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Influence of sintering parameters on the properties of duplex stainless steel

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Properties

ABSTRACT

Purpose: of this paper was to examine the influence of sintering parameters like time, temperature, atmosphere and gas pressure under cooling stage on the mechanical properties of duplex stainless steels.
Design/methodology/approach: In presented study duplex stainless steels were obtained through powder metallurgy starting from austenitic, martensitic base powders by controlled addition of alloying elements, such as Cr, Ni, Mo and Cu. In the studies behind the preparation of mixes, Schaeffler's diagram was taken into consideration. Prepared mixes have been compacted at 800 MPa and sintered in a vacuum furnace with argon backfilling at temperatures from 1200°C to 1285°C for 0.5, 1 and 2 h. After sintering different cooling cycles were applied using nitrogen under pressure from 0.6 MPa to 0.002MPa in argon atmosphere. Produced duplex stainless steels have been studied by scanning and optical microscopy and EDS chemical analysis of microstructure components. Mechanical properties have been studied through tensile test.
Findings: Mechanical properties of sintered stainless steels are strictly connected with the density and the pore morphology present in the microstructure too and especially of cooling rate directly from sintering temperature. The lowest cooling rate - applied gas pressure, the mechanical properties decrease due to precipitation of sigma phase. Mechanical properties of studied steels depends on austenite/ferrite ratio in the microstructure and elements partitioning between phases too.
Research limitations/implications: Applied fast cooling rate seems to be a good compromise for mechanical properties and obtained microstructures, nevertheless further tests should be carried out in order to examine it influence on corrosion properties.
Originality/value: The use of elemental powders added to a stainless steel base showed its potentialities, in terms of fair compressibility and final sintered density. In addition a good microstructural homogeneity and first of all good mechanical properties was achieved, also working with cycles possible for industries.
Keywords: Mechanical properties; Powder metallurgy; Duplex stainless steel

1. Introduction

The powder metallurgy stainless steels, especially ferritic grades, have found applications in mounting brackets for the rear view mirrors, the tone wheels for the antilock brake systems and also in automotive exhaust applications like exhaust flanges and mounting unit of HEGOS. The automotive market introduces newly designed sintered parts in large amounts in produced cars. Stainless steel is the preferred material for powder metal flanges because of its resistance to corrosion and oxidation. The fact that the powder metal parts can be made in high material densities for the optimum combination of properties has encouraged their use at biggest users of powder metal exhaust system flanges in the world [1-3].

Duplex stainless steels are characterized by a two-phase microstructure consisting of approximately equal amounts of ferrite and austenite and thus combine some of the features of the two major classes, austenitic and ferritic grades. In recent years those steels are extensively used for many applications because of excellent chloride stress corrosion cracking resistance, pitting and crevice corrosion resistance, yield strength, ductility, impact toughness and weldability. Wide interest of scientist and numerous publications testify that sintered duplex stainless steel seems to be very promising in fast introduction to industrial production [4-15].

This paper presents the results of researches carried out on the sintered duplex stainless steels obtained by mixing of elemental powders to an either martensitic or austenitic powder and their comparison with steel obtained trough mixing austenitic and ferritic powder in equal quantities. The work has been focused towards the evaluation of influence of sintering parameters like time, temperature and atmosphere as well as backfilling gas pressure under cooling on the mechanical properties especially tensile strength.

2. Experimental procedure

To produce sintered duplex stainless steel different compositions have been tested, using austenitic X2CrNiMo17-12-2 (AISI 316L) and martensitic X6Cr13 (AISI 410L) as starting base water atomized powders of Hoganas Corporation with the characteristics presented in Table 1.

Table 1.
Average composition of starting powders

Grade powder		Elements concentration, wt. %						
PN-EN10088	AISI	Ni	Cr	Si	Mn	Mo	C	Fe
X2CrNiMo 17-12-2	316L	13	17	0.8	0.2	2.2	0.02	bal.
X6Cr13	410L	0.14	12.2	0.88	0.09	-	0.02	bal.
X6Cr17	430L	-	16	1.14	0.19	-	0.09	bal.

