Surface remelting of 316 L+434 L sintered steel: microstructure and corrosion resistance

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Abstract This study presents arc surface remelting of three types of sintered stainless steels carried out in order to constitute a homogeneous microstructure in the surface layer which is free from open and interconnected porosity. The main aim of this treatment was to improve functional properties of the sinters analysed, especially their resistance to pitting corrosion. The sinters were obtained from powders of 316 L and 434 L steels. The PM austenitic-ferritic stainless steels are used mainly in the automotive industry, but their general application is still limited due to relatively poor corrosion properties when compared to casts or wrought components. This study used the gas tungsten arc welding (GTAW) process as a method of economical surface treatment. The effect of surface treatment was evaluated based on macro- and microstructural observations, energy-dispersive X-ray spectroscopy (EDX) analysis, X-ray phase analysis, measurements of surface roughness and electrochemical examinations. It was found that a cellular or mixed cellular and dendritic structure was formed in the remelted zone of the sinters after remelting. X-ray analysis demonstrated that application of remelting contributes to formation of the austenitic phase in the surface layer. The corrosion resistance of the remelted surface layers was evaluated using polarization tests in 0.5 M NaCl solution. It was found that arc surface remelted layers exhibit much better anticorrosive properties than sinters without surface treatment. Microstructural observations of the surface of specimens after electrochemical tests showed only a few single pits

in the remelted layer, while the surface of initial sinters was much more corroded.

Keywords Sintered steel · Surface treatment · Pitting corrosion · Electrochemical method

Introduction

Sintered steels obtained by means of powder metallurgy have been widely used in a number of industrial sectors, especially due to the opportunities for manufacturing of components with complex shapes and small dimensions, whereas using the conventional methods is difficult, expensive or even impossible [1, 2]. Corrosion-resistant sintered steels are used, for example, in the automotive industry, especially in manufacturing of parts for exhaust and braking systems which operate under corrosive and oxidative conditions [3].

Resistance of steels to aggressive environments is determined by a number of factors, especially chemical composition, microstructure, porosity and state of surface, which is particularly important in the case of sinters [4]. Most researchers agree that porosity is the main cause of deterioration of the anticorrosive properties in sintered steels compared to conventional cast and wrought grades. The presence of open porosity helps increase the surface of the material exposed to direct contact with the corrosion agents. Some authors, e.g. [5, 6], argue that pore morphology plays more important role in corrosion processes compared to the presence of pores. A particularly unfavourable effect might be caused by interconnected porosity. Channels that connect several pores help the corrosive medium propagate to the inside of the material. Furthermore, air access into pores is limited, which results in discontinuity of the passive layer. A particularly detrimental effect on corrosion resistance is from the electrolytic stagnation in the interconnected pores, with the formation of

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concentration cells. Flow of current through local cells occurs with intensive metal dissolution within pores. Consequently, rapid changes in pH are observed in the electrolyte, with increasing concentration of chloride and hydrogen ions inside the pores, which subsequently leads to transition of the material from a passive to active state. The phenomena that occur in sintered stainless steels usually lead to pitting or crevice corrosion [5–9].

Improved corrosion resistance can be obtained for high-density sinters and through minimizing open and interconnected porosity, especially in the surface layer. Another method is to stimulate changes in pore morphology and eliminate channels that connect pores [10].

Opportunities of thickening and elimination of porosity in surface layers are offered by, e.g. remelting process. The interest in the methods of surface modification using concentrated sources of energy has increased over the past two decades. Previous studies have demonstrated that constitution of the surface layer in corrosion-resistant sintered steel using laser has a positive effect on the functional properties of this type of materials [11–13]. Laser technologies are characterized by high precision, while a stable and concentrated laser beam, combined with dynamic cooling, allows for obtaining remelted layers with fine crystals [14–17]. Remelting represents a particularly justified type of surface modification in sintered alloy steels as it does not change the chemical composition of the material [18]. This leads to the presumption that sinter properties that result from chemical composition and sintering parameters should not be deteriorated. Laser remelting offers a number of advantages. However, for economic reasons, it belongs to expensive processes. Studies [19–23] discussed gas tungsten arc welding (GTAW) process as cheaper yet comparably efficient method of surface modification in engineering materials. Opportunities to control parameters such as current type and intensity, arc voltage, surface scanning rate and the choice of material and diameter of the electrode significantly affect the quality and properties of the surface layer modified. Arc method might represent an alternative solution compared to the laser technique.

