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Effect of Particle Size of a Powder upon the Properties and Microstructure of Boron-modified Fe-Ni-Mo-Cu Sinters

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Abstract

The article discusses the effect of different particle fractions of prealloyed iron powder on the microstructure, density and hardness of sintered material. Each particle fraction (apart from 160–200 µm, which is a trace fraction) was modified with boron, its contents being, respectively, 0.2 wt.%, 0.4 wt.% and 0.6 wt.%. Next, the powder mixtures were pressed under a pressure of 600 MPa, and the final compacts were subject to sintering at 1200°C for 60 min in a hydrogen atmosphere. It was observed that the higher values of density and hardness were found in samples made from finer fractions of powder. A higher homogeneity of the microstructure and the highest degree of compactness was obtained in sinters from powder of 40–56 µm particle size, with 0.4 wt.% B. Due to the use of small particle fractions of prealloyed powder, a higher degree of compactness in sinters was obtained with lower boron content. Also indicated was which particle fraction of Fe-Ni-Mo-Cu powder should be applied to obtain density in sinters with boron addition equal to almost 100% of the relative density of the analyzed alloy. The presented studies have both scientific and technological aspects.

Keywords:

processing, powder metallurgy, Distaloy SA powder, activated sintering, particle fraction

1. INTRODUCTION

Powder metallurgy is a technology aiming at the production of bulk parts without reaching melting temperature, which in the case of iron mixtures is usually well above 1000°C. Thus, it provides the opportunity to introduce elements and compounds with high melting points, as well as controlling the distribution of these phases. However, in order to successfully proceed in producing a bulk material from an iron-containing powder or powder mixture, there is a need for a liquid phase during sintering. It was observed by Madan and German [1] that boron addition acts sufficiently well as a liquid-forming element, improving sintering by lowering its temperature and forming eutectic regions on the inter-particle boundaries. An explanation of the sintering mechanism for Fe-based alloys can be found in [2–7]. The thermodynamic approach, including regions of phase diagrams where various borides can be formed, are presented in [7, 8].

The formation of the eutectic regions in the sintered samples plays also an important role in decreasing porosity which may increase mechanical properties. The effect of porosity on mechanical properties in Fe-Mo-Ni alloys in tensile and fatigue tests is described in [9]. The addition of boron to iron based sinters (Fe-Mo-B) results in a change in the deformation behavior from ductile to brittle with the increase of boron content [10]. It was found that boron activates a sintering process through a liquid phase formation as a result of a reaction between matrix elements (Fe, Mo, Ni) and borides of the $(Fe, Mo, Ni)_2B$ type [4, 5, 7]. In alloys containing a sufficient amount of boron, the liquid phase may form as quickly as 1176°C. The amount of the liquid phase is affected by copper which may decrease the temperature of the liquid phase formation [7]. Both processes increase the consolidation level and mechanical properties of the sintered samples. It has been proven that the chemical composition of Distaloy SA powder allows one to obtain appreciably higher mechanical properties for sinters alloyed with a lower boron addition.

Additionally, to understand this sintering mechanism better, a dilatometric study brings valuable information. The dilatometric study upon heating and cooling of Fe-Mo-B sinters was presented in detail with respect to the composition of specimens and to a heating rate in [11]. This liquid sintering process increases the consolidation level and provides higher mechanical properties of the sintered samples. It has been proven that the chemical composition of the alloyed powders allows appreciably higher mechanical properties to be obtained [10, 12–18].

The mechanical properties of sintered steels with boron and borides were analyzed in [9–20]. In order to enhance the mechanical and surface properties, especially the hardness and wear resistance of sintered alloys modified by boron, a heat and chemical treatment was applied [21, 22].

Moreover, applying finer particle fractions of alloyed powders makes it possible to acquire a higher consolidation level of sinters with a lower boron content. In this paper, the influence of different particle fraction of boron modified Fe-Ni-Mo-Cu powders on the density, hardness and microstructure of sintered samples was analyzed.

2. MATERIALS AND METHODS

A prealloyed Distaloy SA powder supplied by the Höganäs company with the composition Fe 1.75%Ni-1.5%Cu-0.5%Mo was used in the studies. The particle size of Distaloy SA powder (general particle fraction) is 20–180 μm. By means of screen analysis (PN-EN 24497, July 1999) using sieves with a mechanical shaker, the following particle fractions were determined and are listed in Table 1.

