



Chapter 42

Analysis of Interfaces in AA7075/ Recycled WC Particles Composites Produced via Liquid Route

Marina Ferraz Viana and Maria Helena Robert

Abstract Casting of composites is a simple and low cost route to produce composites, which allows numerous possibilities in terms of reinforcement location in the cast part and high geometry flexibility of the products. However, interfaces between reinforcement and metal depend on the behaviour of the particles in liquid metal during processing. The work investigates the effects of parameters such as temperature and time of contact between WC particles and liquid aluminium, and reinforcement content, in the production of cast AA7075/WC composites. WC particles coming from recycled cutting tools are used as reinforcement for aluminium alloys AA 7075, widely used in aeronautical domain. Reinforcement contents of 15 and 30 wt %, temperatures of 700, 740 and 780C, and contact time of 1, 2, 3 and 4 h were the values of the investigated variables. Microstructure of cast composites produced under these conditions were analysed by OM, SEM and EDS microanalysis. A reaction layer was found in the interface between metal and WC, with composition and micro constituents depending on the studied parameters. A mechanism of formation of the reaction layer is proposed.

Keywords Composites · Aluminium · WC particles · Casting · Interface

42.1 Introduction

Casting of composites is one simple and low cost way of producing metal-matrix composites reinforced with ceramic particles. Such processes are not only flexible in terms of part geometry, reinforcement type and location in the product, but also favour adhesion between matrix and reinforcement, when compared to solid state processes. Generally, it is possible to incorporate up to 30% of particles by those methods. Depending on the nature of matrix and reinforcement materials, chemical reactions may occur while the reinforcement is in contact with the liquid metal – this is favoured by high temperatures and long contact times, and may be desirable when it leads to the formation of in situ reinforcement phases or chemical bonding between the matrix and the reinforcement [1–6].

The interface between matrix and reinforcement is a function of the nature of both materials and the behaviour of particles during processing [1, 2, 7]. In the specific case of particle reinforced composites produced by casting techniques, the ability of the liquid metal to wet the surface of the reinforcement and the chemical affinity between both materials are key in their integration [1, 7, 8]. Generally, ceramic phases whose chemical bonds have a strong metallic character, as WC and the TiC are more easily wet by the liquid metal [7, 8].

Poor adhesion between reinforcing particles and matrix hinders interface quality and is a common issue in the production of such composites. Low wettability and lack of chemical affinity are generally blamed as the root causes of such problems [1, 4, 8–10]. On the other hand, it has been observed that cemented carbides, such as the ones used for producing cutting tools, are not only exceptionally hard and wear resistant [11, 12] but also have great chemical affinity with aluminium [8, 13, 14]. Thus, those are an interesting reinforcement material for wear resistant aluminium composites.

Therefore, the aim of this work is to investigate the effects of parameters such as temperature, time of contact between liquid matrix and reinforcement and amount of reinforcement in the casting of aluminium matrix WC composites.

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42.2 Experimental Procedures

AA 7075 alloy (90.79% Al; 5.12% Zn; 2.19% Mg; 1.50% Cu; 0.22% Cr; 0.07% Si; 0.07% Fe; 0.02% Ti; 0.01% Mn) was used as matrix.

Cemented carbide granules, issued from recycled machining inserts, were used as reinforcement material. The granules, shown in Fig. 42.1a, are irregular and polygonal, and their dimensions range from 300 to 800 μm . Some of their surfaces have distinct colorations, which might be attributed to vestiges of coating materials. Fig. 42.1b shows the surface of one granule with greater magnification. It is possible to see the granules are comprised of WC microparticles whose dimensions range from 0.5 to 3 μm . The chemical composition of the granules is presented in Table 42.1.

The microstructure of one representative granule is shown in Fig. 42.2. As recycled material was used, there may be some variation between granules. Figure 42.2a shows three points where EDS micro analysis were effectuated; the numerical results of the analyses are presented in Table 42.2.

The microanalyses of points 2 and 3 show a high Co content in regions between the WC particles. Co is commonly used as a binder in the sintering of cemented carbides [11, 12]. Fe was also detected in those regions; its presence is probably due to contamination during the machining of ferrous materials. Machining inserts may contain different types of carbides in their composition, such as WC, TiC, TaC, NbC and CrC [11, 12]. Besides, they might be coated with TiCN, TiN, Al_2O_3 , etc [11–13]. Indeed, there is a high content of Ti and Ta at Point 1. The high percentages of WC detected in all points refer to the neighbouring regions of the analyses points.

