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Properties of stainless steel based on ferritic steel X6Cr13 made by powder metallurgy

L.A. Dobrzański, M. Kujawa*

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

* Corresponding e-mail address: magdalena.kujawa@polsl.pl

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ABSTRACT

Purpose: of this paper is to define influence of different concentration of reinforcement in form of X2CrNiMo25-7-4 steel powder on the properties of sintered ferritic stainless steel X6Cr13.

Design/methodology/approach: In presented study, material for investigation was manufactured by powder metallurgy from ferritic powders with appropriate contribution of austenitic-ferritic stainless steels powders. Prepared mixes have been pressed by cold isostatic pressing (CIP) at 350 MPa and sintered in vacuum furnace at 1250°C for 1 h. Obtained samples were examined by scanning electron microscopy (SEM), X-ray diffraction analysis and confocal microscopy for depth of the wear traces observation. Properties like porosity, density, wear resistance, macro- and microhardness were evaluated.

Findings: Researches show that addition of 5-15% powder duplex stainless steel caused the significant increase in hardness and microhardness of X6Cr13 steel. Small amount of reinforcement powder improved the value of wear resistance of base material.

Research limitations/implications: Contribute to study the properties of materials with the base of ferritic stainless steel reinforced by X2CrNiMo25-7-4 particles using powder metalurgy. The next step for research will be to examine tensile properties and corrosion resistance which is a fundamental property required for the described steels.

Practical implications: Development of a new type of materials applicable in the industry which are characterized by a higher wear resistance and good mechanical properties.

Originality/value: The obtain results show the possibility of creation the newest materials base on X6Cr13 steels with better properties caused by proper volume fraction of reinforcement.

Keywords: Properties; Stainless steel; X6Cr13, Powder metallurgy; Hardness, Wear resistance

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PROPERTIES

1. Introduction

Due to the popularity of the sintered stainless steels in industry which are widely used thank to their high corrosion resistance, but due to low hardness their mechanical properties are still very weak, it is necessary to conduct systematic research on the modification their properties, by means of laser surface remelting, alloying of stainless steel with hard particles, heat treatments or structure modification by powder metallurgy, leading to obtain material with the most favourable mechanical properties. In addition, during modification of stainless steel should also take into account economic considerations and ease of use of technology in industrial practice [1-11].

Among the available methods for producing and modifying structure and properties of corrosion-resistant steels special place take up powder metallurgy which is one of the modern technology of metallic materials and subject of has a constant interest from researchers and companies, because of competitivity but also complementarity to the traditional methods. Despite the limitations (e.g. problems with the production of products of complex shape, high porosity), powder metallurgy occupies a special place among other methods of production and processing of materials. Automotive industry are particularly interested of PM technology and the demand for parts made of metal powders are increasing every year [12-15].

Subject of study of this work are ferritic stainless steels which compared to austenitic steels are characterized by similar properties of strength and corrosion resistance, but at a reduced cost of manufacturing due to lower usage of expensive alloying elements. Because of its properties are used in many industries, in particular, are used for the element exposed to the environment of water vapor, nitric acid and acetic acid [5, 8-10, 16,17].

Review of domestic and foreign literature has shown that much attention is given to the modification of surface of PM stainless steel by laser surface remelting or alloying of stainless steel with hard particle [2,4,5] but less is about metal matrix reinforced by harder particles. It seems to be the development of a composite material with introduced into the structure of much harder particles, could lead to produce a new material exhibiting a higher resistance to abrasion while maintaining high strength properties and corrosion resistance comparable with respect to the material comprising in 100% of the base [9,10].

2. Materials and methodology

2.1. Materials

The aim of the present work is structure characterisation, properties (hardness, wear resistance) and density investigations of initial powders and sinter X6Cr13 steel with addition of duplex stainless steel powder.

The investigations were made on the composite materials obtained with the powder metallurgy methods of ferritic stainless steel X6Cr13 reinforced with the super duplex stainless steel X2CrNiMo25-7-4.

The based material used in this research is a powder of low carbon ferritic stainless steel AISI 410L (X6Cr13) which are all available from Höganäs, Sweden and have been obtained by gas atomization and show a spherical morphology (Fig. 1) [17].

The standard and studied chemical compositions of investigated powders are given in Table 1.

