

An Overview of the Interactions Between Reinforcements and Al Matrices with Si, Cu And Mg as Alloying Elements in Aluminum Matrix Composites: Case of Oxide Reinforcements

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Oxides (Al_2O_3 , SiO_2 , TiO_2 , ZrO_2 , Y_2O_3 , CeO_2 , MgO) are among the most used reinforcements for Aluminum Matrix Composites (AMC); while the combination of Al with Si, Cu and/or Mg excels among the alloying systems used as matrices. Some works in literature study the effects of the reinforcements and the alloying elements on the composites manufacturing, microstructure and mechanical properties. Nevertheless, it is necessary a recompilation of the interactions oxide reinforcement-alloyed matrix, including the reciprocal effects between them. Our search revealed that not only reactions occur at the interfacial regions, but also other phenomena depending on the reinforcement characteristics and the matrix composition, which affect mechanical properties. These phenomena include modifications in the matrix microstructure and its precipitation process, diffusion of elements through the interfaces, change in the reinforcement wettability by the liquid metal, loss of alloying elements, and deterioration of the reinforcement. This work presents the occurrence of these phenomena for Al matrices with different contents of Si, Cu and Mg reinforced with the most used oxides. Its novelty lies in exploring these combinations of conditions, which could serve as a benchmark study and help for a better understanding and selection of the matrix-reinforcement system.

Keywords: Al alloys, Al-Si-Cu-Mg, reinforcement, oxide, composite, interface.

1. Introduction

Aluminum alloys are essential for a wide variety of applications in industries such as transportation, structural, packing, electronic, food and chemical. They present a combination of properties including low density, recyclability, corrosion resistance, high specific strength, high temperature strength and good tribological behavior^{1,2}. Among these alloys are those with the presence of Si, Cu and/or Mg as alloying elements. Each one of these elements contributes to the microstructures and properties of the resulting alloys, which are used for applications according to their properties. Silicon is the most used alloying element in aluminum casting alloys^{1,3-6}, providing abrasion resistance, decreasing density, significantly increasing castability, and slightly improving mechanical properties. Regarding Cu, its addition increases the strength and hardness of these alloys, also improving machinability by increasing matrix hardness, although reducing the corrosion resistance⁷⁻⁹. On the other hand, Mg provides

important strengthening and improvement in work hardening, also imparting good corrosion resistance^{1,10-14}. The addition of these alloying elements to Al can lead to the formation of micrometric second phases or nanometric precipitates, which can be respectively obtained from the solidification process, or precipitate from a solid solution due to a heat treatment. Some of them are eutectic Si, $\beta\text{-Mg}_2\text{Si}$, $\theta\text{-Al}_2\text{Cu}$, Al_2CuMg , and quaternary Q phase ($\text{Al}_5\text{Cu}_2\text{Mg}_8\text{Si}_6$)¹³⁻¹⁹, and in some cases they could be considered reinforcements of the alloys and part of in-situ composites (e.g. Mg_2Si and Al_2Cu ²⁰⁻²⁴). Second phases, precipitation hardening and solid solution strengthening are among the most important hardening mechanisms for these alloys^{4,7,16,25-26}, which highly depend on the alloying elements content. Nevertheless, the strengthening reached by these mechanisms is not as high as desired. In search of better properties these alloys have been reinforced with particles, whiskers, fibers and nanotubes of different materials, obtaining Aluminum Matrix Composites (AMC)²⁷⁻³³. These better mechanical properties are possible by adding reinforcements with elastic moduli significantly

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higher than the matrices, such as Al_2O_3 ($E = 400$ GPa) or SiC ($E = 480$ GPa). It is important to remember that the elastic modulus for an aluminum matrix is 70 GPa^{1,27-33}. Elastic modulus, tensile strength, and high temperature stability of AMCs are higher than those of unreinforced alloys; while their ductility, fatigue and fracture toughness are significantly lower²⁷⁻³¹. When aluminum and its alloys are combined with ceramic reinforcements intermediate properties are obtained. It is well known that metals are ductile and present low stiffness; while ceramics are stiff and with very high strength, but also are brittle and their fracture can be catastrophic. AMCs combine the great strength of the ceramics while avoiding their brittle failure, also reducing the deformation in the part of the matrix which is close to the reinforcements. The role of the aluminum matrix consists on binding and transferring load, giving its shape to the composite component; while the degree of strengthening depends on strong bonding at the matrix–particle interface^{28,29,34,35}. AMC have been the most used matrix due to their lightweight and higher mechanical and thermal properties²⁷⁻²⁹. Al has a density of 2.7 g cm⁻³, an elastic modulus of 70 GPa and compressive strength of ~ 450 MPa (e.g. 380 alloy)¹, while some of their most used reinforcements have similar densities (SiC 3.21 g cm⁻³, B₄C 2.52 g cm⁻³ and Al₂O₃ 3.92 g cm⁻³) but very high elastic moduli (SiC 430 GPa, B₄C 450 GPa and Al₂O₃ 350 GPa) and compressive strengths (SiC 2800 MPa, B₄C 2800 MPa and Al₂O₃ 350 MPa)³⁶. This wide variety of physical and mechanical properties lead to applications in industries such as transport, aeronautic, aerospace and electric, besides in a wide variety of recreational products³⁷. Examples of these applications are rocker arms, brake rotors and pistons, drive shafts, core of overhead electrical conductors, energy storage flywheels, and sporting goods for golf, cycling, baseball and skiing^{38,39}. Composites include Metal Matrix Syntactic Foams (MMSFs), formed by porous reinforcements or fillers⁴⁰⁻⁴⁵, which are used in applications requiring higher mechanical properties than the obtained using conventional foams. Hollow fillers used in these materials reinforce the matrix while decreasing its density. Some of their applications are in marine equipment, sandwich parts in other composite materials, and structural components in the aerospace industry⁴⁵. Mechanical properties of both MMC and MMSF depend on details such as the matrix composition and the reinforcement volume fraction, distribution, aspect ratio, size, orientation and interfacial bonding. For MMSF characteristics of the hollow reinforcements such as porosity percentage and wall thickness also affect the mechanical properties⁴⁵. Among the most reported reinforcements for AMC are carbides (SiC, B₄C, WC, TiC)⁴⁶⁻⁵², oxides (Al₂O₃, SiO₂, TiO₂, ZrO₂, Y₂O₃, MgO, CeO₂)⁵³⁻⁵⁸, nitrides (Si₃N₄, BN)⁵⁹⁻⁶¹ and Carbon Nanotubes (CNTs)⁶²⁻⁶⁷. For the case of syntactic foams there have been mainly reported metallic or ceramics hollow spheres, being Fe, SiC, SiO₂ and Al₂O₃ the most used^{40-45,68-72}.

Ceramic oxides represent an important part of these reinforcements, being essential the study of this particular group and their interactions with aluminum alloy matrices. Al is a very active metal, reacting with different reinforcements, fact that could be enhanced with the presence of alloying elements⁷³. Due to the variety of matrices and reinforcements,

the microstructural analysis of these materials is essential, including matrix-reinforcement interactions and their interfaces. Characterization techniques for this purpose include Optical Microscopy (OM), Scanning and Transmission Electron Microscopy (SEM and TEM, respectively), X-Ray Computed Micro-tomography (XCT) and X-Ray Diffraction (XRD)⁷⁴⁻⁷⁷. These studies grow in importance for matrices with more alloying elements, as the case of Al alloys with Si, Cu and Mg. The information in literature about the interfacial reactions for this alloy system with oxides in AMC is little and disperse. Researchers have mainly focused they works in analyzing the effect of the kind and percentage of reinforcements, with some works related to the study of the interfacial reactions, but few works including the effect of the alloying elements as a variable. It is reported that for AMC containing different alloying elements the improvement of the mechanical properties of the composite depends not only on the reinforcement but also on the matrix strengthening due to the alloying elements (solid solution and precipitation strengthening), and on the interfacial reactions and other interactions matrix-reinforcement⁷⁸. Besides, most of the works found in literature only include binary or ternary alloys, and mainly with the most used reinforcements (i.e. SiC, Al₂O₃)^{41,79}. This is accentuated by the fact that majority of the reviews deals only with aspects such as the effect of the reinforcement, its volume fraction and distribution. Then, it is necessary a compilation of the effect of these alloying elements on the matrix-reinforcement interactions. Except for the particular case of silicon in SiO₂, few works in literature include the analysis of the effect of the alloying elements on these interactions, being also considered that much of them do not have a significant effect on the reactions matrix-reinforcement. Maybe Mg is the second most studied element in these composites, reported as a wettability improver of the reinforcement by the liquid metal^{80,81}. That is why in this review we tried to gather and compile information about these interactions and their effect on the mechanical properties of the composites. There are different mechanisms contributing to the composites yield strength (σ_y)⁸², which include: the Al matrix strength (σ_m), which depends among other factors on the alloying elements forming second phases; the Hall-Petch strength (σ_{H-P}) due to the effect of grain boundaries in refined grains; the strengthening by solid solution (σ_{ss}), which depends on the alloying elements content; the precipitate strengthening (σ_{pp}) due to the precipitation of fine particles; and the strengthening due to the reinforcements (σ_r), directly affecting MMCs. In addition to this last mechanism, matrix-reinforcement interactions sum other mechanisms, as the load transfer (σ_{LT}); and thermal (σ_{TM}) and Elastic Modulus (σ_{EM}) mismatches⁸³⁻⁸⁶. Some of these mechanisms can be affected by the interactions produced when a reinforcement is added to a matrix, modifying for instance grain sizes, elements in solid solution, precipitates and their formation mechanisms, etc. That is why the aim of the present review is to analyze the information in literature describing the role of Al alloys with Si, Cu and Mg on the interaction mechanisms and interfaces formation in Aluminum Matrix Composites reinforced with different oxides, and their subsequent effect on the mechanical properties. This study could contribute

to a better selection of reinforcements depending on the alloying elements content in the aluminum matrix.