The study presents the results obtained during the investigations of the effect of remelting using the GTAW process on

the microstructure and functional properties of the surface layer in sintered multiphase steels obtained from powders of 316 L austenitic and 434 L ferritic steels.

Material and methods

Examinations were carried out for specimens of sintered steels (316 L and 434 L). Mixtures of powders were prepared with three different proportions (see Table 1). The specimens were compressed at 720 MPa and then sintered at the temperature of 1,250 °C for 30 min in the dissociated ammonia medium (75 % H₂:25 % N₂) and cooled at the cooling rate of 0.5 °C/s. Table 1 presents the chemical composition of individual powders and sinters obtained in the study.

Porosity of 80A-20 F, 50A-50 F and 20A-80 F sinters evaluated using microstructural tests of unetched metallographic cross-section and the software for image analysis (ImageJ) was respectively 7.95 ± 1.61 , 2.52 ± 0.89 and 10.96 ± 2.75 %.

The surface of the sinters was remelted using a welding method (GTAW). The layout of the GTAW apparatus was presented in a study [23]. Remelting of the sample surface occurred at a constant scanning rate (340 mm/min) and current intensity of 30, 35 and 40 A. The most important variable parameter of remelting process was welding current intensity, which significantly determines arc heat energy. Table 2 presents the parameters used in the remelting process.

The effect of remelting on the quality and microstructure of surface layer was evaluated based on macro- and microscopic observations of remelted surfaces and etched metallographic cross-sections. Geometry of the remelted zone (depth and width of remelting) was measured. Optical microscopy examinations were carried out by means of Axiovert 25 microscope.

Analysis of chemical composition was carried out on the surface sections using scanning electron microscope Jeol JSM5400 with an energy-dispersive X-ray spectroscopy (EDX) device.

Hommel T1000 profilometer was used to determine the parameters that characterized surface topography of the specimens. Three measurements were carried out in contact with

Table 1 Chemical composition of the 316 L-434 L sinters studied (wt%)

Powder grade/sinter	Cr	C	Ni	Si	Mn	Mo	N	O	S	Fe
316 L (A)	16.70	0.025	12.30	0.90	0.1	2.20	0.060	0.30	0.005	Balance
434 L (F)	16.20	0.015	–	0.80	0.1	0.98	–	–	–	Balance
80 % 316 L+20 % 434 L (80A-20 F)	16.60	0.023	9.84	0.88	0.1	1.96	0.048	0.24	0.004	Balance
50 % 316 L+50 % 434 L (50A-50 F)	16.45	0.020	6.15	0.85	0.1	1.59	0.030	0.15	0.003	Balance
20 % 316 L+80 % 434 L (20A-80 F)	16.30	0.017	2.46	0.82	0.1	1.22	0.012	0.06	0.001	Balance

Table 2 Parameters of arc remelting of sinters

Sinter	Current intensity I [A]	Arc voltage U [V]	Total arc power q_c [W]	Arc linear energy E [kJ/m]	Scanning rate v [mm/min]	Gas type and feed rate
80A-20 F	30	10.2	306	54.06	340	Argon~10 l/min
	35	10.0	350	61.84		
	40	10.9	436	77.03		
50A-50 F	30	10.2	306	54.06		
	35	10.2	357	63.07		
	40	9.2	368	65.02		
20A-80 F	30	11.2	336	59.36		
	35	10.0	350	61.84		
	40	11.2	448	79.15		

the surface examined through coupling of the stylus with a differential measurement system. The averaged values are presented in Table 3.

X-ray phase analysis of sinters in the initial state and after remelting was carried out using Seifert XRD-3003 diffractometer with a cobalt lamp.