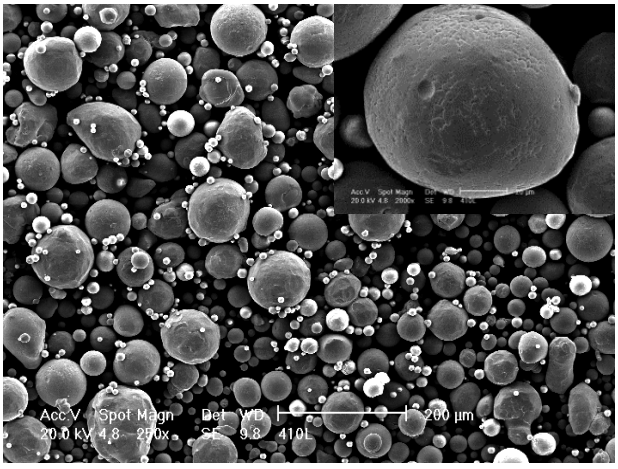

Fig. 1. Morphology of ferritic stainless steel X6Cr13 powder

The second material that used in this research is powder of AISI-F53 (Zeron 100, X2CrNiMo25-7-4) super duplex stainless steel, produced by Carpenter, a highly alloyed super duplex stainless steel with highly resistant to corrosion provided by the combination of elements such as Cr, Mo, W, and N. X2CrNiMo25-7-4 is sensitive to the precipitation of α' in the temperature range 300 to 600°C and secondary phases such as χ-phase, σ-phase at the temperature range 700 to 1000°C (Fig. 2). The hardness of duplex stainless steel is higher than ferrite steel, which is directly associated with a higher strength of two-phase structure. Higher hardness causes that the duplex steels have good resistance to wear resistance and erosion [18].

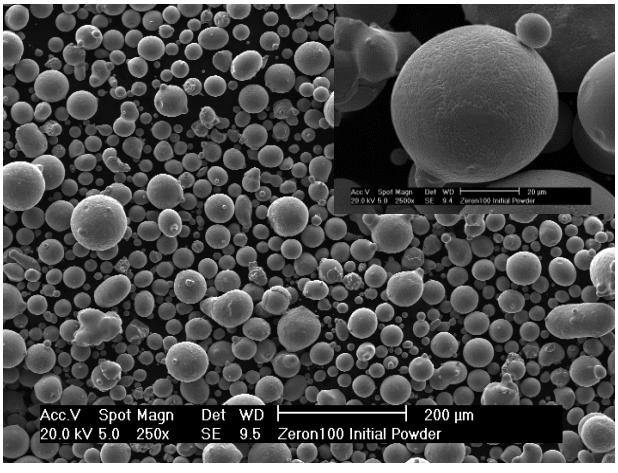

Fig. 2. Morphology of as-atomized super duplex stainless steel X2CrNiMo25-7-4 powder

Table 1.
Chemical compositions of the powder X6Cr13 (wt.%)

		C	Cr	Ni	Mn	Si	N	Fe
X6Cr13	Standard *	0.02	12.4	-	-	0.8	0.03	Bal.
	Initial powder **	0.02	12.9	0.59	0.98	0.86	0.03	Bal.

* Company standard ** Semiquantitative analyse the composition

Table 2.
Chemical compositions of the powder X2CrNiMo25-7-4 (wt.%)

		C	Cr	Ni	Mn	Si	Mo	Cu	N	W	Fe
X2CrNiMo25-7-4	Standard *	max 0.03	24.0-26.0	6.0-8.0	max 1.0	max 1.0	3.0-4.0	0.5-1.0	0.2-0.3	0.5-1.0	Bal.
	Initial powder **	0.03	25.72	6.94	1.09	0.41	3.0	1.17	0.2	1.89	Bal.

* Company standard ** Semiquantitative analyse the composition

The standard and studied chemical compositions of investigated powders are given in Table 2.

2.2. Experimental procedures

Powder X2CrNiMo25-7-4 were subjected to heat treatment to obtain precipitation of sigma phase. In order to obtain secondary precipitates in the steel in the form of sigma phase it was necessary to apply thermal aging treatment. Based on the literature references [18] identified the optimal annealing temperature and time for duplex stainless steel which amounted to 900°C and 10 hours. Selection of parameters for the duplex steel is quite high due to a fairly wide temperature range in which it is possible to obtain a sigma phase. Ageing treatments were performed by holding in the vacuum furnace for 10 hours at temperatures 900°C following cooling with furnace.

After aging the powder X2CrNiMo25-7-4 has been milled (dispersions by comminution by foreign body) in a planetary mill Pulverisette 6. The powder was placed in a drum with a diameter 75 mm and milled in four batches of 40 g. Steel balls of 15 mm in diameter were used, with a ball-to-powder mass ratio of 10:1. The powder was milled for 8 hours, each 15 minutes of milling was separated via 15 minute break. Rotation speed was 400 rpm, ensure a proper process of milling. The powder in the drum was protected by the inert atmosphere of argon, which prevents the increase of oxygen concentration in the powder. As a result of milling there is a change in shape, particle size and the parameters characterizing the powder as the apparent density, flowability and thus changed the geometric and structural activity of the powder.

