MICROSTRUCTURE AND PROPERTIES OF SURFACE REMELTED PM STAINLESS STEEL

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Abstract

The aim of this study was to examine the effects of surface treatment with high energy sources on the microstructure and properties of austenitic-ferritic stainless steel sintered in dissociated ammonia. The multi-phase sinters examined in the study were prepared from two types of water atomized steel powders: 304L and 434L. Surface remelting was conducted using two devices: electric arc (GTAW method) and Nd:YAG pulsed laser. The following research methods were used in order to determine microstructure and basic properties of sintered steel after remelting: optical microscopy, X-ray phase analysis, microhardness and surface roughness measurements, tribological and electrochemical tests. It was found that remelting process leads to formation of the homogeneous structure of surface layer without porosity. The microstructure of laser remelted surface layer was finer than after arc remelting but in both cases X-ray analysis revealed only the presence of austenitic phase. The microhardness of laser-treated layer was slightly higher (322 HV 0.05) than after arc process (312 HV 0.05). The measurements of surface geometry parameters (Ra, Rz, Rmax) showed that roughness obtained after pulsed laser remelting is much higher compared to the sinter in the initial state as a consequence of relief. Wear resistance in the sintered steel after arc remelting was better compared to laser-treated specimens. The major wear mechanism in sinters after remelting was adhesion while in the case of sinter in the initial state, wear was caused by abrasion. Corrosion resistance was higher for sinters after surface treatment as a result of porosity elimination and structure homogenization.

Keywords: Sintered stainless steel, surface layer remelting, Nd:YAG laser treatment, GTAW method

1. INTRODUCTION

Nowadays, surface engineering represents one of the most resilient and most promising fields of science and technology. This results from the fact that in practice, for many construction materials, functional properties cannot be improved any more through only development of the microstructure and chemical and phase composition. Modification of surface layer allows for a significant improvement in properties of already known materials, thus elongating their functional life. In some applications, this offers real opportunities for replacing expensive materials with special properties with cheaper materials with adequately formed surface [1-3].

A dynamic development of methods of surface treatment of materials that use concentrated heat sources has been observed in recent years. The most frequent lasers used in surface treatments are continuous wave lasers (e.g. CO_2) or pulsed lasers (usually Nd: YAG). The remelting treatment is performed using a beam or pulse affecting a small surface, which leads to local heating, melting and quick, often directional crystallization [4, 5]. The cost of the apparatus for laser treatment is relatively high [2]. However, the use of laser methods offers several advantages, including precision, good surface quality, repeatability and opportunity of process automation [6, 7]. This allows for remelting of the surface layer also in the case of small components with complex shapes. Studies in the field of remelting treatment of metallic materials [e.g. 8-10] have reported that remelting of metal surface can be successfully performed also by means of much cheaper heat sources used in welding (such as GTAW).

The studies available in the literature have demonstrated that remelting of steel allows for improvement of properties of the surface layer (such as anticorrosion, tribological and fatigue properties) through fragmentation and homogenization of the structure or stimulation of other favourable changes without affecting core properties [5, 11, 12]. In the case of sintered steel dedicated for working in contact with corrosion media, e.g. in cars, porosity represents a serious problem. Open porosity increases the surface of material exposed to direct effect of the aggressive environment. Furthermore, the pores connected with each other by means of channels are also observed in sinters. The complex shape of pores and uneven access of air is conducive to corrosion factor and increase in chloride ion and hydrogen ion concentration inside voids. Consequently, creation of a tight passive layer on the surface is impossible and steel is transformed into the state of active dissolution [13-15].

The focus of the examinations discussed in this study was sintered corrosion-resistant austenitic-ferritic steel. The most of previous publications on surface remelting of stainless steels have focused on solid materials. There are only few studies [e.g. 16, 17] that have discussed the effect of surface remelting on the microstructure and properties of corrosion-resistant sintered steel. This study attempted to effectively use concentrated heat sources (laser method Nd: YAG and arc method GTAW) to obtain a continuous surface layer with homogeneous microstructure and improvement of basic properties of sintered austenitic-ferritic steels.

