High Density P/M Steel

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ABSTRACT

One of the challenges in the ferrous P/M industry has been achieving densities higher than 7.2 g/cc in a single press and sinter process. This study has incorporated new and existing technologies to achieve near full density in Fe – Ni – Mo – C – B – Al steels with minimal distortion. Boron contents as low as 0.1 wt % were found to enhance densification. In previous studies, boron contents were too high, creating continuous grain boundary films that degrade mechanical properties. Molybdenum was prealloyed with the base iron to contribute to solid solution strengthening of the matrix. Aluminum was added to react with entrapped gas to help eliminate final porosity.

INTRODUCTION

Current trends in the field of P/M development of steels indicate a marked interest in obtaining high strength steels that can be processed at relatively low temperature. The Fe-B system, with boron as an activating agent has sustained the interest of powder metallurgists. Boron, similar to other activating agents such as phosphorous and carbon, forms a eutectic with iron [1]. The low solubility of the boride phase in iron results in its segregation at the grain boundary [2,3]. Presence of boron leads to rapid densification when sintered above 1175°C, the temperature of formation of Fe-Fe_2B eutectic [1,2]. Unlike phosphorous and carbon, the grain boundary cohesion energy of the boride is relatively high [4,5], resulting in sintered products, which have high density and strength.

Madan and German [2] were the first to present a systematic study on the effect of boron content on the density and mechanical properties of the Fe-B alloy system. Subsequent studies by different researchers focused on the effect of alloying elements on the mechanical properties of the alloy. The properties of the Fe-B-C system has been studied and reviewed recently by Semel [6]. Carbon, when added in the form of graphite, did not have a vast impact on the densification. Xiu et al., [7] compared the effect of carbon on the sintering behavior and the properties of iron prealloyed with molybdenum when added as elemental powder and as Fe-C master alloy (containing 4.3 wt% C).

The properties of material processed using prealloyed powder compared to admixed powders, was studied by various authors [7-10]. Dudrova et al., [8] reported that the amount of eutectic phase in the prealloyed Fe-1.5%Mo was less than that of Fe for the same amount of boron (added as Fe-18%B), indicating the boride forming capability of Mo and its effect on the microstructure. In these experiments, sintering was conducted in cracked NH_3 gas, an atmosphere not very effective in sintering these materials to high density. Lui et al., [9] in their experiments with water atomized prealloyed Fe-1.5%Mo and water atomized Fe-0.1%C admixed with 1.5% Mo, sintered in H_2 atmosphere, found that the amount of boron required to obtain the same extent of densification decreased by using the prealloyed powder. Their experiments also showed an increase in density with the use of 1.0 wt % Ni, which was attributed to the lower temperature eutectics in the Ni-B system compared to the Fe-B system. Selecka et al., [10] studied the properties of Cr-Mo-V alloyed steel sintered with 0.2% boron, added as ferroboron, and reported a sintered density of 7 g/cc when sintered at 1200°C in H2 atmosphere for 60 minutes.

In the present work, the effect of aluminum addition and use of low boron and carbon contents on the mechanical properties to the water atomized, prealloyed Fe-1.5%Mo powder is investigated. The experiments were based on the assumption that aluminum would control the residual gas porosity and carbon does not contribute to significant strengthening because of its association with boron to form borocarbides at the grain boundaries.

EXPERIMENTAL PROCEDURE

Water atomized, prealloyed iron powder, provided by Hoeganaes Corporation, was used in the present investigation. The powder composition, as reported by the manufacturer, is given in Table 1 and the powder characteristics is given in Table 2.Elemental nickel powder (3μm), boron (<5μm) supplied by Alfa Aesar, graphite powder supplied by Asbury Carbon and aluminum (<3μm) supplied by Valimet were used.

Table 1. Composition of 150HP powder in weight percent.

Fe	Mn	Mo	Cu	Ni	Cr	C	S	O	Si
Balance	0.13	1.44	0.06	0.05	0.04	<0.01	0.01	0.07	0.02

Table 2. 150HP Powder characteristics

$\rho_{apparent}$, g/cm^3	ρ_{tap}, g/cm^3	$\rho_{pycnometer}$, g/cm^3	Flowrate, s/50g	D_{10}, μm	D_{50}, μm	D_{90}, μm
3.3	3.9	7.87	24.4	42.3	102	194

