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Sintering, microstructure and properties
of WC-FeAl-B and WC-Ni₃Al-B
composite materials

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Chapter 5 Discussion

This chapter discusses the results presented in Chapter Four which cover the topics of liquid phase sintering, phase stability in WC-FeAl-B and WC-Ni₃Al-B composites, microstructure of WC-FeAl-B, WC-Ni₃Al-B, and WC-Co composites and the effects of sintering temperature and boron on the microstructure. The hardness, fracture toughness and wear resistance of WC-40vol%(FeAl-B), WC-40vol%(Ni₃Al-B) and WC-Co composites and the effect of boron on these properties were also discussed.

5.1 Liquid Phase Sintering

5.1.1 Hot Pressing Conditions

A range of sintering temperatures, 1450-1550 °C (higher than the melting points of FeAl (1390 °C) and Ni₃Al (1420 °C)), was employed to study the optimum combinations of low porosity and WC grain growth. The results showed that with increasing temperature from 1450 °C to 1550 °C the porosity of the sintered materials decreased from ~5% to less than ~1 % while the densities of both WC-FeAl-B and WC-Ni₃Al-B composites sintered at 1500 °C and 1550 °C were near full and very close to each other but after sintering at 1450 °C density was significantly lower. The results also showed that grain size increased with increasing sintering temperature from 1500 °C to 1550 °C. The decrease in porosity and increase in WC grain size with increase in sintering temperature seem due to increases in the diffusion rate, wetting and solubility of WC in the liquid binder, the amount of liquid and decreases in the liquid viscosity [44, 58]. The reason why the porosity of the WC-Ni₃Al-B is less than the porosity of WC-FeAl-B at 1450 °C might be because of the lower melting point of Ni₃Al (1390 °C) compared to FeAl (1420 °C).

A pressure of 20 MPa was used during liquid phase sintering to produce specimens with near full density. Pressure is necessary during liquid phase sintering to produce intermetallic matrix composites with full density, because the solubility of WC in the aluminide binders and wettability of WC with aluminides are much lower than for Co

[4, 49, 59, 60]. For instance recently Mosbah [50] has shown that under uniaxial hot pressing of WC-FeAl composites can achieve almost full density, while the elimination of porosity could not be achieved without pressure in the processing of WC-FeAl containing a lower volume fraction of binder than 69%. Therefore in this study uniaxial hot pressing of WC composites was carried out at 1500 °C under a pressure of 20 MPa and an argon atmosphere for a short time (4 minutes) to achieve the lowest porosity and WC grain growth.

5.2.2 Densification Behavior

Significant densification of the intermetallic matrix composites occurred on heating at temperatures higher than ~1200 °C but below the melting points of the binders (~1400 °C). This could be associated with the densification of the composites before the formation of liquid by the solid state sintering process. This behavior also have been reported in conventional WC-Co [58], WC-17% Ni₃Al, WC-68% Ni₃Al and TiC-17% Ni₃Al [9] and WC-FeAl composites[50].

At sintering temperatures higher than the melting points of the aluminide binders, both FeAl or Ni₃Al start to melt and they wet the WC particles so porosity is eliminated by capillary action and the final stage of densification results. It should be noted that although the wetting of aluminides with WC is lower than with cobalt [37], it has been established that thermodynamically and experimentally WC has wettability with FeAl and Ni₃Al at 1500 °C [11, 37, 46, 47, 146]. Interestingly in this study it was found that boron could facilitate densification of WC-FeAl and WC-Ni₃Al composites during liquid phase sintering by increasing the WC solubility in the binders and decreasing the melting points of the aluminide binders. The XRD results showed that the grains of aluminide binders are larger than the precursor powder grain size which could be due to melting, re-crystallization and grain growth of the aluminides during sintering.

5.2 Phase Stability in WC-FeAl-B and WC-Ni₃Al-B

5.2.1 Effect of Sintering Temperature

X-ray diffraction results of the hot pressed WC-40vol%(FeAl-0.05wt%B) and WC-40vol%(Ni₃Al-0.05wt%B) composites at different sintering temperatures ranging from 1450 °C to 1550 °C showed no evidence of new phases developed during sintering within the limits of XRD detectability. It seems that WC is thermodynamically stable with FeAl-B and Ni₃Al-B binder alloys at the above mentioned temperature range and under the heat treatment conditions employed. If thermodynamic stability is not guaranteed, reaction products at the intermetallic/ceramic interface may adversely influence interfacial bonding and this may limit the acceptable times and temperatures during service [47]. These results are consistent with the findings of other researches on WC-FeAl and WC-Ni₃Al composites at temperatures under 1600 °C [37, 46, 50].

