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Metallographic observations during the sintering of BM2 type of high speed steels

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Metallographic changes during the sintering of BM2 type of high speed steels have been investigated by scanning electron microscopy, in the range of optimum sintering to oversintering. The primary carbides observed are M_6C and a small quantity of MC; in the oversintered structure an additional carbide with eutectic morphology was seen. It is a chromium and molybdenum rich phase in BM2+ 0 to 4% cobalt (0.9 to 1.2% carbon) alloy, whereas in BM2+ 8% cobalt (0.9% carbon) the eutectic phase is MC. Under certain conditions M_3C was also detected in the post-sintered alloy.

1. Introduction

Powder metallurgical routes for the fabrication of high speed tool steels are now well established [1]. The first stage of the process is the production of pre-alloyed powder, either by water or inert gas atomization. The powder particles can be regarded as ingots of $\sim 10^2$ to 10^1 μm size, because of the microstructural similarity with those of the conventional material; the only difference being the sub-micrometre size of the features in the powder microstructure and this is due to the rapid cooling, 10^3 to 10^6 °C sec^{-1}, during atomization [2–4].

1.1. Phases observed in the cast structure

The primary carbides observed in the solidified BT1 high speed steel (0.8 C, 4 Cr, 0.4 Mo, 1 V, 18 W weight %) are M_6C, MC and $M_{23}C_6$, but in the molybdenum bearing BM2 (0.8 C, 4 Cr, 5 Mo, 2 V, 6 W), in addition to M_6C and MC carbides M_2C is also present; there is some evidence that M_7C_3 and $M_{23}C_6$ carbides may also occur in the latter steel [5]. M represents a group of atoms, e.g. Cr, Fe, Mo, V and W. Some of the primary carbides take the eutectic morphology; in BT1 the eutectic carbide has been identified as M_6C [5], while in BM2 it is reported to be either M_2C or M_6C [6–10].

1.2. Phases observed in the as-atomized powder and sintered material

Kato [2] reported that, in as-atomized JIS SKH57 steel (1.51 C, 10.05 Co, 4.20 Cr, 0.24 Mn, 3.85 Mo, 0.32 Si, 3.72 V, 10.12 W, similar to BT42), the primary carbides are MC and M_2C. On heating above 700° C M_2C decomposes and M_6C appears in the structure; MC is already present in the structure.

Kumar *et al.* [11] examined nitrogen atomized BT15 (1.5 C, 5.22 Co, 4.6 Cr, 4.85 V, 12.19 W) and its cobalt free counterpart Rex 25 (1.79 C, 3.01 Cr, 6.52 Mo, 4.63 V, 13.29 W). Both of these alloys in the atomized state contained M_6C carbide and MC carbide in cubic and hexagonal form. In the hot isostatically pressed samples, the carbides detected were MC, M_6C, and $M_{23}C_6$. $M_{23}C_6$ was attributed to the slow cooling from the processing temperature.

Ozerskii *et al.* [4] found that MC, M_2C, and M_6C are the primary carbides present in the nitrogen atomized R6M5 alloy powder (1.27 C, 3.7 Cr, 5 Mo, 0.053 N, 2.14 V, 6.22 W, similar to BM2). In the oversintered BM2 and BT15 the eutectic phase has been suggested to be M_6C [12].

The carbides in steel can dissolve alloying elements as solid solution [13] and the composition of the carbides can be determined by solid state micro-analytical techniques like energy dispersive X-ray spectroscopy (EDXS), provided that these phases are larger than the resolving power of the instrument, which is typically of the order of ~ 2 to 3 μm. However the phases present in the powder particles are sub-micrometre in size, which precludes this form of composition determination of the phases in the powder particles.

It is generally accepted that around the optimum sintering temperature, liquid phase occurs in the microstructure of the high speed steels; evidences to support this view are based on metallographic observations and are often indirect in nature [14]. The optimum sintering temperature is defined as the temperature at which near full densification takes place without any undesirable coarsening of the microstructure and formation of the continuous grain boundary film. If the sintering is carried out at a temperature close to the macroscopic fusion point evidence of liquid phase can be seen in the microstructure in the shape of the carbides with eutectic morphology. Thus oversintering can simulate a structure transformed from liquid → solid or liquid + solid → solid. The carbides present in the sintered structure are large enough for their composition to be determined by EDXS.