To produce the composites, the AA 7075 alloy was first melted in an electric furnace, in a ceramic crucible, and heated to a predetermined temperature, hereinafter called contact temperature. When the melt reached the contact temperature, it was removed from the oven, its surface was cleaned and a predetermined quantity of granules was added to it under manual agitation. Then the melt was returned to the oven at the contact temperature and kept there for 15 min., after which it was taken out again and agitated for 40 s. This operation was repeated until the contact time was completed; next, the melt was cooled down in air, resulting in a conic part.

Two series of composites were produced. The first one was fabricated as described above, as for the second one, a last was added to the procedure, which consisted of remelting the conic part in a cylindrical steel die coated with alumina, at 740 $^\circ\text{C}$ for 45 min. The experimental conditions which were investigated are shown in Table 42.3.

All composites were sectioned, and prepared for metallographic analysis using polycrystalline 90, 45, 25, 9, 6, 3, 1, and 0.25 μm diamond abrasives and water as lubricant; optical microscopy, SEM and EDS microanalysis were used to analyse microstructures. Quantitative analysis of the area occupied by the carbide granules in the composites was performed in the second series, by analyzing OM images taken with a 50 \times magnification through free image analysis software.

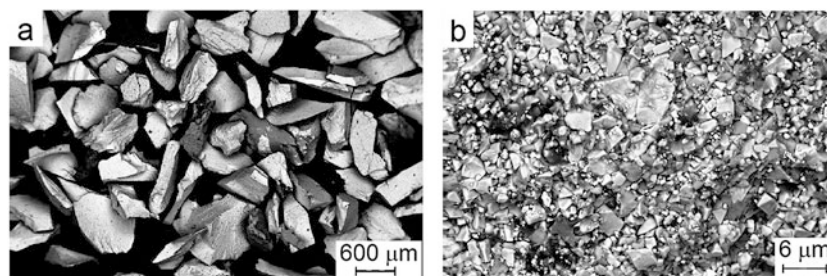


Fig. 42.1 (a) General aspect of the carbide granules used as reinforcement; (b) magnified surface of one granule (SEM)

Table 42.1 Elemental composition of the cemented carbide granules used in the experiments (% wt)

W	Co	Ni	Ti	Ta	Fe	Zn	V	Cr	Ca	Cu	Al	Mn	P	Mo	Nb	Ag	Si
65.30	5.72	0.28	1.51	0.63	10.27	8.28	4.74	0.62	0.50	0.33	0.32	0.32	0.52	0.21	0.16	0.14	0.09

