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PROCESSING AND PROPERTIES OF DISTALOY SA SINTERED ALLOYS WITH BORON AND CARBON

WYTWARZANIE I WŁAŚCIWOŚCI SPIEKANYCH STOPÓW DISTALOY SA Z DODATKIEM BORU I WĘGLA

Prealloyed iron-based powders, manufactured in Höganäs Company, are used in the automotive parts industry. The properties and life time of such sintered parts depend, first of all, on their chemical composition, the production method of the prealloyed powder as well as on the technology of their consolidation and sintering. One of simpler and conventional methods aimed at increasing the density in sintered products is the process of activated sintering, performed, for example, by adding boron as elementary boron powder. Under this research project obtained were novel sintered materials, based on prealloyed and diffusion bonded powder, type: Distaloy SA, with the following chemical composition: Fe-1.75% Ni-1.5%Cu- 0.5%Mo with carbon (0.55%; 0.75%) and boron (0.2%, 0.4% and 0.6%). Distaloy SA samples alloyed with carbon and boron were manufactured by mixing powders in a Turbula mixer, then compressed using a hydraulic press under a pressure of 600 MPa and sintered in a tube furnace at 1473 K, for a 60 minute time, in the hydrogen atmosphere. After the sintering process, there were performed density and porosity measurements as well as hardness tests and mechanical properties were carried out, too. Eventually, analyzed was the effect of boron upon density, hardness and mechanical properties of novel sintered construction parts made from Distaloy SA powder.

Keywords: prealloyed and diffusion bonded powder, carbon, boron, sintering, density, hardness, mechanical properties

Proszki stopowane na osnowie żelaza produkowane w firmie Höganäs znajdują zastosowanie w produkcji części motoryzacyjnych. Właściwości i czas eksploatacji tych elementów spiekanych zależą przede wszystkim od składu chemicznego i metody wytwarzania proszku stopowanego oraz od technologii jego zagęszczania, i spiekania. Jednym z prostszych i konwencjonalnych sposobów podwyższania gęstości wyrobów spiekanych jest proces aktywowanego spiekania np. poprzez wprowadzenie dodatku boru w postaci elementarnego proszku boru. W niniejszej pracy badawczej wytworzono nowe materiały spiekane z proszku stopowanego i wyżarzanego dyfuzyjnie typu Distaloy SA o następującym składzie chemicznym: Fe-1,75%Ni-1,5%Cu -0,5%Mo z dodatkiem węgla (0,25% i 0,55%) i boru (0,2%, 0,4% i 0,6%). Próbki z proszku Distaloy SA z dodatkiem węgla i boru otrzymano metodą mieszania proszków w mieszalniku Turbula, następnie prasowania na prasie hydraulicznej pod ciśnieniem 600 MPa i spiekania w piecu rurowym w temperaturze 1473 K, w czasie 60 min., w atmosferze wodoru. Po procesie spiekania przeprowadzono pomiary gęstości oraz porowatości próbek jak również badania twardości i właściwości mechanicznych. Przeanalizowano wpływ dodatku boru na gęstość, twardość i właściwości mechaniczne nowych elementów konstrukcyjnych z proszku Distaloy SA.

1. Introduction

One of simpler and conventional methods aimed at increasing the density of sintered steel products is the process of activated sintering, performed, for example, by adding boron in the form of elementary boron powder.

Madan and German (1) were the first researchers to have investigated into the boron effect upon the sintering process of iron-based alloys. In their experiments, they proved that boron not only intensifies the consolidation process in samples through a sintering process with participation of a liquid phase, but it also activates sintering processes occurring in the solid state. A 200 ppm addition of boron in the iron powder enables its segregation on grain boundaries and formation of a convenient diffusion layer.

In turn, Ernst (2) discovered that a 200 ppm boron addition will improve the resistance to creep in the case of martensite stainless steels, without affecting their ductility.

Thereafter, Toennes et al (2,3) analyzed the boron effect upon the microstructure and mechanical properties of sintered martensitic stainless steels. They added boron in quantities, respectively, of 50, 100, 150 and 200 ppm to martensitic powders of stainless steels and ascertained that at a boron level of approx. 150 ppm, the density of steels under investigation, subject to a 60 minute sintering at 1623 K, in the argon atmosphere, is equal to the 100% of theoretical density. At the same time, boron-modified martensitic steels distinguished themselves by high values of resistance – around 850 MPa, and of ductility, viz. about 16.5% (2-4).