Alloys of different composition were blended with 0.5 wt% Acrawax in a turbula blender for 30 minutes. The nickel content was maintained at 1.0 wt%, while boron, carbon, and aluminum amounts were varied individually. Flat tensile bars were pressed in a 60 ton hydraulic uniaxial press at a pressure of 600 MPa according to MPIF Standard 10. Green density was measured from sample mass and measured volume. Samples were sintered in a 6 zone CM pusher furnace in pure hydrogen at various temperatures and hold times. Sintered density was measured by the Archimedes method. Tensile testing was performed on a Sintech 20/D machine with a 20 kip load frame and 15 kip fixtures, using a crosshead speed of 6.35 mm/min. Heat treatment was performed by heating in argon at 5°C/min to 850 for 20 minutes and oil quenching, then tempering at 175°C for one hour in argon. Microstructures were studied through standard polishing and etching with 4% nital.

RESULTS AND DISCUSSION

Figure 1 shows the increase in sintered density of Fe-1.5Mo-1.0Ni-0.5C as boron content increases from 0.1 to 0.3 wt%. Also depicted is the decrease in mechanical properties as boron content increases. The decrease in mechanical properties is due to the increase in brittle boride phases at the grain boundaries, allowing an easy path for crack propagation.

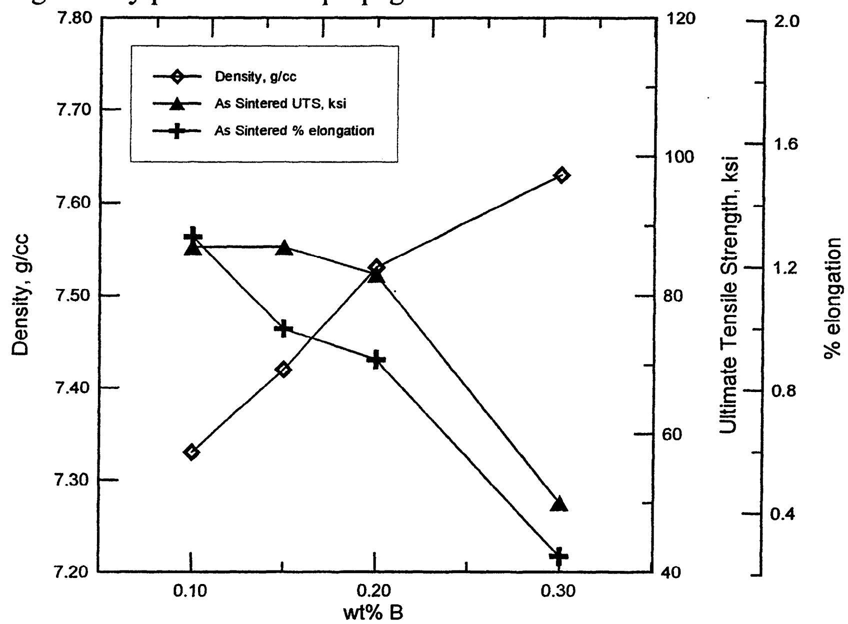

Figure 1. Sintered properties vs. boron content for 150HP+1.0Ni+0.5C samples

Figures 2 and 3 are plots of density and mechanical properties vs. aluminum content for Fe-1.5Mo-1.0Ni-0.5C-0.2B and Fe-1.5Mo-1.0Ni-0.5C-0.3B, respectively. These plots reveal an increase in sintered density and mechanical properties with increased Al content. Too much Al, however is detrimental to properties.