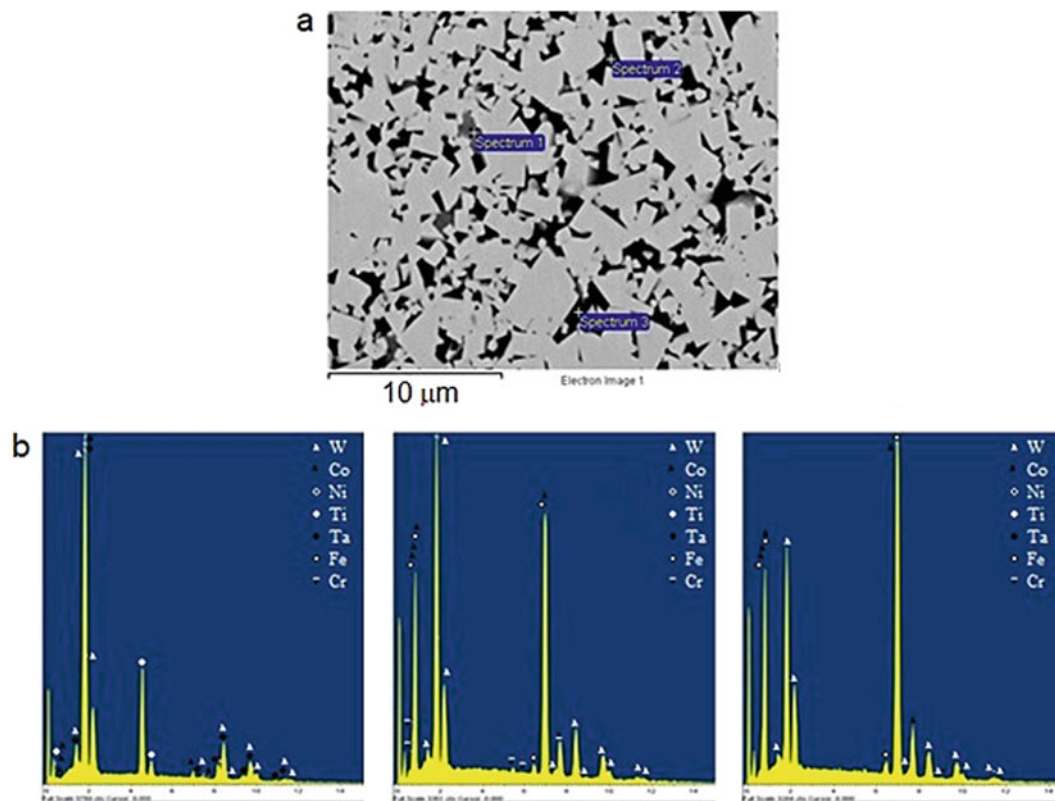


Fig. 42.2 (a) Microstructure of a representative granule with indication of EDS micro analysis points; (b) results from EDS microanalyses of points 1, 2 and 3, respectively (SEM)

Table 42.2 Average compositions of the regions indicated by Spectra 1, 2 and 3 in Fig. 42.2 obtained by EDS analyses (wt. %)

	W	Co	Ni	Ti	Ta	Fe
Spectrum 1	60.42	1.89	–	20.13	17.56	–
Spectrum 2	31.82	66.20	–	–	–	1.97
Spectrum 3	45.40	52.50	–	–	–	1.57

Table 42.3 Experimental conditions investigated in the production of AA 7075 reinforced with cemented carbide granules

	Reinforcement fraction (wt. %)	Contact time (h)	Contact temperature (°C)
First series	30	1	700
	30	2	700
	30	4	780
Second series	30	1	740
	30	3	740
	30	3	780
	15	1	740
	15	3	740
	15	3	780

42.3 Results and Discussions

42.3.1 Microstructure of the Composites.

Typical microstructures of composites produced in the first series of experiments, with the operational conditions described in Table 42.3, are shown in Fig. 42.3. These first composites were produced as a preliminary study of the behaviour of the WC particles in liquid aluminium, in order to allow of an informed choice on the time and temperatures of contact to be

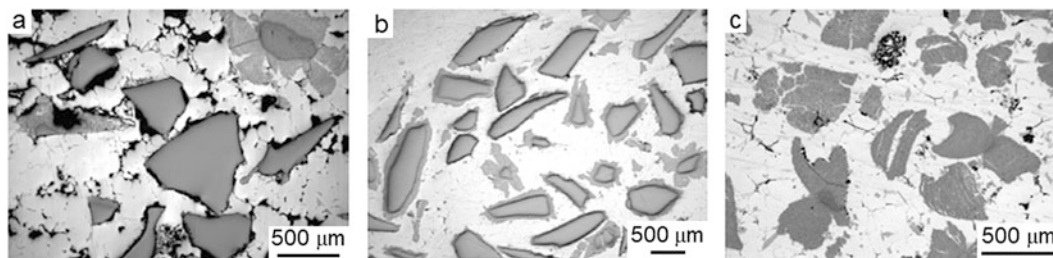


Fig. 42.3 Typical microstructures of composites produced in the first series of experiments, with contact times and temperatures of, respectively: (a) 1 h / 700 °C; (b) 2 h / 700 °C; and (c) 4 h / 780 °C (OM)