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Then, there were also performed investigations aimed at determining the boron effect upon the sintering process of austenitic stainless steels (5-11); instead, paper (12) discusses the process of sintering ferritico-austenitic stainless steels with a boron addition. The 60 minute time sintering process of 0.4% boron stainless steels AISI 316L, performed in the hydrogen atmosphere at 1473 K will lead to a considerable consolidation of material through the sintering in a liquid phase which is generated as a result of an eutectic reaction occurring between the steel matrix and the boride phase (Fe, Cr, Mo)₂B (7, 10,13-16). The mechanical properties of sintered boron-modified stainless steels depend on the density of samples and their microstructure. The quantity of boron added should be the best possible so as to obtain an appropriate microstructure which should guarantee improved properties. The maximum tensile strength (over 450 MPa) was obtained for samples of 0.8% B stainless steel 316L, sintered for a 30 minute time in the hydrogen atmosphere at 1498 K. An analysis of fracture of those samples proved their ductility (4, 17).

Interesting experiments targeted at the effect of boron addition upon the processes of sintering maraging steel 18 Ni (350), with the following analysis: Fe-18% Ni-12.5 % Co-4.2% Mo-1.6% Ti-0.1%Al, were performed by Sercombe (18). Boron was added as Fe-3.9B, its quantity ranging from 0% do 0.4%. Powder mixtures were made by conventional sintering (1413 K – 1553 K), in a 4 hour time, in the argon atmosphere and through selective laser sintering (SLS). A part of samples were subject to heat treatment, viz. annealing, water quenching and ageing.

The said research proved that with increasing boron levels increases as well the density of sinters; the highest density values, close to the theoretical one, were obtained for the samples with 0.4% boron, sintered at 1513 K (4,18).

Boron was also used for sintering steels Fe-Mn-Cr-Mo, Fe-Mn-Mo and Fe-Ni-Mo-Cr-B (19-21). The said research fields are still of interest, which can be corroborated with the publication of new papers (22,23).

The explanation of the mechanism of sintering novel Fe-Mo-B-C construction parts can be found in articles (24-26).

Basing upon the investigations into the microstructure and phase analysis, it was ascertained that while sintering materials like Fe-(1.5-2%)Mo-(0.2-0.6%)B-(0.55-0.85%)C, an eutectic reaction occurring between the alloy matrix (Fe,Mo) and the complex of Fe-Mo-rich borides and carbide borides leads to a liquid phase due to which a consolidation will take place and mechanical properties will be improved. Instead, with increasing Mo levels (3-5%), the quantity of liquid phase on the grain boundaries will decrease, which results from the diffusion of boron and carbon to molybdenum and formation of bigger quantities of borides or carbide borides inside the grains.

The said hypothesis was confirmed by Sarasola et al. (27,28), who proved that the quantity of liquid phase in Fe-Mo-B sinters depends on the B/Mo ratio. In addition, due to the addition of carbon to Fe-Mo-B samples, they noticed yet more liquid phase.

This paper is focused upon making novel sintered materials from prealloyed and diffusion bonded powder, type: Distaloy SA (Fe-1.75%Ni-1.5%Cu -0.5%Mo) with addition, respectively, of carbon (0.55% and 0.75%) and of boron (0.2%,

0.4% and 0.6%). Samples made from powder Distaloy SA with addition of carbon and boron were obtained through a conventional powder metallurgy method. Analyzed was the effect of boron addition upon porosity, density and hardness of new Distaloy SA powder-based construction parts.

2. Experiments

In the investigations hereunder was used a prealloyed and diffusion bonded powder, type: Distaloy SA (Fe-1.75%Ni-1.5%Cu -0.5%Mo), manufactured by Höganäs company. The powder was supplemented then, respectively, with carbon (0.25 wt % and 0.55 wt %), and with boron (0.2 wt % 0.4 wt % and 0.6 wt %).

Carbon was added to the powder Distaloy SA as graphite. Since graphite will be in part burnt out in the sintering process, hence its excess is calculated from the following formula (29):

$$G = C + 3/8 \beta \cdot H \quad (1)$$

where: G – graphite contents, %

C – targeted carbon contents, %

H – hydrogen loss, %

β – coefficient depending on the powder specific area; for the powder SA $\beta = 2$.