5.2.2 Effect of Boron on the Phase Stability

Based on the XRD and TEM electron diffraction results, WC-40vol%(FeAl-B) and WC-40vol%(Ni₃Al-B) composites with boron up to 0.1 wt% did not show new phases at 1500 °C. The reason could be that the present amount of boron is lower than the maximum solubility limit of boron in the FeAl and Ni₃Al alloys. The maximum solubility limits of boron in FeAl and Ni₃Al are approximately 0.2 and 0.35 wt%, respectively which if the boron concentration be higher than those limits new boride phases will be generated [125, 126].

5.3 Solubility of W in WC-FeAl-B and WC-Ni₃Al-B Systems

The semi-quantitative EDS investigations results showed that tungsten can be dissolved in the aluminide binders at 1500 °C. The estimated amounts of the dissolved W in FeAl and Ni₃Al are ~0.7 at% and ~1.2 at%, respectively. Generally the measured tungsten concentration in the binder can be due to the combination of dissolved tungsten and redeposited of milled tungsten [34, 175]. However, it is believed that most of the estimated tungsten concentration in the binders was dissolved because in the present research the TEM specimens were thoroughly cleaned (polished) as a result of using

low ion milling energies during the final stages of ion milling at a very low angle. Because conventional argon ion milling at low energies with very low incidence angles is effective in removing damage layers and removing redeposited material [175]. The obtained results are very similar with the thermodynamic calculations [34] and experimental findings by other researchers [9, 81, 147, 176]. For instance, a study by Draper [81] showed that the FeAl matrix of WC-FeAl, TiC-FeAl and TiB₂-FeAl composites contains less than 1 at% W or Ti, respectively. Moreover, Panov et al. [147] found that there is 2 at% W in the Ni₃Al binder of WC-Ni₃Al sintered at 1500 °C.

Increasing amounts of boron in the aluminide binders up to 0.1wt% resulted in increase the amount of dissolved W in both FeAl and Ni₃Al up to 2.7 and 3.5 at%, respectively, which could be resulted in enhancement of densification during liquid phase sintering [58, 145]. The reason of this behavior of boron might be explained by the effect of boron on the melting point of the aluminide binders. It is established that the melting point of FeAl-B and Ni₃Al-B decreases with increasing amounts of boron [177, 178]. Thus the W solubility in the aluminide binders increased with increasing boron could be due to decreasing the melting points of the aluminides and so allow more time for tungsten to dissolve in the liquid phase. The results also showed that the tungsten content in the Ni₃Al binder is slightly higher than in the FeAl binder for a given level of boron. This is possibly due to the lower melting point of Ni₃Al than FeAl alloy.

Since the solubility of WC in FeAl and Ni₃Al is very low, the beneficial effect of boron on increasing the W solubility in the aluminide binders is very important, as it could result in improvements to the thermodynamic compatibility and densification of the intermetallic matrix composites during liquid phase sintering process.

5.4 Microstructure of WC-FeAl-B, WC-Ni₃Al-B and WC-Co

Scanning electron microscopy investigations showed that WC particles are generally embedded in a continuous Co, FeAl, or Ni₃Al matrix. It was found that sintering temperature and boron level affect the microstructure of the intermetallic matrix composites.

5.4.1 Effect of Sintering Temperature on the Microstructure of WC-FeAl-B and WC-Ni₃Al-B

The results showed that the WC grain sizes in both intermetallic matrix composites at 1450 °C and 1500 °C are very similar, but slightly increased at 1550 °C. Very low WC grain growth could be due to the combinations of significant low WC solubility in the aluminide binders and short sintering time (4 minutes). Because in liquid phase sintering of WC composites, the grain growth of WC occurs by dissolution of small WC particles in the binder and reprecipitate on the large particles which rapid rates of grain growth can occur in composites with high solubility of solid in the liquid [44, 58].

