Chapter 6: Processing AIMgB₁₄

As highlighted in chapter 2, $AlMgB_{14}$ admixed with 30 wt. % TiB_2 has been identified as an ultra-hard material. The processing of $AlMgB_{14}$ and composites of this material will be dealt with in this chapter.

6.1: Introduction

AlMgB₁₄ has an intrinsic hardness of between H_{V100} 27.70 to 28.90 GPa⁵⁶. The addition of 30 wt.% TiB₂ increases the hardness of AlMgB₁₄ to 40 GPa². Although no load was given for the measurement of this hardness, the addition of TiB₂ clearly enhanced the hardness of AlMgB₁₄. No reason for this increase in the hardness of AlMgB₁₄ as a result of the addition of TiB₂ has been offered and it is thus not understood why the addition of TiB₂ increases the hardness.

Part of this chapter is concerned with evaluating the effect of additives, including TiB₂, on the hardness of AlMgB₁₄. The study is not only an attempt to explain the reasons for the enhancement of the hardness, but also to try and understand what additional phases (if any) are formed as a result of the addition of TiB₂, TiC, TiN, TiCN and WC to AlMgB₁₄. For this reason the ternary phase systems Ti-B-C, Ti-B-N and W-B-C will hence also be discussed.

6.1.1: The Ti-B-C Ternary Phase System

A comprehensive account of the Ti-B-C ternary phase diagram has been given by Bittermann *et al*⁹³. An isothermal section of the Ti-B-C system based on the experimental results obtained form the work of Rudy *et al*⁹⁴, taken at 1400°C, is presented in figure 6.1.1. No ternary compounds exist in the Ti-B-C system. Solid solubilities of the binary carbide (TiC) and the binary boride (TiB₂) phases have been found to be negligible⁹⁵⁻⁹⁸. The lattice parameters in the ternary Ti-B-C are increased considerably for TiC with respect to those of the binary Ti-C system as a result of the formation of some solid solution between B and TiC. A very slight solid solubility of

B and C in β -Ti was found by Rudy *et al*⁹⁴. The presence of Ti₃B₄ was confirmed from both binary and ternary alloys annealed at temperatures between 1400 and 1800°C⁹⁹. The solubility of C in Ti₃B₄ was found to be negligible¹⁰⁰. The structural data for the phases in the Ti-B-C system are given in table 6.1.1.



Figure 6.1.1: An isothermal section at 1400°C for the Ti-B-C ternary phase diagram⁹⁴

Phase/Temperature	Crystal System	Lattice Parameters	Reference	
Range (°C)		(Å)	JCPDS	
β-Ti/ < 1670	Cubic	a = 3.3065	44-1288	
β -B/ < 2092	Rhombohedral	a = 10.925, c = 23.814,	31-0207	
C/ <3827	Hexagonal	a = 2.470, c = 6.724	41-1487	
'B ₄ C'/ <2450	Rhombohedral	a = 5.600, c = 12.086	35-0798	
TiB/ <2190	Orthorhombic	a = 6.121, b = 3.061	73-2148	
		c = 4.301,		
Ti ₃ B ₄ / <2200	Orthorhombic	a = 3.259, b = 13.73	19-1368	
		c = 3.042		
TiB ₂ / <3225	Hexagonal	a = 3.0303, c = 3.2295	35-0741	

6.1.2: The Ti-B-N Ternary Phase System

The isothermal section for the Ti-B-N system at 1500° C, reproduced from the work of Smid¹⁰¹ is shown in figure 6.1.2. No ternary compounds form in the Ti-B-N phase system. The formation of the stable TiB-TiN_{1-x} tie line at temperatures below 1500° C highlights the incompatibility of titanium metal and hexagonal BN. The heating of both binary and ternary alloys in the nitrogen rich corner of the ternary system results in the formation of compounds of BN and TiN as determined by the tie line.

The solid solubility of Ti in BN is low up to temperatures of 1500°C. Additionally the solubilities of the titanium borides, titanium nitrides and BN are also low up to 1500°C. Table 6.1.2 gives information about the solid phases.