Table 1
Sieve analysis of Distaloy SA powder

Sieve size [μm]	Retained [%]	Cumulative mass retained [%]
160–200	0.11	0.11
100–160	25.21	25.32
71–100	29.63	54.95
63–71	7.63	62.58
56–63	3.54	66.12
40–56	19.70	85.82
< 40	14.18	100.00

Each particle fraction and a general one (20–180 μm) were modified with boron, respectively, and followed by mixing in a Turbula mixer for 15 minutes. The boron contents used in the studies were: 0.2 wt.%, 0.4 wt.% and 0.6 wt.%. It is worth noting here that 160–200 μm is a trace fraction, therefore it was not used in the further experiments. Additionally, boron-free samples were prepared for reference for each particle fraction. Five specimens were prepared for each composition and fraction. In the next step, powder mixes were pressed under 600 MPa in a mechanical press, and the final compacts were subjected to sintering. No pressure was imposed on samples during sintering. The sintering took place in a tube furnace at a temperature of 1200°C for 60 min in a hydrogen atmosphere as established in [8, 23].

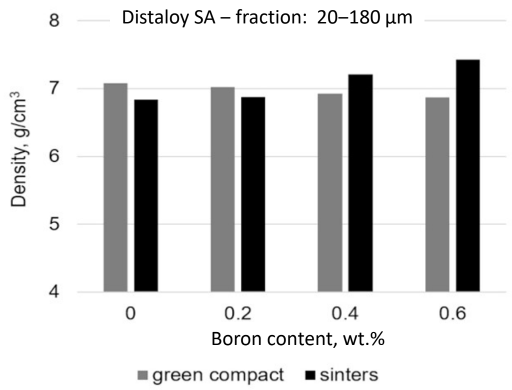
In successive studies, the density of compacts and Distaloy powder sinters per each particle fraction were investigated. The density of compacts and sinters was determined by means of the Archimedes method (PE-EN ISO 2738, December 2001). Samples were polished using Struers polishing machine. A set of abrasive papers of grade 500 to 2000 was used. The final polishing was with a cloth with a 1 μm diamond paste followed by etching with a HNO_3 water solution. Additionally, hardness was measured with Brinell's method (PN-EN ISO 65060-1, 2002). Structural observations were performed with an optical microscope Neophot 32. The effect of particle effect exerted upon the density, hardness and microstructure of boron-modified Distaloy SA sinters was analyzed.

3. RESULTS AND DISCUSSION

3.1. Density of green compacts and sinters

The determined densities of compacts and sinters made from various particle fractions of Distaloy SA powder (Table 1) with boron additions are shown in Figure 1a, b. According to the analysis of the density measurements of compacts modified with boron, irrespective of the boron content and particle fractions of the Distaloy SA powders, the density values of green compacts do not change and are about 6.9 g/cm^3. An example of such measurements for two chosen fractions, general and 40–56 μm ones, are presented in Figure 1a, b. Instead, the densities of Distaloy SA sinters with boron additions showed that particle fractions substantially influenced the degree of compactness of the samples under investigation. In all of the particle fractions used, the degree of compactness increased as boron contents rose (0.2–0.6 wt.%).

a)



b)

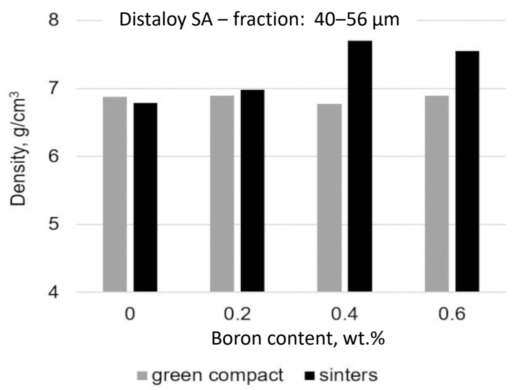


Fig. 1. Effect of particle fraction of powder and boron addition upon density of green compacts and sinters: a) 20–180 μm fraction; b) 40–56 μm fraction

The density of boron-free samples after pressing and sintering are comparable. The small drop of density is caused by the formation of smaller amounts of liquid phase based on copper (melting temperature 1084°C). On the other hand, in the case of lower boron contents (0.2 wt.% B), a thin layer of liquid eutectic is created at the particles' boundaries, which influences the raise of density. The liquid eutectic consists of (Fe, Ni, Mo)-complex borides [7]. However, with higher boron contents, a tendency toward higher densification of sinters due to the larger amount of the liquid phase is observed [7]. This tendency is well illustrated in Figure 2.