Electrochemical tests of corrosion resistance of the sinters were carried out in a three-electrode system by means of CH Instruments measurement station. The reference electrode was Ag/AgCl electrode, and the auxiliary electrode was represented by a platinum wire. The working electrode was sintered in initial state or after remelting. Before the experiment, the exposed slightly polished surface of each sample was cleaned with double-distilled water. The study was carried out in the 0.5 M NaCl solution at room temperature. Measurements were recorded in the $\log(i)=f(E)$ system in the range of potentials from cathode ($E=-0.6$ V) to anode values ($E=1.8$ V) for potentiodynamic curves and from $E=-0.6$ V to $E=1.1$ V for repassivation curves. All measurements were carried out with respect to Ag/AgCl electrode at a scanning rate of 0.01 V s^{-1} . Corrosion parameters were determined using extrapolation method. In order to obtain a comprehensive description of the behaviour of the specimens studied in corrosive media, macro- and microscopic observations of the surface exposed to the effect of the agent were carried out.

Table 3 Roughness in the sinter surface

Specimen description		Ra	Rz	Rmax
80A-20 F	Initial state	2.59	21.13	27.33
	After remelting	2.29	16.41	22.05
50A-50 F	Initial state	2.46	14.04	17.90
	After remelting	2.40	9.93	11.97
20A-80 F	Initial state	2.87	26.28	44.01
	After remelting	2.19	21.03	29.61

Ra arithmetic mean roughness deviation from the mean line, Rz maximum height of the profile, $Rmax$ maximum distance between a profile peak and the lowest valley

Discussion

Macroscopic observations were used to evaluate the effect of remelting on the quality of multiphase sinters. Using such criteria as good surface quality (lack of welding defects) and measurability of the width and depth of the fusion, further studies were carried out based on the sinters remelted at current intensity of 35 A. Welding current intensity of 30 A was too low to obtain fusion of the sinter surface. An irregular remelted band was obtained only in the 80A-20 F sinter, with the surface of two other sinters covered with oxide layer. The bands obtained on the surface of 80A-20 F and 50A-50 F sinters as a result of remelting at 40 A were characterized by substantial waviness and the presence of craters. Furthermore, microscopic observation of cross-sections also revealed the presence of cavities in the remelted layer. Positive macroscopic results were obtained for the bands remelted at 35 A, which were characterized by an even width in the entire section of arc effect and smooth surface without welding defects.

These findings were also confirmed by the profilometric examinations, with the results presented in Table 3. Values of Ra parameter show that, in the case of 80A-20 F and 20A-80 F sinters, the remelted surface was smoothed, whereas 50A-50 F revealed only an insignificant increase in Ra .

The sinters studied at the initial state were characterized by considerable microstructural non-homogeneity and varied porosity. Microscopic observations revealed that none of the sinters studied had a two-phase microstructure typical of conventional duplex steel. The presence of austenite, ferrite and a phase with martensite morphology was found, with its fraction increasing with the content of 434 L steel powder. There are few reports in the literature on the mechanisms that occur during sintering of duplex steels in the dissociated ammonia medium. Previous studies, e.g. [24, 25], have demonstrated that sintering in other media (hydrogen and vacuum) leads to diffusion of, e.g. Cr and Ni. A similar phenomenon was found during sintering of 316 L+434 L in the dissociated ammonia medium. Nickel, which is present in austenite grains

(with 12 % in the cases studied), diffused to ferrite grains, thus causing transformation of ferrite into a new phase with a complex acicular structure. Authors of the studies in [26–28] demonstrated that this phase is martensite (α) or martensite and ferrite ($\alpha' + \alpha$).

In applications where an overriding role is played by a surface layer (mainly the elements used in corrosive conditions), both the non-homogeneity and porosity of the microstructure should be treated as factors that reduce the functional properties of the surface. Figure 1a–c shows that remelting of the surface layer using a GTAW apparatus yielded a homogeneous microstructure of surface layer and eliminated porosity. In 80A-20 F and 50A-50 F sinters, formation of the primary structure occurred through cellular solidification. Nucleation and formation of the primary structure was epitaxial and occurred in partially remelted grains of base material (Fig. 2). Furthermore, melted grains created a narrow transitional heat-affected zone between the base layer and the remelted area. The geometry of the heat-affected zone largely depends on the dimensions of the pool. In our study, heat effect was present in a small part of the surface; therefore, the heat-affected zone was reduced to a narrow band. An increase in columnar crystals occurred consistently with the direction of heat transfer, thus perpendicular to the fusion line