2. MATERIAL AND METHODS

The specimens for the examinations were obtained from water-atomized powders of 304L steel (18.9 % Cr, 11.2 % Ni, 0.9 % Si, 0.1 % Mn, 0.013 % C) and ferritic 434L steel (16.2 % Cr, 0.98 % Mo, 0.8 % Si, 0.1 % Mn, 0.015 % C). Powders mixed with 50:50 with addition of 1% of Acrawax C lubricant were compacted uniaxially with 720 MPa. The molded pieces were sintered at the temperature of 1250 ˚C for 30 minutes in the dissociated ammonia medium and cooled down with cooling rate of 0.5 ^{o}C/s. Sinters in the initial state were characterized by a non-uniform microstructure composed of austenite and multi-phase acicular component.

The remelting treatment of sintered steel was performed by means of GTAW arc method with constant surface scanning rate of 340 mm/min and changing welding current intensity, with its values ranging from 30 A ÷ 50 A. The shielding gas was argon, with the flow set at ~ 10 l/min. Nd:YAG pulsed laser with wavelength of 1064 nm was employed for laser remelting of surface layer of the sinters. The parameters were selected experimentally, by generating the pulses with power of 3.5 kW, 5kW and 7.5kW and changing the degree of pulse overlapping from 50% to 90%. The laser spot size was 1.5 mm.

The basic criterion for selection of the specimens for further examinations was quality of surface of the remelted areas and similar width of the bands obtained, which were evaluated based on the macroscopic examinations and examination of surface topography. The basic roughness parameters i.e. Ra (mean arithmetic deviation of profile ordinates from the mean line), Rz (mean roughness height from 10 points) and Rmax (maximum difference between the highest peak of the profile and the lowest depth) were determined by means of Hommel T1000 profilometer. Length of the measurement section was 4.8 mm. Five measurements were performed along the remelted band.

The microstructure testing was carried out on etched metallographic cross-section. The geometry (remelting depth and band width) was measured for individual remelted bands.

Identification of phase composition of remelted layers and sintered steel without treatment was carried out using Seifert 3003 T-T X-ray diffractometer with a cobalt lamp with characteristic radiation wavelength of $\lambda_{CoK\alpha}$= 0.17902 nm. The measurements were carried out for angle range of 2θ= 30÷120°.

Vickers method (with the load of 490 mN) was employed to measure microhardness of remelted surface layers. Results of measurements were presented in the form of distribution of the values of microhardness vs. distance from the specimen surface.

Examinations of friction wear resistance were carried out using T-05 block-on-ring wear tester. The tests were performed for dry sliding contact. Test parameters were selected experimentally: F_N ≈49.05 N, rotational speed of the roll: 3.55 rps. Evaluation of specimen wear was carried out using an analytical balance with measurement accuracy of 0.00001 g. Test duration was 120 minutes (8 cycles, 15 minutes each). Total friction distance was ~2809 m. Coefficient of friction for each specimen was determined based on the following relationship:

$$\mu = \frac{T}{F_N} \quad (1)$$

where: μ- coefficient of friction, T- friction force, F_N- load.

Electrochemical examinations of corrosion resistance of sinters were carried out in the solution of 0.5 mol*dm^{-3} NaCl at ambient temperature. CH Instruments measurement station with conventional three-electrode measurement setup was employed. Corrosion current density i_{kor} and corrosion potential E_{kor} were determined based on the recorded potentiodynamic curves using the graphical method of curve extrapolation.

3. RESULTS AND DISCUSSION

The remelted bands were then subjected to macroscopic evaluation, with the main criteria including band continuity, comparable width and relatively smooth surface without craters. These requirements were met by the bands remelted using arc method with current intensity of 35 A, whereas in the case of the laser method, this was observed for the bands remelted with pulsed laser with power of 5kW and overlapping of 85 %. **Fig. 1** illustrates the examples of macroscopic images of the remelted bands.