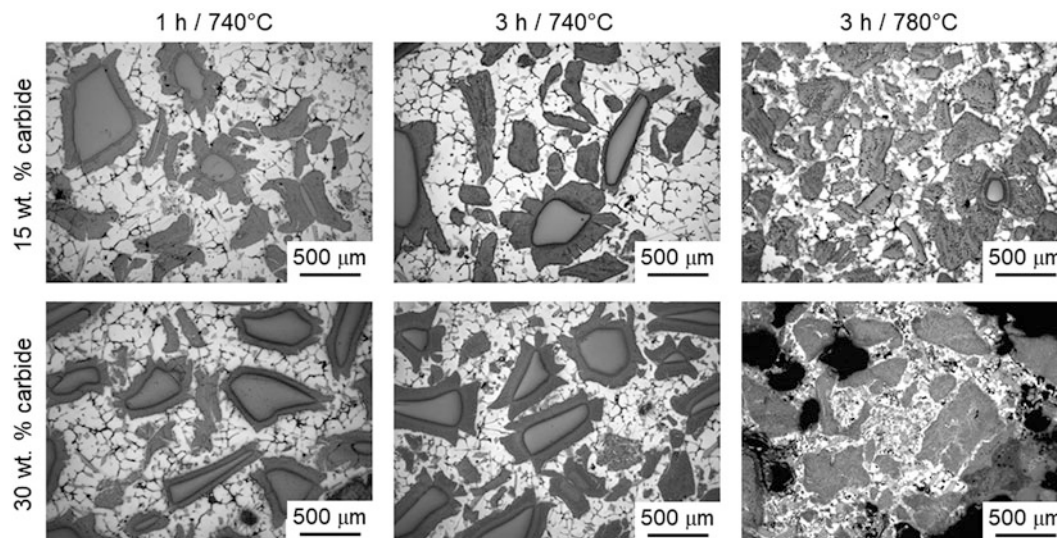


Fig. 42.4 Typical microstructures of the composites produced in the second series of experiments. The contact time (h), contact temperature (°C) and reinforcement fraction (% wt.) are indicated under each image (OM)

studied. Therefore, extreme conditions were chosen: temperatures of 700 and 780 °C, times between 1 and 4 h of contact, and 30%wt. reinforcement. At this scale, an even distribution of the reinforcement particles is observed. For the lowest temperature and contact time, voids are associated to the matrix/metal interface rather than any transition layers, as shown in Fig. 42.3a. When the contact time is increased in 1 h, the formation of a transition layer rather than voids are observed at the interfaces, as seen in Fig. 42.3b. Finally, for the maximum contact time and temperature tested (4 h and 780 °C), the interfaces between reinforcement and matrix become diffuse as the granules change morphology. Therefore, the conditions chosen for the second series of experiments were considered both intermediary in comparison to those tested in the first series, and propitious to the formation of a transition layer between reinforcement and matrix.

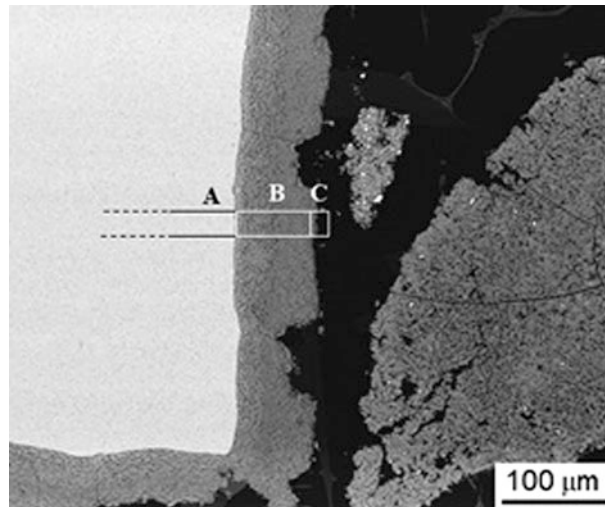
Typical microstructures of composites produced in the second series of experiments, with the operational conditions described in Table 42.3, are shown in Fig. 42.4. At this scale, an even distribution of the reinforcement particles and sound matrix/reinforcement interfaces were observed.

For the composites produced with the contact temperature of 740 °C, no significant difference with respect to the aspect of the carbide granules or the spacing between them was observed. In all composites, the presence of a reaction layer around the granules was observed. Some granules exhibited the same aspect of the reaction layer all over their surface, which suggests that the formation of the reaction layer might have consumed the granules. This last case is the usual situation in the composites produced with the contact time of 780 °C.

The estimated percent area fractions occupied by non-reacted granules, reinforcement layer and matrix are presented in Table 42.4. They were estimated by analysing OM images with the software Image J. These results give a better idea of how the reinforcement fraction and the contact time affect the formation of the reaction layer. For both reinforcement fractions, increasing the contact time from 1 to 3 h decreased the percent area occupied by the granules and increased the area fraction occupied by the reinforcement layer in such a way that the percent area occupied by the matrix remains constant. Thus, the formation of the reaction layer is favoured by the contact time. As the reinforcement content was increased from 15 to 30%

Table 42.4 Area fractions (%) occupied by reinforcement granules, reaction layer and matrix in composites produced with the contact temperature of 740 °C

% wt. reinforcement	Contact time (h)	Granules	Reaction layer	Granules + Reaction layer	Matrix
15	1	7	31	38	62
15	3	5	34	39	57
30	1	14	29	43	57
30	3	9	34	43	57

**Fig. 42.5** Microstructure of composite produced with 1 h contact time and 740 °C contact temperature – regions A, B and C around the reaction layer are indicated (SEM)

wt., the percent area occupied by the non-reacted granules increased, but the area fraction occupied by the reaction layers remained virtually the same – that might indicate that the formation of the reaction layer is not strongly dependent on the weight fraction of reinforcement.

