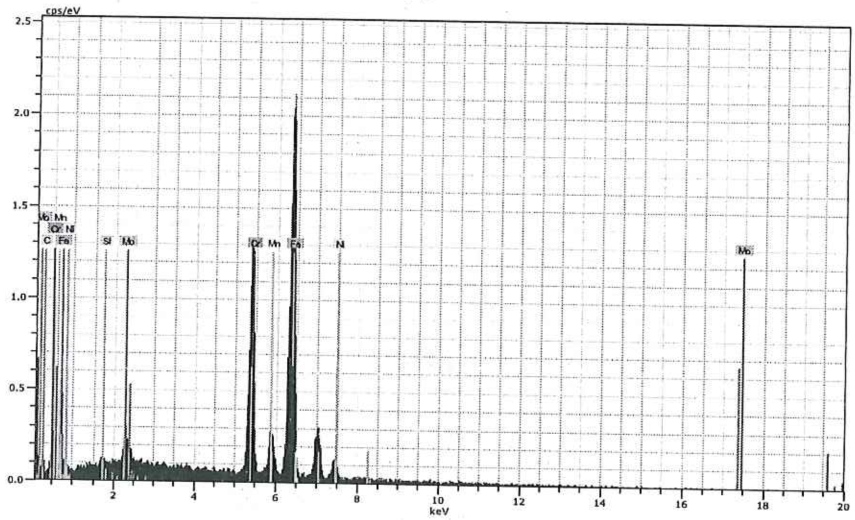


(b)

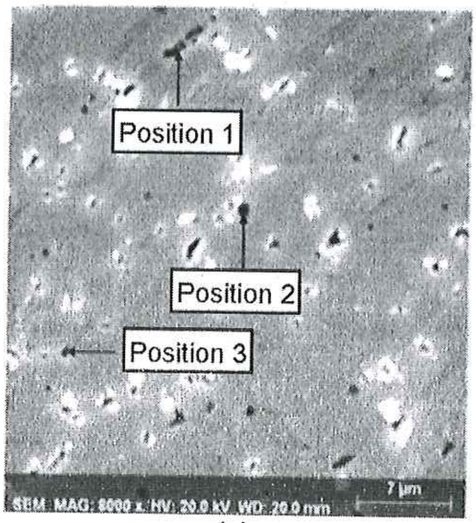
Figure 4. SEM micrograph (a) and EDS spectrum of position 1 (b) of the welded duplex stainless steel X2CrNiMoN22-5-3
BM - base material; HAZ - heat affected zone; WM - weld metal

Table 3. Results of EDX analysis of the welded duplex stainless steel X2CrNiMoN22-5-3, wt. % (positions marked at Fig. 4a)

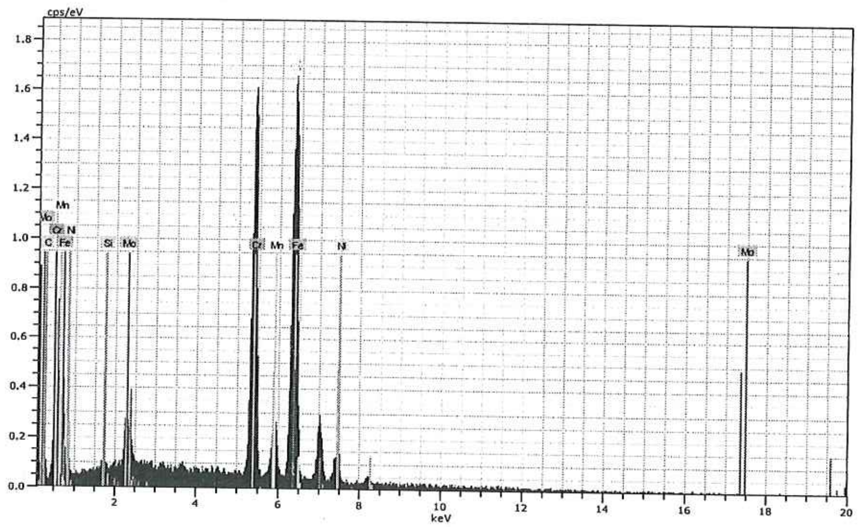
Position	Chemical composition, wt.%						
	Fe	Cr	C	Ni	Mn	Mo	Si
1	65.31	22.01	1.69	4.78	2.81	3.42	0.20
2	63.31	21.77	1.72	7.43	2.56	3.15	0.24