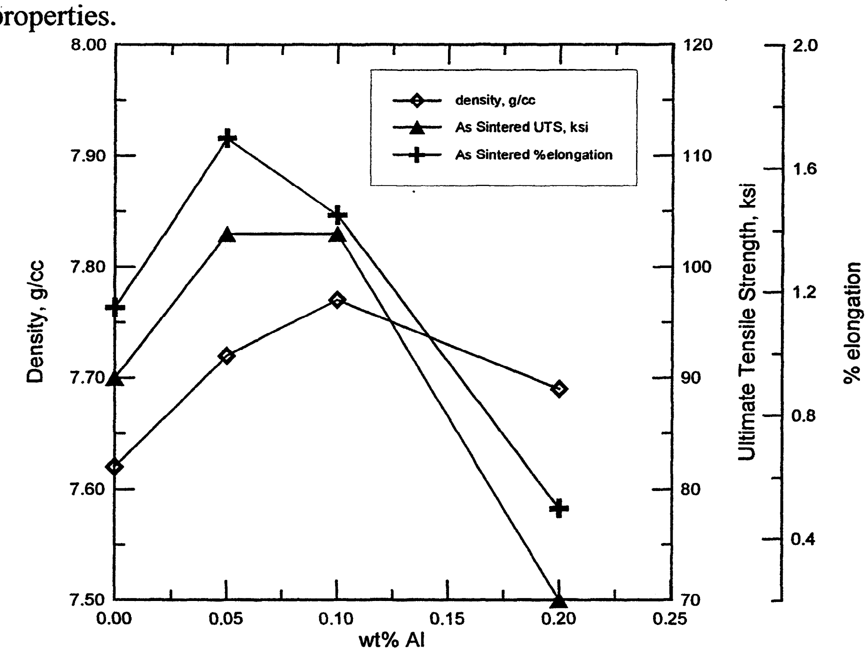

Figure 2. Sintered properties vs. Al content for 150HP-1.0Ni-0.2B-0.5C samples.

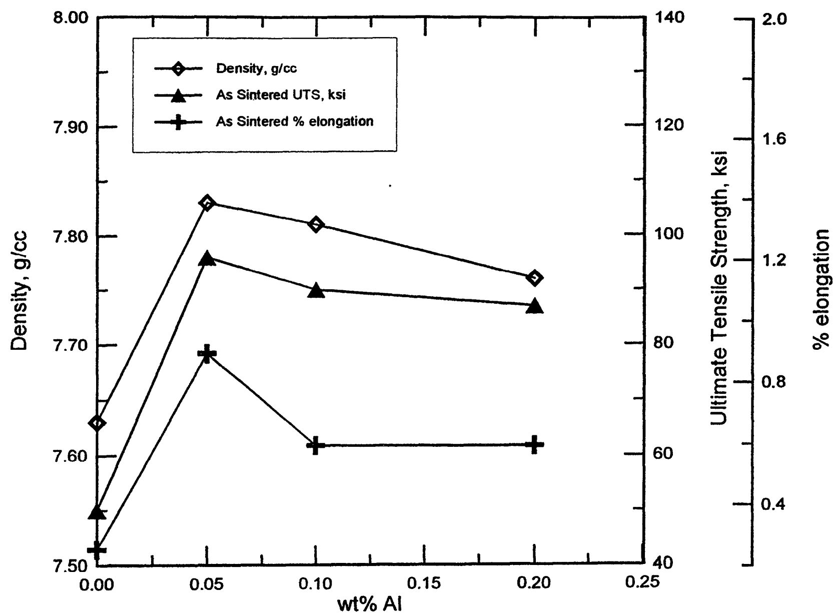

Figure 3. Sintered properties vs. Al content for 150HP-1.0Ni-0.3B-0.5C samples.

Figures 4, 5 and 6 compare microstructures of 150HP-1.0Ni-0.1Al-0.5C samples with 0.3, 0.2, and 0.1 wt.% boron contents, respectively. At 0.3 wt.% boron, nearly complete coverage of grain boundaries with brittle boride phases is observed and the elongation to fracture is 0.6%. At 0.1 wt.% boron, the grain boundary coverage is much lower, and although the density decreases, the elongation to fracture to 2.2%. The best UTS of the three samples is achieved at 0.2 wt.% boron, where a compromise between high density and low boron content is reached. All samples were sintered for 30 minutes, however the 0.1 wt.% boron material was sintered 1215°C, while the 0.3 and 0.2 wt.% boron materials were sintered at 1200°C. Higher temperatures were beneficial for densification of lower boron content alloys.

