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Elastic properties and structural observations of Distaloy SA powder sintered with boron and carbon

Własności sprężyste i obserwacje struktury spiekanego proszku Distaloy SA z borem i węglem

Abstract

One of the methods aimed at increasing the density in PM parts is the process of activated sintering performed by adding boron as elementary boron powder, for example. Under this researchwork novel, PM materials were obtained 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 the addition of 0.55 wt.% carbon and boron (0.2, 0.4 and 0.6 wt.%). Distaloy SA samples alloyed with carbon and boron were manufactured by mixing the powders in a Turbula mixer, then compacting the mixture in a hydraulic press under a pressure of 600 MPa and sintered in a tube furnace at 1473 K for 60 minutes in a hydrogen atmosphere. The densification process of Distaloy SA parts with boron and carbon depends on the sintering mechanism. In order to evaluate the sintering mechanism of the PM samples, structural investigations using SEM/EDS were performed. During sintering of these materials at 1473 K, a liquid phase is generated as a result of the reaction occurring between the alloy matrix and the complex of carbo-borides, which leads to a considerable degree of consolidation. The elastic properties of the sintered samples (such as Young's modulus and the damping coefficient) were measured in a tensile test with a Förster elastometer.

Keywords: Distaloy SA powder, boron, carbon, sintering, elastic properties

Streszczenie

Jedną z metod mających na celu zwiększenie gęstości części PM jest proces aktywnego spiekania, zachodzący np. w wyniku dodania boru w formie elementarnego proszku boru. W badaniach omawianych w niniejszym artykule nowoczesne materiały PM zostały uzyskane ze stopowanych i dyfuzyjnie związanych proszków Distaloy SA o składzie Fe-1,75%Ni-1,5%Cu-0,5%Mo z dodatkiem 0,55% wag. węgla i boru (0,2, 0,4 i 0,6% wag.). Próbki Distaloy SA stopowane węglem i borem otrzymano w wyniku mieszania proszków w mieszalniku Turbula, a następnie zagęszczenia

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na prasie hydraulicznej pod ciśnieniem 600 MPa i spiekania w piecu rurowym w temperaturze 1473 K w atmosferze wodoru przez 60 min. Zagęszczanie części ze stopu Distaloy SA z borem i węglem zależy od mechanizmu spiekania. W celu określenia mechanizmu spiekania próbek PM dokonano obserwacji strukturalnych za pomocą SEM z przystawką EDS. Podczas spiekania omawianych materiałów w temperaturze 1473 K w wyniku reakcji pomiędzy osnową a złożonymi węglano-borkami tworzy się ciekła faza, która prowadzi do znacznej konsolidacji. Własności mechaniczne spiekanych próbek takie jak moduł Younga czy współczynnik tłumienia zostały zmierzone w próbie rozciągania i na elastometrze Förstera.

Słowa kluczowe: proszek Distaloy SA, bor, węgiel, spiekanie, własności sprężyste

1. Introduction

Activated sintering can be regarded as one of the simplest methods aimed at increasing the density of produced steel parts. The first researchers who studied activated sintering initiated by the addition of boron in iron alloys were Madan and German [1]. They proved that boron enhances the sintering with the presence of a liquid phase and activates the diffusion/sintering process in the solid state.

The mechanical properties of steels sintered with boron were analyzed in [2–5], while the explanation of the sintering mechanism for these alloys can be found in [6–9]. It was observed that, during the sintering of Fe-Mo-B-C materials, an eutectic reaction between the alloy matrix and complex carbo-borides decreases the sintering temperature and leads to the formation of a liquid phase, thus improving consolidation and the mechanical properties. Additionally, with an increased Mo content (3–5%), the amount of the liquid phase decreases at the grain boundaries, while larger quantities of molybdenum borides and carbo-borides evolve inside the grains [10].

2. Experimental methods

A prealloyed and diffusion-bonded Distaloy SA powder was used with the following composition: Fe1.75%Ni-1.5%Cu-0.5%Mo. The powder supplied by the Höganäs company was mixed with the addition of carbon and boron in a Turbula mixer for 15 minutes. Carbon in the form of graphite was added to the powder in the amount of 0.55 wt.%; for comparison, samples without carbon were also prepared. Boron was introduced into the mixture in three concentrations: 0.2, 0.4, and 0.6 wt.%. Next, the powder mixtures were subjected to compression in a hydraulic press under a pressure of 600 MPa, followed by sintering in a hydrogen atmosphere at 1473 K for 60 minutes. The relative density of the mentioned powders was about 90%; the highest relative density (94 wt.%) was noted for the sample of Distaloy SA with 0.6 wt.% B.

