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A. ZIEWIEC*, E. TASAK*, M. WITKOWSKA*, K. ZIEWIEC**

MICROSTRUCTURE AND PROPERTIES OF WELDS OF SEMI-AUSTENITIC PRECIPITATION HARDENING STAINLEES STEEL AFTER HEAT TREATMENT

MIKROSTRUKTURA I WŁASNOŚCI SPOIN STALI PÓŁAUSTENITYCZNYCH UTWARDZANYCH WYDZIELENIOWWO PO OBRÓBCE CIEPLNEJ

This paper presents the studies of the microstructure and properties of the welded joints made of 15-7Mo precipitation hardened semi-austenitic stainless steel welded by Tungsten Inert Gas. Microstructural changes in the heat treated welded joints was assessed. It was found that the joints of 15-7Mo steel in as welded state contain martensite, austenite and δ-ferrite. Scanning electron microscope study of the joints was carried out. The sub-zero and destabilization heat treatment were found to decrease or completely eliminate the austenite in the microstructure and increase hardness of the welded joint.

Keywords: semi-austenitic precipitate hardened stainlees steel, heat treatment, welds

W pracy przedstawiono wyniki badań mikrostruktury i własności złącz spawanych stali nierdzewnej półaustenitycznej utwardzanej wydzieleniowo 15-7Mo wykonanych metodą TIG. Oceniono zmiany mikrostruktury w procesie obróbki cieplnej. Stwierdzono, że w spoinach stali 15-7Mo po spawaniu występuje martenzyt, austenit i ferryt δ. Przeprowadzono badania SEM spoin. Obróbka podzerowa i obróbka destabilizacyjna obniżają lub całkowicie likwidują austenit w strukturze i podwyższają twardość.

1. Introduction

The semi-austenitic precipitation hardened stainless steels solidify with formation of the primary ferrite. The ferrite to austenite transformation occurs in these steels at high temperature. However, after cooling down to the room temperature, the ferrite still remains in the microstructure at the level of 5% to 15%. This is also in the 15-7Mo steel [1-3].

There are three stages during heat treatment of the precipitation hardened semi-austenitic stainless steels. The first stage consists of heating the steel to a temperature which results in formation of the austenite. During the second stage the austenite is destabilized for some period at the temperature required for precipitation of carbides from the solution, which causes increasing of the M_s temperature. This is followed by cooling to room temperature. As a result of this cooling the martensitic transformation proceeds nearly entirely. The third step consists of the reheating the steel to 566°C where the precipitation hardening occurs. This enables obtaining very high tensile strength. The alternative way for hardening some of the semi-austenitic stainless steels is solution heat treatment from ca. 950°C. Austenitic microstructure is obtained after cooling. Due to the solving of the carbides the M_s temperature is lowered to -70°C. In order to obtain a structure containing more martensite a cold treatment (sub-zero treatment) is necessary. The aging is carried out at ca 510°C. The other method for additional hardening of the precipitation hardened semi-austenitic steels is plastic deformation after the solution treatment. This results in transformation of the metastable austenite and increases yield strength to ca 1310MPa. Thus, the steel elements can be bent or deformed before aging. In this case only one heat treatment is necessary, i.e. the precipitation hardening with relatively low temperature of aging, usually 480°C. This approach is particularly useful in the production of the thin steel sheets. For these components it is possible to obtain a strength exceeding 1800 MPa. Most previous studies of the precipitation-hardened stainless steels focused on the impact of aging on the microstructure and mechanical properties of base metal [4÷8]. Only few works have reported on the properties of welded joint [9÷11]. For maintaining the mechanical properties of the steels on a good level the knowledge of the phase transformation occurring during rapid thermal cycles is required. Such cycles occur during welding of a steel and heat treatment after welding. The aim of this study was therefore to examine the changes in the microstructure of the 15-7Mo welded steel welded joint after the the different variants of heat treatment.

* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF METALS ENGINEERING AND INDUSTRIAL COMPUTER SCIENCE, AL. A. MICKIEWICZA 30, 30-059 KRAKOW, POLAND

** INSTITUTE OF TECHNOLOGY, PEDAGOGICAL UNIVERSITY OF CRACOW, UL. PODCHORĄŻYCH 2, 30-084 KRAKOW, POLAND

2. Experimental procedure

The samples were prepared from 1.0 mm×300 mm×75 mm sheet made of the precipitation-hardened semi-austenitic 15-8Mo steel. They were welded using TIG method with no binder. The stitch length was 50 mm. Chemical composition of the steel was the following: 0.009%C, 1%Mn, 1%Si, 15%Cr, 7%Ni, 2,5% Mo, 1%Al, 0.04%P, 0.03%S. After the welding the samples were precipitation hardened. Table 1 shows the number of samples and the variants of heat treatment for the particular samples.

TABLE 1

The variants of heat treatment after welding

Sample no	Annealing	Cold treatment	Aging
1	not applied	not applied	not applied
2	not applied	-196°C	not applied
3	760°C/1h	not applied	566°C/1h
4	not applied	-196°C	510°C/1h

The welded joints were sectioned, polished and etched with the two etchants. The first was electrolytic etching in 10% aqueous solution of CrO_3. The etching revealed phase boundaries and microstructural constituents. In order to reveal further details of the microstructure of the samples, they were gently polished and then chemically etched with the Berah II reagent. Its composition was the following: 48g of ammonium acid fluoride, 400 ml of hydrochloric acid, 800 ml of distilled water, 1 g of potassium metabisulfite. Thus the color etching of the microstructural components is obtained. The samples were then studied using light microscopy (LM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and hardness tests were performed. The LM observations was carried out on the samples etched electrolytically and after color etching. The aim of this study was to identify the microstructures formed in the heat affected zone and weld as well as to check the occurrence of cracks after welding. These investigations were performed on samples after welding and heat treatment. The hardness measurements were performed on the entire cross section of the welded joint. XRD analysis was performed in order to determine the quantity of the retained austenite in the welds after cold treatment and without cold treatment as well as after the heat treatment.

3. Results and discussion

3.1. Light Microscope study

Figures 1÷6 presents exemplary microstructures of the base material and the different areas of the welded joint. The microstructure of the base material observed under light microscope consists of δ ferrite surrounded by the austenitic-martensitic matrix (Fig. 1). The larger ferritic areas located at the grain boundaries within the martensite and austenite as well as inside the boundaries of the plate-like austenite are observed in the high temperature region of the heat affected zone (Fig. 2). The microstructure of the weld reveals the larger ferritic areas both in the form of net-like morphology and plate-like morphology as well as substantial flat areas on the martensitic-austenitic matrix (Fig. 3). The heat treatment i.e. the annealing at 750°C and aging at 566°C did not influence the apparent differences in the microstructure observed in the light microscope in comparison to the as-cast state. The cold treatment at -80°C resulted in the heat affected zone (Fig. 4) as well as in the weld (Fig. 5) the formation of the austenitic regions which are visible as the white areas. The heat treatment after cold treatment caused the re-appearance of the austenite (Fig. 6). The microstructure analyzed by the light microscope do not reveals clearly the differences in the microstructure of the welded joints after the different variants of heat treatment. In order to define the influence of the heat treatment on the quantity of the austenite in the microstructure of the weld X-ray diffraction analysis was performed.

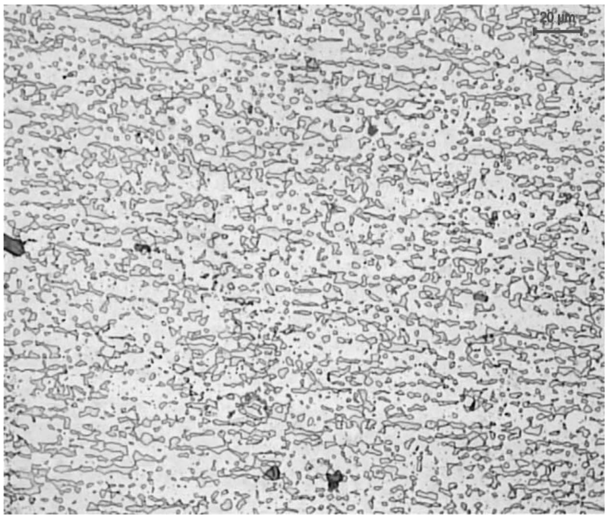

Fig. 1. Microstructure of the base material, δ ferrite in austenite matrix. Sample without heat treatment. Electrolytic etching in 10% CrO_3