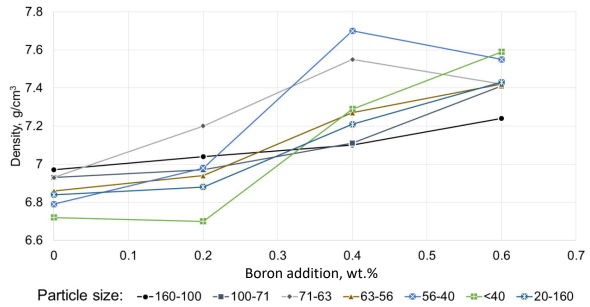


Fig. 2. Density of Distaloy SA sinters for all used powder fractions with boron addition in respect to particle fraction of powder

In the case of Distaloy SA sinters made based upon coarse fractions (100–160 μm, 71–100 μm, 63–71 μm, 56–63 μm) and general fraction (20–180 μm) with 0.6 wt.% B, lower densities were obtained if compared with sinters produced from fine fractions (40–56 μm and under 40 μm) with the same boron content (0.6 wt.% B) (Fig. 2).

During sintering the volume of samples decreases in relation to the green compacts. In the case of boron free sinters, the relative density is in a range of 85% to 89% for fraction under 40 μm and 100–160 μm, respectively. Due to addition of boron, the tendency is reversed and highest densities are achieved by finer powders; e.g. 99% and 98% for 40–56 μm fraction with 0.4 wt.% B or 0.6 wt.%, respectively.

The value of the relative density of Distaloy SA sinters with 0.6 wt.% B made from coarse fractions is 94% (fraction 100–160 μm) and 96% (fractions 71–100 μm, 63–71 μm and 56–63 μm and general fraction 20–180 μm); whereas, for fine fractions (40–56 μm and under 40 μm) – 98% (Tab. 2). In addition, it was observed that it is possible to get the highest densities at a lower boron level with decreasing particle size in sinters, viz. 0.4 wt.%. A conclusion from the analysis of the experiment results is that for each particle fraction used, the highest degree of compactness occurs at the increase in boron of contents from 0.2 wt.% to 0.4 wt.%. However, as the particle size of Distaloy SA diminishes at the same boron content of 0.4 wt.% B kept, the density values increase obtaining a value of 7.70 g/cm³ in case of 40–56 μm fraction (Fig. 1b). Nevertheless, a sinter made from fraction with particle size lower than 40 μm and also with 0.4 wt.% B presents a slightly lower density of 7.29 g/cm³, which corresponds to the relative density of 94% (Tab. 2, Fig. 2).

The highest relative densities were obtained for sinters from Distaloy powders SA, fractions 63–71 μm and 40–56 μm with 0.4 wt.% B (97% and 99%, see Table 2). The increase in density for increased boron contents from 0.4 to 0.6% is appreciably lower. The highest actual and relative density values for Distaloy SA sinters with 0.4 wt.% B were found for fine powders (viz. 40–56 μm and under 40 μm).

Table 2
Density of Distaloy SA sinters with boron addition in respect to particle fraction of powder [16]

Particle fraction [μm]	Boron [wt. %]	Theoretical density [g/cm³]	Real density [g/cm³]	Relative density [%]
100–160	0.0	7.93	6.97	89
	0.2	7.89	7.04	90
	0.4	7.85	7.10	91
	0.6	7.81	7.24	94
71–100	0.0	7.93	6.93	88
	0.2	7.89	6.97	89
	0.4	7.85	7.11	92
	0.6	7.81	7.41	96
63–71	0.0	7.93	6.93	88
	0.2	7.89	7.20	92
	0.4	7.85	7.55	97
	0.6	7.81	7.42	96
56–63	0.0	7.93	6.86	87
	0.2	7.89	6.94	89
	0.4	7.85	7.27	94
	0.6	7.81	7.42	96
40–56	0.0	7.93	6.79	86
	0.2	7.89	6.98	89
	0.4	7.85	7.70	99
	0.6	7.81	7.55	98
< 40	0.0	7.93	6.72	85
	0.2	7.89	6.70	86
	0.4	7.85	7.29	94
	0.6	7.81	7.59	98
20–180	0.0	7.93	6.84	87
	0.2	7.89	6.88	88
	0.4	7.85	7.21	93
	0.6	7.81	7.43	96

3.2. Hardness of sinters

While analyzing the results of hardness for boron-modified Distaloy SA sinters vs. particle size of Distaloy SA powder, it was found that irrespective of the used fraction, as boron content in the samples under investigation increases, their hardness will also increase. Additionally, it was observed that the highest average hardness was obtained for the finest fractions, viz. 40–56 μm (145 HB at 0.2 wt.% B, 190 HB at 0.6 wt.% B, see Fig. 3) and under 40 μm (152 HB at 0.2 wt.% B, and 223 HB at 0.6 wt.% B, see Fig. 3).