The Young modulus was measured in a tensile test (E) and with a Förster elastometer (E_{0F}). The data is collected in Tables 1 and 2.

Table 1. Properties of sintered Distaloy SA powder with boron (without C)

B content [wt.%]	**E [MPa]**	**E_{0F} [MPa]**	**$R_{0.02}$ [MPa]**
0	83,303	149,991	159
0.2	105,385	157,984	197
0.4	118,967	173,336	312
0.6	118,000	185,831	361

Table 2. Properties of sintered Distaloy SA powder with 0.55 wt.% C and boron

B content [wt.%]	**E [MPa]**	**E_{0F} [MPa]**	**$R_{0.02}$ [MPa]**
0	151,615	194,249	143
0.2	96,492	161,442	253
0.4	101,253	173,741	436
0.6	81,129	183,151	95

The ultrasonic Young's modulus E_{0F} was determined based on a frequency of resonant vibrations:

$$E_{0F} = 4Cl^2 \times f^2 \times \rho \times 10^{-7} \; [\mathrm{MPa}]$$

where:

C – constant dependent on the sample's shape,
l – total length of the sample [cm],
f – resonant vibration frequency [1/s],
ρ – density of the analyzed material [g/cm^3].

Additionally, at the mentioned elastometer, damping coefficients $v_{f\Delta}$ and v_{fk} were determined; $v_{f\Delta}$ was determined from the width of the resonant peak at the level of the half resonant amplitude:

$$v_{f\Delta} = \frac{\pi (f_2 - f_1)}{\sqrt{33f}}$$

where:

f_1 – frequency smaller than the resonant one, at the half-resonant frequency,
f_2 – frequency higher than resonant one, at the half-resonant frequency.

Besides, an average logarithmic vibration decrement was established based on the following equation:

$$v_n = \frac{1}{n}$$

where n is the number of cycles of free-damping vibrations with a diminishing amplitude from the resonant one A_0 to $A_{0/e}$.

Additionally, X-ray diffraction spectra were collected for the remnants of the dissolved Distaloy SA with boron as well as with boron and carbon sinters.

Structural observations were conducted with a Hitachi S-3400N scanning electron microscope (SEM) with a Thermo Noran energy dispersive spectrometer (EDS) attachment at an accelerating voltage of 20 kV.

3. Results and discussion

The plastic properties of these sinters are described in detail in research-work [5]. Here, the elastic properties will be discussed. It is observed that Young's modulus value differs upon the measuring method: the E value is lower than E_{0F} by 31 to 49% in the case of the carbon-free samples and by 22 to 56% in the case of the carbon-enriched samples. Young's modulus E increases strongly with the addition of 0.55 wt.% C in the boron-free specimens, from 83 GPa up to 151 GPa (see Tables 1 and 2 and Figure 1). On the contrary, carbon showed a slightly reducing effect on the value of Young's modulus E in the samples with the addition of boron.

In the case of proof stress $R_{0.02}$, the samples with no carbon nor boron content showed higher yield points (see Tables 1 and 2). The samples with 0.2 and 0.4 wt.% B have a higher $R_{0.02}$ value if 0.55 wt.% C is introduced to the mixture. The highest value for $R_{0.02}$ (436 MPa) is observed in the sintered Distaloy SA-0.55 wt.% C-0.4 wt.% B sample, while the smallest (only 95 MPa) is reported for Distaloy SA-0.55 wt.% C-0.6 wt.% B.

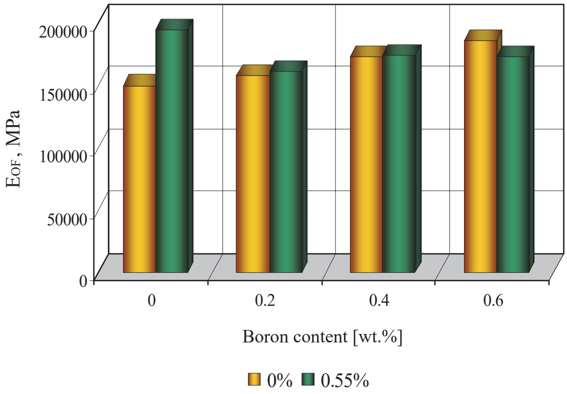

Fig. 1. Effect of boron on Young's modulus E_{0F} of Distaloy SA sinters with carbon and boron additions

The change of damping coefficient $v_{f\Delta}$ is presented in Figure 2. The value of $v_{f\Delta}$ is the highest in the sample with 0.55 wt.% C and decreases then with increasing amounts of boron. In the samples to which graphite was not added, $v_{f\Delta}$ is almost the same for the samples with boron content 0 to 0.4 wt.% B and then decreases for the sample with 0.6 wt.% B.