2. Generalities of Interfaces

Among the most important interactions occurring when a reinforcement is immersed in a liquid metal is the formation of interfaces, which are critical for strengthening and stiffening the composite. Physical properties such as thermal conductivity, expansion, and dimensional stability are also directly related to the characteristics of the interface, which depend on details such as the manufacturing process, constituent phases composition, and time and temperature of contact reinforcement-matrix. Among the most used processes for manufacturing AMC are stir casting, infiltration, squeeze casting, compocasting and powder metallurgy^{79,85-88}. The presence of different alloying elements could modify the interfaces obtained during these processes, affecting the mechanical properties. Such interactions highly depend on the manufacturing temperature, which are different according to the used process, being more determinant the interfacial effects for liquid-state processing due to the use of higher temperatures and the effect of wetting. In these processes temperature is commonly maintained between 700 and 900 °C, required for melting Al alloys.

Interfaces can be just a boundary, presenting a physical nature; or being a layer obtained due to chemical reactions, diffusion or other phenomena. These regions located around the reinforcements are of finite thickness and can contain new compounds, structural deviations, or structures and properties different from those of both matrix and reinforcement^{89,90}. Although there should be avoided some adverse chemical reactions affecting matrix or reinforcement, such as those forming detrimental brittle reaction products³⁴, a high degree of strengthening is directly related to a strong matrix-reinforcement bonding. There are desired interfaces with atomic or molecular interactions to reach optima mechanical properties⁸⁹, being in several cases present only as inhomogeneities in the near-interface region, barely observed even using High Resolution TEM (HRTEM)⁹¹. According to Guo⁹², from a phenomenological point of view interfaces can include physical bonding, chemical bonding, or mechanical keying. That is why the factors contributing to bonding should be analyzed at different levels and with different techniques.

3. Interactions Between Oxides and Al Alloys with the Presence of Si, Cu and Mg

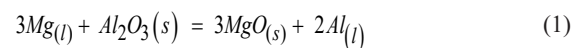
The interaction between a reinforcement and a matrix, and the subsequent mechanical properties of the obtained AMC will depend on various factors as wetting, reactivity, bonding, etc., which at the same time are related to the manufacturing process and the chemical composition of the system. Interfacial reactions mainly occur when the reinforcements are in contact with molten Al alloys, but can be also present at lower temperatures due to diffusion or other processes contributing to solid state reactions.

Microstructural modifications are one of the most important results when a reinforcement is added to a molten alloy. This occurs due to temperature gradients between

the molten matrix and the cooler reinforcements, affecting grain size and second phases morphologies. Besides, the volume of the molten metal decreases with the increase in the volumetric fraction of the reinforcement, leading to a faster solidification compared to the solidification process for unreinforced alloys. The solidification velocity for alloys mainly depends on the mold and not on the presence of reinforcements which could act as nucleation centers if are at temperatures lower than that of the molten metal⁹³. Lower temperature and less latent heat during solidification could cause not only grain refinement but also higher matrix saturation with alloying elements and low wetting^{94,95}. Fine grains can significantly increase the mechanical properties of the matrix due to the Hall-Petch effect contribution, mainly for sizes below 100 nm⁹⁶. On the other hand, matrix composition could be altered by irreversible interfacial reactions involving reinforcement and matrix solutes. This region can influence mechanical properties, being a preferential location for precipitation and alloying elements segregation, with the presence of higher concentration of point defects or residual strains⁹¹. Even if the addition of reinforcements is to an age-hardenable alloy, precipitates nucleation and their growing kinetics may change significantly compared to the unreinforced alloy⁹⁷. Then, besides the interfacial reactions it is also important to analyze the effect of other interactions which could significantly affect the mechanical properties of the composite. In the following subsections the effect of Si, Cu and Mg as alloying elements on the interfacial reactions and on other matrix-reinforcement interactions will be discussed for the most important oxide reinforcements used in AMC. The effect of these phenomena on the mechanical properties of the obtained composites will be also assessed.

3.1. Al_2O_3

Aluminum oxide (alumina, Al_2O_3), presented mainly as $\alpha-Al_2O_3$, has high hardness and specific strength, is chemically resistant to bases and acids, can be used in applications needing high temperature resistant and has excellent tribological properties. It is used in applications requiring refractoriety and heat-resistance, and as abrasive, cutting or coating material^{27,29}. It has been also reported as hollow reinforcement in metal matrix syntactic foams^{68,98}. Al_2O_3 does not react with Al matrix^{41,42}, but it is susceptible to be attacked by alloys containing elements with oxides more stable than Al_2O_3 (e.g. Mg). This could limit the maximum allowable service temperature of the material because Al_2O_3 could be degraded, decreasing mechanical properties⁹¹. It has been reported the formation of MgO by a direct reaction due to the addition of Mg to Al alloys by the following reaction^{99,100}:

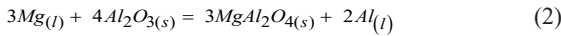


As a result of this reaction in extreme cases the alumina particles can change to MgO, as showed Pai et al.¹⁰¹ for an Al-4.5Mg alloy. In order to promote wetting between aluminum and Al_2O_3 , Mg has been used as external dopant adding it to the molten aluminum, e.g. an Al-2.8Mg-0.81Si alloy¹⁰². This advantageous use was also found adding 1% magnesium powder to a molten 6061 Al-0.85Mg-0.68Si-0.22Cu

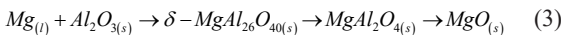
alloy reinforced with Al_2O_3 particles⁹⁵. It is also reported that Mg reduces particles clustering¹⁰³ because it can act increasing the reinforcement surface energy, decreasing the matrix surface tension, and/or decreasing the reinforcement-matrix energy on the interface. This was corroborated by Chen et al.¹⁰⁴, who demonstrated that Mg- and Cu-doped interfaces presented uniform charge distribution, granting excellent tensile properties.

Janowski and Pletka¹⁰⁵ found the presence of a Si-rich oxide-based amorphous phase at the interface for Al-4.4Cu-0.5Mg-Si and Al-0.25Cu-1.0Mg-0.6Si alloys reinforced with 9 and 18 vol.% of Al_2O_3 particles. This was effect of the locally high Si content near Al_2O_3 , and led to diminish mechanical properties due to debonding which occurred as a result of the fracture of the layer surrounding the reinforcement.

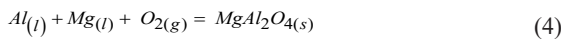
Mg enrichment of the matrix at the interface has been reported, even with appreciable magnesium penetration into the reinforcement. Munitz et al.¹⁰⁶ used Auger Electron Spectroscopy and Electron Diffraction for the analysis of an Al-4Mg reinforced with Al_2O_3 fibers, finding that when Al_2O_3 was added to the molten alloy the formation of the spinel phase MgAl_2O_4 occurred (MgO was also detected), this according to the following reaction¹⁰⁷⁻¹¹¹:



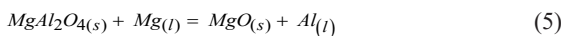
These results show that the formation of MgO or/and MgAl_2O_4 may occur with similar Mg contents (Pai et al.¹⁰¹ obtained MgO using Al-4.5Mg/ Al_2O_3 particles at 850 °C, while Munitz et al.¹⁰⁶ obtained both MgO and MgAl_2O_4 using Al-3.8Mg/ Al_2O_3 fibers stirred at 685°C during 30 min). This demonstrates the importance of the manufacturing process and the control of parameters such as temperature and time of contact matrix-reinforcement. More research is needed on this topic, because in addition to the formation of Mg and MgAl_2O_4 through Equations 1 and 2, both compounds can be obtained following the sequence¹¹²:



MgAl_2O_4 has chemical inertness, high melting point, good thermal shock resistance, and high mechanical strength at elevated temperatures¹¹³⁻¹¹⁵. Its formation has been also reported from de melt due to the presence of oxygen as follows¹⁰⁹⁻¹¹¹:



The formation of the spinel phase through these reactions also includes a reverse reaction, leading to its degradation:

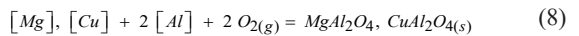
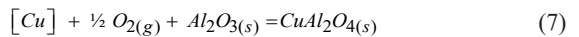