Powder X2CrNiMo25-7-4 with grain size < 160 μm were mixed in appropriate proportions with the powder X6Cr13 with a grain size < 160 μm for 30 minutes in a tubular mixer. Mixing was realized to produce a powder with a uniform and stable distribution of the ingredients in the following proportions:

- 5% X2CrNiMo25-7-4 + X6Cr13 (marked as B);
- 10% X2CrNiMo25-7-4 + X6Cr13 (marked as C);
- 15% X2CrNiMo25-7-4 + X6Cr13 (marked as D).

The study involved also a specimen of the 100% of the powder ferritic stainless steel X6Cr13 (marked as A) for comparative purposes.

The specimens were obtained by cold isostatic pressing (CIP) in wet-bag. Ferritic powder X6Cr13 and three mixtures of powder X2CrNiMo25-7-4 and X6Cr13 with different mass ratio were poured into a containers made of rubber. Samples have been pressed under pressure 350 MPa at 5 min. Selected pressure allowed to obtain compacts of irregular shape, and the same method enabled to prepare the material without having to use lubricants or binders, which are necessary in case of need to reduce friction between the powder and the mould walls, like in the case of cold pressing in dies. It is also beneficial because of the elimination of contaminating problems of compacts. Lubricant is not applied also in order to avoid having to remove it from the compacts before sintering appropriate.

Green parts was free sintered in vacuum furnace Carbolite HVT. During sintering is necessary to ensure a sufficiently high temperature and time, which will sufficiently homogenize the structure, in this case selected following parameters of sintering: 1250°C for 1 h (cooling with furnace).

In order to perform the necessary studies metallographic and mechanical properties sample was prepared by: sectioning and cutting - cutting through water-cooled diamond saw, mounting - in a conductive resin (including the use of test samples for scanning electron microscope), hand grinding on SiC abrasive paper with water-cooled with the following granularity: 180, 320, 600, 1000 at a speed of 300 rpm and polishing - performed using emulsion Al_2O_3 (1.0 and 0.3 μm) on cloth (the polishing speed was 250 rpm).

2.3. Methodology of research

X-ray diffraction analysis has been performed on the initial and heat treated powder on the X'Pert PRO diffractometer to examined the presence of phases. The analysis have been carried out on powder samples prepared by embedding them on a silicon zero background plate and Cu Kα1 radiation (average

λ = 1.54056 Å), with an angular step of 0.02°, measuring angle 10° to 120° and counting time of 0.65 s.

Particle size analysis was carried out on Mastersize 2000 particle size analyzer with Hydro 2000SM (small volume sample dispersion unit) by laser diffraction method (dispersion in water). For each sample three measurements were performed, where the average is presented in the form of grain size distribution curve. In the case of grain-size measurements of X6Cr13 steel it was necessary to apply six droplets of dispersant agent named Dolapix, to help facilitate dispersion and improve the dispersion.

Densities of the sintered samples were determined using the method which measuring dry density ρ_s [g/cm^3] - mass per unit volume of a sintered, non-impregnated P/M part - according to MPIF standard 42. This method consists of measuring the density of sintered metal by weighing it in air and water, and then calculating the density expressed by the Equation (1):

$$\rho_s = \frac{A\rho_w}{B-C} \tag{1}$$

where:
A - mass of the dry test piece, g;
ρ_w - density of distiller water, g/cm^3;
B - mass of the oil impregnated test piece, g;
C - mass of the piece in water, g.

Based on the theoretical and the actual density of sintered materials porosity was calculated according to the formula:

$$P = \left(1 - \frac{\rho_s}{\rho_t}\right) \cdot 100\% \tag{2}$$

where:
P - porosity, %;
ρ_s - actual density of sinter, g/cm^3;
ρ_t - theoretical density of sinter, g/cm^3.

Hardness tests were carried out using a Vickers stationary indenter Wilson Wolpert 930. When testing a diamond indenter was pressed perpendicular to the sample rate of force aggravating 294.2 N. After relieving the length was measured diagonally depth formed on the sample surface.

Vickers microhardness $HV_{0.5}$ tests were carried out on hardness tester Zwick/Roell ZHU 2.5 according to standard PN-EN ISO 14577-1:2005P.

Tribological tests were performed using the CSM Instruments pin-on-disc tribometer with the aid of counter-sample from the aluminum oxide in form of 6 mm ball and process parameters like sliding speed 4.5 cm/s, sliding distance 3 mm, load 10 N. The investigation of abrasive wear resistance was carried out for all four types samples, also determined coefficient of friction. As a measure of friction wear assumed the cross-section area of wear trace, which geometry is measured by profilometer Tylor-Hubson Sutronic 25.

Obtained samples were examined by confocal microscopy on LSM Exciter 5 ZEISS for depth of the wear traces observation.

3. Results and discussion

3.1. Properties of powders

Both the steel powders used in the study are characterized by a spherical shape of the particles. That shape not promotes compressibility during pressing but in the case of sintering is preferable if the powder particles have irregular shape and rough surface, which affect the better contact between particles and increase internal surface area promote sintering. In our case, the use of cold isostatic pressing technique has allowed a proper compressibility in the materials.