SEM analysis carried out after annealing confirmed the presence of the two-phase microstructure (ferrite and austenite), and existence of some precipitates but only in weld metal. Fig. 5a shows a SEM micrograph of the weld metal after annealing with marked positions for EDS analysis. Fig. 5b shows the EDS spectrum for the analyzed position 1. The chemical composition of the investigated positions is given in Table 4. By analysis of data in Table 4 can be obtained that the chemical composition of the position 1 was 55.39% Fe, 29.79 % Cr, 1.35 % C, 6.27 % Ni, 3.10 % Mn, 4.05 % Mo and 0.05 % Si (in. wt.%). The chemical composition of position 2 was 50.65 % Fe, 25.97 % Cr, 1.28 % C, 7.12 % Ni, 12.47 % Mn, 2.51 % Mo, 0.01 % Si, and the chemical composition of position 3 was 63.80 % Fe, 25.24 % Cr, 0.7 % C, 5.60 % Ni, 2.73% Mn, 1.91% Mo, 0.01 % Si. By detailed analysis of the chemical composition of all three investigated positions can be noticed an increase in content of chromium (25, 24-29.79 % Cr). Therefore, it can be concluded that the precipitates formed in the weld metal after annealing represent the intermetallic sigma phase. In stainless steels during welding or prolonged exposure to elevated temperatures can appear intermetallic phases. The sigma phase is most often formed intermetallic phase which occur in the weld metal. The sigma phase is a chromium-rich, hard and brittle non-magnetic intermetallic phase that has a tetragonal body centered crystal structure and its composition in high-alloy steels is often changed. There are about 50 different sigma phases, but the best known is the iron-chromium (Fe-Cr) sigma phase. When the chromium content in steel is less than 14-15%, the sigma phase does not occur if molybdenum, titanium or vanadium are not present [1]. The composition of the sigma phase can also be represented as $(FeNi)_x(CrMo)_yFeCr$ or $(FeNi)_x(CrMo)_y$. Its formation requires the diffusion of numerous elements and is favored by the presence of ferrite forming elements. The tendency to form the sigma phase increases with increasing chromium and molybdenum content, and molybdenum has 4 to 5 times stronger effect than chromium. In contrast, nickel, cobalt, aluminum, carbon, and nitrogen makes sigma phase precipitation difficult. The sigma phase is formed by the eutectoid transformation of δ-ferrite ($\delta \rightarrow \sigma + \gamma$). The sigma phase primarily occurs at the δ-ferrite/austenite phase boundaries and further expands into δ-ferrite [1, 4, 11-13]. In order for the sigma phase to occur, a local increase in the chromium content is required. Since chromium is more soluble in ferrite than in austenite, the precipitation and growth of the sigma phase in δ-ferrite first occur.

The precipitation of the sigma phase in duplex steel, which contains a relatively high chromium content and a low nickel content, is much faster than that of austenitic and ferritic steels. Hrivnak [4] mentioned that in duplex steels the sigma phase mainly contains 29-34 % Cr, and 3-5 % Mo which is similar to the composition of the sigma phase obtained by EDS analysis of the weld metal after annealing in this investigation (Table 4). Jimenez et al. [14] concluded that in duplex stainless steel the formation of the sigma phase from δ-ferrite takes place in two stages, i.e. first the sigma phase with a high content of chromium and molybdenum is formed, and then austenite is formed from the remaining δ-ferrite enriched with iron and nickel.