The presence of Mg not always leads to the formation of these Mg-rich compounds, because it depends on the kind of alumina. For example, Bacciarini and Mathier¹¹⁶ demonstrated that Mg did not react with α - γ -amorphous Al_2O_3 for an Al6061 (~1 wt.% of Mg)/ Al_2O_3 (60% vol.) composite obtained by infiltration. Contrarily to the behavior observed for composites reinforced with α - Al_2O_3 , in this case Mg remained at the matrix and contributed to the composite strengthening by Mg_2Si precipitation. This inertness alumina

consisted of α - Al_2O_3 which was modified into a mixed α - γ -amorphous Al_2O_3 , and made the difference in the interfacial reaction. Its inertness was not explained by these authors, but it could be attributed to differences in wetting. It is reported that γ - Al_2O_3 present a defect structure, surface acidity and high surface which makes it useful in applications as adsorbent and catalyst support^{117,118}. Otherwise, amorphous Al_2O_3 presents chemical, thermal and mechanical stability derived from its non-crystalline nature^{117,118}. Molins et al.¹¹⁹ also found the precipitation of Mg_2Si analyzing the behavior of the alloying elements at the interface of 20% Al_2O_3 /(Al-2Mg-0.3Si) composites through element maps and concentration profiles along line-scans. They found important segregations of Mg and Si at the interfaces when SiO_2 was added as thin colloidal coating. These elements reacted to form Mg_2Si through Equation 6, hardening the matrix although Al-Mg alloys are not known for precipitation hardening:



In the case of Cu additions, Chawla¹⁰⁰ reported the formation of CuAl_2O_4 , while Baik et al.⁹⁴ analyzed the reaction between Al_2O_3 and an Al-12Si-1.0Cu-1.1Mg-1.4Ni alloy reinforced with 20 vol.% of δ -alumina short fibers. They reported that infiltrating an Al_2O_3 preform previously heated at 600 °C led to the reaction between Mg and Al_2O_3 to form the spinel phase, besides reacting Al_2O_3 with Cu. This led to the formation of a reaction layer, corroborating the presence of Cu and Mg in excess on the fiber surface, degrading the strength of the composite. It is important to remark that the formation of the interlayer was not observed for infiltration at lower temperatures, when there was not enough wetting of the Al_2O_3 fibers by the molten alloy. Reactions involving Cu for this system are:



Another reported result derived from the interaction matrix-reinforcement is the modification of the precipitation process, as was observed by Ikeno et al.¹²⁰ using HRTEM for Al_2O_3 reinforcing Al-Cu-Mg alloys. These authors did not observe these modifications for Al-Mg-Si alloys. Otherwise Matsuda et al.¹²¹ reported the decrease of age hardening for an Al-1%Mg₂Si alloy after adding 4% of alumina particles. Using electron diffraction and EDS (X-ray Energy Dispersive Spectroscopy) they found the formation of spinel MgAl_2O_4 at the interface Al_2O_3 -matrix, but not as a thin layer but as plate shaped particles. For an Al-7Si-2.8Cu (in mass%) alloy the addition of Al_2O_3 fibers led to an increase in the age hardening response, significantly decreasing the time required to obtain peak hardness. For the same alloy with 0.4 mass% of Mg the reinforcement with Al_2O_3 led to a decrease in the peak ageing time, also decreasing the age hardening response. This result motivated a decrease in the composite strength, attributed to a lower Mg concentration in the matrix originated by the above commented reaction with Al_2O_3 to form the spinel MgAl_2O_4 phase (Equation 2). Besides, Cu segregation of about 2% at. on the interface

between Al_2O_3 and an Al-1.4Cu alloy has been reported, analyzed by HAADF-STEM (High Angle Annular Dark Field-Scanning Transmission Electron Microscopy) and EDS¹²², leading to heterogeneous nucleation of precipitates at the interfacial zone. Otherwise, Liu et al.¹²³ found that for higher Cu contents (5.3-6.4 wt.%) a soft interfacial layer increased the fracture toughness of the composite in the peak age condition. This interface presented precipitates different to the observed in the matrix, depleting the Cu concentration in the interface.

Figure 1a shows a SEM micrograph for an Al_2O_3 -Al(15Cu) matrix, where Al_2O_3 particles (light) are uniformly distributed¹¹⁴. In this image the eutectic second phase Al- Al_2Cu is also observed. Otherwise, Figure 1b shows a TEM image of the interface between Al_2O_3 and an Al-1.4Cu matrix, with a zigzag morphology and the presence of the intermetallic phase Al_2Cu precipitated at the interface (dark phase) due to Cu segregation¹²⁰. Otherwise, Figure 1c-f shows bright and dark field TEM images and SADP (Selected Area Diffraction Pattern) of the interfacial layer obtained due to the reaction between $\gamma-Al_2O_3$ fiber and a matrix Al-7Si-2.8Cu-0.4Mg (in mass %). This reaction occurred during the manufacturing process, infiltrating an Al_2O_3 preform at 1073 K¹¹². In this figure patterns correspond to aggregates of $\gamma-Al_2O_3$ particles of 10 nm (Figure 1d) constituting the reinforcement, and to the interfacial $MgAl_2O_4$ (Figure 1f) formed due to the presence of Mg (Equation 2). This reaction layer has 100-120 nm in thickness, and its formation leads to a reduction in the concentration of Mg available to form precipitates of λ' phase ($Al_3Cu_2Mg_8Si_8$). Again, the formation of the spinel phase even at low Mg content reveals the narrow limit to

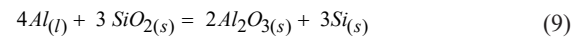
obtain this phase according to the above presented reactions of Equations 1-3.

It has been also found that the grain size of the matrix considerably decreases with the increase in the percentage of Al_2O_3 particles, showing the influence of not only the alloying elements but also of the reinforcements¹²⁴. Grain refinement increases yield strength and wear resistance, decreasing ultimate tensile strength and ductility.

Although important works were found related to matrix-reinforcement interactions for composites reinforced with Al_2O_3 , they are focused to Al alloys with Mg as the main alloying element. The effect of Si and Cu additions needs more research, because the studies including these elements are few and basically deal with the formation of Cu and Si-rich precipitates.

3.2. SiO_2

Silicon dioxide (silica, SiO_2) has been selected as reinforcement due to its high hardness, melting point and availability, although the Al- SiO_2 composites have been barely used because of the high reactivity between these materials. The few studies found in literature reveal that they present high wear resistance and hardness, enabling their applications in severe thermal environments which demand greater mechanical properties, such as automotive parts¹²⁵⁻¹²⁷. Al reacts with SiO_2 through the following reaction¹²⁸:



This reaction is even reported to occur at the interface in the solid state at temperatures as low as 440-550°C¹²⁸,

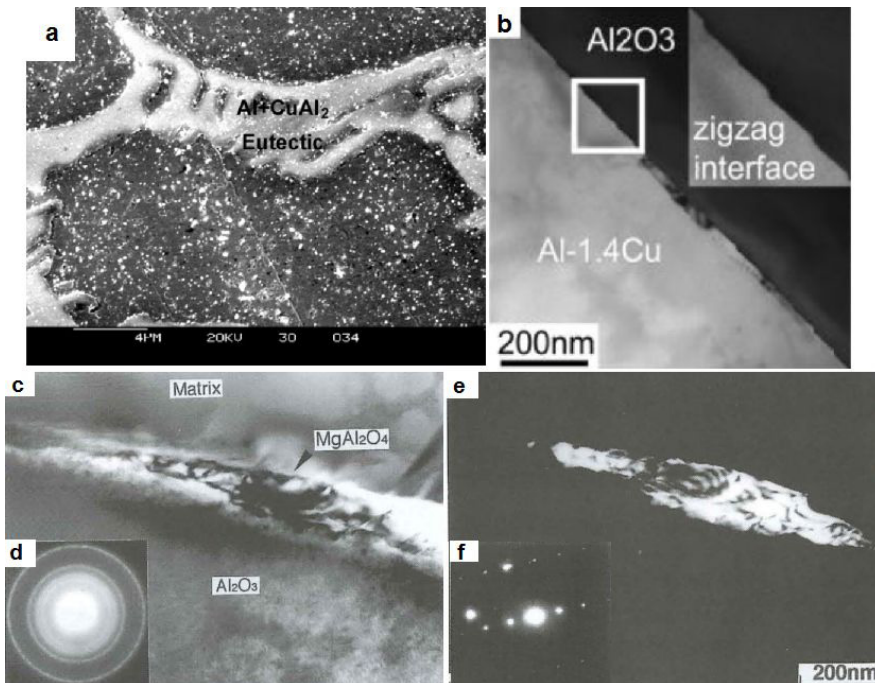
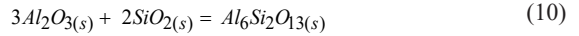


Figure 1. a) SEM image of an Al_2O_3 -Al(15Cu) composite where light grey Al_2O_3 particles are observed (reproduced with permission from¹¹⁴). b) Bright field TEM micrograph for the interface Al_2O_3 -matrix (Al-1.4Cu alloy) (reproduced with permission from¹²²). c-f) Interface reaction layer Al_2O_3 /Al-Si-Cu-Mg matrix: c) bright field TEM image, d) SADP identifying Al_2O_3 , e) dark field TEM image, and f) SADP for the interface ($MgAl_2O_4$). (reproduced with permission from¹¹²).