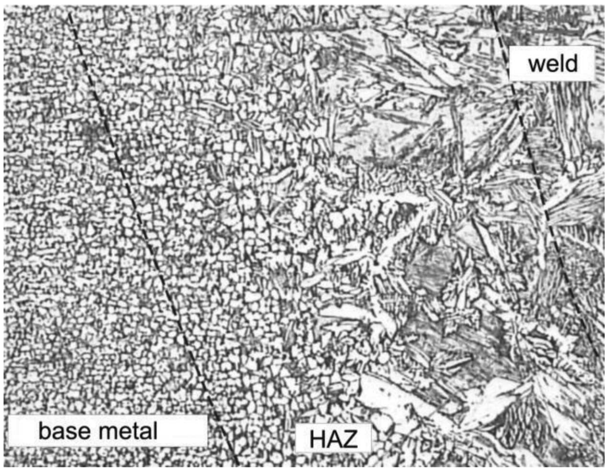

Fig. 2. Heat affected zone microstructure without heat treatment. Electrolytic etching

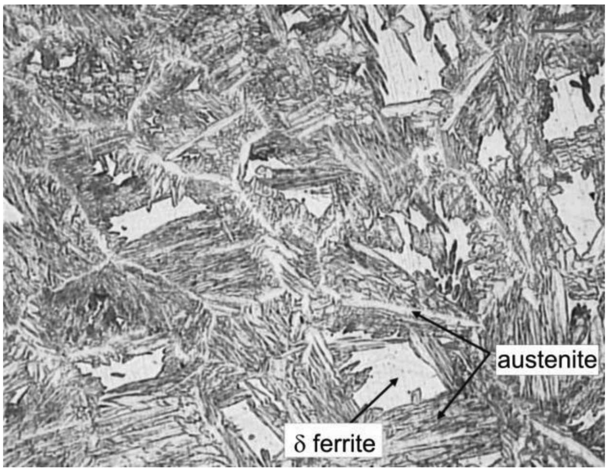

Fig. 3. Weld microstructure without the heat treatment. δ ferrite areas in the martensite and austenite on the grain boundaries as well as on the plate-like austenite inside grains

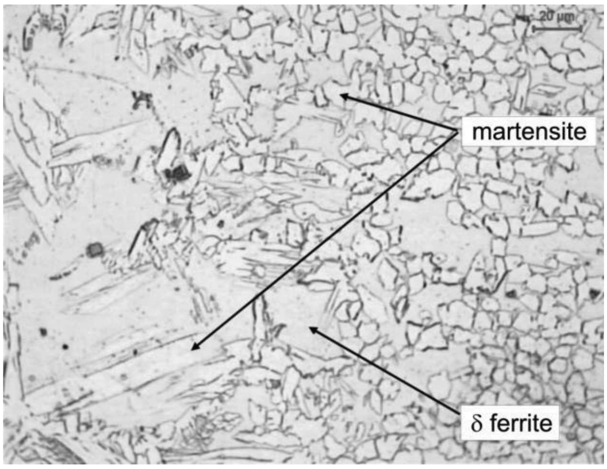

Fig. 4. Heat affected zone microstructure after cooling in the liquid nitrogen and precipitation hardening. Appearance of martensite and δ ferrite

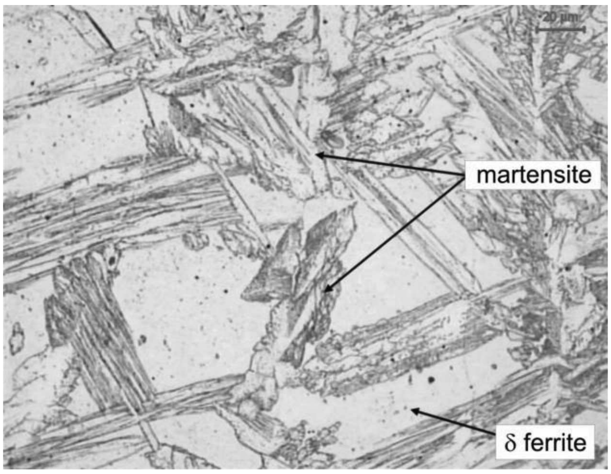

Fig. 5. Weld microstructure after cooling in the liquid nitrogen without heat treatment. Presence of martensite and δ ferrite in the microstructure. Electrolytic etching