Increasing sintering temperature resulted in increase the faceting of WC particles in intermetallic matrix composites. This might be due to increasing WC solubility in the aluminide binders with increasing sintering temperature, because with increasing WC solubility in the binder, rounded WC particles changes to a faceted shape [11]. It thus appears that uniaxial hot pressing of the boron-doped aluminide matrix composites at 1500 °C for 4 minutes produces composites without significant WC grain growth and with faceted WC morphology.

5.4.2 Effect of Boron on the Microstructure of WC-FeAl-B and WC-Ni₃Al-B

The results showed that the WC faceting in intermetallic matrix composites without boron is lower than that of with boron. This is due to very low WC solubility of WC in the binders [44]. For instance, Tumanov et al. [46] reported that the absence of a high faceting of the carbide grains in WC-Ni₃Al may be the result of the significantly lower solubility of tungsten in the Ni₃Al intermetallic in comparison with cobalt. Increasing amounts of boron in the aluminide binders resulted in increase the WC faceting and also decrease the WC contiguity in both WC-FeAl-B and WC-Ni₃Al-B composites could be due to increasing the WC solubility in the aluminide binders. It is suggested that with increasing solubility of WC in the binders, the WC faceting and also anisotropic surface energy could increase [11, 46, 179, 180].

XRD results showed that with addition of boron to the aluminide binders the grain size of the aluminide binders decreased which could result in improvement the ductility and strength of the matrix and also that of the composite [104]. This was expected because it is known that boron addition to FeAl and Ni₃Al decreases the grain size and increases the ductility and strength of aluminides [78, 104, 110].

5.4.3 Comparison of the Microstructure of WC-Co and Intermetallic Matrix Composites

The results of SEM investigations showed that the degree of WC faceting in intermetallic matrix composites without boron is lower than that of WC-Co composites which is consistent with the findings by other researches [72, 180]. This behavior is due to the significant lower of WC solubility in the aluminide binders than that of Co. Boron addition to intermetallic matrix composites resulted in increasing the WC faceting similar to that of WC-Co. This similarity increased with increasing the amount of boron due to increasing the WC solubility in the binders.

The average WC grain size in WC-FeAl-B and WC-Ni₃Al-B composites appears smaller than that in WC-Co composite. This could be due to the much higher WC solubility in cobalt binder than in aluminide binders, which could lead to much higher reprecipitation of WC in Co binder and could result in higher WC grain growth in WC-Co.

5.4.4 The Role of WC/Aluminide Interface Characteristics on the Properties of WC Composites

Transmission electron microscopy of the WC-FeAl-B and WC-Ni₃Al-B composites did not reveal any voids or precipitates at the interfaces and the binders were continuous and filled all the necks and narrow parts. In conditions of good wetting, the liquid phase is pulled by capillary forces into particle necks and small pores [44, 64]. The presence of a continuous intermetallic phase around all the WC particles in the WC/intermetallic composites leads to a low WC contiguity, which is comparable with those of WC-Co composites. Based on the results of SEM and TEM observations, it thus appears that the

FeAl-B and Ni₃Al-B binders sufficiently wet the WC particles during the hot pressing process at 1500 °C and that WC and Ni₃Al-B or FeAl-B are well bonded together, with the aluminide binders forming thin ligaments between the WC particles. It seems that the WC wetting with FeAl or Ni₃Al binders at 1500 °C increases with increasing amounts of boron in the aluminide binders. Because of WC faceting and WC solubility in the aluminide binders increases and melting point of aluminide binders decreases with increasing the amount of boron in the aluminide binders. It is also established that to have the best combination of hardness and fracture toughness, the matrix-grain interface should be free from precipitates, impurities, pores, and reaction products [8, 44]. Therefore, it is reasonable that the WC composites based on boron doped aluminide binders exhibit high performance in mechanical properties.

5.5 Hardness of WC-FeAl-B, WC-Ni₃Al-B and WC-Co

Results showed that the hardness of WC composites depended on the sintering temperature and the type of binder.