(Fig. 1a, b). The remelted layer in 20A-80 F sinter exhibited a fragmented cellular-dendritic structure (Fig. 1c (c-1)). According to the generally accepted model of crystallization of Fe-Cr-Ni alloys, depending on the value of R_{Cr}/R_{Ni} (with R_{Cr} denoting Cr equivalent and R_{Ni} being Ni equivalent), liquid metal can crystallize as a primary austenite ($R_{Cr}/R_{Ni} \leq 1.25$), intercellular and interdendritic ferrite, and austenite ($R_{Cr}/R_{Ni} \leq 1.95$) and primary ferrite ($R_{Cr}/R_{Ni} \geq 1.95$) [29]. This assumptions show that crystallization of the remelted surface layer of the 20A-80 F sinter should lead to the formation of primary ferrite. However, microstructural observations (Figs. 1c (c-1), 2b and 4c) and X-ray phase analysis (Fig. 5) presented in this study lead to the conclusion that solidification caused formation of dendritic structure composed of austenite, primary ferrite and martensite. Furthermore, it was observed that typical cellular-dendritic structure was formed locally, mainly in the areas of higher temperature gradient (i.e., at the contact with base material; see Fig. 2b).

Figure 3 illustrates the values of width and depth of the fusions obtained. In 80A-20 F and 50A-50 F sinters, both the width of the band and remelting depth had similar values, whereas the surface layer of 20A-80 F was remelted to a less substantial degree, with remelting depth being nearly three times smaller compared to that of 80A-20 F and 50A-50 F

Fig. 1 Microstructure of the remelted surface layers in sinters **a** 80A-20 F, **b** 50A-50 F and **c**, *c-1* 20A-80 F; metallographic etched cross-sections

Fig. 2 Microstructure of the transition zone in the sinters **a** 50A-50 F and **b** 20A-80 F, metallographic etched surface sections (magnification $\times 200$)

sinters. The volume of the area affected by remelting should be related to thermal conductivity, which in the case of austenitic steel and duplex steel is low and amounts for both steel

grades to 15 W/m K, whereas this value for ferritic and martensitic steels ranges from 25 to 30 W/m K [30]. This explains the fact of obtaining similar geometry for remelted zones in 80A-20 F and 50A-50 F sinters, where the percentage of austenitic steel powder was 80 and 50 %, respectively. In 20A-80 F sinters, with a microstructure composed mainly of martensite, remelting was observed in considerably lower volume than that in other sinters, thus causing the remelted band to show lower width and depth (Fig. 3).

Figure 4 presents example SEM microstructure images in remelted zones with EDX analysis areas. Analysis of chemical composition (Table 4) revealed insignificant differences in concentration of the main alloy elements between two selected areas: cell's core and intercellular area. In conventional corrosion-resistant steels, solidification of molten metal (e.g. during welding) is accompanied by transitions controlled by diffusion processes. Chromium diffuses to ferrite, whereas nickel diffuses to austenite. Since ferrite in its primary structure forms between austenitic cells, cells' cores are enriched in Ni, with intercellular areas rich in Cr. In the sinters studied, EDX examinations revealed only insignificant desegregation of alloy elements. Cores of primary cells in the sinters studied were poor in Cr, Ni and Mo, with the highest differences found in Cr and Mo. This phenomenon can occur in the case of solidification of the weld with released primary austenite at specific high cooling rates [31, 32]. With regard to the austenitic structures, diffusion processes that occur in solid state are considered a little significant due to the low coefficients of diffusion (by one order lower than those recorded for ferritic structures) [31]. Results of EDX analysis revealed that processing of the surface layers of 80A-20 F and 50A-50 F sinters led to the creation of the austenitic structure. The microstructure of remelted surface layer of 20A-80 F was more complex than that in previous two cases (Fig. 4c). Analysis focused on the central part of cells (dendrites) and discontinuous intercellular areas (Fig. 4c). Table 4 shows that higher concentration

Fig. 3 Geometry of remelted bands (fusion depth/width)

Fig. 4 Analysis of chemical composition of intercellular space (spectrum 1) and cell (spectrum 2) in remelted layer of sinters: **a** 80A-20 F, **b** 50A-50 F, **c** 20A-80 F; surface sections (magnification $\times 3,500$)

of alloy elements can be found in intercellular areas, similar to 80A-20 F and 50A-50 F sinters.

