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MICROSTRUCTURAL CHANGES DURING HEAT TREATMENT OF SINTERED AUSTENITIC NICKEL-FREE STAINLESS STEEL

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Abstract: Production of nickel-free austenitic stainless steels is usually performed using sintering or melting in nitrogen atmosphere, where nitrogen as austenite forming element replaces nickel. These steels can also be produced by additional nitriding in solid state of final ferritic stainless steel components. In this case, properties of the steel do not depend only on casting or sintering parameters, but also on nitriding and post sintering heat treatment parameters. In this regard, effect of subsequent heat treatment parameters on properties of the sintered austenitic nickel-free stainless steel were analysed in this work. Post sintering heat treatments, in nitrogen atmosphere, were performed using dilatometer DIL 402/C/7. Microstructural changes after heat treatment and dilatometer test results are presented in the paper.
Keywords: nickel-free austenitic stainless steel, sintering, nitriding

1. INTRODUCTION

Production of sintered austenitic nickel-free stainless steels starts with manufacturing of appropriate metal powder. Chemical composition of metal powder must be adjusted to allow absorption of sufficient quantity of nitrogen for obtaining completely austenitic microstructure. Alloying with manganese, chromium and molybdenum contributes to increased solubility of nitrogen in iron alloys and steels in liquid state [1,2].

Next step is pressing or injection molding of metal powder into desired shape. If powder injection molding technology is used, green parts are then transferred to debinding furnace, where the most of the binder is removed from the parts, Figure 1. Removing of residual binder is performed at the beginning of sintering cycle. Increasing of temperature, selecting of appropriate nitrogen partial pressure and defining of sintering time are the final steps of the production, Figure 1.

Several phenomenon, very important for production of sintered austenitic nickel-free stainless steels, are taking place during sintering. Nitrogen atmosphere surrounds each powder particle, causing that absorption surface becomes very large. Then, contacts between particles grow and interaction of parts with sintering atmosphere occurs. Sintering mechanisms activated at higher temperatures contribute to increasing of density, reducing of porosity and improving of mechanical properties of the parts [3]. On the other hand, reducing of porosity and closing of pores cause decreasing of the total surface between parts and sintering atmosphere, which significantly affect nitrogen absorption. Also, absorption of nitrogen during sintering changes microstructures ratio and total diffusion coefficient, as well as sinterability of the material [4].

