



Innovative PM duplex stainless steels obtained basing on the Schaeffler diagram

L.A. Dobrzański ^{a,*}, Z. Brytan ^a, M. Actis Grande ^b, M. Rosso ^b

^a Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

^b Department of Materials Science and Chemical Engineering, Politecnico di Torino, Alessandria Campus, Viale T.Michel 5, 15100, Italy

* Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

Received 06.02.2008; published in revised form 01.03.2008

ABSTRACT

Purpose: The purpose of this paper was to describe the sintered duplex stainless steels manufactured in sinter-hardening process and its usability in field of stainless steels and moreover using computer software to calculate the powder mix composition.

Design/methodology/approach: In presented paper duplex stainless steels were obtained through powder metallurgy starting from austenitic or ferritic base powders by controlled addition of alloying elements powder. In the studies besides the preparation of powder mixes, computer software based on Schaeffler's diagram was studied.

Findings: It has been demonstrated that austenitic-ferritic microstructures with regular arrangement of both phases and absence of precipitates can be obtained with properly designed powder mix composition and sintering cycle.

Research limitations/implications: According to the alloys characteristic applied cooling rate and powder mix composition seems to be a good compromise to obtain balanced duplex stainless steel microstructures, nevertheless further tests should be carried out in order to examine different cooling rates and sintering parameters.

Practical implications: Applied producing method of sintered duplex steels and used sintering cycle as well as developed computer software to calculating powder mix composition proves his advantage in case of obtained microstructures and additionally it seem to be very promising for obtaining a balanced duplex structure, also working with cycles easy to be introduced in industries.

Originality/value: The utilization of sinter-hardening process combined with use of elemental powders added to a stainless steel base powder shows its advantages in terms of good microstructural homogeneity and especially working with cycles possible to introduce in industrial practice.

Keywords: Sinter-hardening; Powder metallurgy; Duplex stainless steel; Schaeffler diagram

MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Stainless steels made by powder metallurgy mainly single-phase stainless steels, austenitic and ferritic with desired mechanical properties – ferritic stainless steels and high corrosion resistance – the austenitic one, for many years have stable position on market of sintered components [1, 2]. Utilization of

powder metallurgy for production of complex microstructures such as biphasic ferritic-austenitic stainless steels created chances to manufacture duplex stainless steels in one sintering cycle with no need of the additional heat treatments [3]. Sinter-hardening process applicable to duplex stainless steels ensures proper complex biphasic microstructure with controlled mechanical properties and corrosion resistance [4].

Sintered duplex stainless steels can be made using different methods. Different approaches can be found in literature where biphasic microstructure is manufactured by sintering of atomized duplex powder mixing of fully prealloyed powders or mixing of prealloyed powders with single alloying elements [5-8]. Proper duplex stainless steels structure may be obtained in a single sintering cycle through controlled addition of alloying elements, promoting formation of austenite or ferrite, to single-phase powders either ferritic or austenitic trying to predict the final structure based on Schaeffler's diagram. Alloying element may be added in the form of single elements or in a combined form. The sintering cycle is done in vacuum at argon backfilling using nitrogen under pressure to obtain rapid cooling directly from sintering temperature [9-12].

The development of computer-aided examinations of materials applied in powder metallurgy makes possible fastest and more accurate design of powder mix composition leading to complex biphasic microstructure of sintered stainless steels. There are few constitution diagrams such as Schaeffler, DeLong and WRC-1992 for prediction of stainless steels microstructure after welding and diagrams of Pryce and Andrews for microstructure prediction after plastic forming. The Schaeffler's diagram was taken into consideration in presented study preparation of powder stainless steels mixes [13-15].

