

THE ROLE OF ADMIXED HEXAGONAL BORON NITRIDE IN SINTERED STEELS

1. EFFECT ON SINTERING AND MICROSTRUCTURE

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Abstract

Fe and Fe-C materials containing hexagonal boron nitride (hBN) have been prepared by mixing, pressing and sintering, and the properties and microstructures have been studied. It showed that hBN, especially if added as powder < 1 µm, inhibits the formation of stable metallic bridges during sintering and also the dissolution of graphite in the ferrous matrix. When sintering is done above the Fe-B eutectic temperature in H₂ atmosphere, BN decomposes, and the resulting B forms a liquid phase that activates sintering. This reaction does not occur in N₂-H₂ atmosphere, in this case the sintering temperature being rather irrelevant. Generally, the effects of BN towards mechanical strength and carbon dissolution are both markedly more pronounced with fine grades than with coarse ones.

Keywords: *sintered steel, boron nitride, carbon dissolution*

INTRODUCTION

The addition of boron to PM ferrous materials has been a well known method for activating sintering for many decades. As early as 1955, Benesovsky et al. showed that admixing B to Fe powder results in pronounced densification during sintering of the compacts, although at the expense of toughness [1]. Starting from about 1980, the effect of B was further studied (e.g. [2-7], and the early findings were confirmed. It showed that it is difficult to attain the desired activation process while avoiding embrittlement. There is also a considerable difference between C-free and C containing materials [8] and unalloyed and alloyed steels, [9,10]. There is also a considerable effect of the sintering atmosphere, deboronizing being observed in H₂ [8,11,12] and passivation of B in N₂ containing atmospheres [13].

Compared to this somewhat tricky behaviour of elemental boron, hexagonal boron nitride (hBN) has been regarded as rather easy to handle. This compound is very stable thermodynamically, decomposing in air at T>3000°C [14] and is therefore regarded as more or less inert during the pressing and sintering process. Its role in ferrous PM is mainly that of a machining aid, BN addition reportedly enhancing the usually poor machinability of sintered iron and steels [15]. Furthermore, hBN is a solid lubricant, its lattice structure being very similar to that of graphite. However, in [16,17] it has been shown that in suitable atmospheres BN may decompose at higher temperatures, resulting in a persistent liquid phase that strongly enhances e.g. gravity sintering of austenitic stainless steel powders. Similar effects have been described also for the combination of hBN and nickel [18].

Furthermore, it has been shown that BN addition may affect the dissolution of graphite during sintering [19].

It can thus be stated that the effect of boron addition, also as BN, to sintered steels can be rather complex. In this work, investigations into the sintering behaviour and properties of PM steels sintered with admixed BN are described.

EXPERIMENTAL

The tests were carried out using standard water atomized iron powder ASC 100.29 (Höganäs) and natural graphite UF4 (Kropfmühl). Standard fine BN grade with $d_{50} < 1 \mu\text{m}$ was employed (which, however, proved to be in part agglomerated), and for reference purposes also coarse BN powder (50-180 μm) was used. This grade is not commercially available; it was obtained by crushing HIPed BN compacts (supplied by HTM AG, Biel, Switzerland). 0.5 mass% ethylene bisstearylamine (Microwax C) was used as pressing lubricant. The mixes prepared and the theoretical density values are given in Table 1. Of course a mix with 2.0% BN must be regarded as being unsuitable for industrial use, but is well suited as a model material for studying the effects of BN.

Tab.1. Composition of the mixes prepared and theoretical density (including the lubricant).

Composition [mass%]	Theoretical density [g.cm ⁻³]
Fe	7.600
Fe - 0.5 BN	7.514
Fe - 2.0 BN	7.268
Fe - 0.8 C	7.456
Fe - 0.8 C - 0.5 BN	7.374
Fe - 0.8 C - 2.0 BN	7.137

The powders were dry mixed for 60 min in a tumbling mixer. In particular the batches with coarse BN were found to be very prone to segregation, and therefore these batches were further manually mixed just before filling them by hand into the die cavity. Pouring the powder mixed had to be avoided since it resulted in immediate segregation, the coarse BN particles ending up at the top of the bulk powder.