Figure 5 presents the results of X-ray phase analysis of the sinters studied in initial state and after remelting of the surface layer. The study found that remelting of the surface layer of

Table 4 EDX-analysis of chemical composition of the remelted surface layer of sintered steels: 80A-20 F, 50A-50 F and 20A-80 F

Element	Intercellular areas (spectrum 1)	Cell (spectrum 2)
80A-20 F sinter, wt%		
Cr	18.38	16.07
Ni	10.88	10.19
Mo	3.23	0.98
Si	0.99	0.45
Fe	66.52	72.31
50A-50 F, wt%		
Cr	18.42	16.63
Ni	6.85	6.25
Mo	2.19	1.12
Si	0.99	0.45
Fe	71.56	75.55
20A-80 F, wt%		
Cr	19.05	18.15
Ni	3.69	2.78
Mo	1.00	0.82
Si	0.90	0.60
Fe	76.15	76.46

80A-20 F and 50A-50 F yielded austenitic phase (γ). Among the remelted layers, the substantial peak from ferritic phase was recorded in 20A-80 F sinter. This suggests the presence of primary ferrite (α) and/or martensite (α') in the remelted zone. Furthermore, remelted layers also exhibited minimal shift in peaks towards lower angular values with respect to the diffractograms obtained for the sinters in initial state, which results from the presence of lattice deformations.

In order to characterize the capability of inhibition of electrode processes and resistance of the surface layer to pitting corrosion for multiphase steel (remelted using the GTAW method), polarization curves (Figs. 6 and 8) were recorded in the solution of 0.5 mol dm^{-3} NaCl. In order to carry out electrochemical examinations and analysis of polarization curves (Fig. 6 and 8), the characteristic potentials were determined: corrosion potential, E_{corr} ; breakthrough potential, E_{pr} ; and repassivation potential, E_{rp} (Table 5). An additional indicator was the difference between potentials $\Delta E = E_{\text{pr}} - E_{\text{rp}}$ (size of the repassivation hysteresis loop). Figure 6 shows that remelting of the surface layer in sinters inhibits anode processes, which means the change in electrode potential towards higher values. Corrosion potential after remelting of sinters with a content of 80 and 50 % of 316 L steel sinter is shifted towards positive values compared to the value of corrosion potential recorded for the sinters in initial state. Nucleation potentials for pitting were 0.52 and 0.48 V, respectively. The shape of the polarization curves suggests that the breakthrough potential in remelted layers is similar to the sinters

Fig. 5 X-ray diffractograms of sinters in initial state and after arc remelting

in the initial state, whereas in the sinter containing 80A+20 F with remelted layer, the slope of the polarization curve over E_{pr} is significantly reduced than that of the other two sinters, which means slower growth of pitting.

The microstructure of sinters before and after remelting for 80A+20 F after exposure to the corrosion solution is presented in Fig. 7.

The course of anode polarization in the solution of 0.5 mol dm^{-3} of NaCl (Fig. 8) shows that they represent potentiokinetic curves for the metal that undergoes spontaneous passivation. The potentials E_{pr} , with breaking through the passive layer and nucleation of pitting for remelting the sinters with content of 20A-80 F and 50A-50 F are similar and amount to 0.52 and 0.48 V, respectively. The slope of linear

sections of anode curves with respect to the axis of abscissas over E_{rp} and rapid increase in current density reflect fast formation of pitting, with particular focus on initial sinters and sinters with modified surface (50A-50 F and 20A-80 F). The potentiokinetic studies revealed the correlation between the values of E_{pr} and E_{rp} potentials and the structure and phase composition of steels. The pitting corrosion rate increases with higher differences of ΔE potential. The lowest values of ΔE were found for the 80A-20 F sinter remelted using the GTAW method.