2. Experimental procedure

In present study the investigations were performed on stainless steel samples manufactured using the uniaxial die pressing. Water atomized austenitic stainless steel powder X2CrNiMo17-12-2 and ferritic stainless steel powders X6Cr17, X6Cr13 (Table 1) with diameter of <150 μm and alloying elements powders such as Cr (in form of ferrochromium powder), Ni, Mo and Cu were used. In presented study sintered duplex stainless steels were obtained through powder metallurgy starting from austenitic or ferritic base powders by controlled addition of alloying elements, such as Cr, Ni, Mo and Cu in the quantity determined using Schaeffler diagram.

Table 1.
Average composition of the initial stainless steel powders

Base powder		Elements concentration, wt. %							
PN-EN10088	AISI	Ni	Cr	Si	Mn	Mo	C	Fe	
X2CrNiMo17-12-2	316L	13	16.4	0.9	0.2	2.5	0.03	bal.	
X6Cr13	410L	0.14	12.2	0.88	0.09	-	0.04	bal.	
X6Cr17	430L	-	16	1.14	0.19	-	0.09	bal.	

To make easier calculations of the theoretical chemical composition of hybride mixes of alloyed stainless steel powders and elemental powders, computer software was elaborated where Schaeffler's diagram was taken into consideration. Chemical compositions of produced mixtures were placed in austenitic-ferritic area of the Schaeffler's diagram with various content of ferritic phase (Table 2). Although the proper application of

Schaeffler diagram is in welding, it is possible to extend its use to the field of powder metallurgy. Thus, Cr_E and Ni_E equivalents were obtained using formulas 1, 2 respectively (Table 3).

$$Cr_E = \%Cr + 1.4\% \cdot Mo + 1.5\% \cdot Si \quad (1)$$

$$Ni_E = \%Ni + 30 \cdot \%C + 0.5 \cdot \%Mn \quad (2)$$

Moreover, in order to examine the microstructure derived after sintering the ferritic stainless steel powder X6Cr17 has been admixed to austenitic stainless steel powder X2CrNiMo17-12-2 in equal ratio (composition E).

Table 2.
Chemical compositions of investigated powder mixes

Base powders	Designation	Elements concentration, wt. %							
		Ni	Cr	Si	Cu	Mn	Mo	C	Fe
X2CrNiMo 17-12-2	A	10.52	26.40	0.80	0.80	-	2.02	0.02	bal.
	B	11.50	21.33	0.84	2.00	-	2.21	0.03	bal.
X6Cr13	C	8.10	22.72	0.70	-	0.06	2.00	0.02	bal.
	D	8.09	26.23	0.65	2.00	0.06	2.00	0.02	bal.
X2CrNiMo 17-12-2, X6Cr17	E	6.50	16.20	1.02	0.05	0.10	1.25	0.06	bal.

Table 3.
Chromium Cr_E and nickel Ni_E equivalents of the prepared powder compositions

Composition designation	A	B	C	D	E
Cr_E	30.44	25.68	26.57	30.01	19.86
Ni_E	11.25	12.30	8.97	8.85	9.57

Acrawax was used as lubricant in a quantity of 0.65 wt.% for all compositions produced. Samples were obtained using a hydraulic press applying a pressure of 800 MPa with a floating die. The dewaxing 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 temperature 1260°C for 60min. After sintering the rapid cooling was applied using nitrogen under pressure of 0.6MPa (4.1°C/s) in argon atmosphere.

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. Metallographic specimens of all test materials were analyzed in the unetched as well as etched conditions.

3. Results and discussion

In the powder mixtures preparation computer software was elaborated (Fig. 1) where the calculations of chemical and phase compositions are based on mathematical representation of Schaeffler diagram proposed by Mazurovsky [14]. The main

feature of the developed software is to establish proper proportions between the initial single phase stainless steel base powder and additional alloying elements powders to obtain the theoretical chemical composition in the well defined austenitic-ferritic region. This computer program has defined functions:

- Loading of base stainless steel powder and additional powder compositions,
- Loading of specified individual elements concentrations (Cr, Ni, Si, Cu, Mn and Mo),
- Calculating of powder mixes using the introduced base powder and alloying elements powders composition,
- Calculating of phase composition in austenitic-ferritic region of Schaeffler diagram.