Samples made from the powder Distaloy SA with addition of carbon and boron were obtained by mixing powders in a Turbula mixer, for a 15 minute; then, powder mixtures were subject to compression on a hydraulic press under 600 MPa and sintered in a hydrogen atmosphere in a tube furnace, at 1473 K, for a 60 minute duration time. After the sintering process, there were performed density and porosity measurements. For the sinters under investigation calculated was the consolidation coefficient (by means of Lenel formula), and some hardness tests were carried out, too.

The density of compacts and sinters was investigated by hydrostatic and air weighing – viz. through Archimedes method – in line with the standard (PN-EN ISO 2738, December 2001). For each content of carbon and boron and in identical technology conditions prepared were five samples. The density of compacts and sinters were calculated as an arithmetic mean for five samples with an accuracy of up to 0.01.

Investigations into the hardness of sinters were performed with Brinell method (PN-EN-ISO 65061-1/2002). For each sample there were performed five hardness measurements. The result was determined as an arithmetic mean for five samples with accuracy to a unit. Mechanical properties of sinters were tested on an Instron apparatus, whereas microstructural investigations were carried out on a light and a scanning microscope.

3. Results and interpretation

An analysis of the results showed that an addition of 0.4 wt % B and 0.6 wt % B considerably increases the degree of consolidation of Distaloy SA sinters. The highest relative density – approx. 94% was obtained for Distaloy SA sinters with 0.6wt % B. Parallely ascertained was a reduction of porosity

to 6%. In carbon and boron-modified Distaloy SA samples ascertained was an increase in the value of real and relative density as the contents of those additions went up, but higher densities were obtained for sinters containing 0.55 wt % C and 0.6 wt % B. As boron and carbon contents increased in the samples under analysis, the porosity decreased, and the lowest porosity levels were obtained for sinters having 0.6 wt % B – both for 0.25 wt % C and 0.55 wt % C. The porosity of samples was, respectively, 9% and 8%. With increasing carbon and boron contents an increase in the real density was observed (Fig. 1).

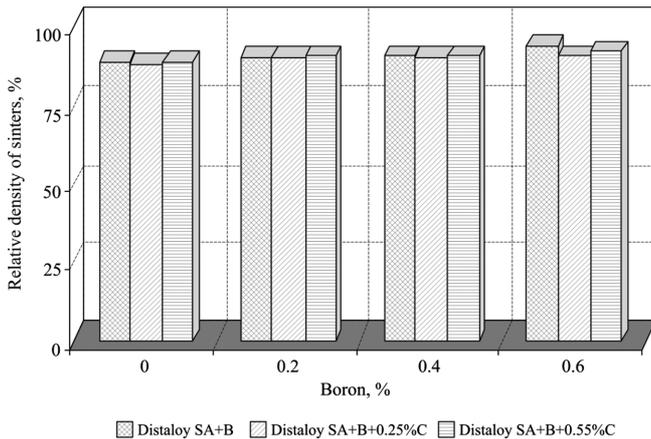


Fig. 1. Effect of boron on the relative density of Distaloy SA sinters with carbon addition. Sintering parameters: 1473 K/60'/hydrogen)

In novel materials sintered on the Distaloy SA powder matrix modified by carbon and boron, a boron addition did not cause such a consolidation as in the case of Distaloy SA sinters only with boron addition. Probably, this effect may be caused by a decrease in the liquid phase required for the consolidation as a result of the formation of carbides and carbido borides. (24, 25).

For the tested samples hardness and mechanical properties (yield strength – Ra and tensile strength – Rm) are presented. Generally, with increasing contents of boron and carbon in the sintered Distaloy SA steels, there were noticed elevated hardness values (Fig. 2). For samples with 0.55 wt % C ascertained were hardness values higher than for the samples with 0.25 wt % C (Fig. 2).

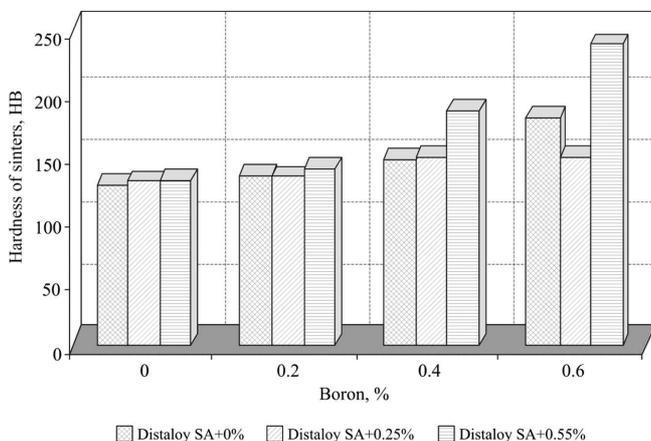


Fig. 2. Effect of boron on the hardness of Distaloy SA sinters with carbon addition. Sintering parameters: 1473 K/60'/hydrogen)

The highest compression rate and hardness were found in all samples with the maximum boron contents. The highest hardness values (186 HB and 239 HB), for a relative density of 91% and 92%, were obtained for Distaloy SA sinters with 0.55 wt % C and 0.4 wt % B and 0.6 wt % B (Figs 1, 2).