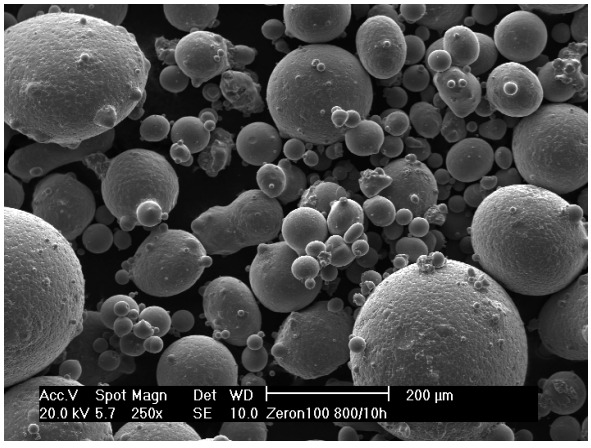

Fig. 3. Morphology of coarse particles of super duplex stainless steel X2CrNiMo25-7-4 after aging at 900°C for 10 h, SEM

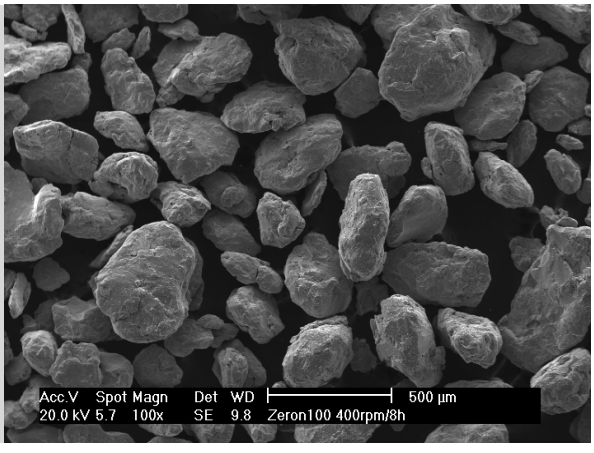

Fig. 4. Morphology of particles of super duplex stainless steel X2CrNiMo25-7-4 after milling

Figure 2 shows morphology of X2CrNiMo25-7-4 powder before aging studied on scanning electron microscopy. Typically spherical morphology as well as clean surfaces were observed

in all powder and this indicates that the powder was produced by gas atomization. Some particles showed evidences of satellite formation, which was caused by attachment of smaller solid powder particles onto larger melt droplets. In Figure 3, it can be seen a view of the powders after the heat treatment. Some of the smaller particles have started to sinter and have formed some agglomerates.

After milling procedure the spherical shape of particles has been modified into the typical sharp (irregular) morphology of the milled materials (Fig. 4). The morphology, size and distribution of the particles was changed.

On the basis of Schaeffler diagram equivalents (Ni-equivalent and Cr-equivalent) were calculated for powder X2CrNiMo25-7-4 based on the results of the analysis of chemical composition (Table 2). Cr- equivalent (29.3) and Ni-equivalent (8.4) define the test steel as steel with a ferritic-austenitic structure. XRD analysis helped clarify the obtained result (Fig. 5). X-ray qualitative phase analysis confirmed the existence of α and γ phases in the structure of powder X2CrNiMo25-7-4.

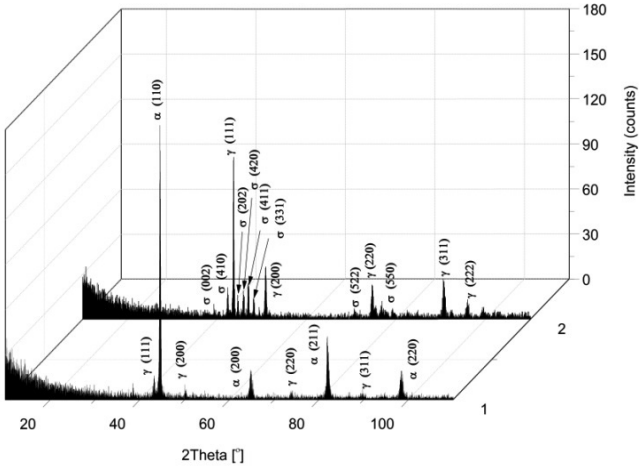

Fig. 5. X-ray diffraction patterns of powder X2CrNiMo25-7-4: 1) initial powder, 2) after heat treatment, 900°C/10 h, precipitation of σ-phase

Analyses of X2CrNiMo25-7-4 powder showed a four peaks (110), (200), (211) and (220), typical for the cubic structure of BCC, e.g. ferrite and four peaks (111), (200), (220) and (311) for the cubic structure of FCC, e.g. austenite.

X-ray diffraction pattern confirmed the existence of two-phase structure of ferrite-austenitic steel powder typical for stainless steel type duplex.