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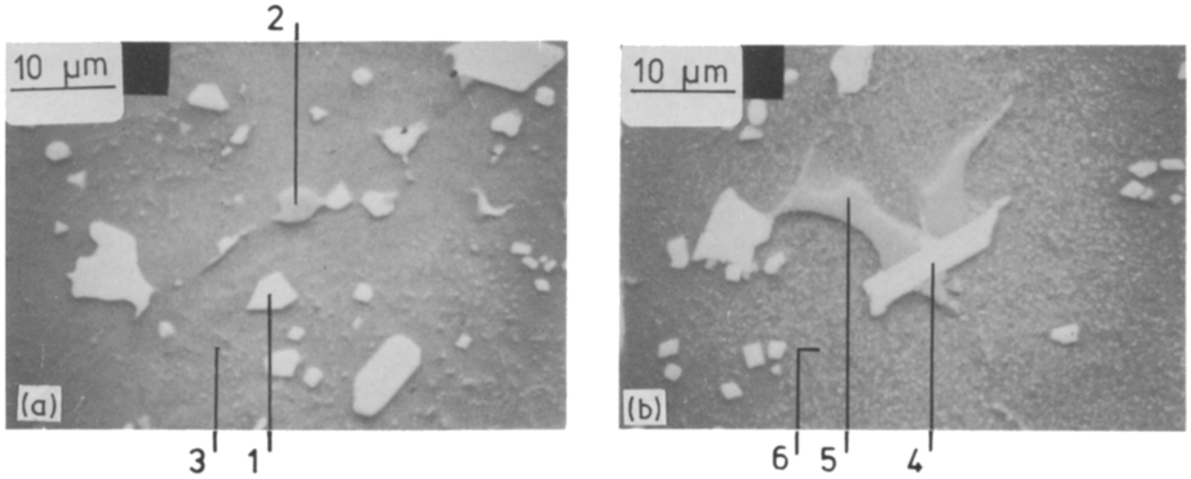

Figure 1 Correctly sintered BM2 (0.92 C, 0.50 Co, 4.10 Cr, 0.05 Mn, 5.15 Mo, 0.15 Ni, 0.50 Si, 1.9 V, 6.50 W); (a) sintered 1250° C, (b) sintered 1250° C and transform annealed 900° C, 0.5 h → 750° C, 5 h → natural cool

Spectrum	Phase	Cr	Fe	Mo	Si	V	W	
1	M_6C	3.24	31.65	19.25	1.33	5.31	39.23	BM2
2	MC	4.74	5.76	15.81	–	47.73	25.66	correctly
3	matrix	4.36	87.63	2.69	0.55	1.45	3.29	sintered 1250° C, 1a
4	M_6C	4.07	31.28	20.27	–	4.54	39.73	BM2
5	MC	5.55	3.75	22.83	–	46.79	20.54	sintered and
6	matrix	4.36	82.96	3.65	–	1.32	6.86	annealed, 1b

–below detection level

The composition of the primary carbides in the sintered BT42 steel over the entire sintering range has been described earlier [14–16]. The present study mainly concerns with BM2 type of steels. The composition of the primary carbides and their genesis have been discussed in the paper.

2. Experimental details

Water atomized annealed powders of different compositions were cold compacted to about 70% theoretical density and sintered under vacuum 13.3 nbar (1.33×10^{-3} Pa, 10^{-5} torr). The thermal cycle was as follows

heat 14° C min^{-1} → sintering temperature, sinter 1 h
→ cool, average rate 14° C min^{-1}
upto 850° C

The sintered samples were sectioned and prepared according to the standard metallographic techniques. Unetched samples were examined under scanning electron microscope (SEM) equipped with a Robinson back scattered electron detector. EDXS was analysed by a Link system using chromium as a standard.