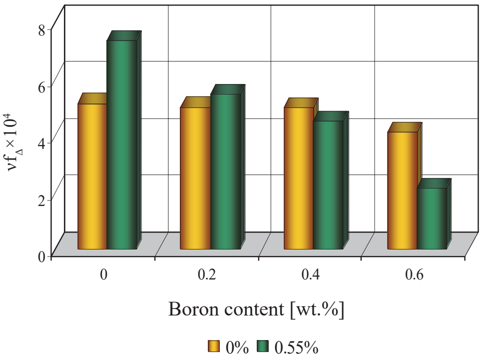

Fig. 2. Effect of boron on damping coefficient $v_{f\Delta}$ of Distaloy SA sinters with carbon and boron additions

Average logarithmic vibration decrement v_n is presented in Figure 3. It is the highest in the sample with 0.55% C and without boron. Then, it rapidly decreases to a value of 12×104 and is kept at this level for the analyzed samples with various boron contents of 0.2 and 0.4 wt.% B; then, it decreases strongly to about 4.6×104 for the sample with 0.6 wt.% B. In the case of samples without the addition of carbon, the vn value increases moderately (up to about 13.7×104 for 0.6 wt.% B).

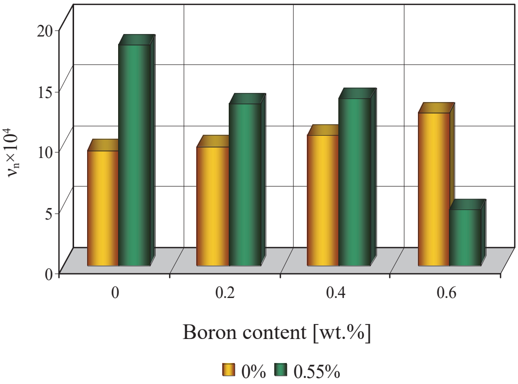

Fig. 3. Effect of boron on average logarithmic vibration decrement v_n of Distaloy SA sinters with carbon and boron additions

Additionally, the sample with the highest content of boron and another one with boron and carbon were analyzed by means of the X-ray diffraction technique to determine compounds formed during the sintering process. The respected spectra are presented in Figures 4 and 5. It was found that, in the activated sintering process, the following compounds are formed in the Distaloy SA with 0.6 wt.% B specimen: Fe_2B, $FeMo_2B_2$, and NiB; while, in the sample made of Distaloy SA with C and B – Fe_2B, NiB, and Mo_2B are present. As mentioned in [6, 11–15], the effective role of the borides is to decrease the sintering temperature and increase the amount of liquid phase. It is also to note that, in the samples containing carbon, cementite Fe_3C is formed.

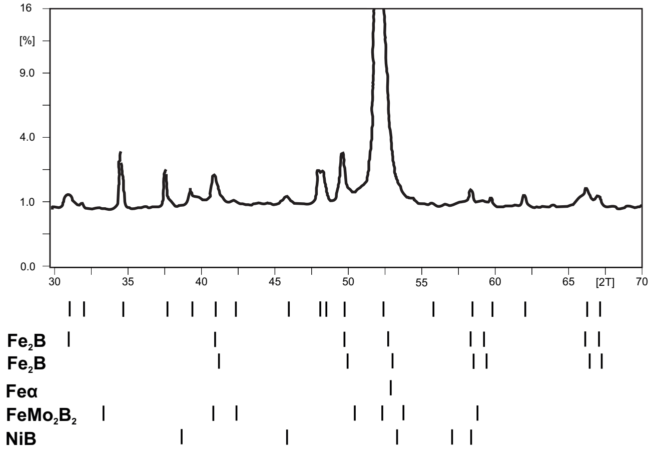

Fig. 4. XRD spectrum for Distaloy SA with 0.6 wt.% B sinter. Position of reference peaks for given phases marked as lines below the spectrum