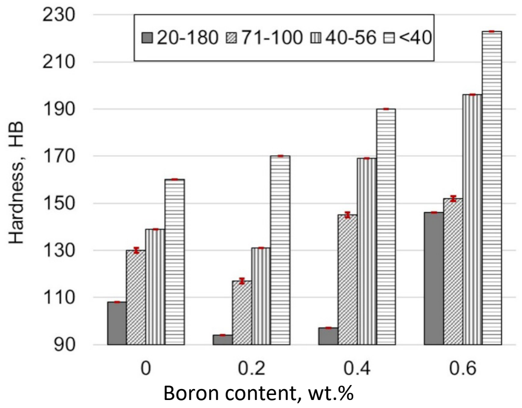


Fig. 3. Effect of powder particle fractions and boron contents upon the hardness of prepared sinters: fraction 20–180 μm, 71–100 μm, 40–56 μm, and < 40 μm

It should be also underlined that sinters, made from the fraction under 40 μm are characterized with appreciably higher hardness irrespective of boron contents; for example: sinters from general fraction (20–180 μm) with 0.2 wt.% and 0.6 wt.% B have the following average hardness values: 130 and 160 HB (Fig. 3). Distaloy sinters made from general fraction (20–180 μm) have appreciably lower hardness values than sinters from undersized fraction (Fig. 3). So, the hardness value of sinters under investigation depends on boron contents and particle size (Fig. 3). As the Distaloy SA particle size decreases, and boron contents increase, the hardness values in sinters under examination were considerably higher. The hardness of Distaloy SA sinters depends on their degree of compactness and the particle size of Distaloy SA powder used – the finer the particle fraction, the higher the density and hardness of the prepared sinters (Tab. 2, Fig. 3).

3.3. Microstructure observations of sinters

Microscopic observations of boron-modified Distaloy SA sinters showed that irrespective of the particle fraction of the powder used, the microstructure consists of an iron matrix with a solidified, non-homogeneous eutectic mixture visible upon iron particles boundaries. In addition, precipitates of fine borides were noticed in the ferrite matrix.

On the other hand, porosity seems to quite noticeable in boron-free samples (Figs. 4a, 5a, 6a, and 7a) and also in the samples with lowest boron content (Figs. 4b, 5b, 6b, and 7b).

