

## Chapter 7: Conclusions and Future Work

In this thesis the temperature dependence of the (Al,Mg)B<sub>2</sub> solid solution boundary has been established. The data obtained show that at temperatures greater than 1000°C, AlMgB<sub>14</sub> is in equilibrium with liquid Al, which can be used to facilitate the production of dense AlMgB<sub>14</sub>.

The existence of a solid solution between AlMgB<sub>14</sub> and MgB<sub>7</sub>, which is known to have the same structure, remains to be answered. The results presented in this thesis indicate that there is no significant change in the lattice parameter of AlMgB<sub>14</sub> in the investigated samples and hence the composition of AlMgB<sub>14</sub> appears to be constant. A more detailed investigation is necessary to shed light on this solid solution since it would be necessary for a deeper understanding of the relationship between the concentration of defects in AlMgB<sub>14</sub> and the synthesis conditions and the resulting influence on the properties.

The amount of spinel phase formed in the final compacts, can be reduced substantially during processing by milling in methanol. It must be stressed that this method only limits the amount of oxide in the starting boron powders and does not remove the oxide phases present in the Al and Mg starting powders. Furthermore, it has been shown that the amount of oxide phases in the starting Al and Mg powders is not increased by the method developed in this work. Although there is strong literature evidence concluding that boron esters are formed by the reaction of B<sub>2</sub>O<sub>3</sub> with organic alcohols<sup>84</sup>, it must still be shown that milling of B<sub>2</sub>O<sub>3</sub> in the various alcohols does in fact produce the esters.

It remains to be seen if the use of the di-alcohols, such as di-ethanol or di-isopropanol or di-propanol may further reduce the oxide content in the starting boron powders. The use of di-alcohols as solvents is less hazardous than the use of methanol and the resulting boron esters could be even more stable than the esters produced from the mono-alcohols.

Dense homogeneous  $\text{AlMgB}_{14}$  was produced from elemental powders and pre-reacted powders at  $1600^{\circ}\text{C}$ , 75 MPa and 1 hour soak time by uniaxial hot-pressing in an argon atmosphere. The initial pre-reacted powders were prepared in an uniaxial hot-press from elemental powders at  $1400^{\circ}\text{C}$ , 20 MPa and 1 hour soak time and then milled to a particle size of less than  $2\ \mu\text{m}$ .

Different microstructures resulted from elemental and pre-reacted powders prepared at  $1600^{\circ}\text{C}$ . The former microstructure indicated a preferential alignment perpendicular to the hot pressing direction whilst the latter microstructure is also more homogeneous indicated no preferential alignment. The density of both types of compacts is enhanced slightly by the presence of additional aluminium.

Addition of more than 3 wt.% Al results in the precipitation of metallic Al which is in agreement with the phase diagram that has been determined from this work. All of the materials produced were composites of  $\text{AlMgB}_{14}$ ,  $\text{MgAl}_2\text{O}_4$  and  $\text{W}_2\text{B}_5$ . Additionally,  $\text{TiB}_2$ ,  $\text{TiC}$ ,  $\text{TiN}$ , Si and additional WC were added to these composites and hot-pressed at  $1600^{\circ}\text{C}$ , 75 MPa and 1 hour soak time.

It was shown that  $\text{TiB}_2$ ,  $\text{W}_2\text{B}_5$  and  $(\text{Al,Mg})\text{B}_2$  are in equilibrium with  $\text{AlMgB}_{14}$ .  $\text{TiC}$ ,  $\text{TiN}$ , WC and Si are not stable with respect to  $\text{AlMgB}_{14}$  and a reaction takes place in which  $\text{TiB}_2$ ,  $\text{AlN}$ ,  $\text{W}_2\text{B}_5$  and  $\text{SiB}_x$  are formed. The identity of the  $\text{SiB}_x$  phase was not determined unequivocally in this study.

In samples containing added  $\text{TiB}_2$  and WC a solid solution phase  $(\text{Ti,W})\text{B}_2$  could be formed which has a core-rim structure. This core-rim structure is not evident when  $\text{TiC}$  or  $\text{TiN}$  is used as the precursors to  $\text{TiB}_2$  or when additional B is added to the  $\text{TiB}_2 + \text{WC} + \text{AlMgB}_{14}$  composites.

Composites prepared from Si,  $\text{TiB}_2$ ,  $\text{TiB}_2 + \text{WC}$ ,  $\text{TiC}$  additions increase the hardness of  $\text{AlMgB}_{14}$  which is in agreement with the literature. However, the  $H_{V10}$  values are lower than those reported by the Ames group but this most likely attributable to the increased load at which the hardness values have been determined in this study. The highest hardness was measured for the  $\text{TiB}_2 + \text{WC} + \text{B}$  additions to  $\text{AlMgB}_{14}$ .

Relatively high fracture toughness values of between 7-9.8 MPam<sup>1/2</sup> were found for all the prepared composites.

Microhardness values from the literature need to be confirmed in order to understand the effect of various loads on the measured hardness of the composites. The macrohardness measurements are not in the same range as those reported in the literature and this is most likely due to the load dependency of hardness.

The role of Si is uncertain and needs to be dealt with. Detailed TEM investigations of the grain-boundary phase and identification of minor secondary phases (e.g. B<sub>4</sub>C) which could not be detected by XRD or SEM needs to be addressed. Minor secondary phases can have a large influence on the properties of the composites.

The precise mechanism for the increased hardness as a result of additions to AlMgB<sub>14</sub> of TiB<sub>2</sub> and Si is unanswered. This must be addressed in any subsequent study. Furthermore such a study should involve the optimisation of the processing to properties relationship using well established statistical methods. To assess the potential applications of this material, extensive tribological testing is required.