The highest yield strength (Ra) was observed by 0.4 wt % B and 0.55 wt % C (Fig. 3) but the highest tensile strength (Rm) by 0.2 wt % B and 0.55 wt % C (Fig. 4).

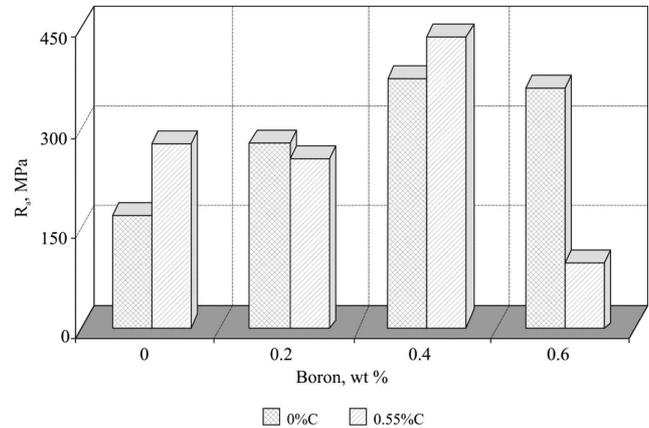


Fig. 3. Effect of boron on the mechanical properties (Ra) of Distaloy SA sinters with carbon addition. Sintering parameters: 1473 K/60'/hydrogen)

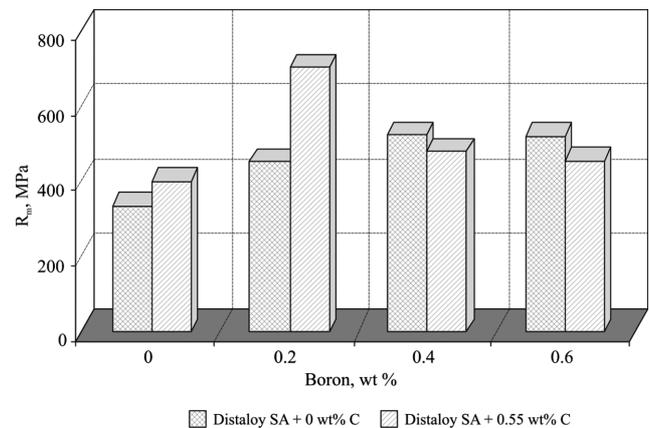


Fig. 4. Effect of boron on the mechanical properties (Rm) of Distaloy SA sinters with carbon addition. Sintering parameters: 1473 K/60'/hydrogen)

The effect related to an increased consolidation, hardness and the mechanical properties in the novel materials sintered from Distaloy SA, observed with increasing carbon and boron contents, is attributable to the sintering process of those alloys. Probably, while sintering those materials at 1473 K, as a result of the reaction occurring between the alloy matrix (Fe-Ni-Mo-Cu) and the complex of borides, type M_2B , carbides like M_3C or carbido borides, there will be generated a liquid phase that leads to a considerable degree of consolidation and improved mechanical properties (24, 27,28). The microstructural investigations showed that effect (Fig. 5, 6). During sintering at temperature 1473 K, a permanent liquid phase was generated as a result of eutectic reaction between iron and Fe_2B . Because of the limited boron solubility in