Figure 6.1.2: An isothermal section at 1500°C for the Ti-B-N ternary phase diagram¹⁰¹

Phase/Temperature	Crystal System	Lattice Parameters	Reference
Range (°C)		(Å)	JCPDS
β-Ti/ < 1670	Cubic	a = 3.3065	44-1288
α-Ti/ < 882	Hexagonal	a = 2.951, c = 4.684	
β -B/ < 2092	Rhombohedral	a = 10.925, c = 23.814,	31-0207
C/ < 3827	Hexagonal	a = 2.470, c = 6.724	41-1487
'B ₄ C'/ < 2450	Rhombohedral	a = 5.600, c = 12.086	35-0798
TiB/ < 2190	Orthorhombic	a = 6.121, b = 3.061 c = 4.561,	73-2148
$Ti_{3}B_{4}/ < 2200$	Orthorhombic	a = 3.259, b = 13.73 c = 3.042	19-1368
$TiB_2/ < 3225$	Hexagonal	a = 3.0303, c = 3.2295	35-0741
$Ti_2N < 1080$	Tetragonal	a = 4.142, c = 8.8055	73-0959
$Ti_4N_{3-x} / < 1066$	Rhombohedral	a = 2.9783, c = 28.9438	39-1015
$Ti_3N_{2-x} / < 1078$	Rhombohedral	a = 2.9809, c = 21.6642	40-0958
δ-TiN _{1-x}	Cubic	a = 4.2417	38-1420

TABLE 6.1.2: Structural data for the phases in the Ti-B-N ternary phase diagram

6.1.3: The W-B-C Ternary Phase System

No ternary compounds exist throughout the W-B-C ternary phase diagram. Solid solubilities of the binary boundary phases are found to be less than 3 at.% or negligible¹⁰². Isothermal sections have been studied experimentally at $1500^{\circ}C^{103}$, $1700^{\circ}C^{104}$ and at $1800^{\circ}C^{105}$. Based on the phase equilibria determined from the isothermal sections a series of isotherms were proposed for temperatures of 1500, 2000, 2150, 2320, 2500, 2700 and $2800^{\circ}C^{103}$.

Figure 6.1.3 is an isothermal section at 1500°C for W-B-C. Table 6.1.3 gives the data for the solid phases present in the W-B-C ternary phase diagram¹⁶.



Figure 6.1.3: An isothermal section at 1500° C for the W-B-C ternary phase diagram¹⁰³

Phase/Temperature	Crystal System	Lattice Parameters	Reference	
Range (°C)		(Å)	JCPDS	
$\beta W/ < 3422$	Cubic	a = 5.050	47.1319	
$\beta B/ < 2092$	Rhombohedral	a = 10.925, c = 23.814,	31-0207	
C/ < 3827	Hexagonal	a = 2.470, c = 6.724	41-1487	
'B ₄ C'/ < 2450	Rhombohedral	a = 5.600, c = 12.086	35-0798	
αW ₂ C/ 1800-1050	Hexagonal	a = 2.9970, c = 4.7279	35-0776	
WC / < 2776	Hexagonal	a = 2.9062, c = 2.8378	25-1047	
$W_2B/<2670$	Tetragonal	a = 5.568, c = 4.744	25-0990	
δWB/ 2170	Tetragonal	a = 3.1166, c = 16.9101	35-0738	
$W_2B_{5-x}/<2365$	Hexagonal	a = 2.9856, c = 13.8723	38-1365	
$W_{1-x}B_3/<2020$	Hexagonal	a = 5.200, c = 6.340	19-1373	

TABLE 6.1.3: Structural data for the phases in the W-B-C ternary phase diagram

6.2: Experimental

The processing of $AlMgB_{14}$ required no additional techniques to those laid out in the experimental chapter. All sintering temperatures were held for 1 hour unless otherwise stated. For a comprehensive explanation of the experimental techniques used for the processing of $AlMgB_{14}$ see chapter 3.

6.3: Results and Discussion

6.3.1 Preliminary investigations of the formation of $AlMgB_{14}$

Powders of Al, Mg and B were milled in the respective molar proportions of 1:1:14, as laid out in the experimental section 2. Green compacts were pressed out at 20 MPa and sintered at 1400°C in a tube furnace, uniaxial hot press and hot isostatic press (HIP) under a constant flow of argon gas.

A comparison of the diffractograms obtained from materials produced in the tube, HIP and uniaxial hot press furnaces are presented in figure 6.3.1.



Figure 6.3.1: A comparison of the diffractograms for AlMgB₁₄ produced in the tube, HIP and uniaxial hot press furnaces at 1400°C The same phases were produced in all three furnaces but the intensity ratio of the major peak for $AlMgB_{14}:MgAl_2O_4$ was different for the materials produced in the three different furnaces. Table 6.3.1 summarises the most important information obtained from the samples produced in the three different furnaces.