In order to verify the phase transformations occurring in the duplex stainless steel under heat to a temperature above 900°C (near to sintering temperature) annealing heat treatment was performed.

X-ray diffraction study show appear of precipitates of sigma phase, the dissolution of the ferrite to the secondary austenite and sigma phase (α→σ+γ').

The aging parameters: temperature 900°C and time 10 hours caused an increase in the intensity of (200), (220) and (311) austenite peaks and the appearances of nine peaks of sigma phase precipitates.

The appearance of sigma phase caused decay all peaks from ferrite, indicating at complete decomposition of the α-phase and simultaneously the intensity of the (111), (200), (220) and (311) austenite peaks considerably increased.

Analyse of X6Cr13 powder showed a five peaks (110), (200), (211) (220) and (310) typical for the cubic structure of BCC, ferrite (Fig. 6).

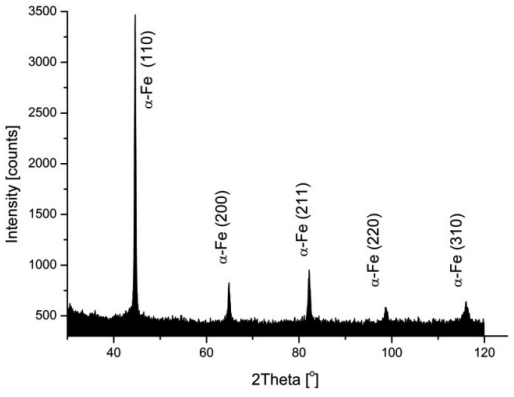

Fig. 6. X-ray diffraction patterns of powder X6Cr13

Particle size distribution

Among the several available methods of particle size analysis (e.g. sieve, image and sedimentation analysis) was use the technique of laser beam due to its accuracy and speed of measurement.

Particle size distribution was examined onto four samples: powder X2CrNiMo25-7-4 before heat treatment, after aging and after grinding as well as the X6Cr13 powder in line with the standards ISO-13320 Particle size analysis - Laser diffraction methods.

The results of research are given in Table 3, while the particle size distribution of X2CrNiMo25-7-4: initial powder, after aging, after milling and X6Cr13 powder are shown on Figures 7 and 8 respectively.

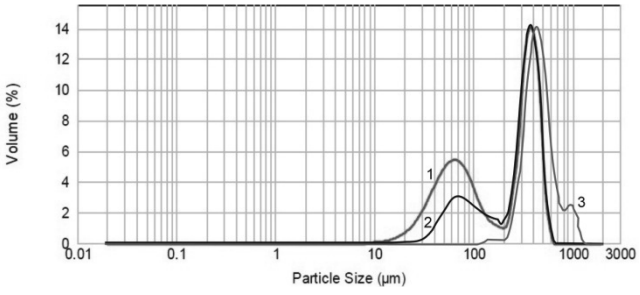

Fig. 7. Particle size distribution of initial powder X2CrNiMo25-7-4 (1), after heat treatment at 900°C/10 h (2), after milling at 400 rpm/8 h (3)

Table 3.
Particle size distribution of super duplex stainless steel X2CrNiMo25-7-4 and ferritic stainless steel X6Cr13

Material		X2CrNiMo25-7-4 IP*	X2CrNiMo25-7-4 HT**	X2CrNiMo25-7-4 M***	X6Cr13
		Particle size distribution (wt.%)			
Particle size [μm]	> 160	54.17	71.62	99.26	-
	160-71	16.81	15.38	0.74	33.95
	71-50	12.07	7.98	-	20.69
	< 50	16.94	3.15	-	45.34
Average grains size [μm]	d (0.1)▪	39.389	63.165	293.642	25.172
	d (0.5)▪	247.822	311.368	423.006	57.876
	d (0.9)▪	439.695	447.434	707.552	105.206
Specific surface area [m^2/g]		0.00811	0.00477	0.00185	0.017

* Initial powder ** After ageing at 900°C/10 h *** After milling at 400 rpm/ 8 h
▪ d (0.1) - particle size, below which there is a 10% vol. sample population, d (0.5) - particle size, below which there is 50% vol. sample population, d (0.9) - particle size, below which is 90% vol. sample population

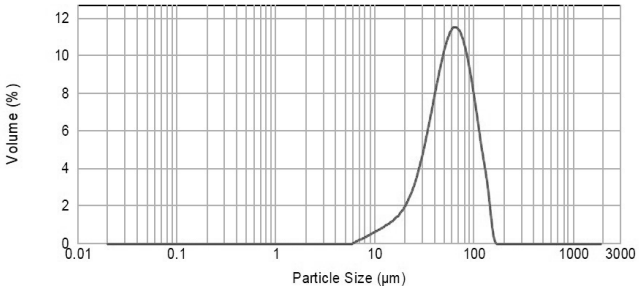

Fig. 8. Particle size distribution of ferritic powder X6Cr13

Powder particle size distributions, shown on Figures 6 (curve marked as 1), showed the existence of two fractions (bimodal distribution). Coarse fraction contains a particle size from 440 microns for initial powder X2CrNiMo25-7-4 and from 447 microns for this powder after heat treated (curve marked as 2).