5.5.1 Effect of Sintering Temperature on the Hardness of Intermetallic Matrix Composites

Increasing the sintering temperature from 1450 °C to 1550 °C resulted in an increase in the hardness of both WC-40vol%(FeAl-0.05wt% B) and WC-40vol%(Ni₃Al-0.05wt% B) composites. Also results showed that with increasing the sintering temperature porosity decreased which this could be the reason of increasing hardness. Because it established that increasing the porosity results in a decrease in the hardness of WC hardmetals [4, 58]. Furthermore, the results showed that the hardness of both the WC-FeAl-B and WC-Ni₃Al-B composites sintered at 1450 °C is much lower than in those sintered at temperatures of 1500 and 1550 °C. This behavior is consistent with the porosity variation of WC/aluminide hardmetals at those temperatures.

5.5.2 Effect of Boron on the Hardness of Aluminides and Intermetallic Matrix Composites

Boron additions up to 1000 wppm resulted in slightly decrease in the hardness of FeAl-B but no significant effect on the hardness of Ni₃Al. It has been found that boron has no significant effect on the hardness of FeAl and Ni₃Al alloys [51, 55]. For instance Skoglund [181] found that the hardness of FeAl-100 wppm B (315 HV10) is slightly less than that of FeAl (320 HV10).

The results showed that the hardness of both FeAl-B and Ni₃Al-B alloys is higher than that of Co. In hardmetals, having a binder with a hardness as high as possible is very important [7] which leads to increase the hardness of hardmetals [58]. It is also expected that hardnesses of FeAl-B, Ni₃Al-B and Co as the binder would be higher than those of bulk alloys due to solid solution strengthening with W and/or C. Engqvist and Wiklund [182] measured the hardness of Co binder phase in WC-Co composite using a nano-indenter which was found that the measured hardness of Co in the form of a binder phase in WC-Co composite is higher than the bulk hardness due to the solid solution of W and C in the binder.

The results also showed that the hardness of WC-FeAl-B is higher than that of WC-Ni₃Al-B at boron contents up to 0.1wt%. This is could be due to higher hardness of FeAl-B binder than Ni₃Al-B, because the matrix properties control the overall properties of composite [51, 58]. Moreover, the hardness of the WC-FeAl-B slightly decreased with increasing amounts of boron, but boron had no significant effect on the hardness of the WC-Ni₃Al-B composites. This behavior is consistent with the effect of boron on the hardness of FeAl-B and Ni₃Al-B, since increasing amounts of boron had no significant effect the hardness of FeAl-B and Ni₃Al-B alloys.

The hardness of WC-40vol%(FeAl-B) is higher than the hardness of WC-40vol%(Ni₃Al-B) and WC-40vol%Co, while it is comparable with the hardness of the commercial WC-10wt%Co (H10F). This is could be due to the higher hardness of FeAl-B than Ni₃Al followed by Co. Furthermore, the hardness of WC-40vol%(Ni₃Al-B) is higher than that of WC-40vol%Co, due to the higher hardness of Ni₃Al-B than Co.

5.6 Fracture Toughness of WC-FeAl-B, WC-Ni₃Al-B and WC-Co

5.6.1 Effect of Boron on the Fracture Toughness of WC-FeAl-B and WC-FeAl-B

Fracture toughness of both WC-40vol%(FeAl-B) and WC-40vol%(Ni₃Al-B) composites increased with increasing amounts of boron in the binders up to 0.1 wt%. It is more pronounced with increasing the amount of boron up to 0.05 wt%. The indentation fracture toughness value of the WC-40vol%FeAl composite is about 5.6 MPa.m^{1/2} which is consistent with the findings of Mosbah (~6 Mpa.m^{1/2}) [50]. The improvement of the fracture toughness of the WC-aluminide composites by boron addition could be explained as follows:

I) Elements such as boron and carbon act as very powerful interstitial solid solution strengtheners in iron and nickel aluminides [41]. It also has been established that microalloying of FeAl and Ni₃Al aluminides with boron leads to significant improvement of ductility [51, 55], due to improvement of the grain-boundary cohesion and strength [37, 42, 60, 78, 104, 110, 127] and also improvement of environmental embrittlement [37, 129, 130]. Also the XRD results in this study showed that microalloying of iron and nickel aluminides with boron resulted in decreased grain size which it improves the strength and fracture toughness of these alloys [51, 78, 109]. As the study of the effects of boron on the intermetallic matrix composites is very new, a very limited number of preliminary studies have been performed. Interesting researches by Inoue et al. [51, 52] showed that the ductility and toughness of Fe-40 at.% Al and Ni₃Al matrix composites including 5-10 vol.% TiC, TiB₂ and β -SiC particles were improved by B doping of the binders. Moreover, Plucknett et al. [12] also found that using a suitable B addition helps to minimize the environmental embrittlement of the TiC-Ni₃Al composites.