42.3.2 SEM and EDS Microanalyses of the Composites

Figure 42.5 shows a representative image of the microstructure observed in the composites fabricated with 1 h contact times and 740 °C contact temperatures. To facilitate the discussion, it was decided to label the three regions around reaction layer as follows: region A is the apparently unreacted carbide granule; region B is the reaction layer around the granules; and region C is the matrix close to the reaction layer.

Region A was analysed by SEM and EDS. The results were very similar to the ones presented in Fig. 42.2 and Table 42.2, and were thus omitted in this paper. However, we must say the portion of the original granules comprised by region A was little affected by the contact with the liquid matrix.

Region B might be divided in three sub regions, B1, B2 and B3, as shown in Figs. 42.6 and 42.7. Those differ both morphological and compositionally. Fig. 42.6 shows how the concentrations of the main elements in the alloy (Al, Zn, Mg and Cu) and in the granules (W and Co) vary across the thickness of the reaction layer. The Al and W concentrations vary especially near the borders of the reaction layer. The Al concentration is at a minimum level in region A, reaches an intermediate, constant, level all across region B and goes to a maximum in the matrix. The W concentration behaves in an opposite manner to the Al concentration. It is at a maximum in region B, at an intermediate level across region B and reaches a minimum in the matrix. As for the Co concentration, it reaches its highest levels in region A and decreases with the distance from the granule along regions B1 and B2. These observations suggest some mass movement from the granules to the matrix. Also, that the reaction layer might have intermediary Al and W concentrations when compared to the granules and the matrix. As for the other alloy elements, Zn and Mg are observed around the borders of the reaction layer, close to the matrix. The Cu concentration does not seem to vary much across the reaction layer.

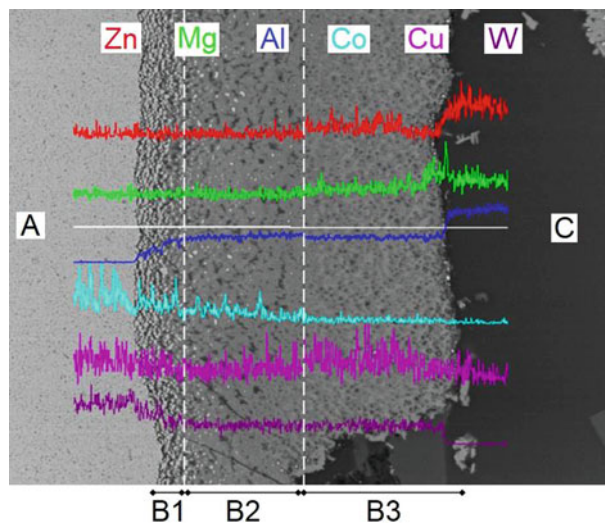


Fig. 42.6 Qualitative variation of the Zn, Mg, Al, Co, Cu and W concentrations across region B – distinct sub regions, B1, B2 and B3, are indicated, as well as regions and C (EDS – 112 μm line. EDS)

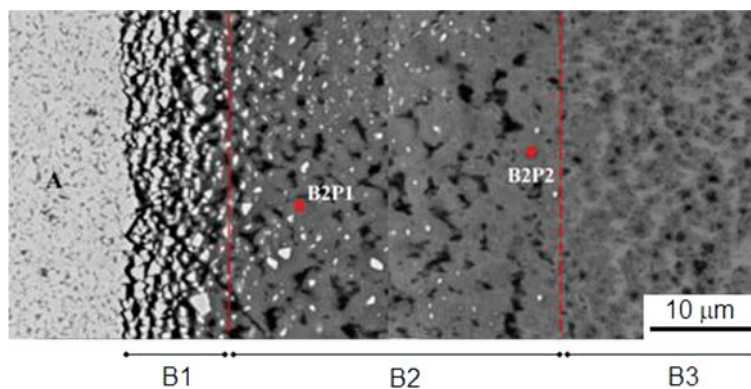


Fig. 42.7 (a) Microstructure of reaction layer with indication of EDS micro analysis points (numerical results from EDS microanalyses of points B2P1 and B2P2 are presented in Table 42.5 (SEM)

