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Properties of vacuum sintered duplex stainless steels

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Abstract: This work presents the possibility of obtaining duplex stainless steels through powder metallurgy technology starting from austenitic X2CrNiMo17-2-2, martensitic X6Cr13 powders by controlled addition of alloying elements, such as Cr, Ni, Mo, Cu in the right quantity to obtain the chemical composition of the structure similar to biphasic one. In the studies behind the preparation of mixes, Schaffler's diagram was taken into consideration. Prepared mixes of powders have been compacted at 800 MPa and sintered in a vacuum furnace with argon backfilling at 1260°C for 1 hour; after sintering rapid cooling has been applied in N₂, with an average cooling rate of 650 °C/min.

Keywords: Duplex stainless steel, Vacuum sintering

1. INTRODUCTION

Powder metallurgy enables producing duplex stainless steels by several methods. The first of those bases, on fully prealloyed powder with a required duplex composition. The principle of the next one is the mixing powders both austenitic and ferritic powder in proper ratios to ensure required duplex microstructure. Researches that have been carried for a few years caused development of third approach where starting from austenitic, martensitic powders by controlled addition of alloying elements, such as chromium, nickel, molybdenum, copper in the right quantity the final chemical composition of the structure similar to biphasic one is achieved. Calculations of theoretical chemical composition of produced mixes is based on Schaffler's diagram. Duplex powders were subject of studies at different universities as well as in the major companies producing powders. Application of powder metallurgy technology for producing biphasic duplex steels enables precise control of their chemical and phase composition of structure as well as elimination of number of technological difficulties that are present during the production of same kind of steels but using traditional methods. In order to reduce production costs of parts made from sintered duplex stainless steels, the heat treatment, so called „sinter-hardening” has been introduced, which relies on rapid convection cooling directly from sintering temperature. Application of this heat treatment method, in case of duplex stainless steels provides to obtain precipitate free structure [1, 2].

2. EXPERIMENTAL PROCEDURE

Different compositions have been tested, using austenitic X2CrNiMo 17-2-2 and martensitic X6Cr13 as starting base powders with the characteristics presented in the Table 1.

Moreover the ferritic stainless steel X6Cr17 has been mixed to austenitic stainless steel in the ratio of 50%-50% in order to exam the deriving structure after sintering. Powders were mixed with single elements using a laboratory tubular 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 2000 kN hydraulic press applying a pressure of 800 MPa. The debinding was done at 550°C for 60 minutes in a nitrogen atmosphere. Samples were then sintered in a vacuum furnace with argon backfilling at 1260°C for 1 h. Rapid cooling was applied, with an average cooling rate of 650 °C/min. Table 2 reports all the prepared compositions.

Table 1.

Average composition of starting powders

Grade powder		Elements concentration, wt. %						Cr _{eq}	Ni _{eq}	PREw*
PN-EN	AISI	Ni	Cr	Si	Mn	Mo	C			
X2CrNiMo 17-2-2	316L	13	17	0,8	0,2	2,2	0,02	20,4	13,7	24,26
X6Cr13	410L	0,14	12,2	0,88	0,09	-	0,02	13,53	0,305	12,21
X6Cr17	430L	-	16	1,14	0,19	-	0,09	13,53	1,385	12,21

* PREw - Pitting Resistance Equivalent number ($PREw = \% Cr + 3.3 \times (\% Mo + 0.5\% W) + 16x\% N$)

In the studies behind the preparation of mixes, Schaffler's diagram was taken into consideration. Although its proper application is in welding, it is possible to extend its use in the field of powder metallurgy. Thus Cr_{eq} and Ni_{eq} ($Cr_{eq} = \%Cr + \%Mo + 1,5 \times \%Si + 0,5 \times \%Nb$; $Ni_{eq} = \%Ni + 30 \times \%C + 0,5 \times \%Mn$) are obtained introducing the wt. % quantity of the corresponding element into the formula which locates all the products in a well defined area, at least in a theoretical point of view. Table 2 presents chromium and nickel equivalent values derived from green composition.

Table 2.