II) The TEM-EDS investigation results showed that with increasing amounts of boron in the WC-FeAl-B and WC-Ni₃Al-B composites the WC solubility in the aluminide binders increased. Moreover, TEM observation showed that the WC-aluminide interfaces are free from precipitates, impurities, pores and reaction products, which is a

necessity for high mechanical properties and resistance to fracture [8]. Since increasing the WC solubility in the binder could result in improvement strength of WC/matrix interfaces [1, 44, 58, 61], it is believed that the other reason for the beneficial effect of boron on the fracture toughness of WC-FeAl-B and WC-Ni₃Al-B composites is the higher WC solubility in the binder without any precipitates or reaction products.

III) The SEM observation results showed that with increasing amounts of boron in the FeAl-B and Ni₃Al-B binders the contiguity of WC decreased which could be led to improved strength and fracture toughness of composites [8, 56, 58]. It appears that the improvement of the microstructure of the WC-aluminide composites with boron addition is another reason that explains the increasing toughness.

Thus it seems that fracture toughness of WC-FeAl-B and WC-Ni₃Al-B composites increased with increasing the amount of boron due to combinations of increasing the strength, toughness and ductility of aluminide binders, increasing WC solubility in the binders, decreasing of WC contiguity and aluminide grain size.

5.6.2 Comparison of Fracture Toughness of WC-FeAl-B, WC-FeAl-B and WC-Co

The results showed that the fracture toughness of WC-40vol%(Ni₃Al-B) is lower than for WC-40vol%(FeAl-B) at any given boron value. This could be attributed to the lower hardness of WC-Ni₃Al-B than that of WC-FeAl-B. This behavior is similar to conventional WC-Co composites which indentation fracture toughness decreases with increasing hardness [72]. According to the obtained results the fracture toughness of WC-40vol%Co boron is slightly lower than that of WC-40vol%Ni₃Al without boron but higher than that of WC-FeAl without boron, which could be due to the very low WC solubility in the aluminide binders and the low ductility and toughness of FeAl and Ni₃Al without boron compared with Co[51, 55]. These results are consistent with the findings of other researchers. For instance, Tiegs et al. [9] also found that the fracture toughness values for WC-17vol%Ni₃Al composite sintered at 1450 °C is comparable to that of WC-17vol%Co. Furthermore, recently it was reported that the fracture toughness of WC-FeAl is lower than that of WC-Co composite at the same binder volume fraction

[11, 50]. Addition of boron in WC-FeAl and WC-Ni₃Al resulted in increasing the fracture toughness higher or comparable to that of WC-Co due to the reasons explained in the previous section.

5.7 Wear Behavior of WC-FeAl-B, WC-Ni₃Al-B and WC-Co

5.7.1 Effect of Boron on the Wear Resistance of WC-FeAl-B and WC-Ni₃Al-B

The results showed that the abrasion wear resistance of WC-40vol%(FeAl-B) is higher than that of WC-40vol%(Ni₃Al-B) at any given boron content up to 0.1wt%. This could be as a result of higher hardness of WC-FeAl-B than WC-Ni₃Al-B in which increasing the hardness can result in an increase in the abrasion wear resistance of the WC composites [158].

The abrasive wear resistance of WC-40vol%(Ni₃Al-B) and WC-40vol%(FeAl-B) increased with increase in the level of boron. This behavior was found to be associated with the effect of boron on the fracture toughness of the composites. Results showed that with increasing boron level in aluminide matrix composites, fracture toughness significantly increased but had no significant effect on the hardness. Normally, hardness is a good first indication of a material's resistance to pure abrasion but when the wear mode changes to impact or gouging mode, toughness of the material controls the wear rate due to the fracture of carbides [165, 183]. The wear mode in the pin-on-drum test seems a combination of abrasive, sliding and impact wear due to using harder and coarser SiC abrasive paper (nominal particle size of 105 μm and hardness 24.0 GPa) compared to the WC particle size (0.7 μm). Thus it seems that the increasing the wear resistance of WC-Ni₃Al-B and WC-FeAl-B with increasing boron level is due to increasing the fracture toughness.