Compacting was done in a tool with floating die for standard impact test bars with a cavity dimension of 55 x 10 mm (ISO 5754) at a pressure of uniformly 600 MPa.

The compacts were sintered in an electrically heated pusher furnace with Mo heating elements (Degussa type „Baby“) in plain H₂ or N₂-20% H₂ mix. The carbon-free specimens were embedded in Al₂O₃ granulate; for the carbon containing steels, getter boats with Al₂O₃-5% graphite getter were used to avoid decarburization. All specimens were investigated and tested in the as-sintered state. Some compacts were sintered in a pushrod dilatometer (Bähr 801) in vacuum and static N₂, respectively. Characterization of the specimens included impact testing, metallography and fractographic studies.

DIMENSIONAL AND MECHANICAL PROPERTIES

The properties of the sintered specimens are listed in Table 2 for the materials prepared using fine BN and in Table 3 for those with coarse BN grade.

Tab.2a. Properties of PM plain Fe containing fine BN (<1 μm). Compacted 600 MPa, sintered 60 min.

Matrix	BN [wt%]	T _{sint} [°C]	Atmosphere	Green density	Sintered density	Dim. Change	Impact energy
Fe	0	1120	H ₂	7.12	7.26	-0.24	>31.4
	0.5	1120	H ₂	7.12	7.21	-0.18	11.5
	0.5	1120	N ₂ -H ₂	7.07	7.00	-0.08	2.8
	2.0	1120	H ₂	6.45	6.37	+0.27	0.78
	2.0	1120	N ₂ -H ₂	6.48	6.36	+0.18	0.9

Fe	0	1250	H ₂	7.12	7.31	-0.13	>40.1
	0.5	1250	H ₂	7.12	7.29	-0.71	>42.5
	0.5	1250	N ₂ -H ₂	7.07	7.08	-0.41	9.5
	2.0	1250	H ₂	6.45	6.57	-0.75	2.54
	2.0	1250	N ₂ -H ₂	6.49	6.47	-0.49	1.6

Tab.2b. Properties of PM Fe-0.8% C containing fine BN (<1 μm). Compacted 600 MPa, sintered 60 min in getter box.

Matrix	BN [wt%]	T _{sint}	Atm.	Green density	Sintered density	Dim. Change	Impact energy
Fe-C	0	1120	H ₂	7.05	7.03	-0.1	11.7
	0.5	1120	H ₂	6.99	7.08	-0.16	3.4
	0.5	1120	N ₂ -H ₂	7.00	6.88	0.09	1.4
	2.0	1120	H ₂	6.24	6.12	+0.61	0.68
	2.0	1120	N ₂ -H ₂	6.38	6.00	+0.56	0.4

Fe-C	0	1250	H ₂	7.19	7.11	-0.02	14.3
	0.5	1250	H ₂	6.98	7.15	-0.73	16.46
	0.5	1250	N ₂ -H ₂	6.99	6.97	-0.35	9.8
	2.0	1250	H ₂	6.03	6.25	-0.76	6.49
	2.0	1250	N ₂ -H ₂	6.42	6.29	-0.1	1.9

Tab.3a. Properties of PM plain Fe containing coarse BN (50-180 μm). Compacted 600 MPa, sintered 60 min.

Matrix	BN [wt%]	T _{sint}	Atmosphere	Green density	Sintered density	Dim. Change	Impact energy
Fe	0	1120	H ₂	7.12	7.26	-0.24	>31.4
	0.5	1120	H ₂	7.07	7.04	-0.10	9.2
	0.5	1120	N ₂ -H ₂	7.18	7.10	-0.08	7.3
	2.0	1120	H ₂	6.84	6.85	+0.06	2.0
	2.0	1120	N ₂ -H ₂	6.94	6.82	+0.13	2.3

Fe	0	1250	H ₂	7.12	7.31	-0.13	>40.1
	0.5	1250	H ₂	7.07	7.14	-0.31	>28.90
	0.5	1250	N ₂ -H ₂	7.17	7.15	-0.21	31.30
	2.0	1250	H ₂	6.79	6.93	-0.23	7.73
	2.0	1250	N ₂ -H ₂	6.93	6.85	-0.06	3.90

Tab.3b. Properties of PM Fe-0.8% C containing coarse BN (50-180 μm). Compacted 600 MPa, sintered 60 min in getter box.