Chemical composition of investigated powder mixes

Base powders		Composition designation	Elements concentration, wt. %						Cr _{eq}	Ni _{eq}
PN-EN	AISI		Ni	Cr	Si	Cu	Mn	Mo		
X2CrNiMo 17-2-2	316L	A	10,52	26,40	0,80	0,80	-	2,02	30,33	11,25
		B	11,51	21,33	0,84	2,00	-	2,21	25,68	12,30
X6Cr13	410L	C	8,10	22,72	0,70	-	0,06	2,00	26,57	8,97
		D	8,09	26,23	0,65	2,00	0,06	2,00	30,01	8,86
X2CrNiMo 17-2-2, X6Cr17	316L, 430L	E	6,50	16,20	1,02	0,05	0,10	1,25	19,48	8,95

Densities were evaluated using the water displacement method. Microstructure observations were carried out using light microscope and scanning electron microscope with EDS microprobe. Evaluations of the phase composition were made using ARL X'TRA 48 X-ray spectrometer, with the filtered copper lamp rays with the voltage of 45kV and heater current of 40mA. Charpy impact test were made according to PN-76/ H-04947 standard using unnotched samples and tensile test according to PN-EN 10045-1 standard. Hardness test was carried out in order to determine HRA value. To determine abrasive wear pin-on-disk test was introduced. The tungsten carbide ball with a diameter of 1,5 mm was used as a pin. The tests were executed with applied load of 5N, speed of 0,44 m/s and various distances.

3. RESULTS AND DISCUSSION

Figure 1 shows the results obtained in terms of green and sintered density evaluated using the water displacement method. As for the martensitic based mixtures sintered densities were included in the range of 7,13 to 7,25 g/cm³. For the austenitic based powders, instead, lower values were obtained, even though starting with green values similar to the other compositions. Greater reactivity of martensitic grade powders when compared to austenitic grades results in higher shrinkage rate of the first one. The smallest shrinkage, 0,5% was obtained for mixture (B) while the highest, 4,1% for mixture (D) based on martensitic powder. The addition of copper results in the formation of a liquid phase during sintering and thereby it influences the growth of sinterability because of faster material transport. This is evident for compositions containing copper with reason of higher sintered density compared to sintered duplex stainless steels without copper addition.

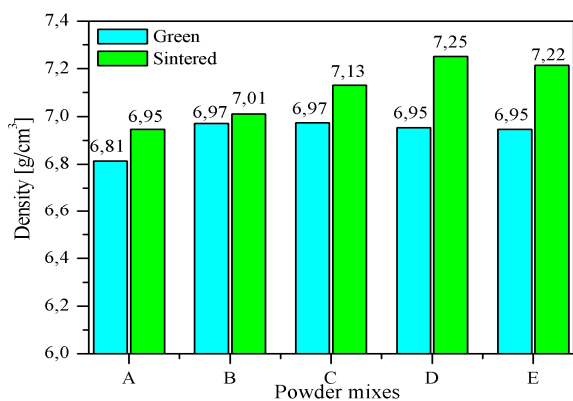


Figure 1. Green and sintered density.

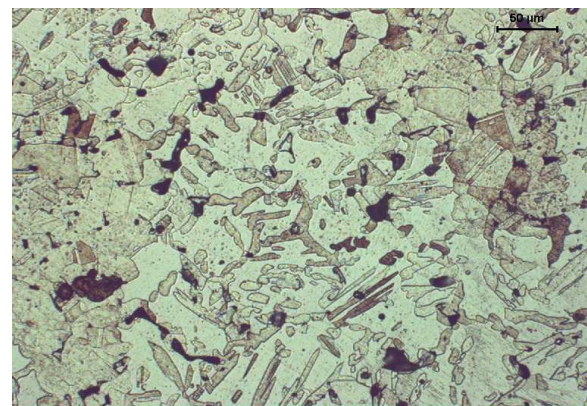


Figure 2. Microstructure of composition (A).