Size distribution for X2CrNiMo25-7-4 powder after milling for 8 h (Fig. 6 - curve marked as 3) showed that the particles have significantly increased their size, this means that they have undergone agglomeration. The sample milled powder was collected for testing at random from the whole volume of powder, however as a result of screening in order to extract the fraction <160 μm was also achieved particles < 75 μm. It would therefore significantly increase the milling time, until the destruction of presence agglomerates.

At each stage of the preparation of duplex steel powder showed an increase in the average grain size in comparison to the size of the particles before aging and milling (Fig. 6 - curves marked as 2 and 3). The average grain size after heat treatment has increased due to sintering powder particles during its execution. The temperature about 900°C caused surface diffusion of alloying elements between single powders particles which led to the formation of agglomerates.

On the other side, the powder steel X6Cr13 shows the homogeneity of grain size, which does not exceed the maximum size of 160 μm and 90% of the share falls below the average grain size of 58 μm.

3.2. Properties of composites

Density

Theoretical density both of materials is follows, for super duplex stainless steel X2CrNiMo25-7-4 $\rho = 7.84$ g/cm^3 and for ferritic stainless steel X6Cr13 $\rho = 7.8$ g/cm^3. Theoretical density of mixtures were calculated from the density of the two steel powders, but the percentage of powder X2CrNiMo25-7-4 does not significantly influence the density of mixtures.

Percentage of the density regarding the theoretical one is shown in Table 8. Taking into account the followed processing route, these values can be considered acceptable.

The measurement results are shown in Table 4.

Sintered densities of the studied samples showed no significant difference. The highest density show samples with 10 (C) and 15% (D) of the X2CrNiMo25-7-4 content. This increasing in the density values can be explained due to the higher activity during sintering of the mechanically milled powders.

Omission the use of wax which facilitate the formation of hard metal powders, especially with spherical morphology and strain hardening during milling or use of too low pressure cold isostatic pressing may be reason of high porosity of obtained materials.

Table 4.
Percent of theoretical density sintered mixtures

	Materials	Theoretical density ρ_t, g/cm^3	Actual density ρ_s, g/cm^3	Porosity, %
A	X6Cr13	7.8	6.33	18.85
B	X6Cr13 + 5% X2CrNiMo25-7-4	7.8	6.47	17.05
C	X6Cr13 + 10% X2CrNiMo25-7-4	7.804	6.59	15.57
D	X6Cr13 + 15% X2CrNiMo25-7-4	7.806	6.5	16.73

Macro- and microhardness

Average macrohardness of the studied composites is between 50 to 71 HV (Fig. 9). The highest hardness shows the material marked as D, which is the result of a large contribution of powder X2CrNiMo25-7-4.

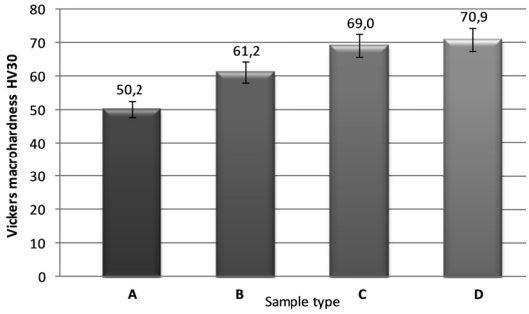

Fig. 9. Vickers macrohardness HV_{30} for sintered materials

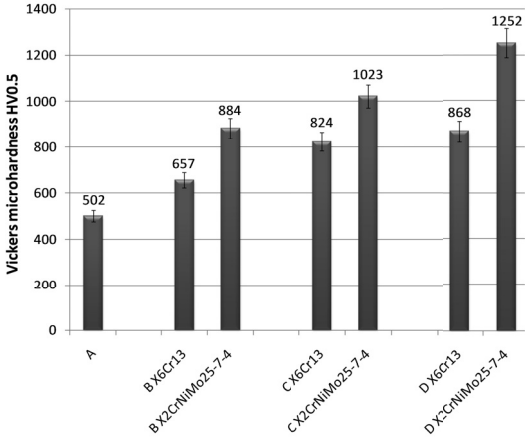

Fig. 10. Vickers microhardness $HV_{0.5}$ for different grain in sintered materials

It can be noted that the increasing of amount of reinforcement particles X2CrNiMo25-7-4 cause increase of micro or macro hardness. Only 5% additive powder steel X2CrNiMo25-7-4 in X6Cr13 stainless steel increases the hardness by 11 HV units.