Matrix	BN [wt%]	T _{sint}	Atm.	Green density	Sintered density	Dim. Change	Impact energy
Fe-C	0	1120	H ₂	7.05	7.03	-0.1	11.7
	0.5	1120	H ₂	6.95	7.01	-0.05	6.1
	0.5	1120	N ₂ -H ₂	7.1	7.02	+0.01	4.5
	2.0	1120	H ₂	6.73	6.70	+0.23	1.8
	2.0	1120	N ₂ -H ₂	6.86	6.72	+0.22	1.8

Fe-C	0	1250	H ₂	7.19	7.11	-0.02	14.3
	0.5	1250	H ₂	6.96	7.12	-0.33	19.4
	0.5	1250	N ₂ -H ₂	7.09	7.08	-0.23	16.1
	2.0	1250	H ₂	6.71	6.88	-0.50	13.1
	2.0	1250	N ₂ -H ₂	6.86	6.76	+0.09	3.6

The results tend to be somewhat irregular, in particular at higher BN contents. This is due to the tendency to the segregation mentioned above, resulting in varying effective BN contents. The occasionally observed disagreement between density change and dimensional change during sintering is at least in part caused by anisotropic dimensional behaviour of the admixed BN, as will be shown in the micrographs.

Nevertheless, there are some very characteristic trends. The compactibility of the mixes is lowered significantly by a 2% BN addition while at 0.5%, the effect is much less pronounced at least with the plain Fe matrix. It might be assumed that with low BN levels the lubricating effect dominates, which at least at the density levels given here enhances densification, while at higher BN levels the volume absorbed by BN strongly inhibits densification, which is also visible from the values for the theoretical density of the respective mixes as given in Table 1, the “jump” between 0.5% and 2.0% BN being markedly larger than between 0.5% BN and the reference materials. This is however contradicted by the results obtained with coarse BN, in which case for the same nominal BN content markedly higher green density levels are attained. Therefore it must be concluded that the lubricating effect of fine BN must not be overestimated and that fine BN adversely affects the compactibility, as do also other fine admixed powders.

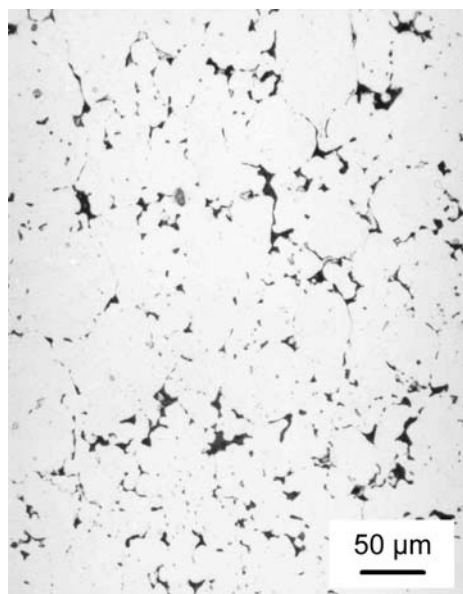
The effect of the BN content on the sintered density and the dimensional change is found to depend strongly on the sintering temperature and the atmosphere. In particular at the higher BN level, low density levels and expansion are observed after sintering at 1120°C in both atmospheres. Sintering at 1250°C in H₂, in contrast, results in marked densification and shrinkage, while in N₂-H₂ the effects are insignificant. The effects can be observed both for fine and coarse BN grades, but they are less pronounced for the coarser BN grade.