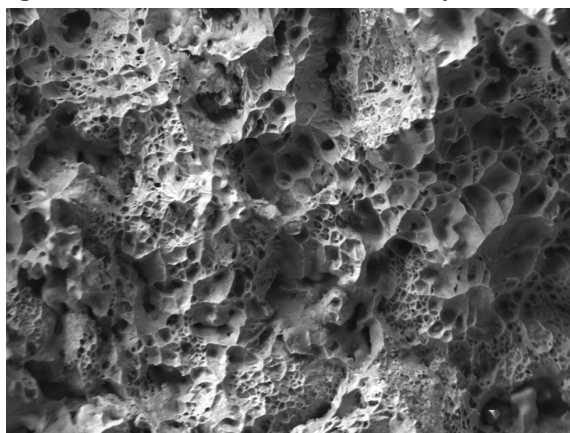
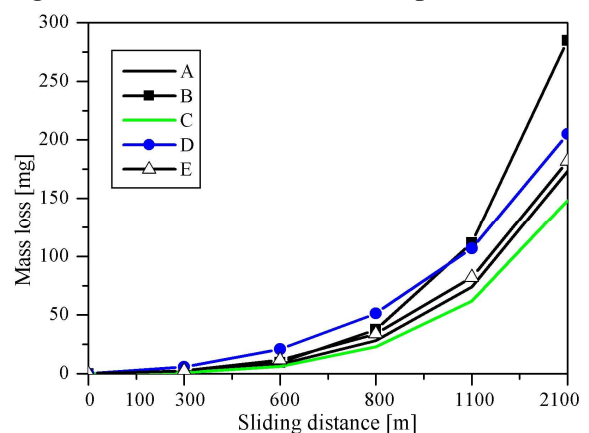


Figure 3. Fracture surface of composition (D), Figure 4. Wear test results obtained by Pin-on-disk method.



X-ray analysis confirm that the structure of obtained sintered steels consists of austenite and ferrite phases. Results of quantity rate calculations of individual structural components in the structure show that the amount of ferritic phase in produced duplex stainless steels is included in the range of 20% for compositions (D) and (E) to 80% for composition (B). Amount of austenite in composition (A) reached about 30% while for composition (C) the approximate balance of ferrite and austenite was achieved. Figure 2 shows microstructure example of studied sintered duplex stainless steels.

Table 3.
Mechanical properties of investigated sintered duplex steels

Composition designation	Yield strength $R_{p0.2}$ [MPa]	Tensile strength R_m [MPa]	Young's modulus E [kN/mm ²]	Elongation A [%]	Impact energy [J]	Hardness [HRA]	Mass loss [mg]
A	373	565	97.6	4.44	87.6	36	173.32
B	246	460	50.5	7.72	99.2	30	284.83
C	371	555	122.5	6.15	151.8	43	148
D	445	581	116.9	3.60	89.6	45	205
E	478	652	176.6	3.05	102	47	182.1

Mechanical properties of investigated sintered duplex steels are reported in Table 3. Impact energy of tested duplex steels reaches from 87 to 99J for compositions based on austenitic powder however for composition based on martensitic powder shows the highest values, 151J of impact energy was measured for composition (C). Obtained results are in accordance with quantity rate of austenite and ferrite in the microstructure. Tensile test analysis shows, that the highest tensile strength $R_m=652$ MPa has been achieved for steel obtained by mixing both austenitic and ferritic powders in equal amounts. Sintered steels based on martensitic powder X6Cr13 achieved optimal results in case of tensile strength and elongation. It must be noted that elongation of all produced steels is in the range of 3% to 7% thus proves greater sintering. Performed fractography analysis demonstrates that fracture surfaces of all steels are a mixed type of ductile and brittle fracture. Fracture surface is composed of wide and deeper dimples (Fig. 3), in case of composition (E) dimples are smallest and shallow.

Results of performed pin-on-disk wear test (Fig. 4) shows the highest mass loss 284 mg for composition (B), while the most resistant on abrasion is the (C) composition where mass loss is equal 148 mg.

4. SUMMARY

Manufactured steels demonstrate the austeno-ferritic structure with regular arrangement of both phases with no presence of precipitates. Mechanical properties of sintered duplex steels strictly depend on the austenite/ferrite ratio in structure. Austenitic phase in these steels assure ductility and impact resistance, on the other hand ferritic phase enlarge tensile strength, yield strength and hardness. Applied producing method of sintered duplex steels and used sintering cycle prove their advantage in case of mechanical properties and additionally it seems to be very promising for obtaining a balanced duplex structure, also working with cycles easy to be introduced in industries.

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