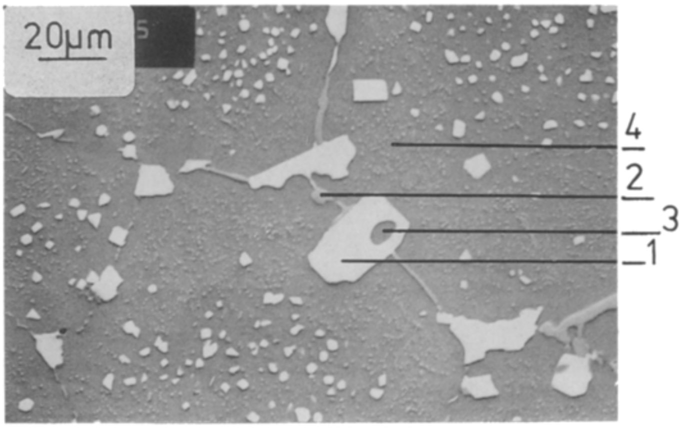

Figure 2 Cobalt modified BM2 (0.85 C, 8.28 Co, 4.04 Cr, 0.20 Mn, 4.75 Mo, 0.14 Ni, 0.32 Si, 1.69 V, 6.23 W), sintered 1260° C, oversintering by about 10° C; areas marked 1 and 3 suggest the occurrence of liquid phase

Spectrum	Phase	Co	Cr	Fe	Mo	Si	V	W
1	M_6C	3.43	2.99	27.69	19.19	0.95	3.97	4.06
2	MC	–	5.22	4.69	22.37	–	44.19	23.48
3	matrix	8.96	4.02	80.47	1.63	0.41	1.06	3.46
4	matrix	8.38	4.06	76.24	3.44	0.66	1.77	5.25

–below detection level

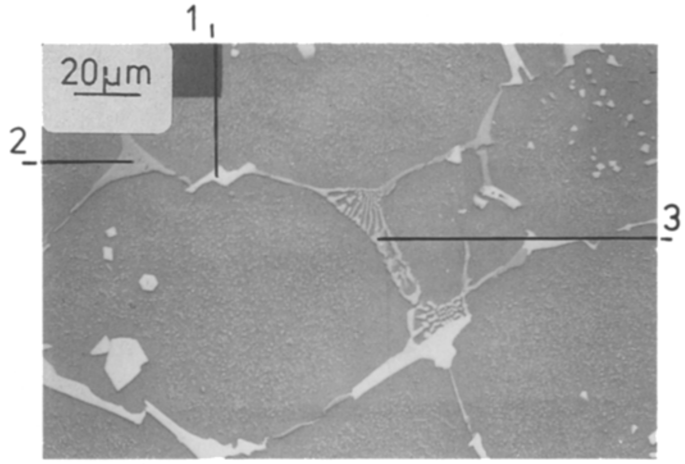

Figure 3 Cobalt modified BM2 (0.84 C, 2.45 Co, 4.50 Cr, 0.05 Mn, 5.20 Mo, 0.25 Ni, 0.40 Si, 1.44 V, 6.20 W), sintered 1275° C, oversintering ~ 10° C

Spectrum	Phase	Co	Cr	Fe	Mo	Si	V	W
1	M_6C	0.69	3.86	30.67	21.66	1.85	3.71	37.56
2	MC	–	4.83	3.34	22.54	1.98	44.94	22.37
3	eutectic morphology	–	18.68	23.46	33.78	–	10.86	12.65

–below detection level

Composition of the BM2 type of steel was modified by the additions of cobalt, carbon and silicon. The cobalt modified alloys contained 2, 4 and 8% cobalt, the carbon modified alloys contained 0, 2 and 4% cobalt; the silicon modified alloy contained about 1.5% carbon and 2% silicon. The detailed composition of the cobalt and carbon modified alloys are given elsewhere [17] and that of the silicon modified alloys is given in the next section.