This effect of the temperature and atmosphere is also observed for the impact energy. While all BN containing materials sintered at 1120°C show KC values that are significantly lower than for the reference materials, after sintering at 1250°C in H₂ at least for the specimens with 0.5% BN, the same or even better KC values as for the reference materials are found, indicating quite effective sintering. Also here the differences are more pronounced for the fine BN grade than for the coarse one.

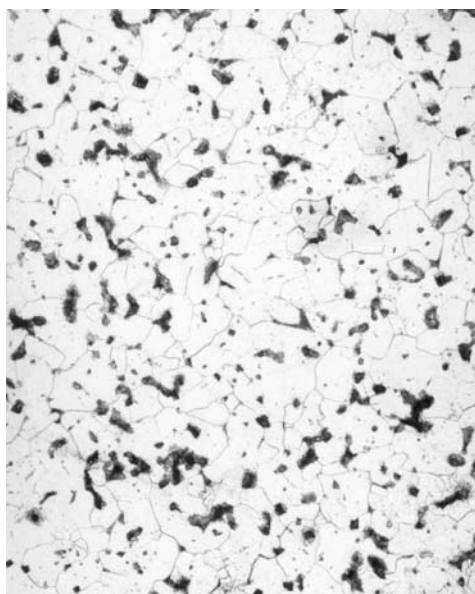
MICROSTRUCTURES

In order to explain these in part surprising results, metallographic sections of the specimens were examined. First of all, it was found that also in those specimens prepared from fine BN, at least in the case of 2% BN added, fairly coarse inclusions were present that were not markedly different from those originating from coarse BN. This indicates that the very fine BN used had a considerable tendency toward agglomeration during mixing.

In Figure 1, micrographs of Fe-BN specimens sintered in H_2 at different temperatures are given. It is evident that the pore structure varies considerably. While after sintering at $1120^\circ C$, very angular pores can be found and the specimen rather resembles a green compact, after sintering at $1250^\circ C$ well rounded structures are observed, and the material is comparable to plain Fe sintered at $T > 1300^\circ C$, e.g. soft magnetic materials. This indicates that also here, the chemical behaviour of BN controls the sintering process: At the lower temperature BN remains stable during sintering and, as indicated by the very low KC values, inhibits formation of stable sintering contacts, just as undissolved graphite does in low temperature sintered Fe-C [20,21]. At temperatures above the Fe-B eutectic, i.e. at $T > 1174^\circ C$ [22], BN decomposes in N_2 -free atmospheres, the N_2 thus generated being removed by the flowing atmosphere and boron reacting with the Fe matrix to form a persistent liquid phase. This explains the pronounced pore rounding and also the excellent impact energy values obtained at the lower BN level. If more B is available, the well known grain boundary embrittlement occurs, and the KC values drop [5,8] (although recent findings indicate that this drop can be avoided by adding getter elements to the boride melt [23]). I.e. the reaction here takes place in a very similar way as described in [17], resulting in boron activation of the sintering process.