Average microhardness values of all the phases present in the different materials are shown in Figure 10.

In all the cases can be seen the lower value of the soft phase (on the X6Cr13 former powder), and the hard phase (on the X2CrNiMo25-7-4 former powder). The value in the microhardness in the materials with higher amount of additives could be explained by a possible interdiffusion on alloying elements among both materials. In some particular cases, some of the micrograins of the former particles of X2CrNiMo25-7-4 show microhardness values higher than 1250 HV0.5.

Wear resistance

Figures 11-14 (a) shows the shape and depth of cross-section wear tracks of tested materials and Figures 10-13 (b) shows changes of topography of the investigated samples after the abrasion wear tests.

The smallest abrasive wear characterized sample with 5 and 10% of the reinforcement powder addition for which the cross-section area and depth of wear tracks were significantly less compared to the sample without and with 15% X2CrNiMoN25-7-4 powders addition.

The average volume of the abrasion trace developed in contact of the investigated surface layer with the counter-specimen material (Al_2O_3 ball with diameter 5.55 mm) was calculated based on the abrasion wear resistance tests. The average value of surface area of samples without and with 5%, 10% and 15% of reinforcement particles amount to respectively:P_0 = 102091 μm^2, P_5 = 41493 μm^2, P_{10} = 36245 μm^2, P_{15} = 120385 μm^2.

Small amount of reinforcement powder improved the value of wear resistance of base material but 15% additive caused reduction of wear resistance presumable because of the change the bond between the reinforcement and the matrix.

4. Conclusions

This work present the characterisation of materials using to obtain new composite and influences of different concentration of reinforcement in form of X2CrNiMo25-7-4 steel powder on the properties of sintered ferritic stainless steel X6Cr13.

Based on observations and researches formulated followings conclusions:

1. Five-percent mass fraction of X2CrNiMo25-7-4 powder in obtained sinter materials causes violent increase of macrohardness to average value 61 HV, while next increase of mass fraction of this powder to 10 and next 15% causes increase the hardness to 69 and 70HV respectively. Given this, addition of X2CrNiMo25-7-4 powders to X6Cr13 steel shouldn't exceed 10%, because further increasing the mass fraction of additive don't influence on hardness increase.
2. The researches of microhardness of single particles of X2CrNiMo25-7-4 and X6Cr13 powders, after mixing and annealing in high temperature confirm the increase their average values. Nature of increase is linear and dependent on mass fraction of X2CrNiMo25-7-4 steel, which equal 5, 10 or 15%. This effect is presumably connected with interdiffusion between particles of both steels.

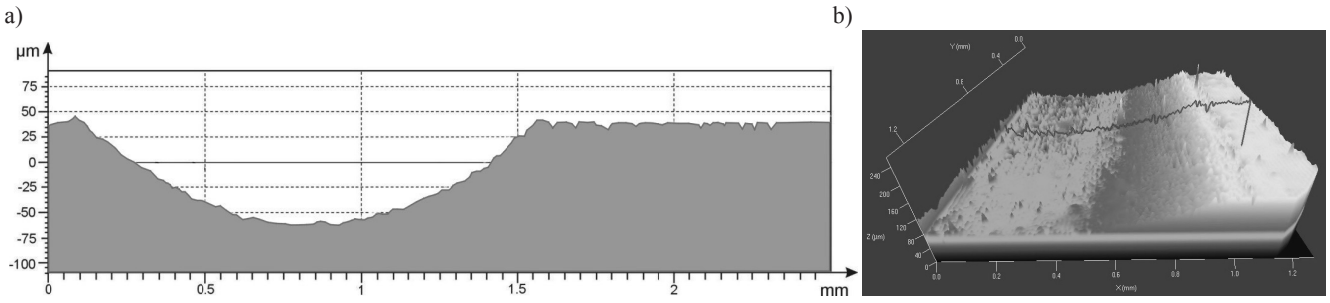

Fig. 11. Shape and depth (a) and half topography (b) of the wear trace after the abrasion wear test of X6Cr13steel