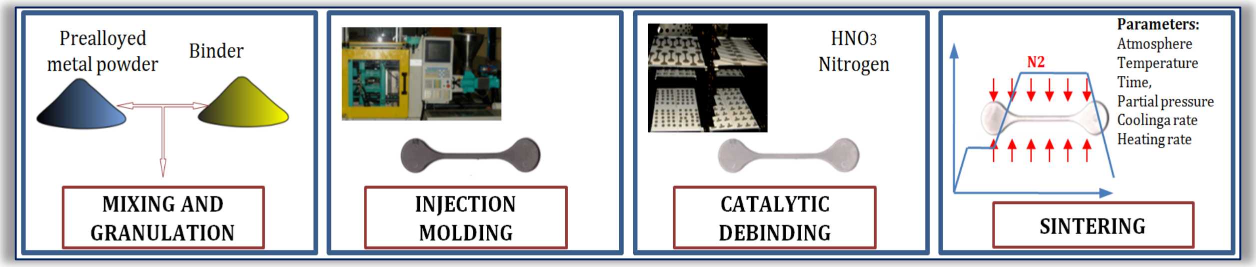


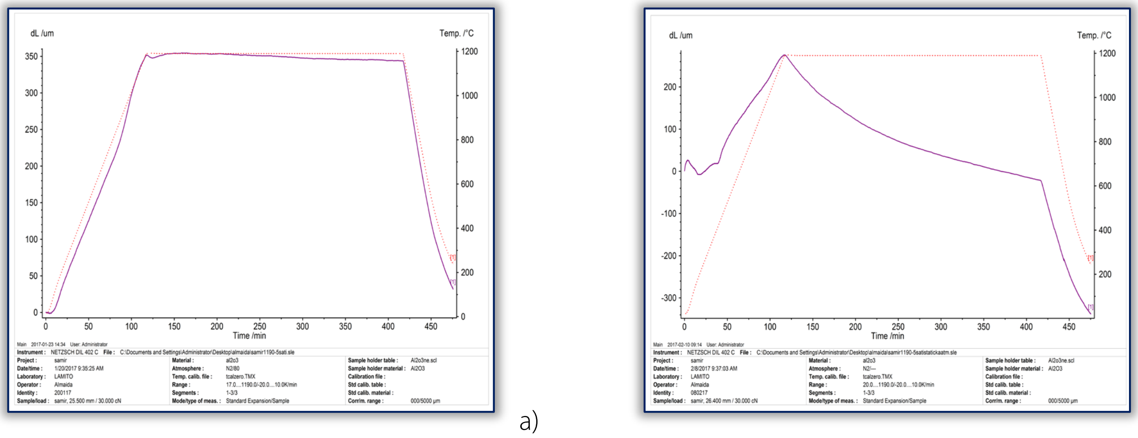
Figure 1. Production of austenitic nickel-free stainless steel using MIM technology

Interaction of parts and sintering atmosphere depends on temperature, heating and cooling rate, partial pressure of sintering atmosphere, atmosphere composition and sintering time [4].

In addition to sintering, production of high nitrogen stainless steels can be done using melting in nitrogen atmosphere [5]. Nitrogen containing alloys or compounds can be added to melt in order to increase nitrogen content [6]. Also, it is possible to produce austenitic stainless steel by nitriding, in solid state, of final ferritic parts, where absorption of nitrogen takes place through the surface of the part. Transformation of microstructure begins at the surface and progress to the centre of the part. Final properties of the parts are highly dependent on cooling conditions and nitriding parameters. Formation of nitrides during cooling phase makes cooling conditions very important for final properties of the material. In this regard, effect of subsequent heat treatment parameters on properties of the sintered austenitic nickel-free stainless steel were analysed in this work.

2. EXPERIMENTAL WORK

Sintering of Fe-17Cr-11Mn-3Mo alloy, as a basis for production of austenitic nickel-free stainless steel, in argon atmosphere reduces sintering time and sintering costs [4]. However, absence of nitrogen in atmosphere, as well as in parts, results in microstructure composed of austenite and ferrite, Figure 2, a). Post sintering nitriding is necessary to attain austenitic microstructure. Parts used in experiments were sintered for 3 h at temperature of 1200ºC. First sample was sintered in argon with starting microstructure containing high amount of ferrite. Second part was sintered in nitrogen atmosphere with partial pressure of 800 mbar. Cooling of parts was done in sintering furnace supported with fan at 770ºC. Density of parts sintered in argon are significantly higher than those sintered in nitrogen, while residual porosity is lower [4].



a) b)

Figure 2. Dilatometric curves for a) sample sintered in argon and b) sample sintered in nitrogen

Post sintering nitriding and solution annealing for production of appropriate nickel-free austenitic stainless steel cause changes in microstructure, starting from grain size, porosity characteristics to nitrogen redistribution between solution and compound. In order to observe aforementioned changes post sintering heat treatments of components sintered in nitrogen and argon atmosphere were performed in very controlled conditions, using dilatometer DIL 402/C/7. Heat treatment was performed in nitrogen atmosphere at temperature of 1190ºC with heating rate of 10ºC/min and cooling rate of 20ºC/min. During heat treatment, dilatometric analysis were performed. Results of dilatometric analysis are presented in Figure 2. Comparison of microstructures after sintering and subsequent heat treatment is presented in Figure 3.

3. RESULTS AND DISCUSSION

Dilatometric analysis revealed significant differences in behaviour of steel sintered in argon and nitrogen atmosphere. Both samples are heated to 1190ºC with dwell time of 5h. In first case, argon sintered sample showed emphasized expansion during heating, while dwell time did not significantly affect shrinking intensity. Analysis of the curve presented in Figure 2. a) showed very complex dilatometric behaviour with few sudden changes in length and small shrinking during dwell time.