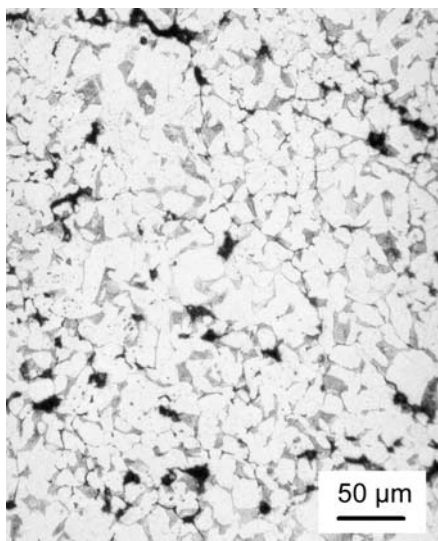
Fine BN, sintered at $1120^\circ C$



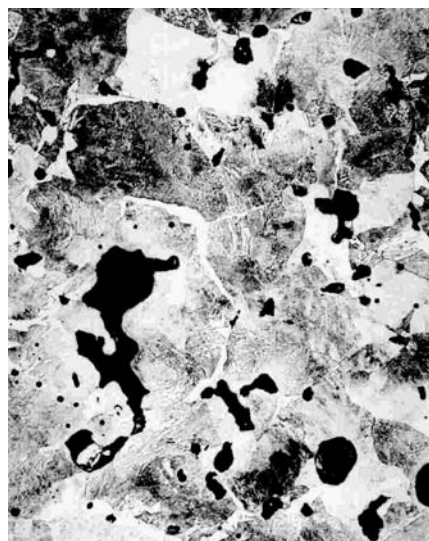
Fine BN, sintered at $1250^\circ C$

Fig.1. Metallographic sections of Fe-0.5% BN, compacted at 600 MPa, sintered 60 min at different temperatures in plain H_2 .

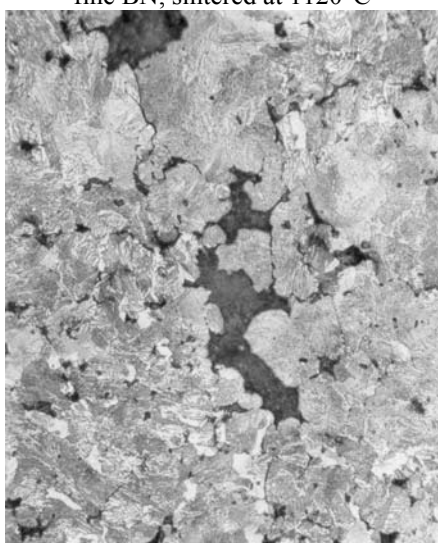
The same phenomenon can be observed also for the carbon containing steels (see Fig.2); the pore rounding and coarsening during sintering at 1250°C is even more pronounced than for the plain Fe matrix, which is not surprising since the ternary eutectic Fe-C-B is reported to be found at about 1100°C [24] compared to 1174°C for Fe-B, i.e. 1250°C is a higher relative temperature here. The fact that BN dissociation does not occur at 1120°C indicates that the carbon level here is not sufficient to depress the Fe-B eutectic right to the ternary invariant, which in [24] is given at 1.25 wt% C and 3.24 wt% B.



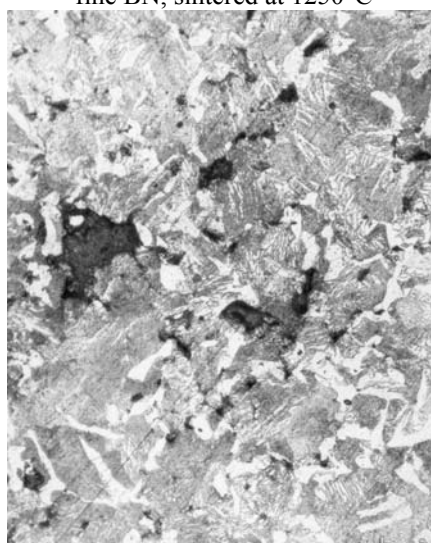
fine BN, sintered at 1120°C



fine BN, sintered at 1250°C



coarse BN, sintered at 1120°C



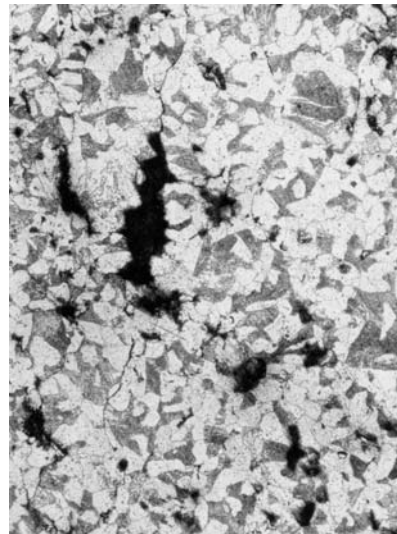
coarse BN, sintered at 1250°C

Fig.2. Metallographic sections of Fe-0.8%C-0.5%BN, compacted at 600 MPa, sintered 60 min at different temperatures in plain H₂.