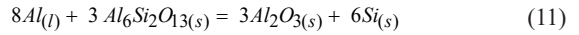
and can be used for fabricating Al_2O_3 reinforced AMCs¹²⁹. Gregolin et al.¹³⁰ found that an Al-5SiO₂ (in wt.%) fiber composite changed into another composite after being heat-treated at 600 °C. The original fibrillar morphology was retained but its composition changed from SiO₂ to Al₂O₃. Orbulov et al.¹³¹ found that for hollow SiO₂ spheres used in syntactic foams this reaction also occurs. These authors revealed that the reaction degraded SiO₂, but did not occur for matrices with about 12 wt% of Si, where SiO₂ remained stable. Then, a possible route for manufacturing these composites without the degradation of the reinforcement could be the use of high content of this alloying element. The same behavior will be further observed for other reinforcements, where adding as alloying element the same element of the oxide minimizes or even avoids the reduction reaction. More research is necessary for establishing in each case the correct combination of alloying elements content and processing conditions, such as temperature and time. Figure 2a-c shows¹²⁹ the kinetic model of the reaction present in Equation 9, indicating that when the temperature increases to the melting point of the aluminum alloy, solid Al melts and surrounds SiO₂, forming the interfaces Al-SiO₂ (Figure 2a). As the temperature increases the reaction continues with the formation of Al₂O₃ and solid Si at the interfaces (see Figure 2b). These new phases then leave the interfaces and diffuse into the liquid Al matrix, being obtained Si blocks and Al₂O₃ particles as final products of the reaction (Figure 2c)¹²⁹. This is shown in Figure 2d-f for a real composite, where first Si is displaced by the reaction between SiO₂ particles and the Al matrix, surrounding the particles (Figure 2d); followed by the formation of an outer layer of Al₂O₃ (Figure 2e); and the final total transformation from SiO₂ to Al₂O₃¹³². This reaction not only leads to modify the reinforcement, but also to obtain a different matrix with the presence of Si, which could affect properties such as wear resistance and strength.

Although as it was observed in Fig. 2d-f Wang and Shi¹³² did not obtain other reaction products apart from Al₂O₃ and Si, it is reported that besides Equation 9 another possible

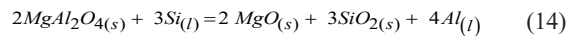
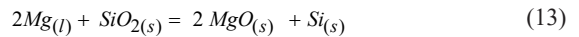
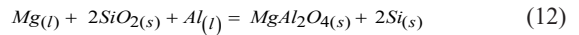
reaction occurs at solid state due to the interaction of alumina and silica, being obtained Al₆Si₂O₁₃ as follows¹³²:



This reaction may be favored at low temperatures, although eventually Al₆Si₂O₁₃ will transform to Al₂O₃ as follows:



Microsegregation of second phases in the interfaces has been reported for an Al-4Cu-1Mg-0.04Si alloy AMC reinforced with Al₂O₃, with the presence of SiO₂ as a binder in the manufacturing process. This study of Cayron⁸⁷ revealed that Mg reacted to form MgO and spinel MgAl₂O₄, which was determined using EDS and SAED (Small Angle Electron Diffraction) through TEM⁸⁷. This author reported the following reactions:



SiO₂ is found to be an important oxygen source in the formation of MgAl₂O₄ in Al alloys with the presence of Mg. The reactivity of SiO₂ is higher than other oxygen sources such as Al₂O₃, MgO and TiO₂^{113,116}. Low Mg content favors the formation of MgAl₂O₄ according to the reaction in Equation 12, while high Mg content tends to form MgO according to the reaction in Equation 13^{133,134}. In various works Mg content is considered to be low when it is lower than 4 at.%, favoring Equation 10¹³⁴⁻¹³⁹ and the formation of the spinel phase. Nevertheless, Sato and Mehrabian¹⁴⁰ reported that the spinel MgAl₂O₄ is obtained if the concentration of Mg in the melt is in the range 0.04-1.7 wt% Mg, while MgO

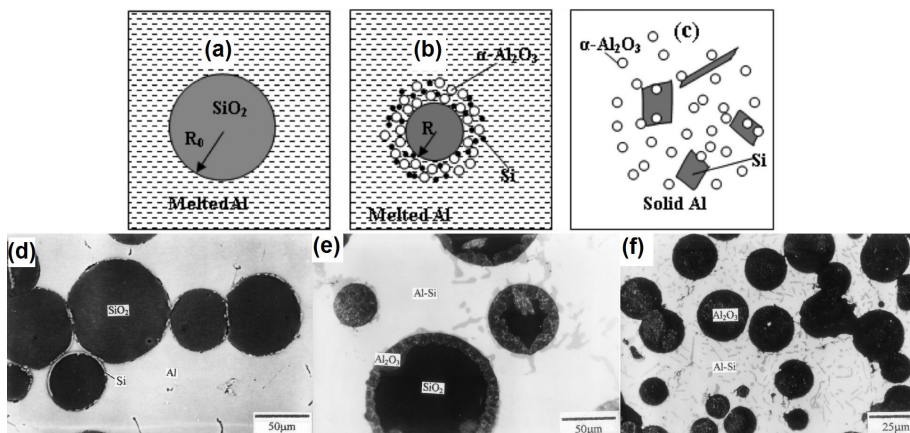


Figure 2. Models illustrating the reaction between SiO₂ and Al matrix to obtain Al₂O₃ and Si. (a) before the interfacial reaction; (b) during the reaction; (c) at the end of the interfacial reaction. (Reproduced with permission from¹²⁹). (d-f) Microstructures for real composites at different stages of the SiO₂-Al interfacial reaction: (d) SiO₂-Al composite, where a Si layer is observed surrounding SiO₂; (e) SiO₂-AlSi composite, where the Si layer changed to Al₂O₃; and (f) Al₂O₃-AlSi composite after the final transformation from SiO₂ to Al₂O₃. (Reproduced with permission from¹³²).

is obtained for concentrations higher than about 1.7 wt%. That is why special attention must be paid in order to adequately control these reactions.

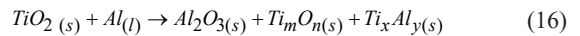
In some cases, most of Mg within the matrix could be consumed as a result of the interfacial reaction with SiO₂, leading to the absence of Mg-rich precipitates, e.g. Al₂CuMg. These precipitates are responsible of the matrix strengthening, and their absence it is reported to decrease mechanical properties¹³⁴. Besides, the bonding strength between MgAl₂O₄ and Al is higher than that between Al₄C₃ and Al^{134,141}, modifying the fracture mechanism of the particles from pull-out to tensile loading-induced fracture. Then, Mg content in the matrix needs to be controlled, being reported a substantial increase in the hardness of both the composites and the matrices by increasing the amount of Mg¹³⁴. Mg and Si released from the interfacial reactions can combine to form Mg₂Si according to Equation 6, hardening the Al-Mg matrix in not heat treatable alloys. The original Al-Mg matrix are reported to changes to Al-Mg-Si due to the reduction of SiO₂ and consumption of Mg, precipitating Mg₂Si mainly along the grain boundaries^{80,142,143}.

The addition of Mg into the aluminum melt helps to improve the wettability of SiO₂ with matrix by increasing the surface energy of solid, decreasing surface tension of liquid, and decreasing the particle/alloy interfacial energy. Besides, this element increases the interface bonding strength^{78,80,144}.

No interfacial products are reported involving Cu, but only the precipitation of Cu-rich phases such as Al₂Cu and Al₂CuMg, according to the composition of the matrices^{87,134}. This shows that Cu presents a reactivity with this reinforcement even lower than the observed for Al₂O₃. Nevertheless, more research is needed to support this asseveration, because as was above mentioned Al-SiO₂ composites have been barely used.

3.3. TiO₂

Titanium dioxide (titanium (IV) oxide, titania, TiO₂) has high tensile strength, impact strength and hardness, being used for reinforcing AMC in automotive applications²⁷. Almost all the works found in literature mention the use of this ceramic combined with other reinforcements such as SiC¹⁴⁵. Otherwise, the preferred manufacturing process is powder metallurgy¹⁴⁶, with some articles including the TiO₂ addition using welding¹⁴⁷ or spray formed, as the case of Al-2Mg-TiO₂ composite studied by Chaudhury and Panigrahi¹⁴⁸, who found good interfacial bonding. Venugopal and Karikalan¹⁴⁵ used stir casting to obtain hybrid metal matrix composites AA6061-TiO₂-SiC and also reported good interfacial strength, but did not make an in depth study of the interfaces. The most important reported reaction for this system is¹⁴⁹⁻¹⁵¹:



This reaction it is reported to occur by the extraction of oxygen anions from TiO₂¹⁵². The most commonly obtained intermetallic Ti_xAl_y phases are Ti₃Al, TiAl and TiAl₃; while titanium Ti_mO_n oxides include Ti₂O, TiO, Ti₂O₃ and Ti₃O₅. Saboori et al.¹⁴⁹ found that between 800 and 1000 °C for a 6060 Al alloy (max. 0.5Mg and 0.6Si) the final products of this reaction also were TiAl₃ and α-Al₂O₃, while Shin et al.¹⁵³ reported that the formation of this interfacial layer has beneficial effects on the composite strength. These authors studied the evolution of the interfacial layer and the formation of reaction products due to the reaction of Equation 16, as can be seen in the schematic representation in Figure 3a-d for the morphological variations of TiO₂, resulting in the