Austenitic base powder X2CrNiMo 17-12-2 were mixed with addition of alloying elements powders such as Cr (in form of ferrochromium powder), Ni, Mo and Cu in the right quantity to obtain the chemical composition similar to biphasic one - mixtures A and B. Powder mixtures signed as C and D were produced starting from martensitic powder X6Cr13. Moreover, the ferritic stainless steel X6Cr17 powder has been mixed to austenitic stainless steel powder in the ratio of 1/1 in order to examine the microstructure derived after sintering (mixture E). In the preparation of powder mixtures, Schaeffler's diagram was taken into consideration (Fig. 1). Although its proper application is in welding, it is possible to extend its use in the field of powder metallurgy. Thus Cr_E and Ni_E equivalents were obtained using formulas: $Cr_E = \%Cr + \%Mo + 1.5\%Si + 0.5\%Nb$ and $Ni_E = \%Ni + 30\%C + 0.5\%Mn$ respectively.

The weight quantities of the corresponding elements in percent were introduced in those formulas which locate all prepared powder mixtures in a well defined area, at least from a theoretical point of view. Chemical composition of produced mixtures were placed in austenitic-ferritic area of the Schaeffler's diagram with various content of ferritic phase in the range from 20 to 80%.

Powders were mixed with single elements using a laboratory turbula mixer. Acrawax was used as lubricant in a quantity of 0.65 wt.% in excess 100 for all compositions produced. Samples were obtained using a hydraulic press applying a pressure of 800 MPa with a floating die. The debinding process was done at 550°C for 60 minutes in a nitrogen atmosphere. Samples were then sintered in a vacuum furnace with argon backfilling at temperatures 1200°C, 1260°C, 1285°C, for 30, 60 and 120 min. After sintering different cooling cycles were applied using nitrogen under pressure 0.6, 0.2, 0.042, 0.002 MPa and 0.002MPa in argon atmosphere. Table 2 presents all the prepared compositions according to Schaeffler's diagram.

Densities were evaluated using the water displacement method. Microstructure observations were carried out using light microscope and scanning electron microscope equipped in EDS. Evaluations of the phase composition were made using ARL X'TRA 48 X-ray spectrometer, with the filtered copper lamp rays with 45kV voltage and heater current of 40mA.

Table 2.
Chemical composition of investigated powder mixes

Base powders	Composition designation	Elements concentration, wt. %						
PN-EN10088		Ni	Cr	Si	Cu	Mn	Mo	Fe
X2CrNiMo 17-12-2	A	10.52	26.40	0.80	0.80	-	2.02	bal.
	B	11.51	21.33	0.84	2.00	-	2.21	bal.
X6Cr13	C	8.10	22.72	0.70	-	0.06	2.00	bal.
	D	8.09	26.23	0.65	2.00	0.06	2.00	bal.
X2CrNiMo 17-12-2, X6Cr17	E	6.50	16.20	1.02	0.05	0.10	1.25	bal.

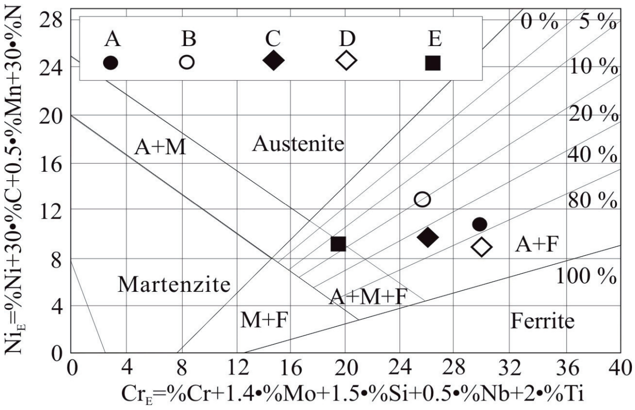

Fig. 1. Schaeffler's diagram. The marked points on the graph determine the forecast microstructure of the compositions