3. Results

Fig. 1 records the typical SEM back scattered micrograph from a correctly sintered material, composition of the alloy and those of the metallic constituents of the phases, in weight %, have also been included. The large grain boundary primary carbides are M_6C and MC – the former being the major constituent. With the increase in the sintering temperature the carbides coarsened and there is a tendency towards the formation of grain boundary film, consisting mainly of MC, as shown in Fig. 2; this sample has been oversintered by about 10° C. The island-like feature marked 1 and 3 occurred mainly with the M_6C carbides and has been observed in other systems as well [14]. Microstructures of the specimens sintered close to the macroscopic fusion point (oversintered by ~ 20 to 25° C) are given in Figs 3 to 5. Some distortion was noted in these samples. *It is important to stress that, in the materials for practical applications these structures are avoided and only studied here because on sintering in this*

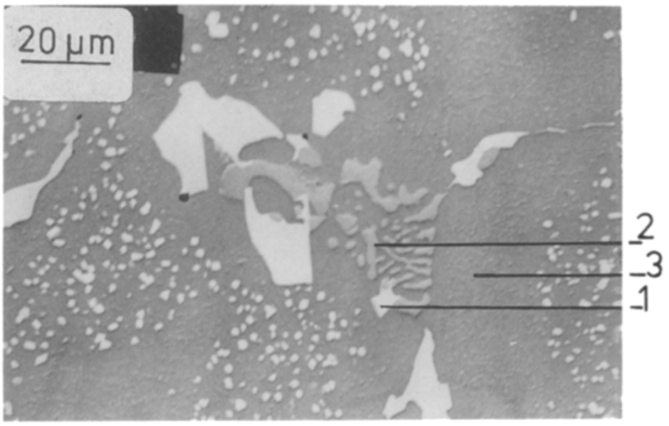

Figure 4 Cobalt modified BM2 (composition same as Fig. 2, 8.28 Co), sintered 1270° C, oversintering ~20° C; only MC eutectic was observed

Spectrum	Phase	Co	Cr	Fe	Mo	Si	V	W
1	M_6C	3.31	3.75	27.68	19.37	0.71	4.14	41.09
2	MC eutectic morphology	–	5.93	5.68	23.61	–	42.01	22.43
3	matrix	8.49	4.24	78.10	2.90	0.58	1.13	4.38

–below detection level

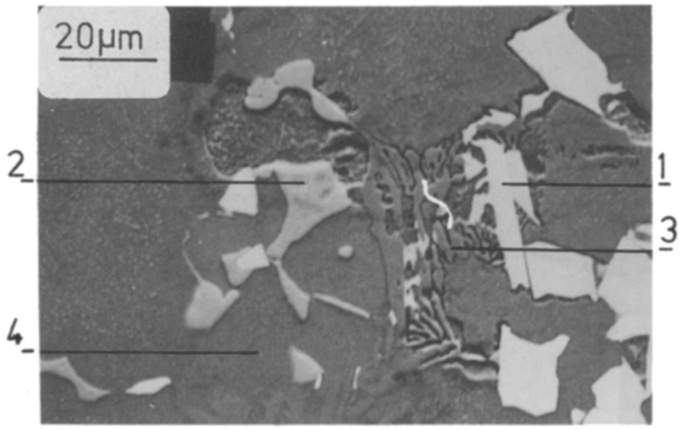

Figure 5 Experimental alloy based on the composition of BM2 (similar to Fig. 1, except 1.52 C, 2 Si) sintered 1250° C, oversintering ~ 30° C, resulted in fusion

Spectrum	Phase	Cr	Fe	Mo	Si	V	W
1	M_6C	3.09	29.27	19.50	2.18	3.34	41.47
2	MC	5.09	5.90	18.65	-	45.57	26.79
3	eutectic morphology	22.99	58.00	9.17	-	4.34	4.39
4	matrix	5.38	90.29	1.34	2.02	0.37	2.05

-below detection level

temperature range carbides of eutectic morphology can be seen. Within the limit of the experimental scatter the composition of the phases does not appear to be dependent on the sintering temperature and carbon content. This also appears to be the case with BT42 [14]. The average composition of the phases (Table I) falls in the range as observed in other laboratories on conventional BM2 [6, 7, 18, 19]. The present study also confirms that cobalt is present in the matrix and M_6C carbide [14, 16, 19, 20]. In the current series of alloys two types of carbides with eutectic morphology have been seen in the highly oversintered alloys.