Table 42.5 Average compositions of the regions indicated by Points B2P1 and B2P2 in Fig. 42.7 obtained by EDS analyses (wt. %)

Point	Element (wt. %)				
	W	Co	Al	Zn	Mg
B2P1	53.42	2.38	43.57	0.42	0.22
B2P2	70.16	0.63	28.17	0.75	0.29

The sub regions B1, B2 and B3 are shown in more detail in Fig. 42.7. In the sub region B1, individual WC particles are observed – those particles seem to have turned themselves loose from the granule. Close to the granule, the WC particles seem to have the same dimensions as the particles inside it, but appear surrounded by the matrix. In sub region B2, both the quantity and the dimensions of the WC particles decrease as the distance from the granule grows. Also, a new, grey coloured phase appears and predominates as the distance from the granule increases. In region B3, more distant from the granule, no more WC particles are observed and the new phase seems thicker.

Results of the microanalyses points indicated in Fig. 42.7 are shown in Table 42.5. The analysis at point B2P1 allows evaluating the composition of the new phase that appears in B2; it is mainly composed of Al and W, contains a significant amount of Co and minor amounts of Zn and Mg. This result suggests that this new phase might have been formed by a reaction between the Al matrix and the WC particles; this reaction, apparently, consumed the WC particles. This new phase, for simplicity's sake called AlW phase from this point, seems to be partially continuous in region B2. At point B2P2, the EDS analysis indicated a high concentration of W at point, probably because there is a fragment of a WC particle at that point (the high concentration of Al and the presence of alloy elements Mg and Zn might be explained by the reduced size of the analysis region).

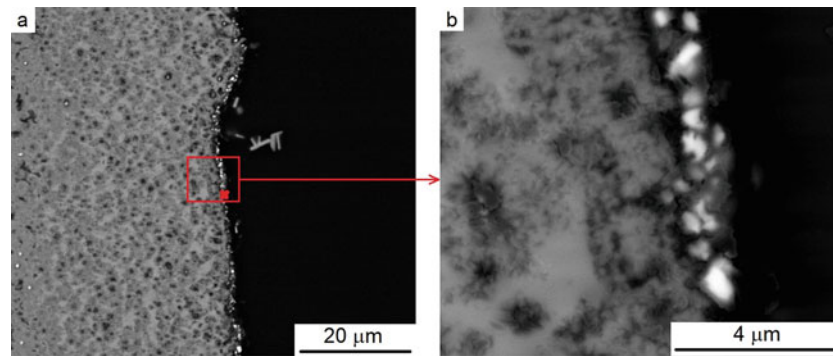


Fig. 42.8 (a) Microstructure of sub region B3 in the reaction layer with indication of a point where EDS micro analysis was performed. The analysis showed a composition of (in wt. %) 71.90% W; 028% Co; 25.22% Al; 1.98% Zn; 0.62% Mg. (b) Detail of the region indicated by a rectangle in a (SEM)

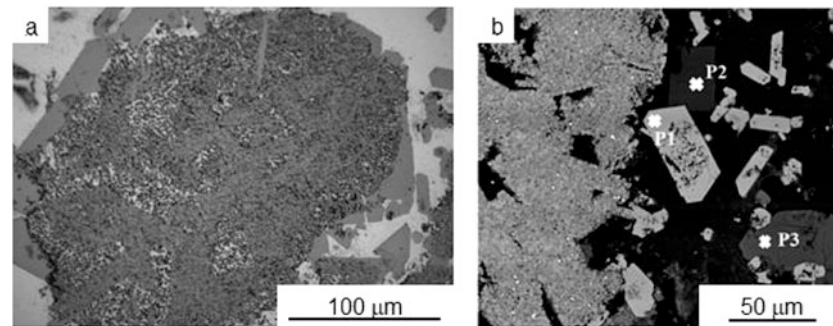


Fig. 42.9 (a) Typical aspect of the reinforcement in composites produced with 3 h contact time and 780 °C contact temperature (OM) (b) Region between the reinforcement and the matrix; EDS microanalyses were performed at points P1, P2 and P3, their numerical results are presented in Table 42.6 (SEM)

The microstructure of sub region B3 of the reaction layer, which is more distant from the granule, is presented with more detail in Fig. 42.8. In Fig. 42.8a, individual WC particles are observed just next to the matrix. The composition of that region, obtained by EDS microanalysis, is (in wt. %): 71.90% W; 028% Co; 25.22% Al; 1.98% Zn; 0.62% Mg. This confirms the particles are very similar to the particles that have turned themselves loose from the granule in sub region B1, as seen in Fig. 42.7, with the difference that the particles in Fig. 42.8 seem smaller and their contours seem less sharp. Their presence in the front of the reaction layer might be explained by the fact that they were not entirely consumed by reactions with the liquid Al, either because they were too large, or because of the presence of coating materials on their surfaces, which significantly reduce wettability [13].