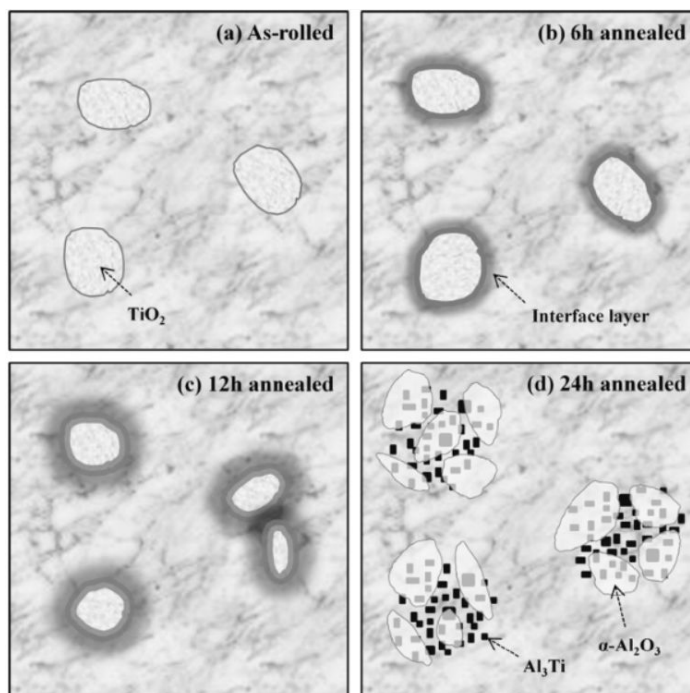


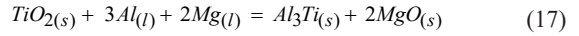
Figure 3. Schematic representation of the morphological variations of TiO₂ during annealing at 500 °C for an Al-TiO₂ composite. (a) aluminum matrix with a clearly observed interface. (b,c) O and Ti atoms decomposed from the TiO₂ diffused out to the matrix, enlarging the interfacial layer and decreasing TiO₂ size. (d) α-Al₂O₃ and Al₃Ti obtained as final reaction products. (Reproduced with permission from¹⁵³).

formation of new reinforcements¹⁵³. They found that this reaction can occur at temperatures as low as the annealing temperature (~ 500 °C).

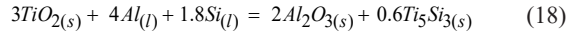
Part of the above commented reaction can be observed in the TEM images of Figure 4a-d. This work of Chao et al.¹⁵⁴ showed that Al and TiO₂ (25% vol.) reacted during hot press sintering at 933 K, obtaining a composite Al₃Ti+Al₂O₃/Al. These authors obtained Al₃Ti of two sizes: micrometric and nanometric (~ 100 nm), as can be seen denoted as A and B in Figures 4a and 4b, respectively. The formation of these particles indicates that Al₃Ti presented nucleation, growth and coarsening. The electron diffraction pattern included in Figure 4a corroborated the formation of Al₃Ti. On the other hand, Figure 4c shows the interface Al-Al₃Ti, which is clean and without the formation of precipitates or other reaction products. This kind of well bonded interface favors strength and ductility. Finally, Figure 4d shows the formation of α -Al₂O₃ particles of ~ 100 nm in size, another reaction product of Equation 16. XRD studies of these authors showed that part of the TiO₂ still remained in the final composite, completing the observed in Equation 16.

The reduction of TiO₂ to Ti was found by Ghanaraja et al.¹⁵⁵ for a composite with TiO₂ contents from 3 to 12 wt.% manufacturing by stir casting at 900 °C. These authors added 2% Mg to Al for increasing wettability of the reinforcement. They found that the resulting composite was Al (Mg, Ti)-Al₂O₃ (TiO₂), starting from a matrix of Al-Mg-Ti alloy reinforced with oxide particles consisting of un-reacted TiO₂ and Al₂O₃. Other expected phases due to this reaction are MgAl₂O₄, MgO, MgTi₂O₄ and Al₂TiO₅, which act as reinforcements

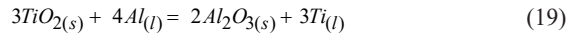
in the matrix. In presence of Mg the following reaction is reported for the formation of MgO¹⁵⁶:



The Al-TiO₂ system reaction has been used for the in-situ fabrication by powder metallurgy of Al₃Ti and Al₂O₃ particles¹⁵⁴. The use of an Al-Si alloy modified the reaction products, being them Ti₅Si₃ besides Al₂O₃¹⁵⁷:



These products can be also obtained by two reactions:



Otherwise, for Al alloys with Cu it has been reported the presence of Al₅CuTi₂, Al₄Cu₉ and Al₃Ti as reaction products¹⁵⁸. This shows that as was observed for Al₂O₃, TiO₂ also presents reaction products with Cu, but works reported in literature are few. Again, more research is needed for the interaction of this alloying element with oxide reinforcements in Al matrix composites.

3.4. ZrO₂

Zirconium dioxide (Zirconia, ZrO₂) is considered one of the most important ceramic materials for manufacturing MMC, although its density is considerably high (5.68 gcm⁻³). It presents high flexural strength, good fracture toughness and

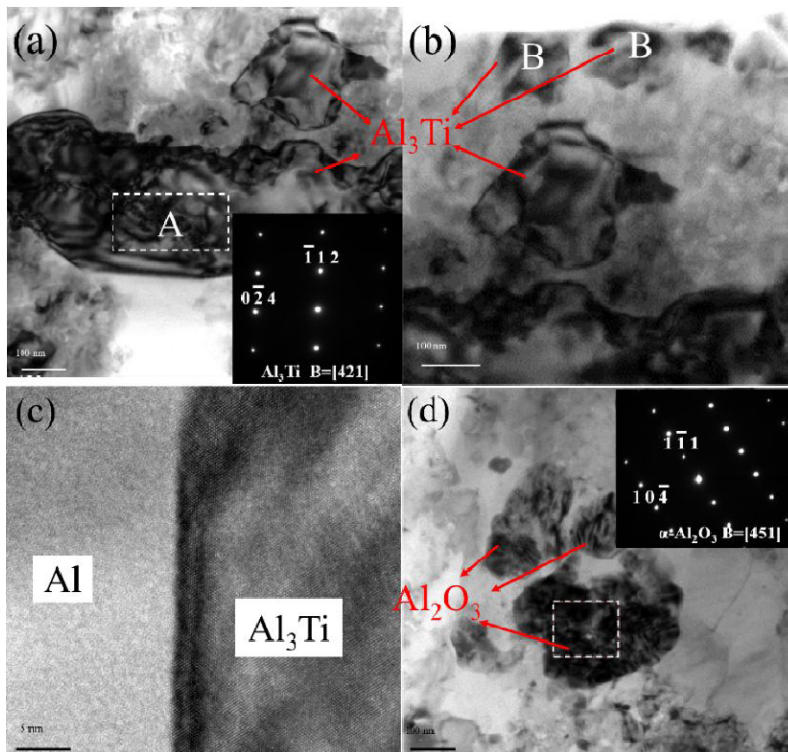
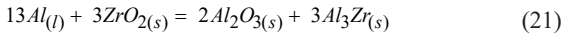
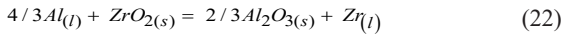


Figure 4. TEM images of: (a) Al₃Ti and its SAD pattern of region A, (b) Al₃Ti with nanometric size (B), (c) HRTEM image of the interface Al-Al₃Ti, and (d) α -Al₂O₃ particles of nanometric size and the corresponding SAD pattern. (Reproduced with permission from¹⁵⁴).

stability at high temperature. Also has wear and corrosion resistance, reinforcing Al alloys in applications such as abrasives, support material for catalysis, ceramic tubes, connecting rods, and thermal insulations^{27,159}. Different reactions are reported between zirconia and Al. Zhu et al.¹⁶⁰ reported that at 750 °C Al_3Zr phase could be formed through the following exothermic reaction:



A similar reaction has been reported at temperatures higher than 800 °C, but without the formation of Al_3Zr ¹⁶¹:



Rostami and Tajall¹⁶² also reported the formation of Al_3Zr in an Al-9.7Si-2.1Cu-1.0Mg alloy reinforced with ZrO_2 and Al_2O_3 , obtained by stir melting at 750 °C. These authors revealed that Al_3Zr remained undissolved even at elevated temperatures, and its presence enhanced the wettability of Al_2O_3 particles obtained through Equation 22, which also reinforce the material. Besides, the presence of Al_3Zr reduced porosity and led to increase tensile and yield strengths. These authors also identified precipitates as $(Al,Si)_3Zr$. No other precipitates or interfacial products were reported although Mg and Cu are present in the matrix, only the precipitates which are characteristics for this alloy system (i.e. Al_2Cu , $Al_3Cu_2Mg_8Si_6$, Mg_2Si). Baghchesara et al.¹⁶³ found Si nucleation on the surface of ZrO_2 particles for Al-7.23Si alloys reinforced with 5, 10 and 15 vol. % ZrO_2 using stir casting at 950 °C, but Si-rich products were not detected. These authors agree in the fact that Al_3Zr acts as hard pinning

points in the matrix inhibiting dislocation motion, affecting the strength of the composite¹⁶⁴. Moya et al.¹⁶¹ also studied an Al-5.2Si-3.5Cu-0.5Mg alloy, but the presence of Cu and Mg did not cause the formation of other phases.