(a)



(b)

Figure 5. SEM micrograph (a) and EDS spectrum of position 1 (b) of the duplex stainless steel X2CrNiMoN22-5-3 weld metal after annealing 850°C/60`/air

Table 4. Results of EDX analysis of the duplex stainless steel X2CrNiMoN22-5-3 weld metal after annealing 850 °C/60`/air, wt. % (positions marked at Fig. 5a)

Position	Chemical composition, wt.%						
	Fe	Cr	C	Ni	Mn	Mo	Si
1	55.39	29.79	1.35	6.27	3.10	4.05	0.05
2	50.65	25.97	1.28	7.12	12.47	2.51	0.01
3	63.80	25.24	0.70	5.60	2.73	1.91	0.01

From the detailed analysis of measured values of microhardness can be observed that after annealing there is an increase in microhardness, Fig. 6. The largest increase in microhardness values is obtained in weld metal. Before annealing, the microhardness of the weld metal was 294.9 HV1 and after annealing was 357.0 HV1. This increase in microhardness may be associated with the formation of the sigma phase in microstructure.

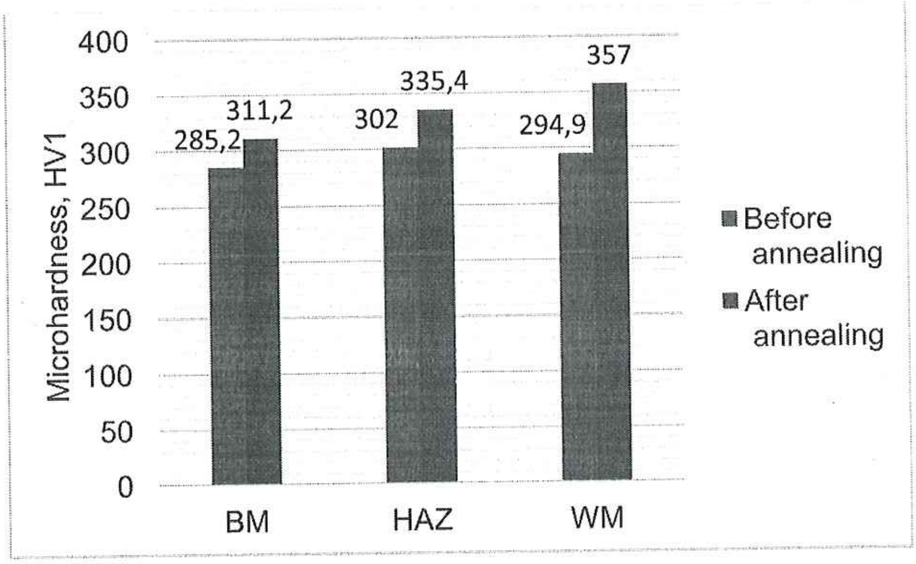


Figure 6. Average values of microhardness of the welded duplex stainless steel X2CrNiMoN22-5-3 before and after annealing 850°C/60`/air
BM – base material, HAZ – heat affected zone, WM – weld metal

CONCLUSIONS

On the basis of the microstructural characterization and the microhardness analysis of the welded duplex stainless steel X2CrNiMoN22-5-3 before and after annealing (850 °C/60`/air), the following can be concluded:

- The microstructure of the base material, the heat affected zone and the weld metal before annealing consisted of ferrite and austenite.

- Optical microscopy analysis showed that after annealing precipitates were sporadically observed in the weld metal.
- SEM analysis confirmed the presence of precipitates in the weld metal after annealing. EDS analysis showed that the precipitates formed in the weld metal represent the intermetallic sigma phase. The composition of the sigma phase was: 50.65-63.80 % Fe, 25.24-29.79 % Cr, 5.60-7.12 % Ni, 2.73-12.47 % Mn and 1.91- 4.05 % Mo.
- The microhardness of the base material, the heat affected zones and the weld metal were similar (285.2-302 HV1) before annealing. After annealing, the microhardness values increases. The higher increase was obtained in the weld metal (from 294.9 to 317.0 HV1), and this can be caused with the formation of the sigma phase.

Acknowledgements
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