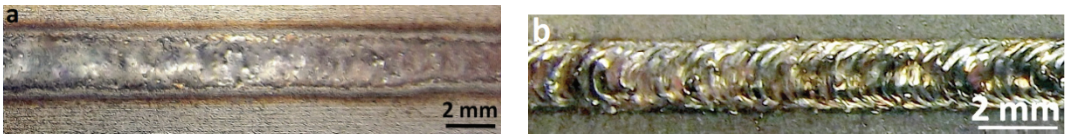

Fig. 1 Macroscopic images of remelted bands: (a) GTAW method; (b) laser method

Microstructural examinations revealed that in the case of arc surface treatment, the surface layer of the sintered steel showed a homogeneous cellular-dendritic structure, typical of the alloys that crystallize with inclusions of primary austenite. Presence of columnar crystals oriented according to the direction of heat transfer and cells with the shape similar to equiaxed (**Fig. 2a**) was observed. A transient zone with thickness of less than 50 µm was formed at the contact of the remelted layer with core material. This zone (see **Fig. 2b**), obtained through melting the core material, was the location of nucleation of primary structure crystals. In the case of laser treatment, a cellular-dendritic structure was also formed in the surface layer. Cooling rate in the case of laser treatment was substantially higher compared to the arc method, with natural consequence being formation of much finer structure (**Fig. 3a**). Similar to GTAW method, nucleation and growth of cells was also of epitaxial character, although a distinct transient zone was not observed (**Fig. 3b**). Furthermore, it was found that both methods ensured development of a continuous surface layer without voids.

X-ray phase analysis (**Fig. 4**) demonstrated homogenization of the microstructure in the area of remelted surface layers. Reflexes corresponding to angular locations of ferritic and austenitic phases were observed in the X-ray image recorded for the sinter in initial state, whereas X-ray images for the remelted layers did not show presence of ferritic phase as its formation was limited through rapid crystallization. Furthermore, substantial reinforcement of maximum for (002) surface was observed for the surface layer obtained with pulsed laser, which suggests texturization of the primary structure and preferred increase in crystals according to the crystallographic direction.

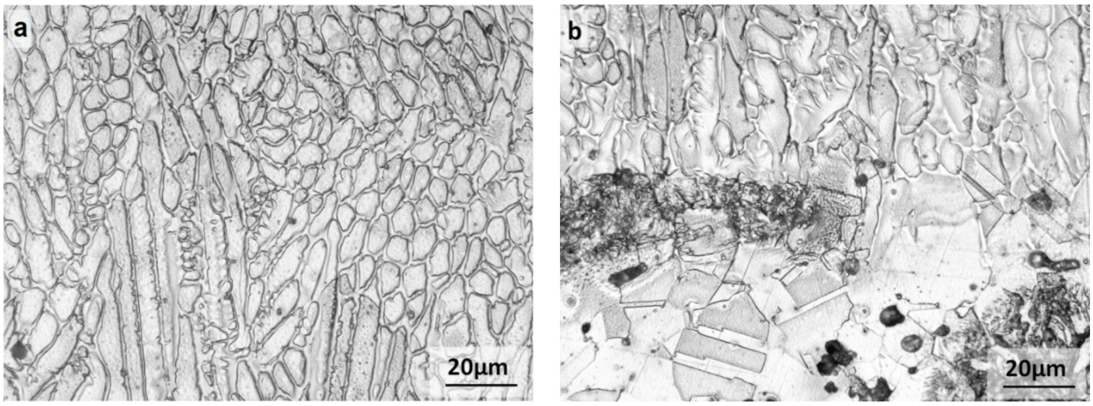

Fig. 2 Microstructure of the layer remelted using GTAW method: (a) central part of the band; (b) transient zone