Related to microstructural modifications, Sharma et al.¹⁶⁵ studied the effect of ZrO_2 nanoparticles added into a molten Al-12Si-20Cu alloy at 750 °C, and homogenized at 1100 °C. They found that the addition of ZrO_2 led to affect the morphology of the second phases present in the alloy, but did not cause the formation of Zr-rich phases. Second phases were Si particles, Al_2Cu , and eutectics of Al-Si, Al-Cu, and Al-Si-Cu. Daoud et al.¹⁶⁶ also found an important modification of the microstructure due to the addition of 5% ZrO_2 particles with irregular shapes, as it can be observed in Figure 5a,b. This figure shows that silicon phase in the matrix of the composites (Figure 5b) is significantly finer than that of the unreinforced alloy. Khalili et al.⁸¹ studied an Al-1Mg-0.55Si-0.26Cu (in wt.%) alloy reinforced with 3 and 6% of 30 μm in diameter ZrO_2 particles, stir melted at 700 °C. They found that Mg acted as a surface-active agent, increasing the wettability of the ZrO_2 particles. Besides reported the presence of Mg_2Si , Al_8Fe_2Si , CuO, and Al_2O_3 , but no chemical reactions matrix- ZrO_2 to originate new phases. As can be observed in Figure 5c-e, the addition of ZrO_2 particles to this alloy led to significantly decrease the grain size, attributed to the presence of ZrO_2 as nucleation sites. It could be thought that the increase in the ZrO_2 percentage from 3 to 6 wt.% would originate a reduction in the grain size, but its average increased from 62 to 93 μm due to the agglomeration of the ZrO_2 particles, decreasing the surface

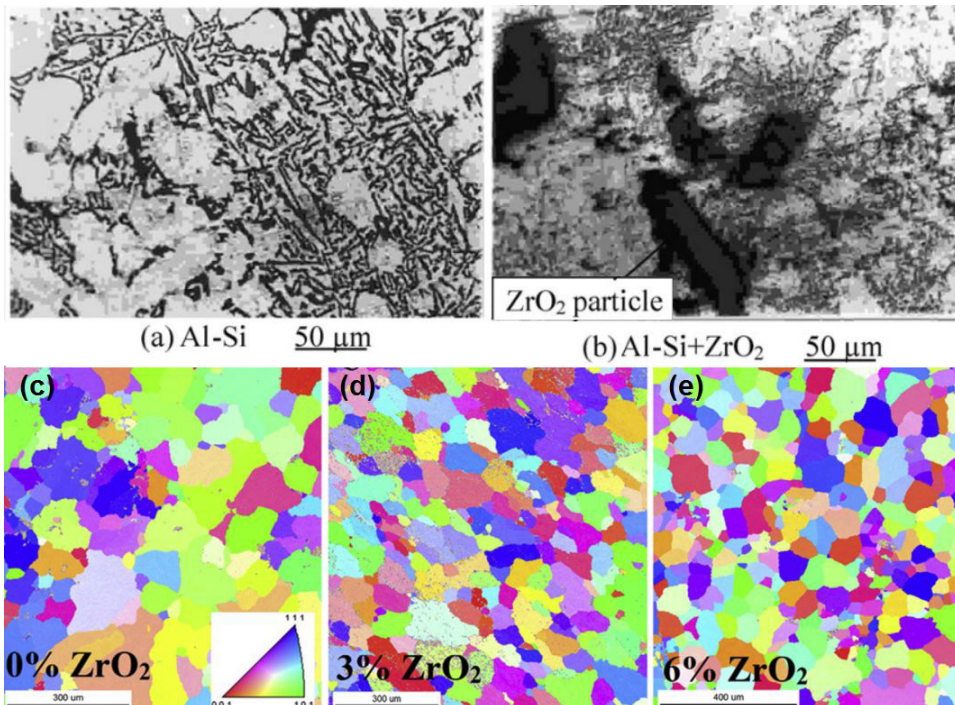
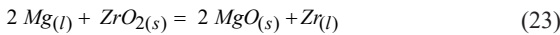


Figure 5. OM images of the microstructures for an Al-Si alloy (a), and for the same alloy reinforced with 5% of ZrO_2 particles (b). (Reproduced with permission from¹⁶⁶). (c-e) Inverse pole maps for: c) unreinforced 6061 alloy, d) 6061 alloy reinforced with 3 wt.% ZrO_2 , and e) 6061 alloy reinforced with 6 wt.% ZrO_2 . (Reproduced with permission from⁸¹).

area of the particles. Agglomeration is derived from the increase in the viscosity of the melt, also originating pores and other detrimental defects for the mechanical properties. These results show that care must be taken in the correct selection of the size and percentage of the reinforcement.

Although the above presented works did not show reactions between Mg and ZrO_2 , Liu et al.¹⁶⁷ reported the formation of other phases with the presence of Mg. They studied a $ZrO_2/Al-10.8Mg$ composite manufactured at 910 °C, and found an interfacial reaction which led to the formation of Al_3Zr , $Zr_{0.875}Mg_{0.125}O_{1.875}$ and $Al_{0.1}Zr_{0.9}O_{1.95}$, being the Mg content the highest among all the elements present at the interface. This can be observed in the SEM image and EDS mappings of Figure 6, where a ZrO_2 particle is surrounded by a reaction layer rich in Mg and O, demonstrating the formation of MgO according to the following reaction due to the high Mg content in the alloy:



The formation of polygonal Al_3Zr in the outer area of the reaction layer is also corroborated in Figure 6¹⁶⁷. This reaction product is obtained through the following reaction, starting from Equation 23:



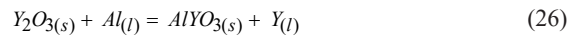
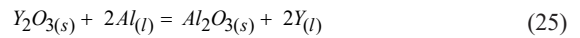
Other reactions products have been reported for composites obtained with alloys presenting high Mg and Si contents. Guo et al.¹⁶⁸ found the presence of different Zr-rich phases during the pressureless infiltration of 30 vol.% of mixtures $Al_2O_3-ZrO_2$ of weight ratios 1:9, 3:7, 5:5, 7:3 and 9:1 with an Al-12Si-10Mg alloy. They observed multiple chemical

reactions, being the main reaction products $(Al_{1-m}Si_m)_3Zr$, Al_2O_3 and $ZrSi_2$. On the other hand, Gao et al.¹⁶⁹ reported that with the increase of Si in an Al-Si-Zr alloy, the primary phase changed from Al_3Zr to (Al,Zr,Si) and further to $ZrSi_2$. These phases increased the compressive strength but decreased toughness.

The above presented results show that the addition of Cu to Al alloys reinforced with ZrO_2 does not cause the formation of any reaction product, while Si and Mg additions lead to reactions involving these elements, obtaining a wide variety of reaction products. Although these reactions are generally reported as beneficial for the mechanical properties of the composites, works found in literature for Al matrices with Si or Mg are relatively few, even for the case of Mg. This element as wetting improver and its reactions with the reinforcements presented in the previous sections were more reported than for ZrO_2 .

3.5. Y_2O_3

Yttrium oxide (yttria, Y_2O_3), exhibits remarkable stability against liquid aluminum. Nevertheless, different research works report the formation of reaction products through the following reactions^{128,170,171}:



Examples of the formation of $YAlO_3$ due to reaction of Equation 26 were presented in the works of Barzilai et al.^{170,171} for the study of the metal- Y_2O_3 interface at 1423 K, using Al, Al-Cu and Al-Cu-Y matrices. They reported that Y_2O_3 decomposed and Y was transferred into the molten metal,

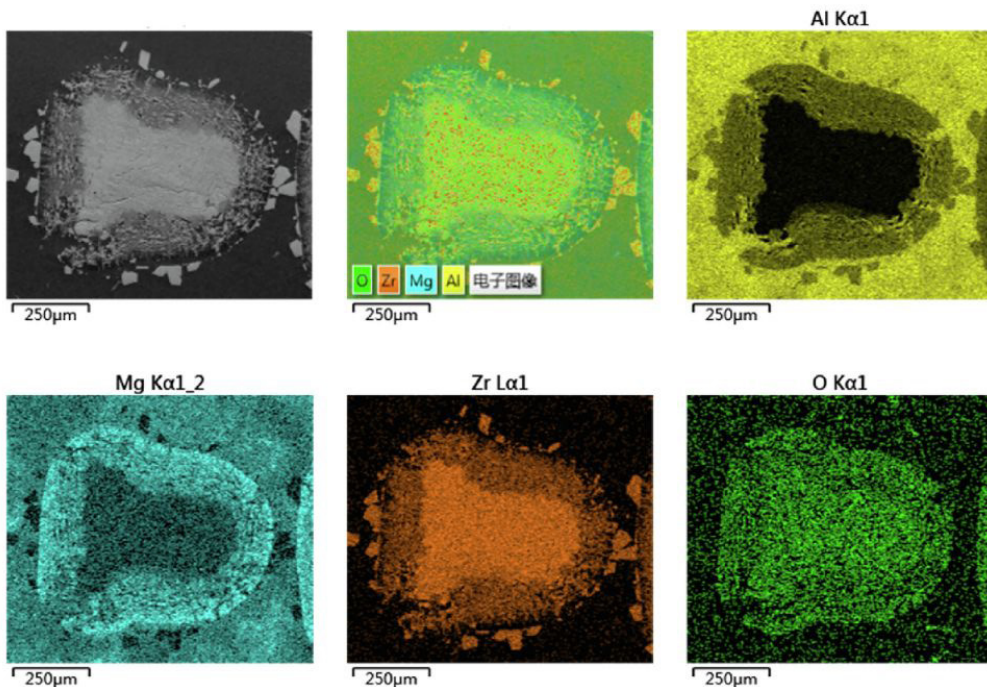
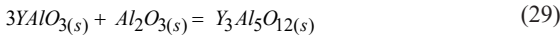
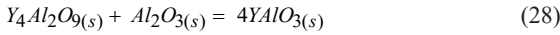
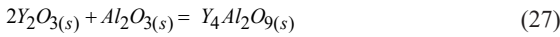