Figure 42.9 presents typical aspects of the reinforcement phase in composites produced with 3 h contact time and 780 °C contact temperature. Most of the cemented carbide granules were consumed in reactions with the liquid metal. Instead of the compact granules surrounded by a reaction layer, discrete phases, composed of polygonal particles are seen, as in Fig. 42.9b. Those phases are present in the matrix as well. Generally, the increase in the contact temperature from 740 to 780 °C not only favoured more extensive reactions between the reinforcement granules and the matrix but also promoted the formation of other complex phases that were not observed in the composites processed at 740 °C.

In Fig. 42.9b, points where EDS microanalysis was performed in different phases are indicated. The numerical results of the analyses are shown in Table 42.6. Point P1 indicates a phase whose composition, as shown in Tables 42.5 and 42.6, is very similar to the composition of the AlW phase observed in the composites processed at 740 °C. Differently from the other phases indicated by points P2 and P3, that phase is ubiquitous all over the composite, its presence is much more extensive, and it has the same aspect as the previously observed AlW phase – therefore, it was concluded that it indeed that phase. However, it was able to develop more extensively, with a polygonal morphology, at higher temperature. The phase indicated by P2 presents high concentrations of Co and Fe in Al, while the phase indicated by point P3 presents a complex composition, where Al predominates but there are also high contents of W, Cr and some Fe and Ti. The Fe, Ti, and Cr might be from the cemented carbides, which contained those elements, as shown in Tables 42.1 and 42.2.

Table 42.6 Average compositions of the regions indicated by Points P1, P2, and P3 in Fig. 42.9 obtained by EDS analyses (wt. %)

Point	Element (wt. %)						
	W	Co	Ti	Fe	Al	Zn	Cr
P1	56.98	–	–	–	43.02	–	–
P2	–	20.35	–	11.63	68.01	–	–
P3	14.32	0.99	1.28	4.77	66.01	2.34	10.30

42.3.3 Phenomena Involved in the Formation of the Reaction Layer.

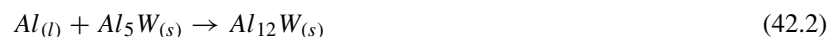
A mechanism of formation for the reaction layer is proposed based on the microstructures of the composites produced with contact temperature of 740 °C, and the observation of regions B1, B2 and B3 of the reaction layer. The sub region B1, which is adjacent to the granule, is characterized by the presence of disaggregated WC particles, which loosened themselves from the cemented carbide granule. They are morphologically similar to the particles found within the cemented carbide granule. In the transition between regions B1 and B2, the size of the carbide particles is progressively reduced as a new, platelike phase, which was called AIW, appears. In sub region B2, the carbide particles eventually disappear. That can be seen in Fig. 42.7. Finally, in the sub region B3, the AIW phase forms thicker agglomerates. In the interface between region B3 and the matrix, some remaining carbide particles that loosened themselves from the granules, but were either not entirely consumed or pushed away from the granule as the reaction layer was formed, are found; as shown in Fig. 42.8.

The disaggregation of the carbide particles from the granules might be explained by the high affinity between Al and Co [13, 14]. Al and Co form various intermetallic compounds even at low temperatures [15]. A solid solution of Al in Co, with Al concentrations around 1.5 wt. % Al, undergoes a eutectoid reaction around 350 °C, which is described in Eq. 42.1. The eutectoid product AlCo has a lower density (6.08 g/cc) than any of the allotropic forms of Co (8.84 and 8.79 g/cc for ϵ and α Co, respectively), and thus the transformation is accompanied by a volumetric expansion [14, 15]. Co is present as binder between the particles in machining inserts [11–14]. The Al would diffuse to the Co rich areas in the machining insert granules and form the phase AlCo, which is fragile and has a smaller density than any of the allotropic forms of the Co (and thus a greater specific volume) [14, 15]. The formation of that phase would cause local stresses to arise. As the AlCo is fragile, it would crack, separating the WC particles from the rest of the granule, as schematized in Fig. 42.10.