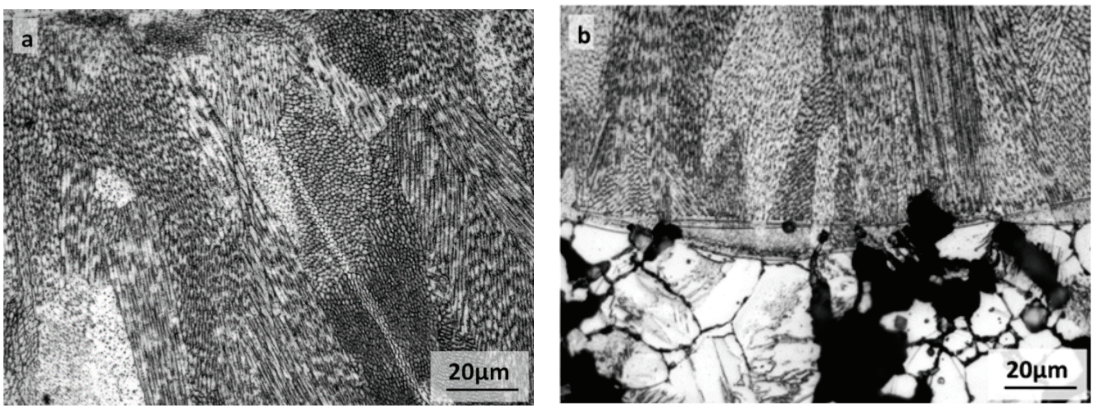

Fig. 3 Microstructure of the layer remelted using laser method: (a) central part of the band; (b) transient zone

Microhardness of the remelted layers (**Fig. 5**) was comparable both in the case of arc and laser method, while its mean value was 312 HV0.05 and 322 HV0.05, respectively. Higher microhardness of the layer modified with laser is likely to have been caused by substantial fragmentation of the structure, thus greater contribution of cell borders which, as a result of crystallization with austenite inclusions, show usually elevated content of alloy elements (Cr, Mo, Si) that increase hardness. Microhardness measurements also revealed increased microstructural homogeneity in the area of the remelted layers compared to the core material.

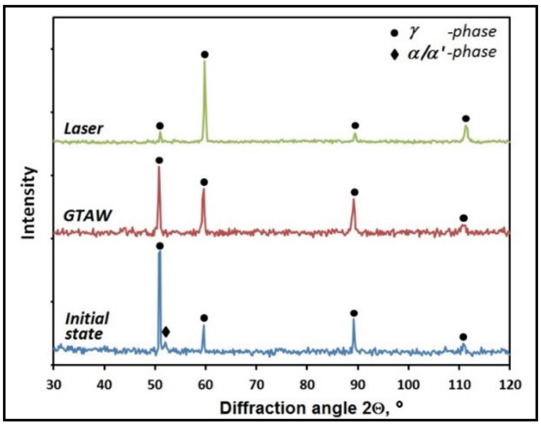

Fig. 4 X-ray diffractograms of sinter in initial state and after surface treatment

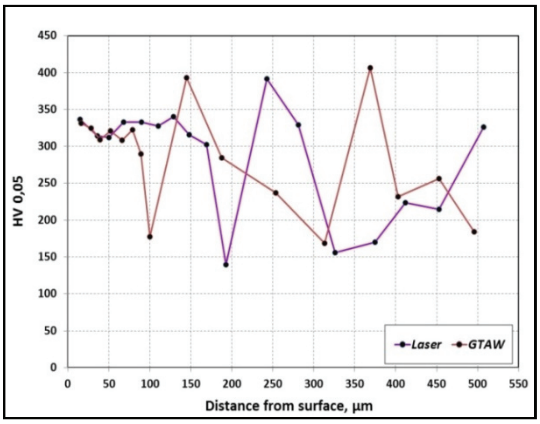

Fig. 5 Distribution of microhardness vs. distance from the surface

Profilometric examinations of specimens' surface showed that, after remelting treatment using GTAW method, roughness was comparable with the sinter in the initial state, which was demonstrated by similar values of Ra, Rz and Rmax for both cases studied (see **Table 1**). A substantial increase in roughness was found after laser treatment, which was connected with presence of relief on the surface. This development of surface is typical for pulsed laser treatment, where individual remelting points overlap on each other and form flash. In the case of the arc method, continuous operation mode of the device guaranteed equal distribution of liquid metal in the remelted band.