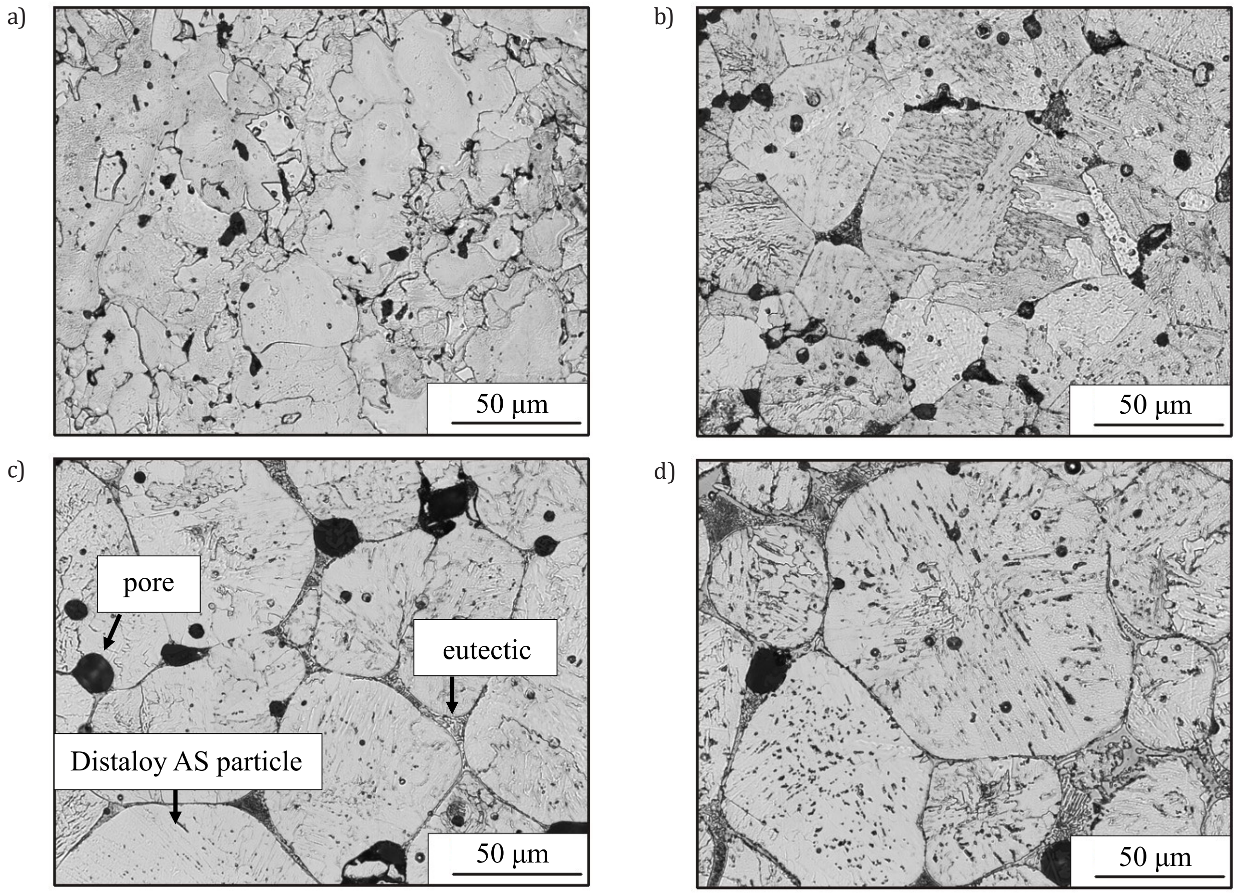


Fig. 4. Microstructure of Distaloy SA sinters alloyed by boron for 20–180 μm particle fraction: a) 0.0 wt.% B; b) 0.2 wt.% B; c) 0.4 wt.% B; d) 0.6 wt.% B [7]