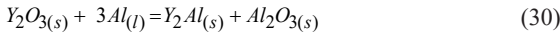
Figure 6. SEM image of a ZrO_2 block-like phase and EDS mappings showing the presence of a MgO layer and Al_3Zr particles in the outer region. (Reproduced with permission from¹⁶⁷).

depending the reaction extension on the alloying elements content. If Y content in the alloy is high (>10 at.%) the formation of $AlYO_3$ decreased while high Cu contents (>10 at.%) decreased wettability and $AlYO_3$ formation. This can be observed in Figure 7a for an Al pure matrix, where Y_2O_3 almost disappeared due to the formation of $AlYO_3$. The addition of 15 at. % Y led to a decrease in the thickness of the $AlYO_3$ layer (Figure 7b), while an Y content of 75% completely avoided its formation, being obtained Y_2O_3 . This oxide is different from the initial Y_2O_3 due to stoichiometric changes¹⁷¹. These results show that the interactions between matrices and reinforcements are complex, not only obtaining different reaction products but also modifying the starting reinforcements without the detection of new products, as the case of the stoichiometric modification of Y_2O_3 .

For the Al-yttria system it has been also reported that when Al oxidizes according to Equation 25 and 26, three different reactions can occur, which are¹⁷²:



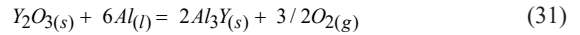
Otherwise, Al_2Y as reaction product was reported by Yu et al.¹⁷³ due to the interfacial reaction in a 6061Al alloy reinforced with Al_2O_3 particles coated with Y_2O_3 . This product, different of the above mentioned, improved wettability and bonding strength of the interface, leading to better mechanical properties. Al_2Y was originated due to the reaction between Al liquid and Y_2O_3 :



Kim et al.¹⁷⁴ analyzed the effect of adding Y_2O_3 to pure aluminum (99% purity) inside the mold at 900 °C, and found that Y_2O_3 nanoparticles were uniformly distributed in the matrix, not reporting particles degradation or reactions with molten Al. The presence of 2% of Y_2O_3 led to increase hardness by 1.2 times and tensile strength by 1.55 times. Zhang et al.¹⁷⁵ used 1% of Y_2O_3 for reinforcing pure Al, manufacturing the composite by conventional casting, and

showing that the dispersed yttria particles are effective in enhancing both the mechanical properties and corrosion resistance of the Al matrix.

The presence of alloying elements can lead to the formation of different interfacial compounds. Moussa et al.¹⁷⁶ reported another reaction for an Al-7.36Si-0.23Mg-0.103Cu alloy at which was added Al-30wt.% Y_2O_3 powder when melt reached 750 °C. This led to the formation of Al_3Y , which segregated at the edge of eutectic Si restricting its growth and leading to its modification. These authors did not report the formation of Y_2SiO_5 , but the EDS presented in their research could indicate its formation. Bouaeshi and Li¹⁷⁷ also used premixed powder of Al- Y_2O_3 followed by melting in an arc-melting furnace. Using XRD they demonstrated the formation of Al_3Y , while this technique and SEM/EDS examinations showed the absence of Y_2O_3 . This suggests that this phase completely decomposed due to the high temperature (up to 3700 °C), which is higher than the melting point of yttria. Hardness, mechanical and electrochemical properties of Al were improved due to the Al_3Y phase, residual yttrium in Al matrix and finer microstructure. Figure 8a-b presents backscattered SEM images of these composites studied by Bouaeshi and Li¹⁷⁷, where microstructures consisted of aluminum dendrites and Y-rich eutectic domains. As can be seen, the increase in the Y_2O_3 addition made the microstructure finer. Besides, Y_2O_3 melted or decomposed during the manufacturing process due to the high temperature, forming Al_3Y according to Equation 26:



Al_3Y particles can be better observed in the SEM image of Figure 8c arrowed B (at higher magnifications). The XRD diffractogram of Figure 8d shows the increase in the intensity of the peaks for this phase with the increase in the Y_2O_3 quantity used in the manufacturing process, and the absence of Y_2O_3 due to the reaction of Equation 25.

Oliveira et al.¹⁷⁸ studied the interaction of a sintered mixture of various ceramics ($90Si_3N_4-5Y_2O_3-5Al_2O_3$) with molten Al, and found that sintering originated the formation of Y_2SiO_5 . Then, it could be thought that if Y_2O_3 is in contact with a molten Si-rich Al alloy this compound could be also formed. This could confirm the above commented

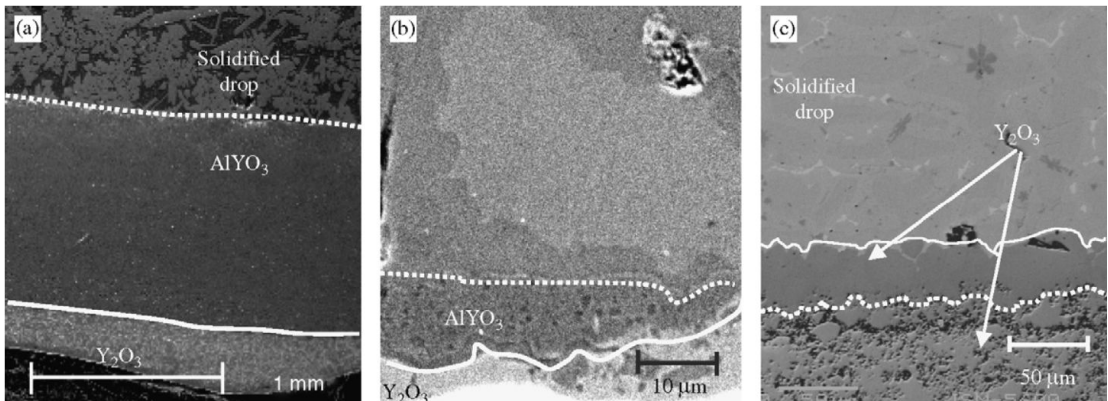


Figure 7. SEM images for Y_2O_3 /Al matrix interfaces for: (a) Pure Al matrix, (b) Al-15Y matrix, and (c) Al-75Y matrix. (Reproduced with permission from¹⁷¹).

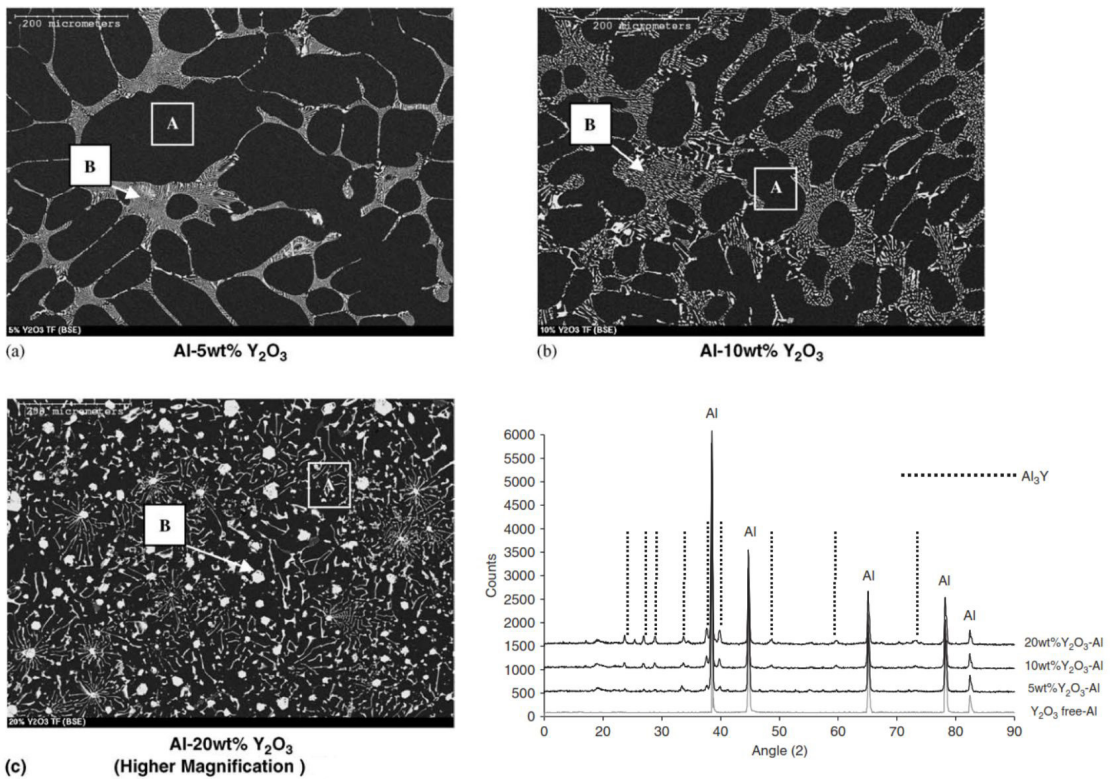


Figure 8. SEM images of Al- Y_2O_3 composites with: a) 5 wt.% Y_2O_3 , b) 10 wt.% Y_2O_3 , and 15wt.% Y_2O_3 , (d) XRD diffractograms of unreinforced Al and with different contents of Y_2O_3 . (Reproduced with permissions from¹⁷⁷).

report about EDS findings in the work of Moussa et al.¹⁷⁶, and shows that research about this topic is insufficient and needs more attention.