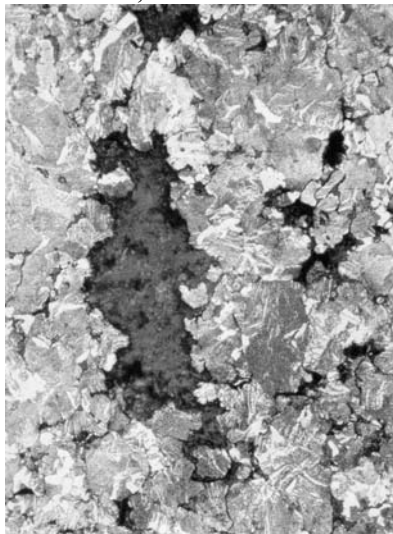
With the carbon steels, there is however a second interesting effect: as indicated also in [19], BN seems to inhibit the dissolution of graphite during the sintering process. As visible in Figure 2a, the ferrite-pearlite ratio in the section resembles that of steel with 0.2 to 0.3% C, although 0.8% C have been admixed and can also be found in the sintered material by combustion analysis. Part of the carbon therefore remains in the material as undissolved graphite. The retarding effect of BN is lost immediately if BN dissociates and forms liquid phase, as clearly visible from Fig.2b, depicting a material sintered at 1250°C; in this case, mainly a pearlitic structure with only small amounts of ferrite can be found, just as would be expected for this carbon level.



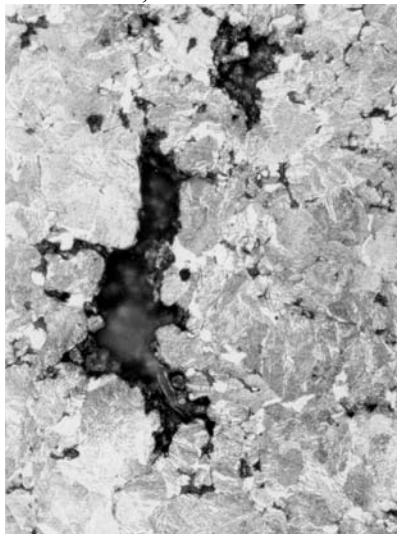
fine BN, sintered at 1120°C



fine BN, sintered at 1250°C



coarse BN, sintered at 1120°C



coarse BN, sintered at 1250°C

Fig.3. Metallographic sections of Fe-0.8%C-0.5%BN, compacted at 600 MPa, sintered 60 min at different temperatures in N₂-20%H₂.

If the materials prepared with fine and coarse BN, respectively, are compared, it is immediately visible that the latter grade does not have such a pronounced effect on carbon dissolution as has the fine BN. Fe-C with coarse BN exhibits an almost fully pearlitic microstructure with only small amounts of ferrite. There is even less ferrite than in the equivalent material sintered at 1250°C, which can however be explained by the slightly higher decarburization occurring at higher temperatures. Coarse BN does not cause the pronounced sintering activation at 1250°C either, apparently due to the much lower interface area available for the reaction between BN and Fe that generates the eutectic melt.

If the materials sintered in N₂-H₂ atmospheres are studied, it is immediately visible that here the differences between sintering at 1120° and 1250°C are virtually negligible, and only the particle size of the BN is of relevance, Fig.3. This corroborates that in atmospheres containing N₂, decomposition of BN is thermodynamically unfavourable and the admixed BN remains stable up to high temperatures, resulting in poor mechanical properties and incomplete carbon dissolution if BN is present within the particle contacts. This latter precondition is much more probable to occur in the case of fine BN than with the coarse one, and therefore the former is significantly more detrimental. For carbon containing specimens it should also be considered that undissolved graphite adversely affects the interparticle strength, [20] as does hBN.