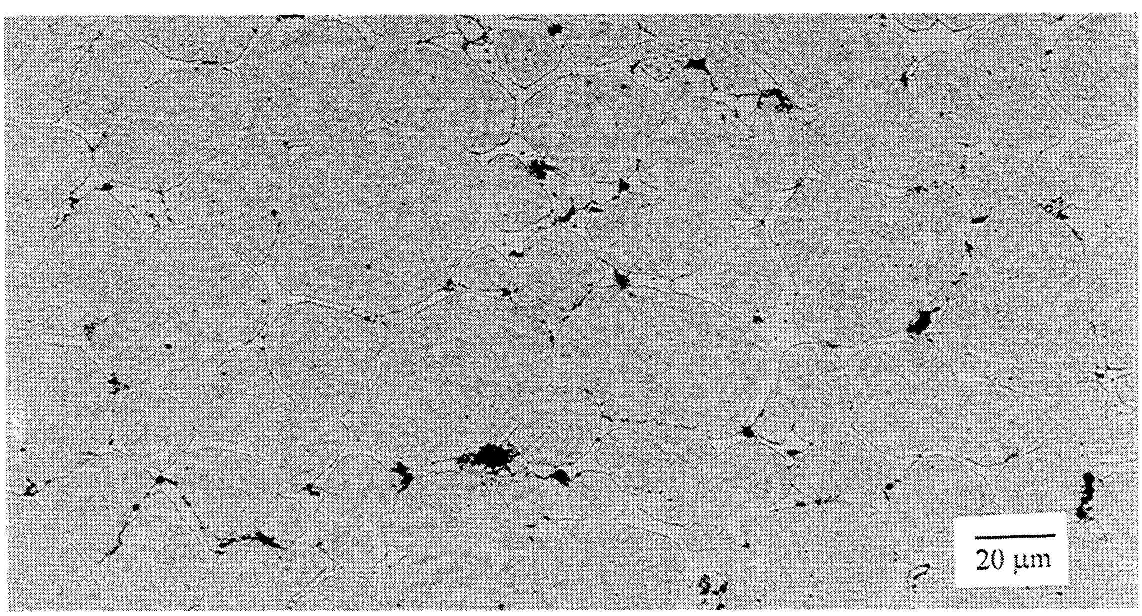

Figure 4. 150HP-1.0Ni-0.1Al-0.5C-0.3B sintered at 1200°C for 30 minutes. Density = 7.81 g/cc, UTS = 90 ksi, % elongation = 0.6.

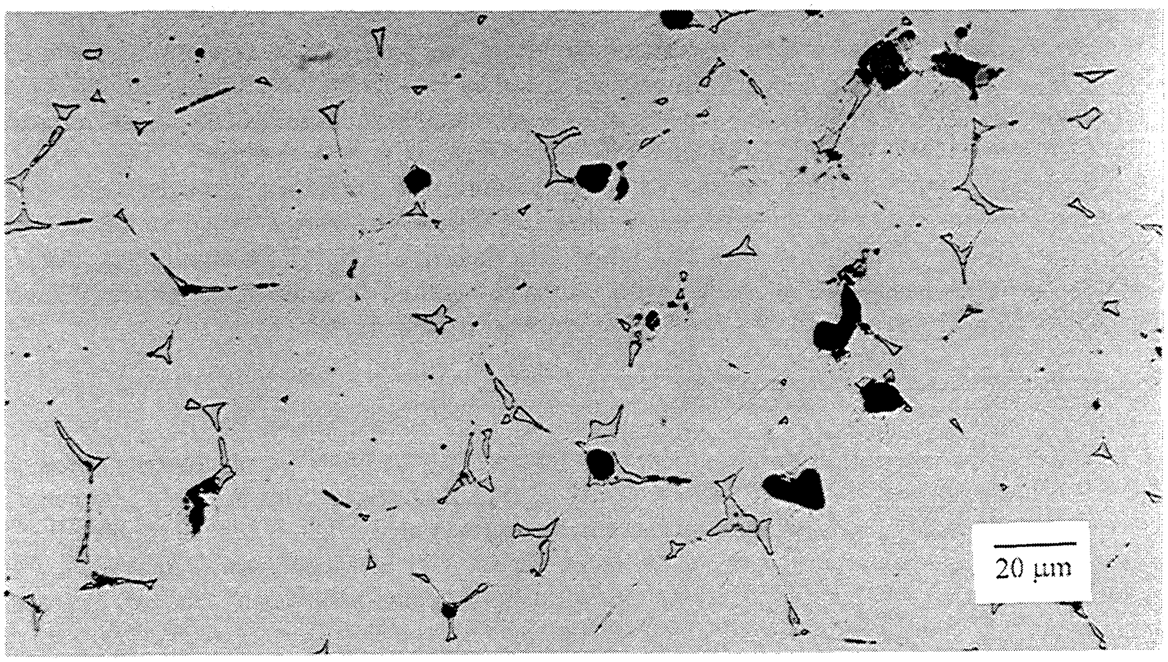

Figure 5. 150HP-1.0Ni-0.1Al-0.5C-0.2B sintered at 1200°C for 30 minutes. Density = 7.77 g/cc, UTS = 103 ksi, % elongation = 1.5