Obtained calculations results of individual powders were then used to calculate the weight respective powders in grams to produce final powder mixes. For each composition 1000g of powder mix was prepared.

Calculating of phase composition in austenitic-ferritic region of Schaeffler diagram is implemented according to Mazurovsky et al. [13, 14] and following formulas (3-5) were introduced.

The amount of ferritic phase is dependent of nickel equivalent Ni_E and when formula (3) is valid the ferrite content is calculated according to formula (4), otherwise according to formula (5). The austenitic phase amount is given as difference of ferritic phase from total amount.

$$r_f = Ni_{eq} + 0.4455Cr_E / 1 - 0.1908Cr_E \leq -6.4 \quad (3)$$

$$Q_{ferrite} = 10.142r_f + 85.071 \quad (4)$$

$$Q_{ferrite} = -13.972r_f^2 - 112.22r_f - 125.38 \quad (5)$$

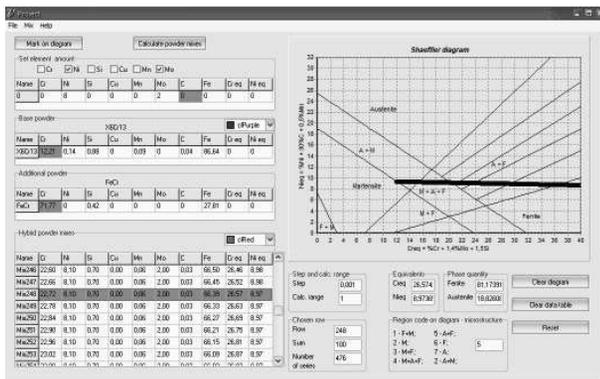


Fig. 1. Example on screenshot of developed computer software used for computer aided powder mix preparation calculations of mixture C

In present study to produce sintered duplex stainless steel different compositions have been calculated and then tested, using austenitic X2CrNiMo17-12-2 and ferritic X6Cr13 as starting base stainless steel powders. Compositions A and B were based on austenitic powder with addition of alloying elements powders such as Cr (in form of ferrochromium powder) and Cu in the right amount to obtain the chemical composition similar to biphasic one. Powder mixtures designed as C and D were produced starting from ferritic powder X6Cr13 in the same manner with addition of Ni, Mo and Cu powders. Composition E was produced mixing both austenitic and ferritic X6Cr17 powder in equal amounts.

Density measurements have revealed that as for the ferritic based mixtures densities were close to 7.2 g/cm³. For the austenitic based powders, instead, lower values were obtained, close to 7.0 g/cm³, even though starting with green values similar to the other compositions. It is remarkable to notice that, in case of composition B, an approximate dimensional stability was obtained but after sintering a biphasic microstructure were not obtained in this case. Composition B shows microstructure with relatively large island of ferritic grains in austenitic matrix. In this case a non-homogenous microstructure may be caused by insufficient portion of alloying powder Fe-Cr in base powder mixture which activates sintering process and formation of ferrite.

Analyzing the microstructures of prepared compositions in green state it was confirmed then alloying powders particples exhibit a uniform arrangement in the matrix of stainless steel powder what can guarantee a uniform biphasic microstructure after sintering (Fig. 2).