(i) Alloys with 0 to 4% cobalt, 0.9 to 1.2% carbon and high silicon alloy: In the former material the phase with the eutectic morphology contains about 20% chromium and 36% molybdenum (Figs 3 and 5), where as the chromium level in other phases is about 4 to 5%. A similar carbide occurs in the oversintered BT42 [14]. In the high silicon alloy the eutectic carbide contains about 23% chromium and 9% molybdenum. Recently a high chromium containing M_2C carbide has been observed in ASP [20] and BM42 alloys [21]. The composition of the eutectic carbides in Table I does not conform to that of M_2C.

(ii) Alloy with 8% cobalt and 0.9% carbon: In this system vanadium-rich MC carbide has been found to occur as the eutectic type of carbide (Fig. 4) and no chromium-rich carbide was observed. This is rather surprising, since the solubility of cobalt in the MC carbide is negligible and in the high cobalt bearing BT42 steel MC eutectic was not seen under similar conditions [14]; however a limited amount of MC carbides of eutectic morphology was seen in BT42 when sintered to fusion [16].

Throughout the sintering range studied, in the post-sintered structure, pearlite containing M_3C was observed in BT42, BM2 + 8% cobalt and BM2 + 0 to 4% cobalt, 1.2% carbon alloys. Only a small quantity of M_3C occurred if the carbon content is less than 0.9%. This phase was inferred from the nital etching characteristics [17]. It should be pointed out that the reliable estimation of silicon by EDXS may not be possible in the presence of high tungsten content.

4. Discussion

It has been mentioned earlier that MC, M_2C, and M_6C are the carbides present in the atomized and cast BM2 steels. The maximum annealing temperature of the water atomized powder is about 1050° C [22]; at this temperature, although the decomposition of M_2C according to the reaction $M_2C + \gamma \rightarrow MC + M_6C$ may not reach completion in the cast structure, in the as-atomized structure, because of the fine carbide size, total decomposition M_2C can be expected. Hence it is concluded that MC and M_6C are only primary carbides present in the annealed atomized BM2 powder. This appears to be the situation in BT42 [2].

From the results of Fedricksson *et al.* [7] and Lenta *et al.* [10] it is likely that around the optimum sintering temperature (~ 1250° C), M_2C should not be present in the microstructure and majority of the MC and M_6C should be in solution especially because of their fine size in the original powder particles. Their results also suggest localized liquidation in this temperature range, but the present study does not show any clear evidence of fusion near the optimum sintering temperature.

Development of the final microstructure during sintering need not follow the same pattern as that by the conventional material on heating, namely if a liquid phase occurs during sintering the surface energies control the morphology of the phases [23], while prolonged heating leads to the spheroidization of the phases [9]. In the present series of experiments at the majority of sites MC and M_6C carbides occurred together, but their morphology does not suggest that their genesis is

TABLE I Composition of the phases observed in BM2 and cobalt modified BM2 alloys, determined by EDXS, some of the published results by other workers are also included