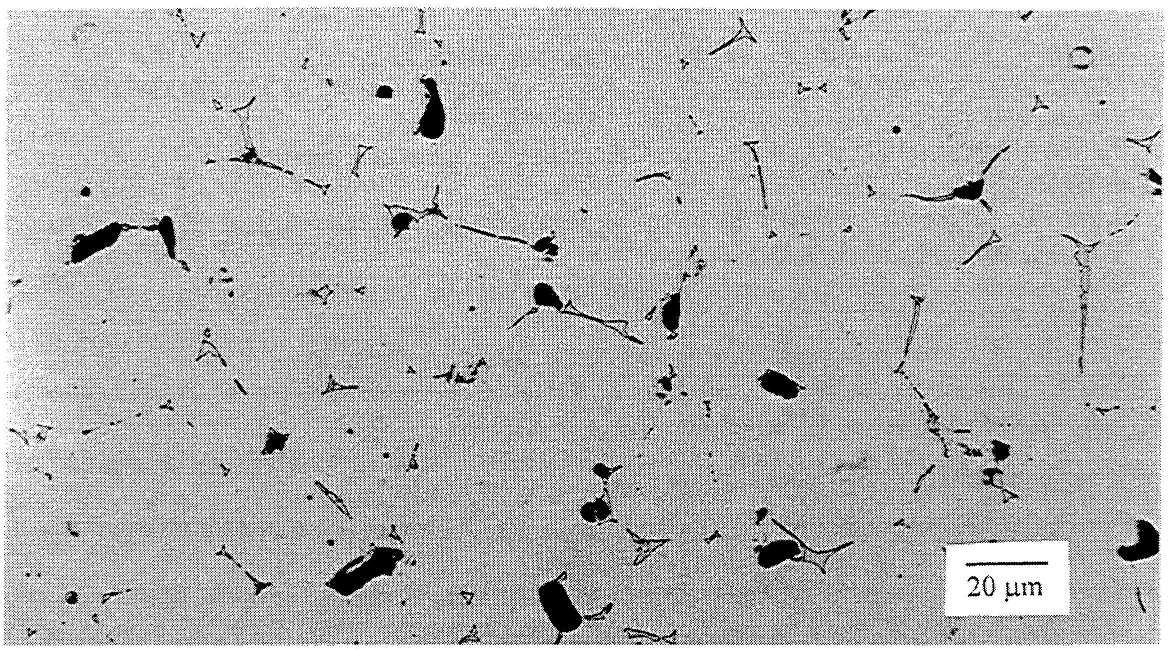

Figure 6. 150HP-1.0Ni-0.1Al-0.5C-0.1B sintered at 1215°C for 30 minutes. Density = 7.55 g/cc, UTS = 90 ksi, % elongation = 2.2.

Sintering at temperatures higher than 1200°C and/or holding for longer times than 30 minutes produced mixed results. In alloys where the higher temperature and/or longer time produced an increase in density, properties were improved. In alloys where density was not increased, results were inconclusive.

Samples processed with low (0.05 wt.%) carbon levels displayed much higher ductilities, which were on the order of 4.5% elongation in the as-sintered state. Ultimate tensile strengths were roughly 95 ksi, and densities were 7.6 g/cc, lower than the high carbon samples. It is possible that the low carbon content softens the matrix, which could cause crack tips to blunt as they enter the grains. Alternatively, less borocarbides may be present at the grain boundaries, increasing the ductility of the grain boundary phase.

In general, response to heat treatment in all alloys was poor, despite apparent hardness increases from 25 to 50 HRC. In most alloys, UTS only increased by 10 – 25 ksi with heat treatment. In comparison, the UTS of FN0208 at 7.4 g/cc typically increases from 95 ksi to 190 ksi and apparent hardness increases from 78 HRB to 40 HRC (55 HRC matrix) with heat treatment. These results indicate that although the matrix of the boron steels is hardened during heat treatment, properties are largely controlled by the extent of the grain boundary phases. The best properties attained for a heat treated sample was 150HP-1.0Ni-0.5C-0.1B-0.2Al, which was sintered at 1215°C for 40 minutes. After heat treatment, the alloy has a UTS of 142 ksi, with a 0.5% elongation. In general, higher heat treated elongations were observed in the 0.1 and 0.15% boron compositions.

CONCLUSIONS AND FUTURE WORK

Mechanical properties of boron steels are improved by the use of boron contents in the range of 0.1 - 0.2 wt.%, compared to 0.3-0.5 wt.% typically used in these steels. This is due to a decrease in brittle grain boundary boride phases. Additions of aluminum in the range 0.05 – 0.2 wt.% can be benificial to density and mechanical properties, from aluminum reaction with entrapped gas. Future work will focus on the effects of time-temperature combinations for different compositions, and study of microstructures by nanohardness, microhardness, optical microscopy, and microprobe analysis to determine the role of each element and composition of matrix and grain boundary phases.

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