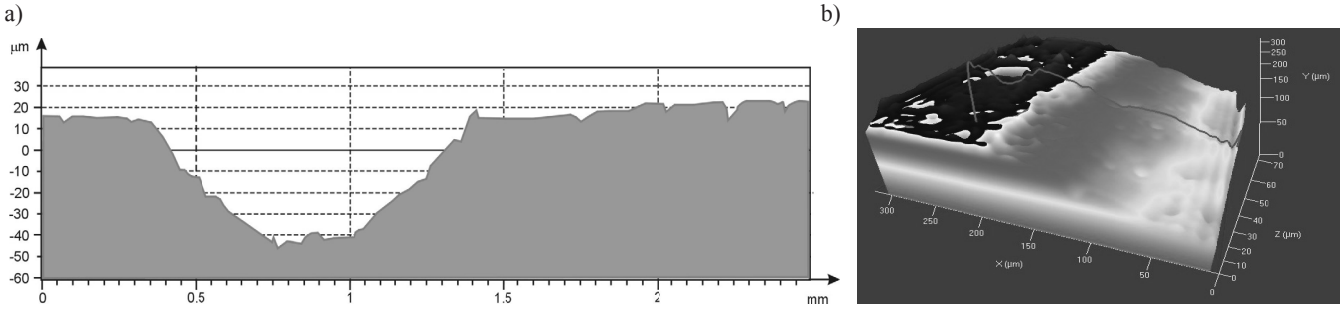

Fig. 12. Shape and depth (a) and half topography (b) of the wear trace after the abrasion wear test of X6Cr13steel with 5% addition of X2CrNiMo25-7-4 steel powder

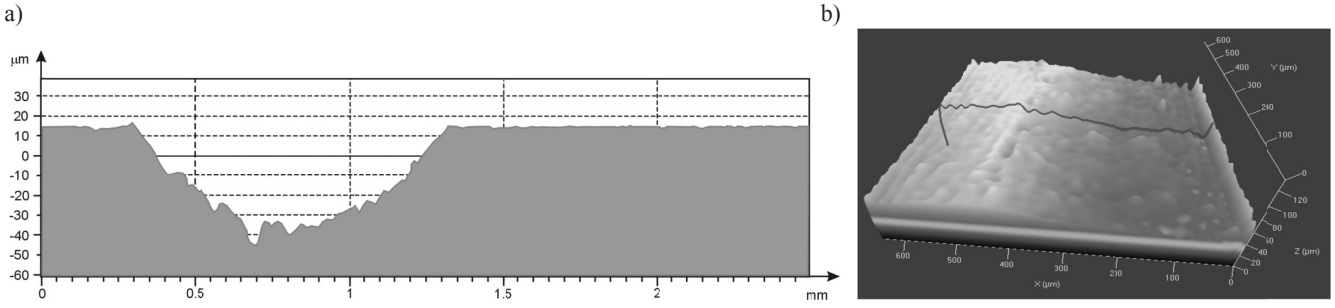

Fig. 13. Shape and depth (a) and half topography (b) of the wear trace after the abrasion wear test of X6Cr13steel with 10% addition of X2CrNiMo25-7-4 steel powder

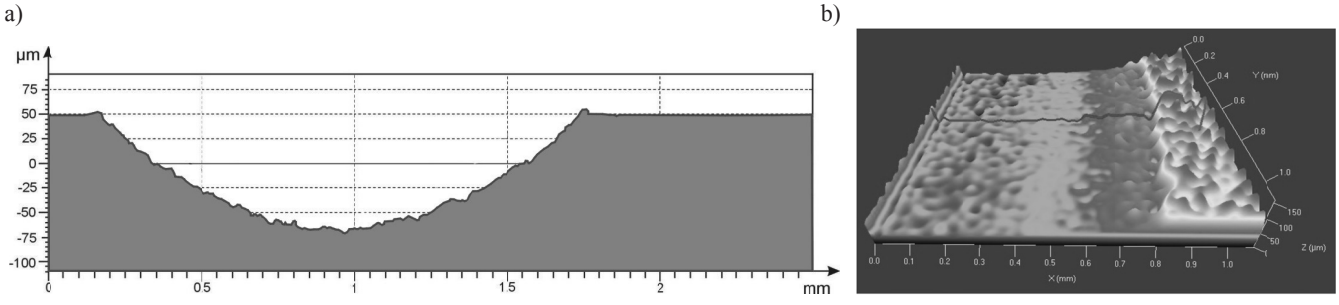

Fig. 14. Shape and depth (a) and half topography (b) of the wear trace after the abrasion wear test of X6Cr13steel with 15% addition of X2CrNiMo25-7-4 steel powder

3. Increase of hardness influences on wear resistance which confirm investigations. Materials containing 5 and 10% of X2CrNiMo25-7-4 powders be characterised by decrease the surface area of wear track. Only 5% addition of X2CrNiMo25-7-4 powders, causes decreases of surface area wear track about 59.4%, while 15% addition of X2CrNiMo25-7-4 powders decreases of wear resistance presumable by reason of pull out of hard particles from matrix.
4. Cold isostatic pressing of powders of X6Cr13 steel reinforced by X2CrNiMo25-7-4 powders under a pressure of 350 MPa for 5 minutes and subsequent sintering in vacuum (1250°C/1 h), allowed to obtain a density about 80% theoretical density. Forecasted that further researches of selection of forming and sintering conditions of those mixtures will allow increase the density.
5. Obtained investigations indicate on advisabillity applying of X6Cr13 and X2CrNiMo25-7–4 mixtures and heat treatment to obtain sigma phase, which causes increases in hardness of sintering materials compare to X6Cr13 steel.

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