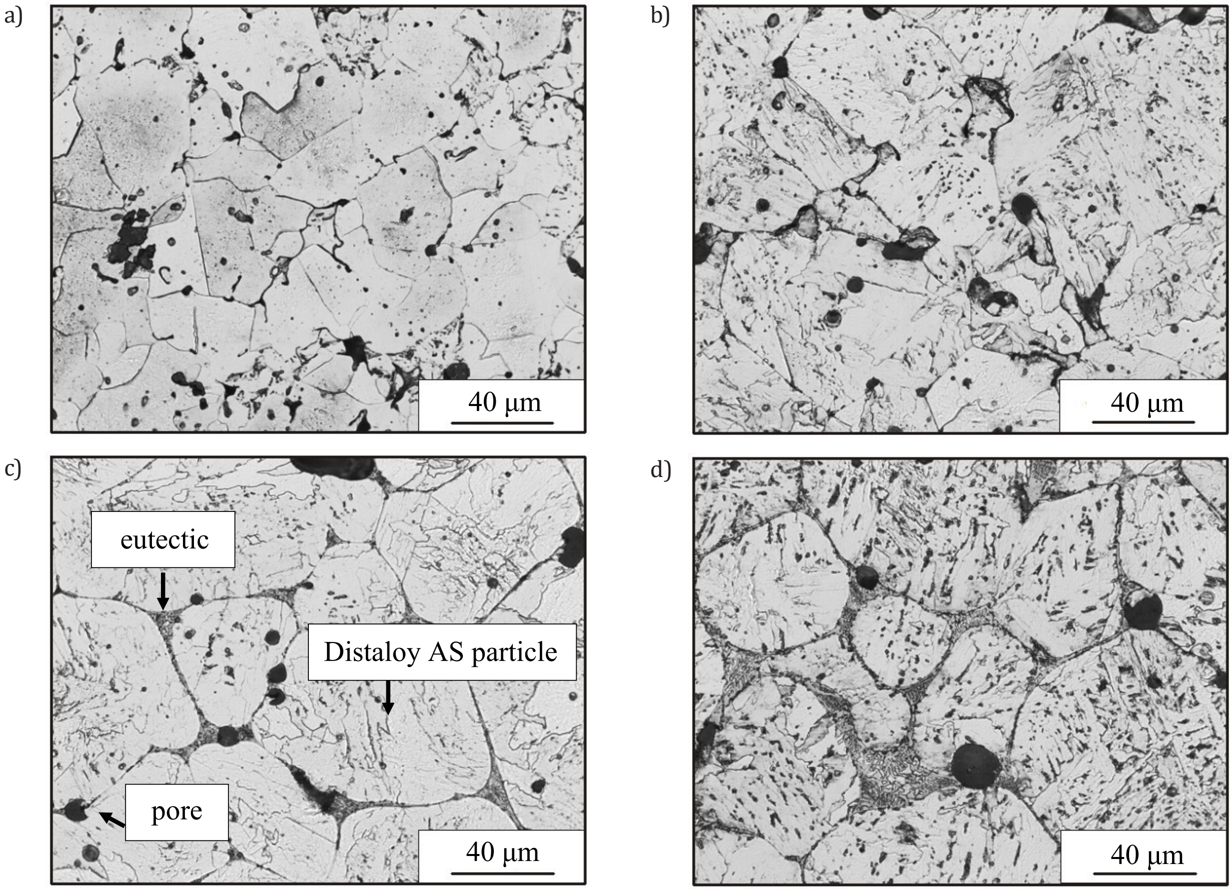


Fig. 5. Microstructure of Distaloy SA sinters alloyed by boron for 70–100 µm particle fraction: a) 0.0 wt.% B; b) 0.2 wt.% B; c) 0.4 wt.% B; d) 0.6 wt.% B

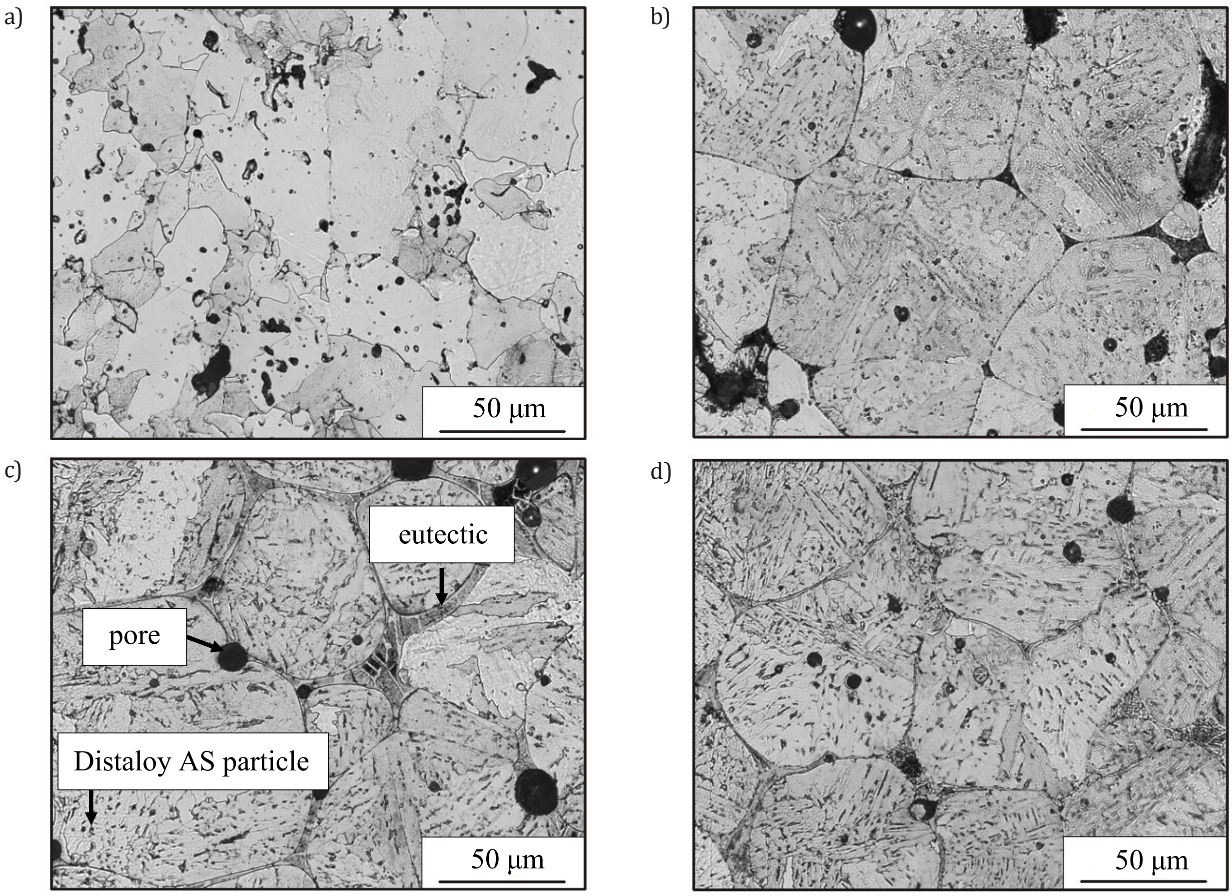


Fig. 6. Microstructure of Distaloy SA sinters alloyed by boron for 40–56 µm particle fraction: a) 0.0 wt.% B; b) 0.2 wt.% B; c) 0.4 wt.% B; d) 0.6 wt.% B