The review of the literature reveals that one particular benefit of commonly applied austenitic steels, e.g. 316 L compared to ferritic steel, lies in its high corrosion resistance, which improves with higher content of chromium, nickel and molybdenum [1, 2, 33]. The potentiokinetic tests of the sintered steels (initial state) showed that the sinters manufactured using the highest content of austenite powder have the best corrosion resistance. Similar pattern occurs for the sinters remelted using the GTAW method, with the best

Fig. 6 Potentiodynamic curves recorded for sinters in the initial state and after remelting. Electrolyte, 0.5 mol dm^{-3} NaCl; scan rate, 10 mV s^{-1}

Table 5 Results obtained for electrochemical measurements of the sinters studied

Material designation	E_{corr} , V	i_{corr} , mA cm^{-2}	E_{pr}	E_{rp}	ΔE
Initial state					
80A-20 F	−0.35	0.015	−0.05	−0.15	0.10
50A-20 F	−0.36	0.025	0.05	−0.08	0.13
20A-80 F	−0.39	0.030	0.18	−0.20	0.38
Remelted layer					
80A-20 F	−0.23	0.009	0.28	0.20	0.08
50A-20 F	−0.27	0.005	0.48	0.18	0.30
20A-80 F	−0.30	0.006	0.52	−0.10	0.62

E_{corr} , corrosion potential; i_{corr} , corrosion current density; E_{pr} , break-through potential of passive layer; E_{rp} , repassivation potential; ΔE , difference between potentials (size of repassivation hysteresis loop)

Fig. 7 Microstructure of surface of 80A-20 F after exposure to 0.5 M solution of NaCl: **a** initial state, **b** after remelting

anticorrosion properties recorded for the specimen with 80 % of austenite.

With regard to the increased resistance to corrosion, the most basic benefit of the sinters modified with the GTAW method is reduction in porosity of the surface after remelting. The attack begins in the pores and becomes more aggressive because it moves towards the inside of the material. Nucleation and propagation of crevices is associated with the creation and development of local solutions inside cavities and pores. The presence of porosity favours a differential aeration in crevices and differential concentration of protons.

There are active conditions inside the pores, and active-passive cells are created. The beginning of crevice corrosion in sintered materials is initiated with enrichment of metal ions, mainly chromium, inside the pores, which leads to acidification as a result of hydrolysis. High concentration of chlorides and acidic hydrolysis in the pores is likely to cause local metal dissolution.

In sintered duplex stainless steels, pitting occurs in the open porosity and tends to propagate towards the inside of the material [34–36].

Fig. 8 Repassivation curves recorded for sinters in the initial state and after remelting (*inset*). Electrolyte, 0.5 mol dm⁻³ NaCl; scan rate, 10 mV s⁻¹

Conclusions

This study presents the results of the investigations of the effect of remelting using the GTAW method on the microstructure and anticorrosive properties of surface layer in multiphase 316 L+434 L sintered steel. The results obtained during the experiments lead to the following conclusions:

- Application of arc remelting reduced surface roughness and contributed to thickening of the surface layer and elimination of the open porosity, which helped to improve corrosion resistance of the sinters studied.
- Rapid solidification led to the formation of homogeneous cellular structure (20A-80 F, 50A-50 F) or cellular-dendritic structure (20A-80 F). It was also found that alloying elements migrated towards cell boundaries rather than cell centres, which resulted in slight differences in the chemical composition between these areas.
- The microstructure of the remelted surface layer of 80A-20 F and 50A-50 F sinters exhibited the presence of the austenitic phase, while austenitic and ferritic/martensitic phases were found in the 20A-80 F sinter.
- Improvement of pitting corrosion resistance of superficial remelted sinters was caused by the elimination of porosity in the remelted layer; the best corrosion resistance was observed for the 80A-20 F sinter after surface treatment (the highest potential and the lowest ΔE).
- The results obtained from electrochemical examinations show that remelting of the sinters studied significantly improved corrosion resistance: the passive range was extended, the potential was shifted towards positive values and the value of corrosion current was reduced.

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