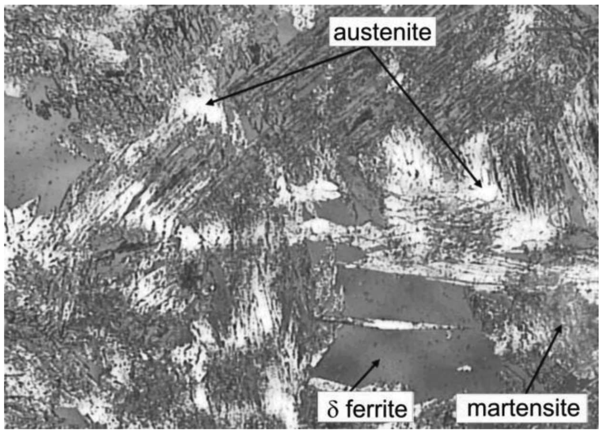

Fig. 6. Weld microstructure after cooling in the liquid nitrogen after precipitation hardening. The presence of martensite, δ ferrite and austenite on the grain boundaries. Etching in Berach II etchant

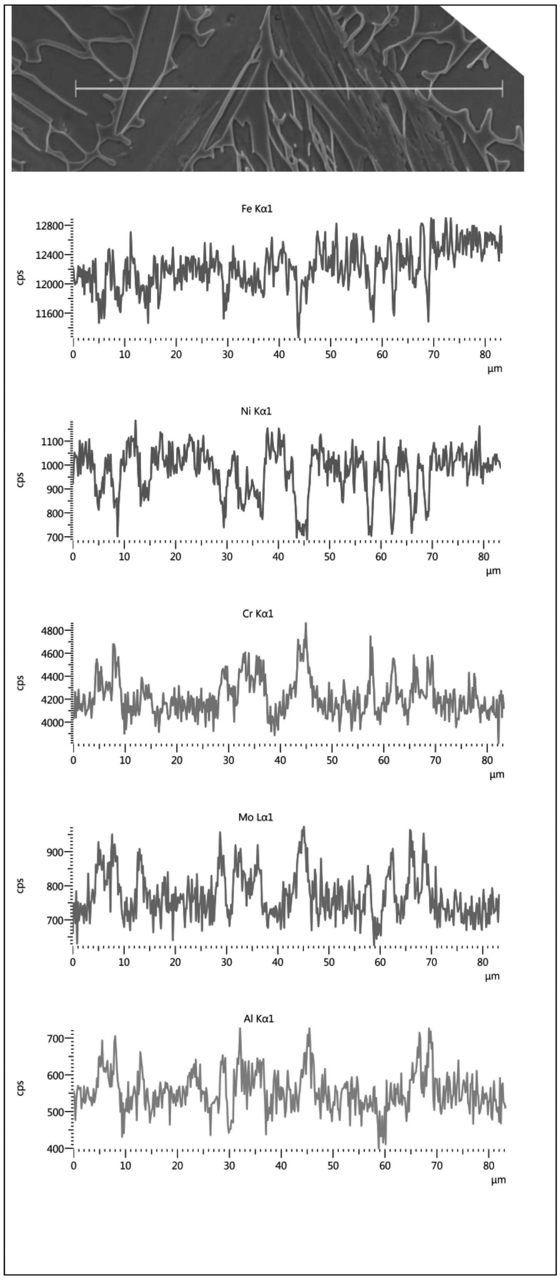

Fig. 7. The weld metal microstructure for the sample without the heat treatment. The places of EDS scan are marked with rectangles

3.2. SEM study

The line scan EDS analysis was carried out from the particular regions of the weld metal in as-welded state (Fig. 7). The brighter regions of the micrograph are enriched in chromium, molybdenium and aluminium. On the other hand the darker regions are enriched in nickel and iron. The EDS analysis of the regions from Fig. 11 shown in Table 2 indicates that the ferrite (areas B and C) contain ca 17.5% Cr, ca. 5% Ni, 3% Mo and 1.5% Al. The matrix is austenitic (areas A and D) and its exemplary compositions is 15.2%Cr, 7.5%Ni, 1.7%Mo and 0.9% Al.