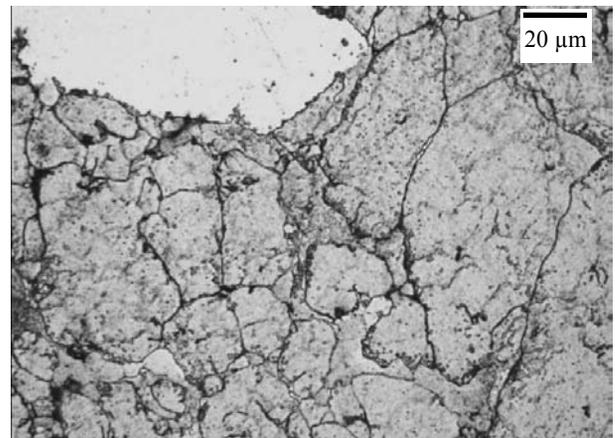


Fig. 2. Microstructure after debinding process of green compacts - composition C

According to metallographic examinations of sintered stainless steels, the presence of a fine microstructure with no recollection of precipitates could be observed using sintering with rapid cooling rate. Obtained microstructures were confirmed using X-ray diffraction thus bi-phase microstructure of manufactured sintered stainless steels composed of austenite and ferrite with variable amount of both phases which is in good accordance whit calculated using elaborated software. In the case of composition A based on austenitic powder X2CrNi17-12-2 the microstructure is composed of $\alpha + \gamma$ where austenitic phase is twined and uniformly distributed with ferritic grains. The microstructure of compositions based on ferritic powder X6Cr13 is well formed and composed of mixture $\alpha + \gamma$ where individual grains are fine and well mixed. In both cases, the formation of lenticular austenite was revealed. The microstructure of duplex stainless steel obtained from mix of austenitic X2CrNiMo17-12-2 and ferritic X6Cr17 powders exhibits a coarse grained microstructure with characteristic subgrains of duplex composition identified as an interdiffusion zone - a sharp and thin microstructure. This intermediate phase, shows chemical composition and microhardness values between those characteristic of the surrounding austenite and ferrite as those reported by Campos in [5].

Element concentrations of respective austenitic and ferritic region were evaluated too, using EDS analysis (Fig. 3). Basing on its results the concentration of ferrite former elements like Cr and Mo in ferrite region is higher while concentration of Ni is lower than in austenitic region and the element partitioning between both phases is consistent with the stabilizing effect of each element on the respective phase (Table. 4).

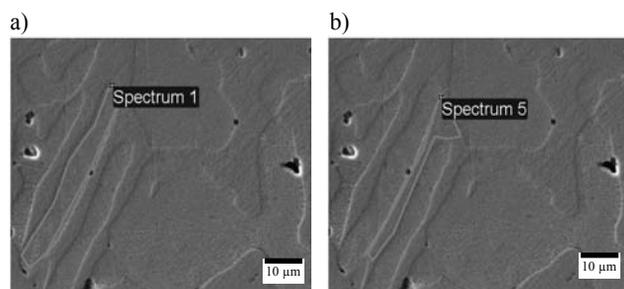


Fig. 3. EDS analysis of selected regions in composition C, a) austenitic, b) ferritic

Table 4. Results of EDS analysis of selected austenitic and ferritic region in sintered duplex stainless steel – composition C

Phase	Spectrum	Element concentration, wt. %					Total
		Si	Cr	Fe	Ni	Mo	
austenite	Spectrum 1	0.76	24.19	65.68	7.39	1.98	100.00
ferrite	Spectrum 5	0.77	29.32	62.28	4.28	3.35	100.00

4. Conclusions

It has been demonstrated that austenitic-ferritic microstructures with regular arrangement of both phases and no presence of precipitates can be obtained through a properly designed powder mix composition and sintering cycle. Main conclusion deriving from microstructures of manufactured materials is the possibility of application of the sinter-hardening process with rapid cooling as well as powder mixtures preparation and utilization of developed computer aided materials design software to ensure desired balance between phase concentration and elements partitioning between phases in sintered duplex stainless steels. The addition of alloying element powders, promoting formation of either ferritic or austenitic phases to initial stainless steel alloy powder, makes possible the formation of microstructures and therefore properties of sintered duplex stainless steels. The utilization of computer aided materials design in stainless steels powder metallurgy connected with application of sinter-hardening process can considerably shorten the time of alloy designs and guaranty proper bi-phases microstructure and properties.