TABLE 6.3.1:	Phases,	density	and	porosity	for	AlMgB ₁₄	prepared	the	three	differer
furnaces										

Furnace	Phases (XRD)	Density	Porosity	
*		(gcm ⁻³)	(%)	
Tube	MgAl ₂ O ₄ ,	1.532	41.5	
(atm)	$(MgAlB_{14})^t$			
HIP	MgAlB ₁₄ ,	1.913	27.7	
(20 MPa)	MgAl ₂ O ₄			
Hot Press	MgAlB ₁₄ ,	1.772	33.7	
(20 MPa)	MgAl ₂ O ₄			

*Samples prepared at 1400°C, 1 hour soak time

t = trace amounts only

The lowest porosity obtained, and expected, are for encapsulated samples prepared in the HIP at 20 MPa and 1400°C and the greatest porosity obtained from samples prepared in the tube furnace. The data can be explained since the application of an external load to a material shortens diffusion distances between the reacting particles. Hence, the HIP furnace which offers an isostatic external pushing force produces the samples with the lowest porosity. The tube furnace that applies no force at all produces samples with the highest porosity. AlMgB₁₄ is formed only in trace amounts in the tube furnace at 1400°C.

Although no detailed quantification of the phases has been done on these samples the intensity of the spinel (MgAl₂O₄) peak was much larger than the intensity of the boride (AlMgB₁₄) peak for the samples prepared in the tube furnace. This suggests that the tube furnace is not entirely air tight and hence a greater oxide product was produced. Oxidation is further limited in the HIP and the hot press since the

application of an external load to the samples produced in the HIP and the hot press result in samples with lower surface areas.

Figure 6.3.2 shows the microstructure obtained from the sample prepared in the hot press at 1400°C and 20 MPa. The presence of the spinel and boride phases as determined by EDS analysis are labelled in the figure. The resin in which the sample was mounted for polishing is present because it (the resin) filled the pores of the sample during sample mounting. Further information as regards the densification of AlMgB₁₄ and the composites to be presented is given in section 3.3.4.



Figure 6.3.2: Microstructure of AlMgB₁₄ made at 1400°C and 20 MPa in a hot press

6.3.2: A synthesis of AlMgB₁₄ from AlB₁₂ and MgB₂

The density of the compacts prepared at 1400°C from elemental powders is quite low, $1.76 \pm 0.10 \text{ gcm}^{-3}$. The low density associated with high levels of porosity, $35 \pm 1 \%$

for compacts prepared from elemental powders at 1400°C make hardness measurements meaningless.

In order to produce denser sintered compacts it is important to start with as small a starting particle size as possible. As detailed in the experimental section the particle size of Al and Mg reaches a minimum value of about 30 μ m in the planetary mill. It was found that the milling of these metals together with boron in a planetary mill yielded starting powders that were between 7-15 μ m in size.

The use of AlB_{12} and MgB_2 , which are brittle powders, should upon milling produce starting powders with smaller particle sizes. Milling of AlB_{12} and MgB_2 in 1:1 molar proportions yielded a starting powder of between 2-5 µm particle size in the planetary mill. The proposed reaction scheme during thermal treatment is shown in equation 6.1.

$$AlB_{12} + MgB_2 \rightarrow AlMgB_{14}$$
 6.1

Pure AlB₁₂, MgB₂ and the powder mixture AlB₁₂ and MgB₂ were all pressed into green compacts. The green compacts were then sintered at 1400° C under a load of 20 MPa. Figure 6.3.3 shown is the observed diffractogram for the sintered compacts.



Figure 6.3.3: Diffractogram for $AlB_{12} + MgB_2$ prepared at 1400°C for 1 hour

The peaks for AlMgB₁₄ at 13° and 41.5-43° 2 θ are matched in the diffractogram for AlB₁₂ + MgB₂. The peaks at 41.5-43° 2 θ overlap with both MgB₂ and AlB₁₂ but the peak at 13° 2 θ can be unambiguously assigned to AlMgB₁₄. Therefore, it is possible to produce AlMgB₁₄ as shown in equation 6.1. Furthermore, W₂B₅ is present due to the contamination of the powders during the milling cycle. The XRD results show that the reaction is much slower than from the elemental powders.

Table 6.3.2 compares the densities and porosities of the sintered compacts with elemental $AIMgB_{14}$.