Metallographic specimens of all test materials were analyzed in the unetched as well as etched conditions. Unetched metallographic samples were utilized to evaluate stereological parameters of the pore structure such as pore size and pore shape factors $f_s = 4\pi A/P^2$ and $f_e = D_{min}/D_{max}$, where A and P are the area and the perimeter, respectively, of the metallographic cross-section of the pore and D_{min} and D_{max} are the minimum and the maximum Feret diameter of a pore. The shape factor of 1 represents a circular pore in the plane of analysis and as the number decreases from 1, the elongation and degree of irregularity increases. Pore shape factor f_s determine profile irregularity of a pore while pore shape factor f_e represent the pore elongation. This was accomplished with a Leica Qwin image analysis system.

Mechanical properties were evaluated basing on the tensile test performed according to PN-EN 10002-1 standard on samples prepared according to ISO 3928 standard.

3. Results and discussion

Density results show that for the martensitic based mixtures higher sintered densities were obtained (7.13÷7.15 g/cm^3). For the austenitic based powders, lower values were obtained (6.95÷7.01 g/cm^3), even though starting with green values similar to the other compositions. Mixture obtained by mixing ferritic and austenitic powders in equal amounts (composition E) shows good density after sintering cycle (7.22g/cm^3). Greater reactivity of martensitic grade powders when compared to austenitic grades results in higher shrinkage rate of the first one. Moreover, the addition of copper has resulted in the formation of a liquid phase during sintering and there through it influences on growth of sinterability caused by faster mass transport. This is evident for compositions containing copper with reason of higher sintered density when compared with sintered duplex stainless steels without copper addition.

Plot of pore shape factor f_s indicate that for all prepared mixtures his value is much the same and the major part of pores (about 50%) achieve approximately 0.7 in the case of sintering cycle in 1260°C with cooling from sintering temperature under gas pressure of 0.6MPa. For this sintering cycle pore shape factor f_e demonstrate major variety of pores shape and is including in the range of 0.45-0.7. There appears to be no significant change in the pore shape for all the materials that were evaluated in given cycle but analyzing influence of gas pressure under cooling stage of sintering cycle on the pore shape factors (Fig. 2 and 3) become more spherical due to elongated temperature affect. This effect is more evident for lowest gas pressure and for composition (E). Increase of sintering temperature and prolonged sintering time influence on the pore morphology provoking their rounding.

Tensile test analysis shows, that the highest tensile strength R_m=650 MPa has been achieved for steel obtained by mixing both austenitic and ferritic powders in equal amounts for sintering in higher temperatures. Increase of sintering time result in increase of tensile strength from 600 to 650 MPa (Fig. 4). Analyzing influence of backfilling gas pressure (Fig. 5) on mechanical properties the decrease of tensile stress were noted except composition E and D. Rest of examined compositions shows decrease of tensile stress due to precipitation of intermetalic sigma phase, rich in Cr and Mo which precipitate on ferrite-austenite boundaries and inside ferritic grains what was confirmed by X-ray analyses.

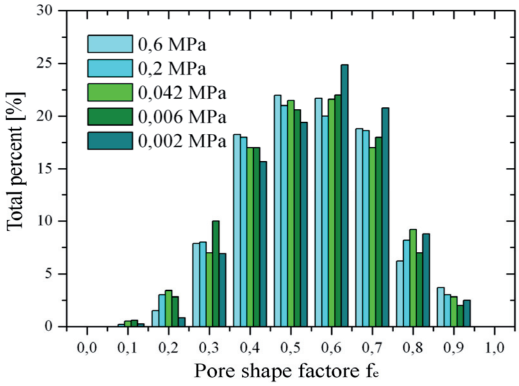

Fig. 2. Influence of backfilling gas pressure on the pore shape factor f_e of composition (C) sintered in 1260°C for 60min