On the other hand, sample sintered in nitrogen atmosphere with significantly lower starting density showed emphasized shrinking during 5 h, Figure 2, b). Reheating of parts sintered in nitrogen atmosphere by 10ºC /min to 1190ºC and holding for 5 h may initiate grains boundaries to move, causing changes in porosity characteristics. Thus, residual porosity makes difference between dilatometric behaviour of sintered components and cast or wrought materials.

It is very important to be aware that dilatometric behaviour of sintered samples, for the same materials sintered under same conditions can be significantly different. Samples from different positions of the same part may have different starting density and porosity, mostly caused by pressing and non-uniform density distribution before sintering (green part). Comparison of porosities in one tensile test specimen, produced by MIM technology, taken from different positions are presented in Figure 3, e), f).

Microstructure of the part sintered in argon and etched with Kalling's reagent is presented in Figure 3. a). It was observed that microstructure, through complete cross section of the part, consists of austenite and ferrite. Heating of this part to 1190ºC for 5 h in nitrogen atmosphere, resulted in significant microstructure changes. Surface of the parts experienced the most pronounced changes because of the longest exposure to nitrogen atmosphere, Figure 3. b),c).

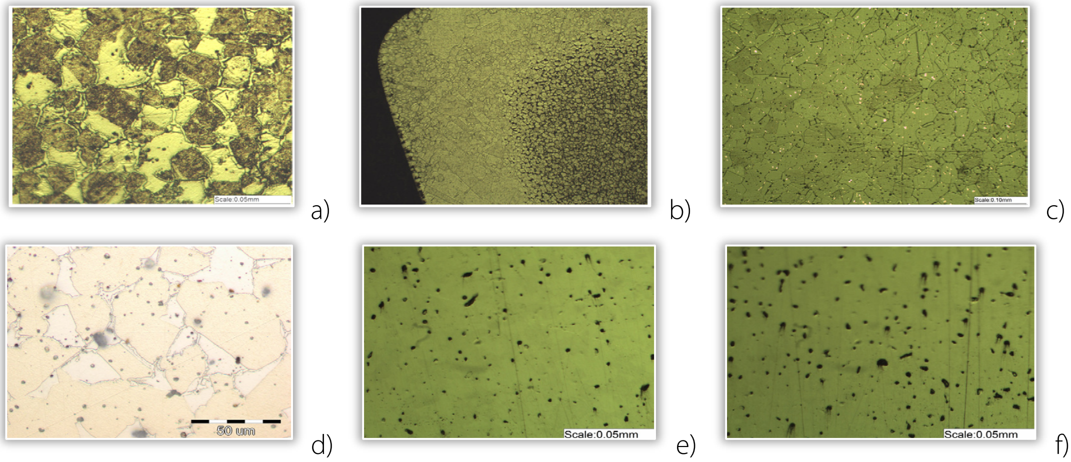


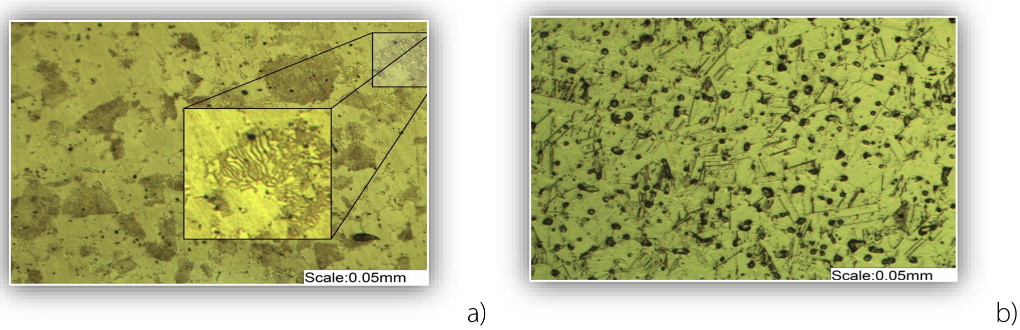
Figure 3. Optical micrographs: a) sample sintered in argon, b) sample sintered in argon and reheated in N_2, c) sample sintered in argon and reheated in N_2-near to surface, d) sample sintered in argon and reheated in N_2-centre, e)and f) polished N_2 sintered sample taken from different positions of tensile test specimen

Absorption of nitrogen from the surface of the part and diffusion to its core may be a reason for microstructure transformation at the surface of the part. This behaviour of the argon sintered parts with subsequent heat treatment in nitrogen atmosphere needs additional investigation. Moving to the centre of the part microstructure more resembles original sintered microstructure. Core of the part experienced changes in grain size because of long exposure to high temperature.