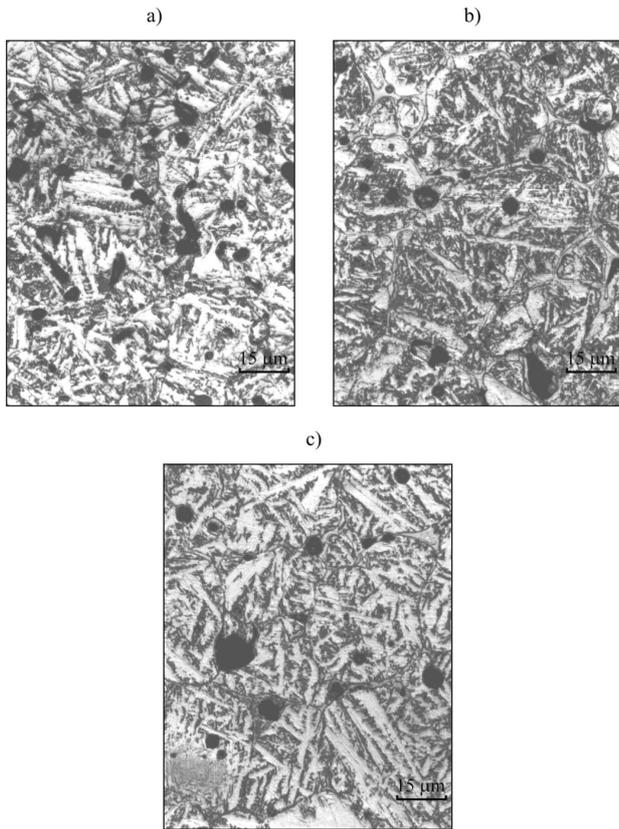


Fig. 5. Microstructure of Distalloy SA sinters with boron and carbon. Light microscope: a) 0.2 wt% B and 0.55 wt% C, b) 0.4 wt% B and 0.55 wt% C, c) 0.6 wt% B and 0.55 wt% C

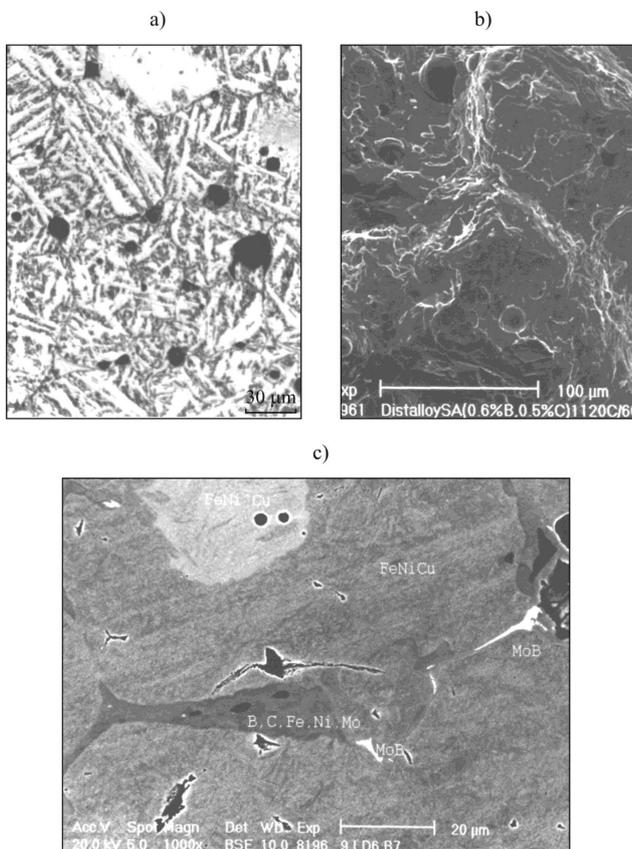


Fig. 6. Microstructure of Distalloy SA sinters with boron (0.6 wt%) and carbon (0.55 wt%): a) Light microscope, b) fracture, c) SEM/EDX

iron, a liquid phase was continuously present during the entire sintering process, and it influences on the morphology of the porosity and the increase in density (30), Fig. 5. In Distalloy SA samples containing 0.6 wt% B and 0.55 wt% C, very high eutectic contents deposited at grain boundaries (Fig. 6 a,c). The boride phase in the eutectic network was brittle (Fig. 6b) and affected the decrease in the ductility of examined samples and also blocked a further increase in yield and tensile strength (31).

4. Conclusions

1. Porosity, density, hardness and mechanical properties of novel, Distalloy SA matrix-based sintered materials depend upon the contents of carbon and boron.
2. With increasing contents of carbon and boron in the samples under investigation, ascertained was an increase in the values of density, hardness and mechanical properties whereas the porosity was reduced.
3. The said relationships are related to the mechanism of sintering process of novel sintered materials. While sintering those materials at 1473 K, as a result of the reaction occurring between the alloy matrix (Fe-Ni-Mo-Cu) and the complex of borides, carbides or carbide-borides, a liquid phase is created and leads to a considerable degree of consolidation and improved mechanical properties.

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