Material	Density	Open Porosity
(1400°C, 20MPa)	(gcm ⁻³)	(%)
AlB ₁₂	1.642	37.5
MgB_2	1.968	30.1
$AlB_{12} + MgB_2$	1.758	34.9
AlMgB ₁₄	1.772	33.7

TABLE 6.3.2: Density and porosity of AlB_{12} , MgB_2 and the composite $AlB_{12} + MgB_2$

The electron densities of each of the phases AIB_{12} and $AIMgB_{14}$ are similar. Therefore it is difficult to contrast these phases in back scattered mode in the SEM.

The amount of unreacted material is large since the major peaks in the XRD are that from unreacted MgB₂. The major peaks for AlMgB₁₄ are small in comparison to those peaks from the unreacted MgB₂. No Rietveld analysis was done to confirm the amounts of the respective phases as the production of AlMgB₁₄ was not high yielding. The formation of AlMgB₁₄ by this route is low yielding. The reason for this is most likely due to the slow kinetics of solid state reactions. This route, although novel was not pursued further. Also a dense material was not produced by this method. Thus, the optimisation of the processing was based upon the synthesis of AlMgB₁₄ from the elemental powders.

6.3.3: Additives in AlMgB₁₄

The role of additives in AlMgB₁₄ has been shown, by the Ames group, to have very significant influence on the hardness of AlMgB₁₄. In this study 20% by mass Al₂O₃, TiN, TiC, TiCN and WC and 30 wt.% TiB₂ was added to the AlMgB₁₄ elemental composition and the each mixture was sintered in the hot press at 1400°C and 20 MPa. The additives were chosen in order to understand which of the above phases are stable with respect to AlMgB₁₄.

Table 6.3.3 indicates the density and the porosity associated with all of the composites produced in the hot press.

Sample	Density	Porosity	Phases
$(AlMgB_{14} + wt.\%)$	(gcm ⁻³)	(%)	(XRD)*
+ 30% TiB ₂	2.204	29.1	TiB ₂ ; AlMgB ₁₄ ; MgAl ₂ O ₄
+ 20% TiN	1.972	33.7	TiB ₂ ; AlMgB ₁₄ ; MgAl ₂ O ₄ , AlN
+ 20% TiC	1.381	52.7	TiB ₂ ; AlMgB ₁₄ ; MgAl ₂ O ₄
+ 20% TiCN	1.959	32.5	TiB ₂ ; AlMgB ₁₄ ; MgAl ₂ O ₄ , AlN
+ 20% WC	2.139	26.9	W ₂ B ₅ ; AlMgB ₁₄ ; MgAl ₂ O ₄

TABLE 6.3.3: Density, porosity and major phases for AlMgB₁₄ composites

* (Al,Mg)B₂ could be present in all the phases but can't readily be identified from XRD due to peak overlap with other phases

Very porous samples are produced for all the additives and thus polishing the samples in order to determine the hardness of the materials was not successful. The major phases identified in each of the composites indicated that TiN, TiC, TiCN and WC are not stable with respect to their corresponding borides, i.e. each one of the preceding additives preferentially forms the corresponding boride. The evidence, as based on XRD and SEM analysis, for all of the composites is presented below.

6.3.3.1 AlMgB₁₄ + 30 wt.% TiB₂

Figure 6.3.4 a compares the diffractograms obtained for 30 wt.% TiB_2 with pure TiB_2 and $AlMgB_{14}$.



Figure 6.3.4a: Identified phases in AlMgB₁₄ + 30 wt.% TiB₂

The high electron density associated with TiB_2 results in very dominant peaks for the TiB_2 phase. The presence of $AlMgB_{14}$ can be seen as a broad peak in the region of $41.5^{\circ}-43^{\circ}$ 20. A more detailed XRD analysis, of that region $(41^{\circ}-44^{\circ})$ indicates sharp peaks for $AlMgB_{14}$, figure 6.3.4 b. The presence of the spinel phase is related to the starting oxide content in the aluminium, magnesium and boron powders, see chapter 5. The peak indicated by U₁ has not been identified.

From this data TiB_2 is stable with respect to the formation of $AlMgB_{14}$. Additional phases are not produced as a result of any chemical reaction of TiB_2 with any of the starting components. Figure 6.3.4 c shows a SEM image of the microstructure of the composite obtained at a magnification of 5000 times in back scattered mode.



Figure 6.3.4 b: AlMgB₁₄ + 30 wt.% TiB₂ expanded 2 θ region. The experimental data are compared to a simulated scan from the JCPDS data



Figure 6.3.4 c: The microstructure of $AlMgB_{14} + 30$ wt.% TiB₂

The appearance of W_2B_5 particles arises from the reaction of WC with B to make W_2B_5 . WC is an impurity phase that is present due to the contamination from the milling vessel during the milling cycle.