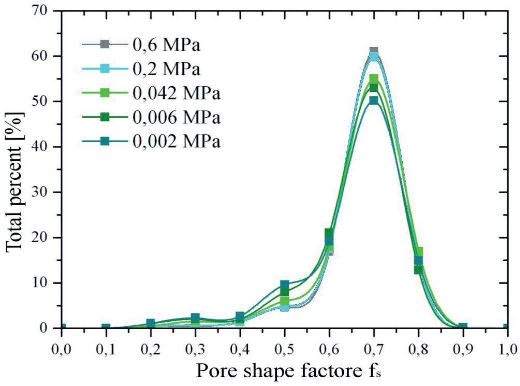

Fig. 3. Influence of backfilling gas pressure on the pore shape factor f_s of composition (C) sintered in 1260°C for 60min

During cooling with slow rate, using low gas pressure, ferrites decompose on sigma phase and secondary austenite causing impoverishment of surrounded zones in alloying elements. Chemical composition analysis (EDS) of individual structural components in studied steels shows that the concentrations of elements such as Cr and Mo in ferrite phase is higher, while Ni concentration is lower than in austenitic phase. The element partitioning between ferritic and austenitic phase is consistent with the stabilizing effect of each element on the respective phase. Precipitation of phases causing impoverishment of mechanical properties strictly depend of respective elements concentration in analyzed composition especially Ni which stabilize austenite and counteracts of precipitates.

Executed X-ray analyses confirm that the structure of the obtained sintered steels in the case of fast cooling directly from sintering temperature, using high gas pressure e.g. 0.6MPa, consists of austenite and ferrite phases. Phase quantities in the microstructure were evaluated and in composition (A) and (D) reaches the ferrite content about 75% while composition (B) 18%. For composition (C) the approximate balance of ferrite and austenite was archived. Steel marked as (E) reach ferrite content about 67%. Performed analyses do not demonstrate other secondary phases like sigma phase, carbides or nitrides precipitations in the microstructure of steels sintered in this conditions.

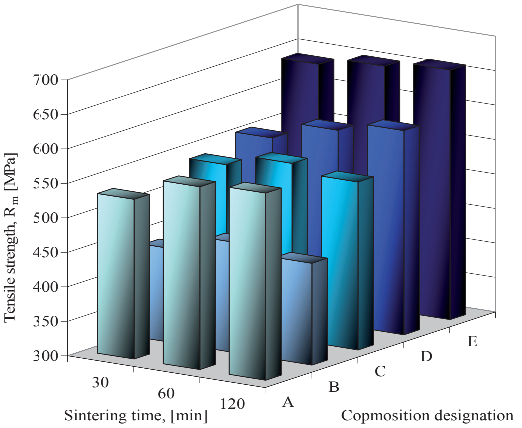

Fig. 4. Influence of sintering time on tensile strength of compositions sintered in 1260°C and cooled under pressure of 0,6MPa

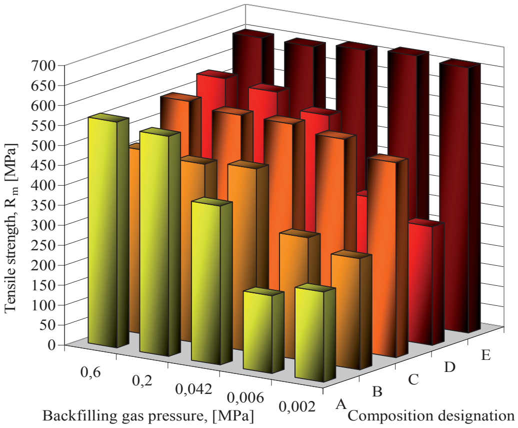

Fig. 5. Influence of backfilling gas pressure on tensile strength of compositions sintered in 1260°C for 60min

4. Conclusions

According to achieved results, it was affirmed that applied sintering method as well as powder mixes preparation allows for manufacturing the sintered duplex steels with good mechanical properties which depends on austenite/ferrite ratio in the microstructure and elements partitioning between phases. Mechanical properties are strictly connected with the density and the pore morphology present in the microstructure too and especially of sintering parameters: time, temperature and cooling rate directly from sintering temperature. The lowest cooling rate - applied gas pressure, the tensile strength decrease. Precipitation of phases causing impoverishment of mechanical properties strictly depend of respective elements concentration in analyzed composition especially Ni which stabilize austenite and counteracts of precipitates.

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