Areas E and F represent plates of martensite formed during austenite-martensite transformation. Their composition is the same as the composition of austenite. The similar investigations were carried out on the remaining samples after the heat treatment. The EDS analysis for the particular regions indicated that the similar contents of the elements were found as in the as received state. Only after the heat treatment at the temperature range 760°C ÷560°C only ferrite and martensite are present in the microstructure.

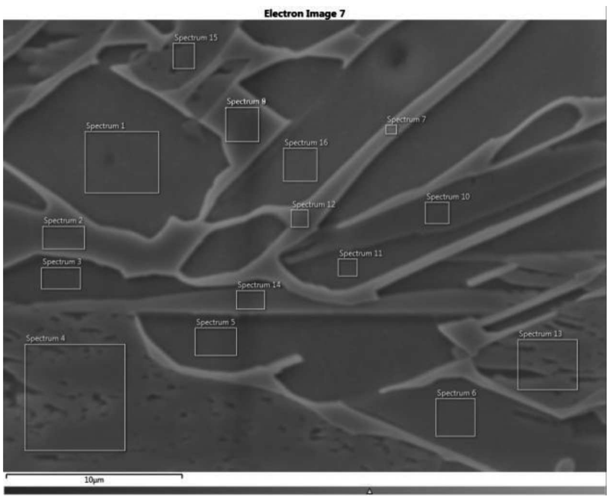

Fig. 8. Appearance of the microstructure observed using SEM with the analysed areas marked with rectactangles

TABLE 2

Chemical composition of the areas marked on the Fig. 8

Area	Cr	δ_{Cr}	Ni	δ_{Cr}	Mo	δ_{Cr}	Al	δ_{Cr}	Si	δ_{Cr}	Fe
	% wt.										
A	15,0	0,1	7,5	0,1	1,7	0,1	0,9	0	0,5	0	Bal.
B	17,6	0,1	5,0	0,1	3,0	0,1	1,3	0	0,6	0	Bal.
C	17,5	0,1	5,1	0,1	2,7	0,1	1,2	0	0,6	0	Bal.
D	15,3	0,1	7,3	0,1	1,8	0,1	0,9	0	0,5	0	Bal.
E	15,3	0,1	7,5	0,1	1,9	0,1	1	0	0,6	0	Bal.
F	15,1	0,1	7,3	0,1	1,6	0,1	0,9	0	0,5	0	Bal.

3.3. X-ray diffraction study

In comparison with the phase composition of the as-welded sample (sample no 1) the cold treated sample immersed in the liquid nitrogen (sample no 2) it could be clearly stated that in the sample no 2 there are traces of austenite. Therefore, the cold treatment caused transformation of austenite into martensite (Fig. 9). It is clearly seen that there is no austenite in the sample no 3 after the heat treatment at 760°C and at 566°C. However in sample no 4 cold treated and precipitation hardened at 510°C the austenite is appearing repeatedly.

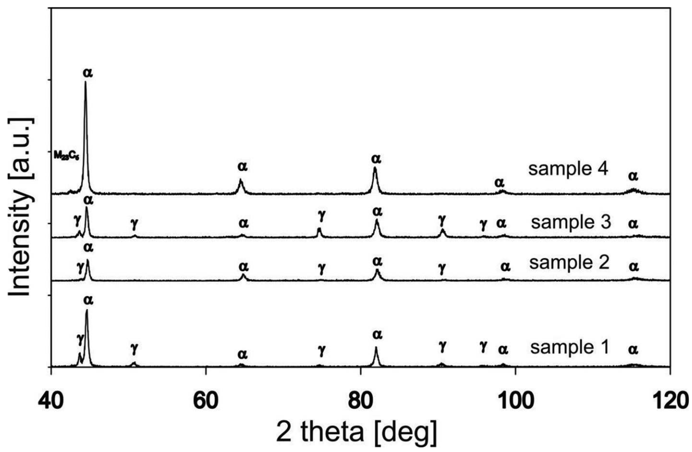

Fig. 9. X-ray diffraction of the welds. The ferrite and austenite occur in the weld immediately after welding (sample no 1). After the cold treatment (sample no 2) the presence of martensite, δ ferrite and traces austenite is observed. The microstructure of the sample no 3 consists only of the martensite and the ferrite. The presence of austenite is observed in the microstructure of the sample no 4