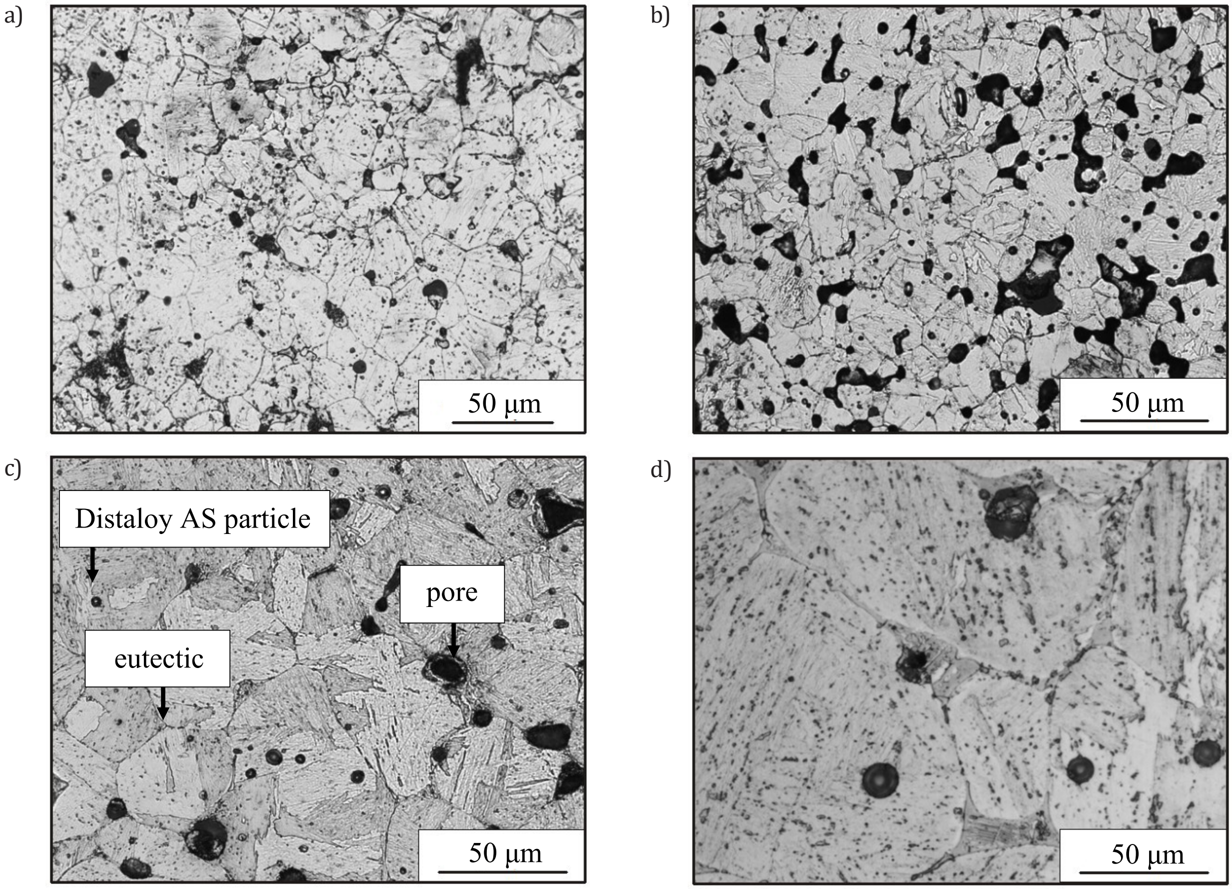


Fig. 7. Microstructure of Distaloy SA sinters alloyed by boron for <40 μm particle fraction: a) 0 wt.% B; b) 0.2 wt.% B; c) 0.4 wt.% B; d) 0.6 wt.% B

A small number of pores occurs in the microstructure of sinters under observation with higher boron content (0.4 wt.% and 0.6 wt.%) (Figs. 4c, d; 5c, d; 6c, d, and 7c, d). Thus, in general, one may say that the more eutectics is formed, the lower porosity. Those are only spheroidal pores, in particular in sinters produced from finer powder fraction: 40–56 μm and under 40 μm (Figs. 6 and 7).

As particle fractions in Distaloy SA powders decreased and boron contents increased, a smaller non-homogeneity of fine borides was observed in the microstructure of sinters under investigation – uniform precipitations of fine borides inside particles (Figs. 5c, d; 6c, d, and 7c, d) and more uniform precipitations of eutectics between particles boundaries, especially for fractions 40–56 μm and under 40 μm (Figs. 6d, 7d). In all microstructures observed, there was discernible increase of particle size with boron concentration – the bigger the particle, the higher the boron contents in sinters (Figs. 4–7). Thus, the formation of a large amount of the liquid phase favors the growth of particles at the expense of the smaller ones.