References

[1] P.K. Samal, J.B. Terrell, Mechanical properties improvement of PM 400 series stainless steels via nickel addition, *Metal Powder Report* 1 (2001) 28-34.

[2] A.J. Rawlings, H.M. Kopech, H.G. Rutz, The effect of service temperature on the properties of ferritic P/M stainless steels, *Proceedings of the International Conference on Powder Metallurgy & Particulate Materials PM2TEC'97*, Chicago USA, 1997, 33-40.

[3] M. Rosso, M. Actis Grande, D. Ornato, Sintering of duplex stainless steels and their properties, *Powder Metallurgy Progress* 2 (2002) 10-17.

[4] W. Brian James, What is sinter-hardening?, *Proceedings of International Conference on Powder Metallurgy & Particulate Materials PM2TEC'98*, Las Vegas, Nevada USA, 1998, 55-60.

[5] M. Campos, A. Bautista, D. Caceres, J. Abenojar, J.M. Torralba, Study of the interfaces between austenite and ferrite grains in P/M duplex stainless steels, *Journal of the European Ceramic Society* 23 (2003) 2813-2819.

[6] P. Datta, G.S. Upadhyaya, Sintered duplex stainless steels from premixes of 316L and 434L powders, *Materials Chemistry and Physics* 67 (2001) 234-242.

[7] J. Kazior, T. Pieczonka, A. Molinari, Properties of AISI 316L, AISI 434L and duplex stainless steel, *Proceedings of the 8th Scientific Conference „Achievements in Mechanical and Material Engineering” AMME'1999*, Gliwice – Pawłowice – Rydzyna – Rokosowo, 1999, 289-293.

[8] M. Campos, P. Sarasola, J.M. Torralba, Sintering evolution of duplex stainless steels obtained from austenitic and ferritic stainless steels powders mixtures, *Proceedings of the 9th Scientific Conference „Achievements in Mechanical and Material Engineering”*, AMME'2000, Gliwice – Sopot – Gdańsk, 2000, 83-86.

[9] L.A. Dobrzański, Z. Brytan, M. Actis Grande, M. Rosso, E.J. Pallavicini, Properties of vacuum sintered Duplex Stainless Steels, *Journal of Materials Processing Technology* 157-158 (2004) 312-316.

[10] L.A. Dobrzański, Z. Brytan, M. Actis Grande, M. Rosso, Corrosion resistance of sintered duplex stainless steel evaluated by electrochemical method, *Journal of Achievements in Materials and Manufacturing Engineering* 17 (2006) 317-320.

[11] L.A. Dobrzański, Z. Brytan, M. Actis Grande, M. Rosso, Properties of duplex stainless steels made by powder metallurgy, *Archives of Materials Science and Engineering* 28/4 (2007) 217-223.

[12] M. Rosso, M. Actis Grande, 'High density sintered stainless steels with improved properties', *Journal of Achievements in Materials and Manufacturing Engineering* 21/2 (2007) 97-102.

[13] V. Mazurovsky, M. Zinigrad and A. Zinigrad, Mathematical representation of a modified Schaeffler diagram, *Proceedings of the Second International Conference on Mathematical Modelling and Computer Simulation of Metal Technologies*, Ariel, Israel, 2002, 3/129-139.

[14] V. Mazurovsky, M. Zinigrad and A. Zinigrad, Predicting weld structure using modified Schaeffler constitution diagram *Proceedings of the International Conference Mathematical Modeling and Simulation of Metal Technologies*, Ariel, Israel, 2000, 540-545.

[15] V. Mazurovsky, M. Zinigrad and A. Zinigrad Mathematical Model of Weld Microstructure Formation. *Proceedings of the 12th International Conference TWI Computer Technology in Welding and Manufacturing Conference*, Sydney, Australia, 2002, 79/1-9.