3.4. Hardness tests

The hardness tests were performed at mid-height of the weld. Figure 10 shows comparison of the results of hardness measurements for the samples without cold treatment that were subsequently heat treated and not heat treated. Figure 10 shows that the heat treatment increases hardness of the weld to 300HV5. This value is slightly higher than the hardness of the welded material that was annealed and had the hardness ca. 280 HV_5. Cooling the weld to the temperature of the liquid nitrogen causes transformation of the austenite into martensite in the heat affected zone and increases hardness number ca. 20 HV5. The precipitation hardening at 510°C causes further increase of the hardness level to ca 290 HV5 (Fig. 11).

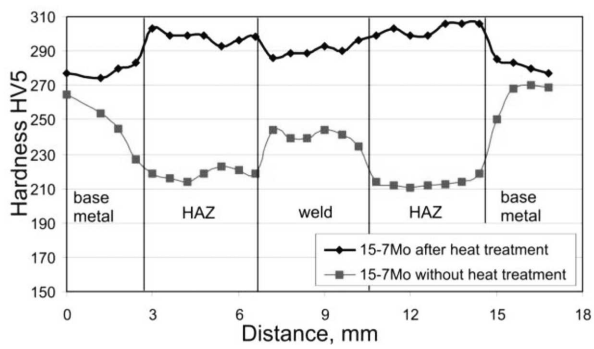

Fig. 10. Hardness distribution on the cross-section of the joint after welding without cold treatment and after heat treatment that consisted of annealing at 760°C and aging at 566°C

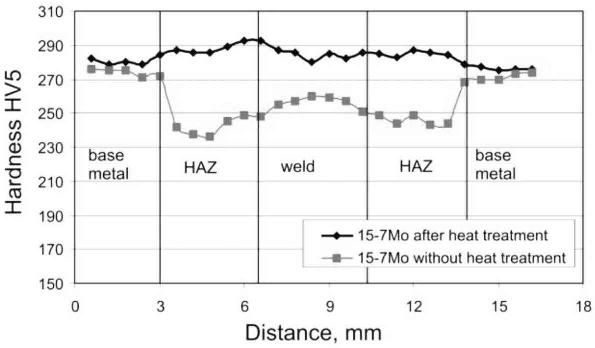

Fig. 11. Hardness distribution on the cross-section of the joint after cold treatment and aging at 510°C

4. Conclusions

The study showed that weld of the 15-7Mo semi-austenitic steel are not prone to hot cracking. They have austenitic microstructure with δ ferrite with hardness number 240 HV_5. Cold treatment in the liquid nitrogen causes transformation of the austenite into martensite with only traces of the retained austenite. The following process of the precipitation hardening carried out at 510°C causes formation of the reverted austenite. The presence of the austenite after annealing at 510°C exceeding the Ac_1 temperature and proceeding the inverse transformation of martensite into austenite at the regions where segregation occurred. The resulting austenite is enriched with the elements that segregate during solidification (carbon and nickel) and during the repeated cooling. Thus the austenite does not transform into martensite. The austenite is revealed in the microstructure using Berah II etchant. The hardness of the weld after cold treatment and precipitation hardening is ca. 285 HV_5. After the heat treatment destabilizing the austenite at 760°C and precipitation hardening at 566°C there is no austenite in the microstructure, in spite of the fact that the temperature of the treatment is higher. After such treatment, the lack of the austenite in the weld can be explained by the fact that during heat treatment at 760°C the precipitation of the chromium carbides and decreasing the carbon content at the grain boundaries and the decreasing segregation of nickel. The factors caused the increasing of the M_s and A_{c1} temperatures. The precipitation hardening at 566°C was below A_{c1} and did not caused formation of the austenite. The presence of the austenite in the microstructure after aging at 510°C only slightly influences the hardness of the weld in comparison with the hardness obtained after the heat treatment at 760°C + 566°C, after which there is no austenite in the microstructure. The drop of hardness associated with the formation of austenite may be compensated by the strong influence of the high dispersion hardening phases.

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