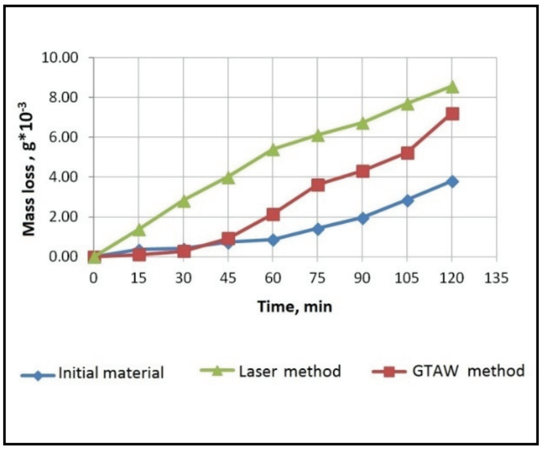

Fig. 6 Kinetics of mass loss of sintered steel

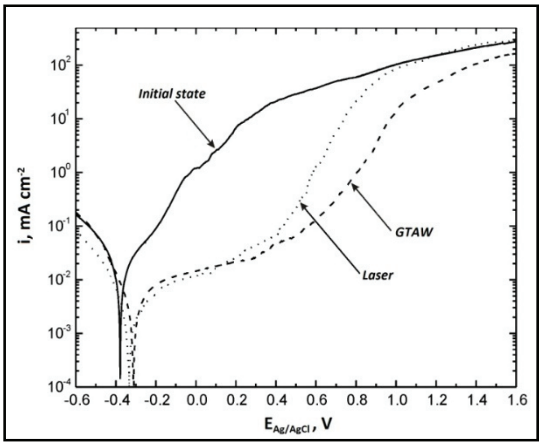

Fig. 7 Potentiodynamic curves recorded for sinter in the initial state and after remelting. Electrolyte 0.5 mol dm^{-3} NaCl; scan rate 10 mV s^{-1}

It was found based on tribological examinations that the treatment used in the study causes a change in the wear mechanism in the sinter, which results from homogenization of the structure of the surface layer and changes in its phase composition. In the case of the sintered steel without treatment, the wear of material occurred through abrasion connected with chipping and transfer of particles, whereas the dominant pattern after remelting was adhesion, manifested in delamination and grinding of material. Deterioration of the friction wear resistance after remelting (**Fig. 6**) was mainly connected with presence of austenitic phase in surface layers. In the case of the sinter in initial state, a hard and non-homogeneous acicular component was limiting the depletion of material. Values of the coefficient of friction were comparable for all the specimens tested (see **Table 1**).

Table 1 The value of surface roughness, wear resistance and electrochemical parameters

Surface state	Roughness parameter, µm			Wear process parameter		Corrosion parameter	
	Ra	Rz	Rmax	Total mass loss, g*10^{-3}	Coefficient of friction	i_{corr}, mA cm^{-2}	E_{corr}, V
Initial	1.68±0.29	15.32±3.30	23.49±2.40	3.81	0.62	0.01	-0.37
Remelted using GTAW method	1.63±0.31	14.34±5.13	20.76±7.99	7.20	0.58	0.003	-0.31
Remelted using Nd:YAG method	6.16±0.9	34.63±3.84	45.15±3.80	8.56	0.57	0.002	-0.34

Electrochemical examinations revealed an improvement in corrosion resistance of sintered steel after remelting with both GTAW and laser method. As results from the potentiometric curves recorded in the study

(**Fig. 7**), corrosion current densities i_{kor} were much lower compared to the sinter in initial state, whereas values of corrosion potential E_{kor} were moved towards positive values (see **Table 1**). Furthermore, the curves recorded for the remelted specimens were characterized by longer passive range compared to the sinter in initial state for which a rapid increase in current density occurred almost directly after exceeding the value of $E_{kor.}$, which is reflected by quick transition of the material into the active dissolution state. Improvement in resistance to corrosion medium in the case of steel after remelting treatment was connected with surface layer remodelling: disappearance of porosity and formation of single-phase structure.

4. CONCLUSIONS

Remelting treatment used in the study led to formation of a surface layer with homogeneous cellular-dendritic structure without voids. The microstructure of the laser-remelted layer was substantially finer than in the case of the layer remelted using GTAW method. Presence of only austenitic phase was found in the surface layers modified in the study. The layers obtained were characterized by similar microhardness. Due to homogenization of the structure in the remelted layers, the wear mechanism of the sinter was changed, which contributed to deterioration of tribological properties. Furthermore, a substantial improvement in corrosion resistance was observed, which is attributable to changes in phase composition and disappearance of porosity.

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