From the micrographs it is also clear that the nominally fine BN contains quite a few agglomerates, see Fig.3b, that are similar in size to the particles of the coarse BN. However, the finely dispersed particles seem to dominate the effect on the properties and the reactivity, i.e. the fine particles present in the sintering contacts and covering the iron powder particles are more effective than the large agglomerates which can be regarded as inert inclusions; from the very low basic strength of the material it can be concluded that agglomerates of that size are well below the threshold given by the Kitagawa-Takahashi concept, even for fatigue loading [25].

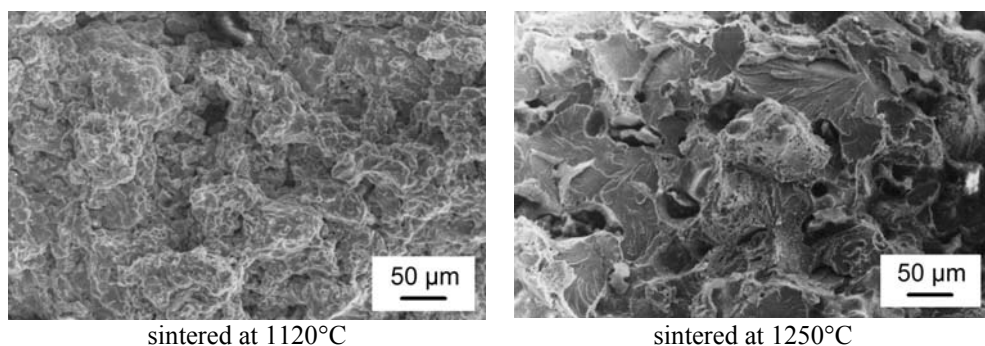


Fig.4. Fracture surfaces of Fe-0.8%C-0.5%BN, compacted at 600 MPa, sintered 60 min in H₂.

The adverse effect of fine BN present in the material is also clearly visible from the fracture surfaces. If Figures 4a and b are compared it can be stated that Fig.4a looks almost like the fracture surface of a green or at best presintered compact, the original powder particles still being clearly discernible, while the material sintered at 1250°C in H₂, depicted in Fig.4b, - in which BN has decomposed during sintering - shows excellent interparticle bonding, as indicated by the considerable amount of cleavage facets visible.

DILATOMETRY

In order to study the effect of BN on carbon dissolution in more detail, dilatometric runs were carried out in vacuum with a well defined temperature-time profile, and the dimensional changes were recorded. The graphs are shown in Fig.5a for Fe-0.8% C(-0.5% BN). From the graphs it can be seen that the BN containing compact shows an α - γ transformation that occurs at markedly higher temperatures compared to plain Fe-C. This corroborates that in the BN containing material, carbon dissolution occurs only very slowly and incompletely. Furthermore, there is considerably more shrinkage below the phase transformation than in the reference material. This can in part be explained by the lower green density of the BN containing compact, but also to the more pronounced effect of α sintering, ferrite, with its higher sintering activity, being retained up to higher temperatures. It is however surprising that this marked shrinkage does not result in sufficient interparticle bonding.

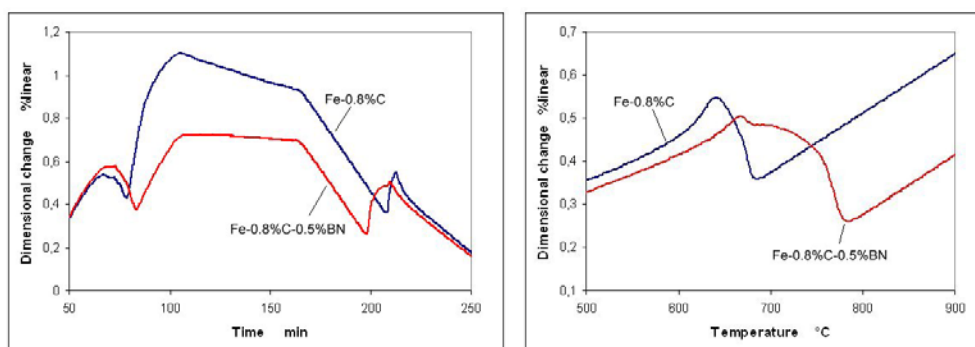


Fig.5. Dilatometric graphs for Fe-0.8%C-0.5%BN and Fe-0.8%C, respectively. Heating / cooling rate 10 K/min, 60 min 1120°C isothermal, rotary pump vacuum.