One of the very important steps in production of sintered austenitic stainless steels is solution annealing after sintering in nitrogen atmosphere or after additional nitriding. Formation of nitrides during slow cooling of parts from the sintering temperature significantly reduces mechanical properties [4].

Sample for this experiment was taken near the surface of the part, where potentially, concentration of nitrogen is the highest because of the longest exposure to nitrogen atmosphere. Polishing and etching with Kaling's reagent revealed significant quantity of precipitated nitrides. It can be observed that precipitate formed during slow cooling are in lamellar form, Figure 4, a. Heating of parts to temperature which causes decomposition of nitrides increases nitrogen content in form of solution.

Heating this parts to 1090 °C and holding for 20 min results in complete dissolution of nitrides, while subsequent fast cooling prevents its reformation. Metallographic sample etched with Kalling's reagent reveals austenitic microstructure with emphasized twin boundaries, Figure 4, b).



a) b)

Figure 4. Nitrogen sintered part, etched with Kalling's reagent:
a) surface after sintering, b) surface after solution annealing at 1090 °C

4. CONCLUSIONS

Porosity in material, which remains after sintering, is important factor in analysing of shrinking behaviour of reheated components. Porosity distribution of sintered parts is strongly related to density gradient of green part and injection molding parameters. Thus, samples taken from the same part can show different dilatometric behaviour. If there is phase change, as seen during reheating of parts sintered in argon using nitrogen atmosphere, dilatometric curves are even more complex. In this case, nitrogen absorption at 1190°C caused phase transformation, which progresses from the surface to the centre of the part.

Surface of the parts sintered in nitrogen atmosphere, compared to centre, is longer subjected to nitrogen atmosphere causing nitride rich area. Lamellar structure of nitrides in austenite, formed during cooling of nitrogen sintered parts, can be decomposed with appropriate solution annealing followed by fast cooling.

Acknowledgements

The work was supported by Federal Ministry of Education and Science of Bosnia and Herzegovina.

Note

This paper is based on the paper presented at 13th International Conference on Accomplishments in Mechanical and Industrial Engineering – DEMI 2017, organized by University of Banja Luka, Faculty of Mechanical Engineering, in Banja Luka, BOSNIA & HERZEGOVINA, 26 - 27 May 2017.

References

[1] Anne H. Satir-Kolorz, Heinrich K. Feichtinger. (1991). On the solubility of nitrogen in Liquid iron and steel alloys using elevated pressure. Z Metallkde, 82(1991): 689-697.

[2] L.Iorio, M.Cortie, R. Jones, (1994). Solubility of nitrogen in experimental low-nickel austenitic stainless steel. The Journal of The South African Institute of Mining and Metallurgy, Technical note 1994, p.173-177.

[3] Randall M. German (1996). Sintering theory and practice. New York,1996.

[4] Samir Butković. (2013). Sinterability and tensile properties of nickel-free austenitic stainless steel x15CrMnMoN 17-11-3, Journal Technical Gazette, Vol.20 No.2 April 2013.

[5] Woo-Il PARK, Sung-Mo JUNG and Yasushi SASAKI. (2010). Fabrication of Ultra High Nitrogen Austenitic Stainless Steel by NH3 Solution Nitriding. ISIJ International, Vol. 50 (2010), No. 11, pp. 1546–1551.

[6] P.D. Jablonski and J. A. Hawk. (2013). Nitrogen control in VIM melts. Proceedings of the 2013 International Symposium on Liquid Metal Processing & Casting, Edited by: Matthew J. M. Krane, Alain Jardy, Rodney L. Williamson, and Joseph J. Beaman.

ISSN 1584 - 2665 (printed version); ISSN 2601 - 2332 (online); ISSN-L 1584 - 2665