6.3.3.2: AlMgB₁₄ + 20 wt.% TiN

TiN reacts with B to form TiB₂, table 6.3.3, and the addition of 20 wt.% TiN to a composition of Al, Mg and B (1:1:14, mole ratio) forms TiB₂ and AlMgB₁₄. Figure 6.3.5 a shows the diffractogram of AlMgB₁₄ + 20 wt.% TiN prepared at 1400°C. Figure 6.3.5 c is a picture of the microstructure of this composite taken at 3550 times magnification in back scattered mode.



Figure 6.3.5 a: Identified phases in $AIMgB_{14} + 20$ wt.% TiN

Due to the high electron density associated with TiB_2 the peaks associated with this phase dominate the diffractogram. The peak marked with the bold arrow in

figure 6.3.5 a belongs to AlN. The presence of AlMgB₁₄ was confirmed by an XRD trace performed over the range of 40-44° 2 θ . This scan confirmed that the broad peaks in this range of 2 θ is in fact AlMgB₁₄, figure 6.3.5 b. Furthermore, EDS analysis of the microstructure for the AlMgB₁₄ + TiN composite could not identify the nitride phase. A reaction scheme for the possible reactions is presented in equation 6.2.

$$2\text{TiN}_{(s)} + 4B_{(s)} \rightarrow \text{TiB}_{2(s)} + N_{2(g)}$$
 6.2

Additionally, TiB₂ could form from the following reaction scheme, equation 6.3,

$$TiN_{(s)} + AlMgB_{14(s)} \rightarrow TiB_{2(s)} + (Al,Mg)B_{2(s)} + AlN_{(s)} + N_{2(g)}$$
 6.3

Due to the formation of the solid solution $(Al,Mg)B_2$ it is not possible, with the evidence presented, to balance equation 6.3. In figure 6.3.5 a the diffraction peaks due to the presence of a solid solution $(Al,Mg)B_2$ would exist between the indicated pure AlB₂ and MgB₂. The peak marked with a dashed line has not been identified. Because of the reactivity of TiN with respect to B, the opportunity to produce the composite material AlMgB₁₄ + TiB₂ from the addition of TiN exists. Thus, this route can potentially produce the hard composite reported by the Ames group from different starting materials.



Figure 6.3.5 b: AlMgB₁₄ + 20 wt.% TiN expanded 2 θ region. The experimental data are compared to a simulated scan from the JCPDS data



Figure 6.3.5 c: The microstructure of $AIMgB_{14} + 20$ wt.% TiN

6.3.3.3: AlMgB₁₄ + 20 wt.% TiC

The addition of 20 wt.% TiC to a stoichiometric composition of $AlMgB_{14}$ produced a composite with a porosity of 57%. The relatively low temperatures and pressures at which the reaction was performed (1400°C, 20 MPa), as compared to the sintering temperature of TiC (1600°C) is one of the reasons why the porosity of the compact is so high.

As with the additions of TiN, TiC also forms TiB_2 in the presence of excess boron. Possible reaction routes are presented in reaction equation 6.4 and reaction equation 6.5.

$$2\text{TiC} + \text{AlMgB}_{14} \rightarrow 2\text{TiB}_2 + (\text{Al},\text{Mg})\text{B}_2 + 2\text{B}_4\text{C}$$
6.4

$$TiC + 6B \rightarrow TiB_2 + B_4C$$
 6.5

It is not clear from the XRD that B_4C is formed. EDS analysis did not indicate any B_4C either. Figure 6.3.6 a shows the diffractogram for the AlMgB₁₄-TiC composite.

The disappearance of the major peaks for stoichiometric TiC and the appearance of TiB_2 means that the formation of TiB_2 from TiC + B in the prepared composite is possible. The broad peak between 40-44° 20 was again identified to be $AlMgB_{14}$ based on the diffractogram measured only over this region and is shown in figure 6.3.6 b. The peak U₁, marked with a dashed arrow, has not been identified. Figure 6.3.6 c is a SEM image (back-scattered mode) of the microstructure of the $AlMgB_{14} + 20$ wt.% TiC composite.