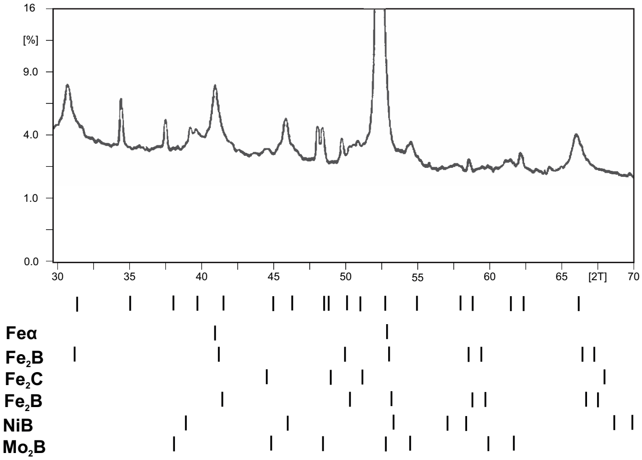

Fig. 5. XRD spectrum for Distaloy SA with B and C sinters. Position of reference peaks for given phases marked as lines below the spectrum

The elastic properties of Distaloy SA sinters with the addition of boron or with the addition of both boron and carbon are strongly connected with the sintering process of the powder's mixture; i.e., with the formation of a liquid phase. The liquid phase forms due to a eutectic reaction between the alloyed matrix, iron borides, and borides of the alloying elements ($FeMo_2B_2$, NiB, and Mo_2B) at a temperature of 1473 K (Figs. 4 and 5). The resulting liquid phase leads to a significant densification of the Distaloy SA sinters and a considerable increase in elastic modulus (E_{0F}) up to a boron concentration of 0.4 wt.% B.

At a higher boron content (0.6 wt.%), a too-big amount of the liquid phase is generated at the grain boundaries, which inhibits the further increase of the elastic properties. The mentioned process is confirmed in structural observation (SEM, XRD, Fig. 6).

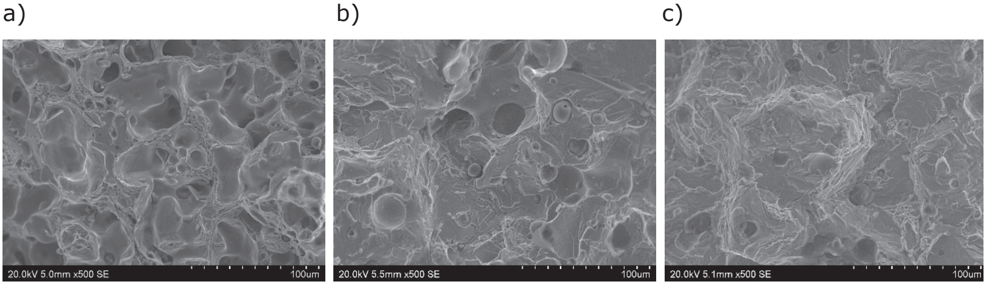

Fig. 6. Fracture surface of Distaloy SA sintered powder with: a) 0.2 wt.% B and 0.55 wt.% C; b) 0.4 wt.% B and 0.55 wt.% C; and c) 0.6 wt.% B and 0.55 wt.% C; magnification of 500×

During the sintering of the Distaloy SA powder with boron or boron and carbon, a reaction of boron with the alloying elements occurs at first, and a boride phase or carbide phase is generated due to the diffusion of iron, molybdenum, and nickel to boron as well as the diffusion of iron to boron and carbon. Iron, molybdenum, and nickel consist of a part of the boride phase that forms eutectics as well as precipitates formed both at the grain boundaries and within the ferrite grains. The liquid phase dissolves iron particles subsequently precipitating at the grain boundaries, giving a hardening effect. In the case of sinters with the addition of boron and carbon, carbon was also identified in the eutectic.

4. Summary

The elastic properties of the studied materials depend on the density and porosity. Sinters with lower density are characterized by a lower initial Young's modulus E_{0F} and higher values of damping coefficients $v_{f\Delta}$ and v_{fk}.

In Distaloy SA sinters with an increase in boron content, the initial Young modulus E_{0F} increases, while the damping coefficients show a decreasing trend. The presented characteristics prove that the damping coefficients depend on such factors as boron content, density, and porosity, where the dominating factors are the densities of the samples. The higher the boron concentration in a specimen, the higher the density and lower the porosity.

At a temperature of 1473 K, the liquid phase forms as a result of the eutectic reaction between the alloyed matrix, iron borides, and borides of the alloying elements, which leads to the densification of Distaloy sinters as well as a considerable increase in elastic modulus (E_{0F}) up to a boron concentration of 0.4 wt.%. The X-ray diffractogram showed the presence of $FeMo_2B_2$, NiB, and Mo_2B compounds in the analyzed sinters.

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