Still, AlCo might be formed even at very low Al concentrations in Co [15]. The authors studied failure of machining inserts during the cutting of Al. Their observations showed that when both Al and cemented carbides are heated together, the Al can diffuse up to 80 μ m under the surface of the insert and form the AlCo phase. That renders the surface of the insert fragile and causes its failure – porosity and disaggregation of the cemented carbide are observed, which results in the loss of some particles. The microanalyses performed in this investigation did not specifically detect AlCo in sub region B1, but both the presence of Al and Co was detected by the EDS microanalyses in that region (see Fig. 42.6). Moreover, an AlCoFe rich phase was detected in regions near the reaction layer in the composites produced with greater contact temperature and time, as shown in Table 42.6.

Once they are loosened from the granule, the carbide particles are more closely surrounded by the liquid Al and reactions between the elements Al and W may form the AIW phase observed in sub region B2 (as shown in Fig. 42.7). For low W concentrations, two peritectic reactions, expressed by Eqs. 42.2 and 42.3, happen at 697 and 661 °C, respectively. EDS microanalyses in specific regions where the AIW phase is observed indicate W concentrations higher than 50% wt. and Al concentrations higher than 40% wt.. A comparison between this data and the approximate composition of the phases in Eqs. 42.2 and 42.3 indicated that, most probably, the phase observed in region B2 is Al₅W.



Other authors have also observed reactions between W and liquid Al as WC particles were added to Al melts [8]. added WC particles to a Al melt at 830 °C and observed the formation of both Al₁₂W and Al₅W. Al₁₂W was formed with less frequency and appeared as polygonal plates. Al₅W was formed with more frequency and appeared as elongated and/or agglomerated plates. This behaviour is similar to the observed in Fig. 42.9 for the composite produced at higher temperatures. Also, the phase formed at lower temperatures exhibited, too, a strong tendency to agglomerate.

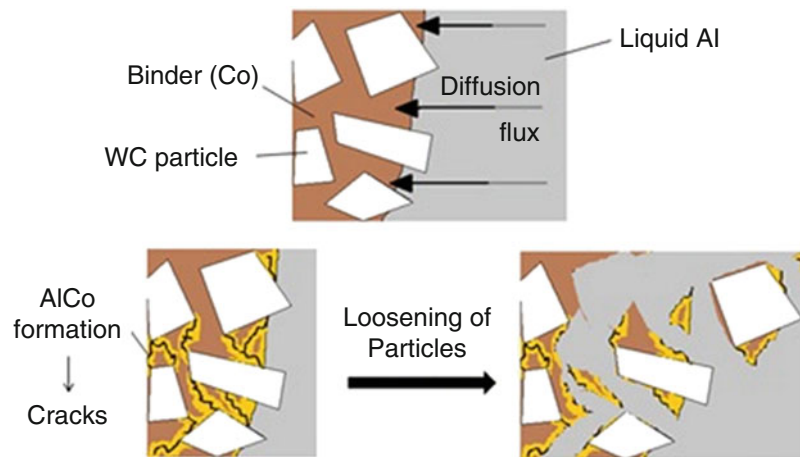


Fig. 42.10 Schema of the mechanism by which the WC particles would be separated from the granule

The formation of the reaction layer between the matrix and the granules might be desirable, as it proportionates a metallurgical bond between the matrix and the reinforcement and, possibly, a more gradual transition between the two materials. Other interesting result was that little difference was observed in the reaction layer of the composites fabricated with contact temperature of 740 °C – for that temperature, similar results are obtained after 1 and 3 h contact times. Therefore, if a reaction layer is desirable, it can be obtained with the blandest mixing conditions, of 1 h/ 740 °C.

42.4 Conclusions

This work investigated the possibility of producing, by casting, Al matrix composites reinforced with cemented carbide granules in such a way to promote metallurgical interaction between the matrix and the reinforcement.

The obtained results indicate that the contact between the liquid metal and the cemented carbide granules propitiates the formation of a reaction layer between granules and matrix at contact temperatures above 700 °C (for the time intervals studied). The formation of that reaction layer consumes the granules, at the same time it forms a metallurgical bond between matrix and reinforcement. For the contact temperature of 740 °C, the contact time (within the 1 and 3 h conditions studied) had little influence in the extension of the reaction layer formed. For the contact temperature of 780 °C, the formation of compact and/or complex phases in the material was observed. Moreover, the cemented carbide granules were mostly consumed by the formation of other phases at that temperature.

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