Interestingly, the wear and fracture toughness results confirmed the earlier conclusions which indicated that the beneficial effect of boron in WC-FeAl and WC-Ni₃Al composites on the microstructure could be resulted in improvement of mechanical properties, such as fracture toughness and wear resistance.

5.7.2 Comparison of Wear Resistance of WC-FeAl-B and WC-Ni₃Al-B with WC-Co

WC-40vol%Co showed the lowest wear resistance compared with WC-40vol%(Ni₃Al-0.05wt%B) and WC-40vol%(FeAl-0.05wt%B). This could be because of the higher hardnesses of FeAl-B and Ni₃Al-B binders compared with that of Co binder and also presence of boron. It should be noted that although the hardness of FeAl and Ni₃Al are higher than Co but the wear resistance of WC-40vol%Ni₃Al and WC-40vol%FeAl without boron are lower and similar to that of WC-40vol%Co, respectively. This indicates that to improve of the wear resistance of WC-Co composites, replacing of Co by the harder binder is not enough but adequate solubility of WC in the new binder is also required. The obtained results confirmed this conclusion which boron addition resulted in increase the solubility of WC in the aluminide binders and consequently wear resistance of boron doped intermetallic matrix composites increased.

The wear resistance of the commercial WC-10wt%Co composite (H10F) was found higher than that of WC-40vol%Co. This is caused by the higher hardness of H10F than WC-40vol%Co which as the Co content in WC-Co cermets decreases, the hardness and abrasion resistance increases [5, 158, 160, 161].

5.7.3 Morphologies of Worn Surfaces of WC-FeAl-B, WC-Ni₃Al-B and WC-Co

The SEM investigations showed that materials seem to be removed from the worn surfaces through ductile processes including binder extrusion, microploughing and microcutting, and brittle processes including pull-out of different phases and cracking of the carbide particles, which have been also found in WC-Co composites by other researchers [35, 153, 155, 158, 159, 161, 183-185]. The roughness of the worn surfaces of WC-FeAl-B composites at any given boron level is higher than those of WC-Ni₃Al-B composites. This is could be consequence of the lower ductility of FeAl-B binder than that of Ni₃Al-B. The morphologies of worn surfaces of WC-FeAl-B and WC-Ni₃Al-B composites changed with increasing amounts of boron in the binders. As the amount of boron in the aluminide binders increased, the removal of material from worn surfaces by

ductile processes such as binder extrusion and microploughing increased and the roughness of the grooves decreased. This could be due to the improvement of the fracture toughness of WC-FeAl-B and WC-Ni₃Al-B composites and also the ductility and strength of the binders when boron is present. Boron addition to FeAl and Ni₃Al alloys leads to significant improvement in their ductility and toughness [37, 51, 55, 78]. Also, the stronger interfacial bonding between the constituent phases due to higher WC solubility in the binder and high fracture toughness will prevent preferential pull-out (plucking) of one of the phases during abrasion [161]. Thus it seems another reason for the increasing abrasive wear resistance of WC-FeAl-B and WC-Ni₃Al-B composites with increasing boron content might be because of decreasing of the removal of material by brittle processes.

The morphology of the worn surface of WC-40vol%(FeAl-1000 wppm B) and WC-40vol%(Ni₃Al-1000 wppm B) composites is very similar to that of WC-40vol%Co and this similarity is more pronounced between WC-40vol%(Ni₃Al-1000 wppm B) and WC-40vol%Co composites. This is could be a result of high ductility of all the binders including FeAl-B, Ni₃Al-B and Co.

It appears that the combination of ductile processes such as binder extrusion and brittle processes such as pull-out of the phases is responsible for the removal of material from the worn surfaces of the WC-40vol%FeAl and WC-40vol%Ni₃Al composites and that increasing the amount of boron results in a decrease in the removal of material by brittle processes.