A characteristic feature for sintering Distaloy with boron is activated sintering resulting in the formation of eutectic containing Fe_2B and Fe_3B phases [7, 19]. In the microstructures of Distaloy SA sinters with 0.6 wt.% boron produced from a general fraction (20-180 μm) and from a coarse fractions (100–160 μm, 71–100 μm, 63–71 μm) larger quantities of eutectic phase located upon grain boundaries were observed (Fig. 3 and 4) than in the microstructures of sinters produced from fine fractions (56–63 μm, 40–56 μm and under 40 μm) (Figs. 6a and 7). Besides, no appreciable grain growth was noticed in the microstructures of sinters made from Distaloy SA powders of many fractions: 100–160 μm, 63–71 μm, 56–63 μm and 40–56 μm, modified with 0.4 wt.% and 0.6 wt.% B, which results from eutectic uniformly distributed upon grain boundaries (Figs. 4c, d; 5c, d; 6c, d). While using finer particle size, the sintering process enables the formation of larger amounts of liquid phase which in turn improves densification. The smaller the particles, the larger the specific surface and thus sintering is enhanced.

It should also be stressed that the highest values of real density (7.55 g/cm^3 and 7.70 g/cm^3) and relative density (about 97% and 99%) were noticed in Distaloy SA sinters produced from finer particle fractions viz. 63–71 μm and 40–56 μm with 0.4 wt.% B content.

The performed structural investigations showed that in sinters made from Distaloy SA powders of finer particle fractions (63–71 μm, 56–63 μm, 40–56 μm and under 40 μm), the non-homogeneity inside ferrite grains decreased as the boron contents increased. Additionally, uniformly distributed borides and an eutectic all along the grain boundaries were observed. The changes noticed in the microstructures of sinters under investigation influenced the degree of their compactness and hardness – they both increased (Figs. 3, 5–7).

It should also be underlined that a smaller non-uniformity of the microstructure and the highest degree of compactness can be obtained in sinters from Distaloy SA powder with 0.4 wt.% B, which belongs to 40–56 μm fraction. Due to the use of small grain fractions of Distaloy SA, one can obtain a higher degree of compactness in sinters and already at lower boron contents.

An additional scientific and technological achievement was the indication of the granular fraction of Distaloy SA powder in order to obtain almost 100% relative density of sinters from Distaloy SA powder with the addition of boron.

The experiments have shown that the application of Distaloy SA powder with a particle fraction of 40–56 μm allows a relative density of 99% to be obtained already at 0.4 wt.% B (Tab. 2) together with high hardness (Fig. 3). The structural investigations reveal, that in Distaloy SA sinters made from finer fractions (40–56 μm), borides are regularly distributed in the structure while eutectic regions are homogeneously distributed on the grain boundaries (Fig. 6).

The application of the given particle fraction of Distaloy SA powder with 0.4 wt.% B will significantly decrease the production costs of sintered elements due to implementing of conventional technologies of pressing and sintering allowing almost 100% relative density to be achieved, which in other cases can only be obtained by applying more expensive technologies of powder metallurgy (hot pressing, isostatic pressing, or finally SPS).

The described method of conventional pressing and sintering can be applied in the production of gearwheels with increased properties (density, hardness) on the basis of a given particle fraction of Distaloy SA powder with boron additions. Thus, the presented studies have both scientific and technological aspects.

4. CONCLUSIONS

The following conclusions were drawn from the research conducted and observations:

- It was observed that the density and hardness of the material depend strongly on the particle fraction of powder; with an increase in density, hardness also rises. Indeed, higher values of density and hardness were observed in samples made from the finer fraction of a Distaloy SA powder (especially 40–56 μm).
- A smaller non-uniformity of the microstructure and the highest degree of compactness can be obtained in sinters from the powder of fraction 40–56 μm, with 0.4 wt.% B.
- Due to the use of small particle fractions of prealloyed powder, a higher degree of compactness in sinters was obtained with lower boron contents.
- The hardness of sinters under investigation depends on boron contents and particle size.
- During sintering, the volume of samples decreases in relation to the green compacts.
- The liquid-phase enhances the sintering process for all fractions but it is the most visible in case of 40–56 μm fraction with 0.4 wt.% and 0.6 wt.% B giving 99 and 98% of relative density.

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