Figure 6.3.6 a: Identified phases in AlMgB₁₄ + 20 wt.% TiC



Figure 6.3.6 b: AlMgB₁₄ + 20 wt.% TiC expanded 2 θ region. The experimental data are compared to a simulated scan from the JCPDS data



Figure 6.3.6 c: The microstructure of 20 wt.% TiC + AlMgB₁₄

The reaction of TiC with $AlMgB_{14}$ will produce $(Al,Mg)B_2$. The presence of this phase was not established convincingly from the XRD and EDS analysis due to the overlapping of the peaks of $(Al,Mg)B_2$ with the main peaks of the other phases.

6.3.3.4: AlMgB₁₄ + 20 wt.% TiCN

The addition of 20 wt% TiCN to stoichiometric AlMgB₁₄ produced a composite material in which the TiCN had reacted to form TiB₂. Figure 6.3.7 a shows the diffractogram obtained for this composite material and matched by the phases shown therein. The dashed line in figure 6.3.7 a indicates a peak in the diffractogram that as not been identified. The major peaks for the boride phase are not clearly revealed in this scan. More detailed XRD analysis of the major boride peak region (40-44° 20) did indicate the presence of AlMgB₁₄, as shown in figure 6.3.7 b. TiCN reacts with B to yield TiB₂. The presence of AlN was determined from XRD. The spinel phase (MgAl₂O₄) is also formed. The dashed line indicates an unidentified peak. Figure 6.3.7 c is a SEM micrograph of the microstructure of the composite AlMgB₁₄ + 20 wt.% TiCN.



Figure 6.3.7 a: Identified phases in $AIMgB_{14} + 20$ wt.% TiCN



Figure 6.3.7 b: Identified phases in $AIMgB_{14} + 20$ wt.% TiCN. The experimental data are compared to a simulated scan from the JCPDS data



Figure 6.3.7 c: The microstructure of AlMgB₁₄ + 20 wt.% TiCN

EDS analysis indicated C to be present in all of the phases indicated in the micrograph. The formation of titanium diboride can be explained from either of the following four reactions:

$$TiCN + 7B \rightarrow TiB_2 + B_4C + BN$$
 6.6

$$2\text{TiCN}_{(s)} + 12\text{B}_{(s)} \rightarrow 2\text{TiB}_{2(s)} + 2\text{B}_4\text{C}_{(s)} + \text{N}_{2(g)}$$
6.7

$$TiCN + AIMgB_{14} + B \rightarrow (AI,Mg)B_2 + TiB_2 + B_4C + AIN$$
6.8

$$2\text{TiCN}_{(s)} + \text{AlMgB}_{14(s)} \rightarrow (\text{Al}, \text{Mg})\text{B}_{2(s)} + 2\text{TiB}_{2(s)} + 2\text{B}_4\text{C}_{(s)} + \text{N}_{2(g)}$$
 6.9





Figure 6.3.8: Comparison of the diffractograms obtained for all the composites prepared from the Ti based additions to $AIMgB_{14}$

The following conclusions can be drawn from the above analyses. AlN is present in the composites prepared from TiN and TiCN. The presence of TiB_2 dominates the diffractograms obtained for all of the Ti based composites. Therefore, the preparation of the composite TiB_2 + AlMgB₁₄ is possible after addition of TiC or TiN or TiCN to AlMgB₁₄. Thus, the synthesis of the "super-hard" TiB_2 + AlMgB₁₄ composite, prepared by the Ames group, should be feasible from different routes starting from various Ti based starting materials.

6.3.3.5: AlMgB₁₄ + 20 wt.% WC

The addition of 20 wt.% WC to $AlMgB_{14}$ resulted in the reaction of the WC with boron to produce W_2B_5 . Figure 6.3.9 a shows the diffractogram obtained for the composite $AlMgB_{14} + 20$ wt% WC.



Figure 6.3.9 a: Identified phases in $AIMgB_{14} + 20$ wt.% WC

In addition to W_2B_5 , EDS analysis indicates the presence of carbon throughout the microstructure, which can be explained by the presence of the mounting epoxy that has set in the pores of the composite, figure 6.3.9 b. There may be some unreacted WC present but XRD assignment is difficult because of the overlapping of the peaks of WC and W_2B_5 . The formation of W_2B_5 is possible from reaction equations 6.10 and 6.11 respectively.

$$2WC + 13B \rightarrow W_2B_5 + 2B_4C$$

$$2WC + AIMgB_{14} + B \rightarrow (Al,Mg)B_2 + W_2B_5 + 2B_4C$$
6.11
6.10

Figure 6.3.9 b is a SEM micrograph of the microstructure of the 20 wt% WC + $AIMgB_{14}$ composite.



Figure 6.3.9 b: The microstructure of $AlMgB_{14} + 20$ wt.% WC

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