The shrinkage during isothermal sintering is rather similar for both materials, but there are differences in the cooling section. In Figure 5b the γ - α transformation is plotted for both materials, and it can be seen that the BN containing steel exhibits a transformation curve that is found commonly with low-carbon ferritic-pearlitic sintered steels, while the reference grade shows the straight transformation typical for fully pearlitic steel. This once more confirms that, despite the nominal carbon content of 0.8%, the matrix of the sintered material prepared with BN in fact contains markedly less combined carbon, most of the admixed graphite being present still as graphite even after 60 min at 1120°C, which has been confirmed also by metallographic investigation.

The reason for the very slow and incomplete dissolution of carbon that is observed in the presence of fine BN can be supposed to be a well adhering layer of BN that virtually shields the iron surfaces against graphite and prevents direct contact. It may be assumed that the iron powder particles are preferentially coated with BN, which is markedly finer than the graphite used, and therefore the direct contact between graphite and iron, which according to [26] is necessary for effective carbon dissolution, is attained only to a minor degree. If coarse BN is used, this coating effect does not occur, and carbon dissolution takes place more or less as common in Fe-C.

When sintering at 1250°C, i.e. above the Fe-B eutectic, the effect of the atmosphere becomes very much evident. Sintering of the BN-containing material in N_2 results in an isothermal shrinkage behaviour that is similar to that of Fe-C in vacuum,

although during the heating section there are considerable differences, for Fe-C the ferrite-austenite transformation once more starting at lower temperatures, and there is also a more pronounced expansion than in the BN containing steel. Both phenomena can be attributed to the faster carbon dissolution in Fe-C than in Fe-C-BN.

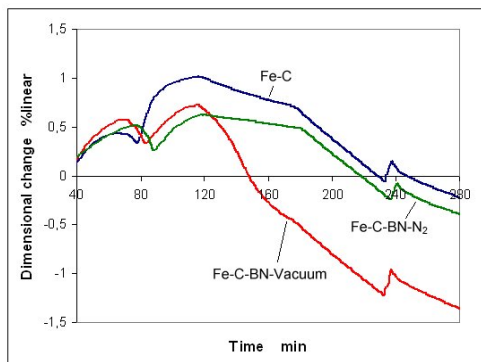


Fig.6. Dilatometric graphs for Fe-0.8%C-0.5%BN and Fe-0.8%C, respectively. Heating / cooling rate 10 K/min, 60 min 1250°C isothermal, rotary pump vacuum / static N₂ 5.0.

Sintering of Fe-C-BN in vacuum, in contrast, results in pronounced shrinkage during isothermal sintering. Up to the isothermal sintering temperature, the graph is similar to that obtained for the same material in N₂, but then shrinkage starts to become very pronounced, which is typical for the well-known sintering activation effect of boron. This underlines that in vacuum, BN can be used as a sintering activator, decomposing to generate free boron.

CONCLUSIONS

The investigations have shown that hexagonal boron nitride (hBN) adversely affects compactibility, sintering and properties if added as a very fine grade. hBN inhibits formation of stable sintering contacts, and also the dissolution of carbon in the iron particles, even for Fe-0.8% C more ferrite than pearlite being found after sintering. If sintering is done in a plain H₂ atmosphere or in vacuum, hBN decomposes at higher temperatures, activating the sintering process and resulting in satisfactory mechanical properties, unless the BN content is so high that grain boundary embrittlement through boride networks occurs. In the case of BN decomposition the carbon dissolution occurs as expected as well. In N₂ or N₂-H₂ atmosphere, BN decomposition does not occur even at 1250°C, and the sintering temperature is of minor relevance. Coarse hBN is much less effective in every aspect than fine grade, its adverse effect on compactibility and mechanical properties being rather small, and also carbon dissolution is hardly affected.

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