Alloy	Phase	Composition (wt %)							Reference
		Co	Cr	Fe	Mo	Si	V	W	
BM2	M_6C carbide	–	4	31	19	1	4	41	present study, as-sintered
BM2 + 2 Co		0.6	4	33	21	0.7	3	37	
BM2 + 4 Co		2	3	29	21	1	4	40	
BM2 + 8 Co		3	3	29	19	1	4	40	
BM2			4.8	29.4	25.5		4.2	31.7	cast [6]
BM2			7	33	22		5	33	cast and reheated [7]
BM2			3	34.1	20.3		4.05	38.3	cast and reheated [9]
BM2			5.1	31.5	25.6		4.7	29.1	deformed and hardened [17]
BM2 + 8 Co		0.2	7.2	7.9	30.3		12.5	39.0	
		3.2	3.5	37.0	21.7		5.3	28.0	
BM2			3.7	35.1	18.9		3.4	36.0	deformed and hardened [18]
BM2	MC carbide	–	4	4	20	–	49	23	present study, as-sintered
BM2 + 2 Co		–	5	5	24	–	45	21	
BM2 + 4 Co		–	4	4	25	–	45	20	
BM2 + 8 Co		–	5	4	22	–	44	24	
BM2		too small for accurate analysis							cast [6]
BM2			8	3	14 – 10		42 – 57	22 – 15	cast and reheated [7]
BM2			5.1	1.1	14.5		46.3	33	cast and reheated [7]
BM2				2.6	9.0		53.1	12.6	deformed and hardened [17]
			6.1	2.7	10.3		45.3	15.7	
BM2 + 8 Co		0.2	3.7	2.7	21.0		40.2	19.5	
BM2		not reported							deformed and hardened [18]
BM2	eutectic carbide	–	18	23	35	–	11	13	present study, as-sintered
BM2 + 2 Co		–	18	20	36	–	12	14	
BM2 + 4 Co		–	16	28	30	–	9	16	
BM2 + 8 Co	MC eutectic	–	6	5	24	–	42	22	
BM2	M_2C carbide		7.0	7.4	30.5		13.7	35	cast [5]
BM2			7	5	34		12	42	cast and reheated [7]
BM2			7.6	7.2	21.3		14.7	49.3	cast and reheated [9]
BM2			not reported						deformed and hardened [17]
BM2 + 8 Co		4.1	6.0	10.0	33.3		22	38.3	
BM2			not reported						deformed and hardened [18]
BM2	Matrix	–	4	86	3	0.6	1	4	present study, as-sintered
BM2 + 2 Co		1.7	4	82	4	0.4	2	4	
BM2 + 4 Co		4	4	82	3	0.7	2	4	
BM2 + 8 Co		8	4	77	3	0.5	2	5	

–below detection level

in the decomposition of M_2C into MC and M_6C as observed by other workers in the conventional material [7, 9, 10].

The matrix structure is due to solid state precipitation and occurs at a relatively low temperature; this compares well with the results of Ghomaschi and Sellers (Fig. 1c, [9]). In the present series of alloys the heterogeneous grain boundary nucleation of the carbides occurs at higher temperature and any unequivocal conclusion if the morphology is due to liquid → solid transformation, is not possible yet. It is appropriate to mention that the morphology of the solidified liquid phase in the conventional material by Fedricksson *et al.* [7] and Lenta *et al.* [10] is not the same as noted by Takajo and Nitta [24] in the powder metallurgical alloy. A liquid phase field is reached on oversintering by about 20 to 25° C and on cooling a phase with eutectic morphology appears in the final microstructure – it is a chromium-rich phase in BM2 + 0 to 4% cobalt alloy and vanadium rich MC in BM2 + 8% cobalt alloy. These carbides are stable in the post-sintered structure.

5. Conclusions

(1) At the optimum sintering temperature $\sim$ 1250° C, the majority of the carbides are expected to be in solution in the present series of alloys.

(2) During cooling from the sintering temperature the primary carbides precipitate at a higher temperature and these occur mainly at the grain boundaries, precipitation of finer carbides within the matrix takes place at a relatively lower temperature.

(3) At higher sintering temperature carbides of eutectic morphology also appear and these do not decompose appreciably during cooling.

(4) EDXS suggests that the typical metallic constituents of the carbides in weight % are

	Co	Cr	Fe	Mo	Si	V	W
M_6C	0–3	4	30	20	1	4	40
MC		5	5	24		45	20

These occurred as primary grain boundary carbides. The composition of the carbides of eutectic morphology in BM2 + 0 to 4% cobalt, 0.9 to 1.2% carbon alloys is 20 Cr, 20 Fe, 36 Mo, 11 V, 13 W; in BM2 + 8% cobalt, 0.9% carbon alloy it is MC.

(5) In high carbon and 8% cobalt alloy M_3C bearing pearlite is a common feature, while in BM2 + 0 to 4% cobalt, 0.9% carbon alloys only traces of pearlite were seen in the post-sintered structure.

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