Corrosion of composite materials quite often begins with the reaction of the reinforcement material, being in general the composites more susceptible to corrosion attack than the matrix alloy. Nevertheless, Anae¹⁷⁹ found that the corrosion resistance of an Al-Si-Cu alloy reinforced with 1% of Y_2O_3 is higher than the unreinforced alloy, although if Y_2O_3 content is increased the corrosion resistance is not improved. This author did not analyze the microstructure of the composites, being impossible to conclude if Y_2O_3 particles remains without significant changes or if another compound was obtained.

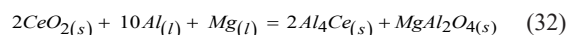
Few works were found in literature about the effect of Mg for Y_2O_3 reinforced Al alloys, but it has been reported that Y_2O_3 exhibits better stability in molten magnesium than other oxide reinforcements such as alumina and zirconia¹⁸⁰. Divakar et al.¹⁸¹ studied an Al-Mg alloy reinforced with 4% Y_2O_3 processed at 600 °C, and found a relatively uniform distribution of reinforced particles. Y_2O_3 presented good interfacial integrity without any damage. These particles significantly increased hardness and tensile strength of the composite.

The above presented results show that the information related to the effect of Si, Cu and Mg on possible reactions of Al- Y_2O_3 composites is limited. From the works about this system found in literature it can be concluded that Y_2O_3 is

less reactive than other oxide reinforcements, being only reported reaction products rich in Si. Although Cu and/or Mg additions did not lead to the formation of interfaces they could affect wettability and the distribution of the reinforcements.

3.6. CeO_2

Cerium Oxide (Ceria, CeO_2) has high stability at elevated temperatures, good behavior against mechanical abrasion and is a cathodic inhibitor. Nevertheless, its study as reinforcement of Al alloys has been limited¹⁸², being used mainly as coating, similar to the case of BN¹⁸³. Skrzekut et al.^{184,185} studied mechanical alloying of Al with 9.2% of CeO_2 particles at annealing temperature ≤ 773 K, reporting high thermal stability of CeO_2 . For an Al-4.88Mg matrix they detected the decomposition of CeO_2 particles forming Al_4Ce and Al-Mg rich oxides as reaction products. This reaction did not occur for the system Al- CeO_2 , but the addition of Mg led to the reduction of CeO_2 like for other MeO particles resulting in Al_xMe_y (Me=metal). The spinel $Mg_{0.4}Al_{0.6}Al_{1.8}O_{0.4}$ was also reported in this work. This reaction could be deduced from similar reactions in systems such as AlMg-SiO₂ or AlMg-TiO₂, being as follows:



Example of the formation of Al_4Ce can be observed in Figure 9a-b from the works of Skrzekut et al.^{184,185}, were the TEM images and the SAD (Selected Area Diffraction) patterns

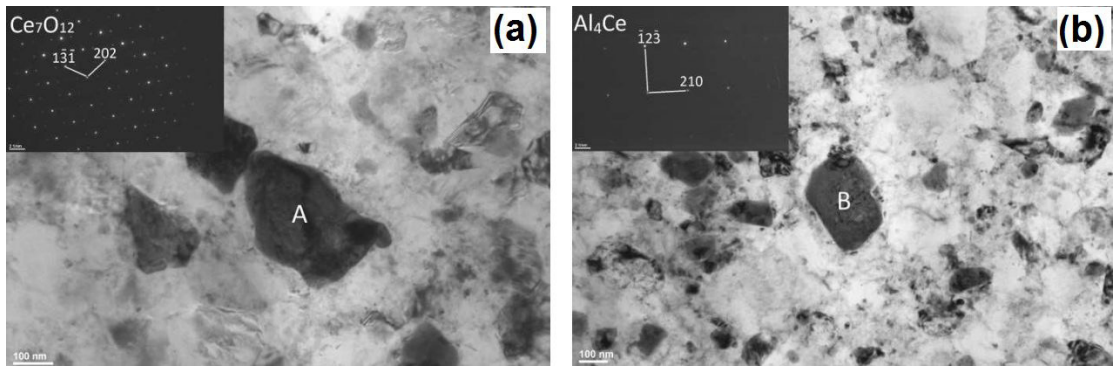


Figure 9. TEM images of an AlMg-CeO₂ composite where the presence of Ce₇O₁₂ and Al₄Ce nanoparticles can be observed, corroborated by their SADPs. (Reproduced with permissions from¹⁸⁴).

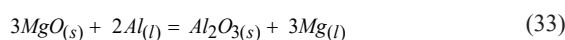
reveal the presence of Ce₇O₁₂ as an intermediate product of the reduction of CeO₂ (Figure 9a). The end of this reaction led to the nucleation and growth of Al₄Ce (Figure 9b). The use of these techniques revealed the similarities of these particles, being necessary this kind of studies for their identification. This reaction took place during mechanical alloying at 673 K, but the presence of Al₄Ce significantly increased when the composite was re-melted at 973 K, almost completely decomposing CeO₂. This was corroborated by XRD studies, which also revealed that MgAl₂O₄ peaks were less intense, meaning that after the complete reaction instead of Equation 32 the formation of small quantities of oxides such as MgO or Al₂O₃ could occur. These authors mentioned that other phases such as CeO_{1.66} and Ce₄O₇ could be also present after re-melting.

Xue et al.⁵⁶ reported the formation of AlSiCe and Al₄Ce for an Al-6.97Si-0.36Mg alloy reinforced with 1% CeO₂. AlSiCe had needle-like or block irregular morphologies and bad bonding with the matrix, which decreased the mechanical properties. The formation of AlSiCe originated the decrease in the concentration of Ce in grain and interdendritic boundaries, also leading to modify eutectic Si.

The bibliographic search of possible reactions between CeO₂ and aluminum alloys revealed that reaction products were only obtained when Si or Mg were present, while there were not found reports of reactions for pure Al matrices. The effect of Cu on the matrix-reinforcement interactions for these composites was also not found. This shows again, as for Al-Y₂O₃ composites, the limited information related to possible interfacial reactions in these systems, being necessary new research about this important topic. This lack of information is even more critical for the case of CeO₂.

3.7. MgO

Magnesium oxide (MgO, magnesia) has a density of 3.58 gcm⁻³ and a melting point of 2800°C. It also has elevated Young Modulus (320 GPa), compressive strength and hardness. Also present excellent thermodynamic stability and high wettability with aluminum¹⁸⁶. Al-MgO composites are used where light weight and high strength-to-weight are needed¹⁸⁷. For the case of the interaction between molten Al and MgO, the following reaction has been reported^{128,188}:



As was already observed for the additions of Si to Al alloys reinforced with SiO₂, and Y to alloys reinforced with Y₂O₃, adding to the matrix high contents of the same alloying element which constitutes the oxide reinforcement allows to avoid or diminish the formation of interfacial reactions. For example, Mohammed and Gamal¹⁸⁹ reported the absence of any intermetallic formation in an Al1100 alloy reinforced with 0-4 wt.% of MgO casting at 750 °C. Yar et al.¹⁹⁰ found that the addition of 1.5 wt.% of MgO particles to an Al-7.23Si-0.32Fe-0.18Cu-0.38Mg A356.1 alloy manufactured at 850 °C led to improve properties such as hardness, strength and toughness. For these conditions, no reactions products were observed. Lin et al.¹⁹¹ also found that the use of MgO as coating avoided the interfacial reaction between an Al-6Mg-0.4Si-0.1Cu alloy and glass microspheres (SiO₂) used in syntactic foams manufactured at 850 °C. These authors reported a large amount of MgAl₂O₄, Si and Mg₂Si at the interfaces when there were used spheres without MgO coating. Calin and Citak¹⁹² studied an Al matrix with 0, 1, 2, 3 and 4% Mg reinforced with MgO by infiltration, and reported that when Mg content and temperature of matrix increased the infiltration process was easier. They found the formation of the spinel MgAl₂O₄ phase, which facilitated the infiltration of Al. The formation of this phase could be directly from the melt due to the reaction between Al, Mg and O₂; or from the interaction MgO-molten alloy. Sun et al.¹⁹³ used TEM to study the interactions between a MgO substrate and molten Al, and found the formation of an intermediate layer of and MgAl₂O₄, as can be observed in Figure 10a. Figures 10b, c show the HRTEM analysis of this layer along the [001] zone axis of MgO, where the lattice structure defined by different planes can be observed. Fast Fourier Transformation (FFT) analysis corroborated that the intermediate layer is MgAl₂O₄, with a face-centered cubic (FCC) structure corresponding to the planes (400) and (220). These images reveal that orientation relationships between phases are [001] (400) MgAl₂O₄// [001] (200) MgO and [001] (220) MgAl₂O₄// [001] (220) Al.

For Al matrices with the presence of Si it is reported that the reaction with MgO could lead to the formation of MgAl₂O₄ and Mg₂SiO₄, although only it is possible for very high Si contents (mass ratios of MgO:Al:Si=70:15:15) and high mixture temperatures (1450 °C)¹⁹⁴. The formation of

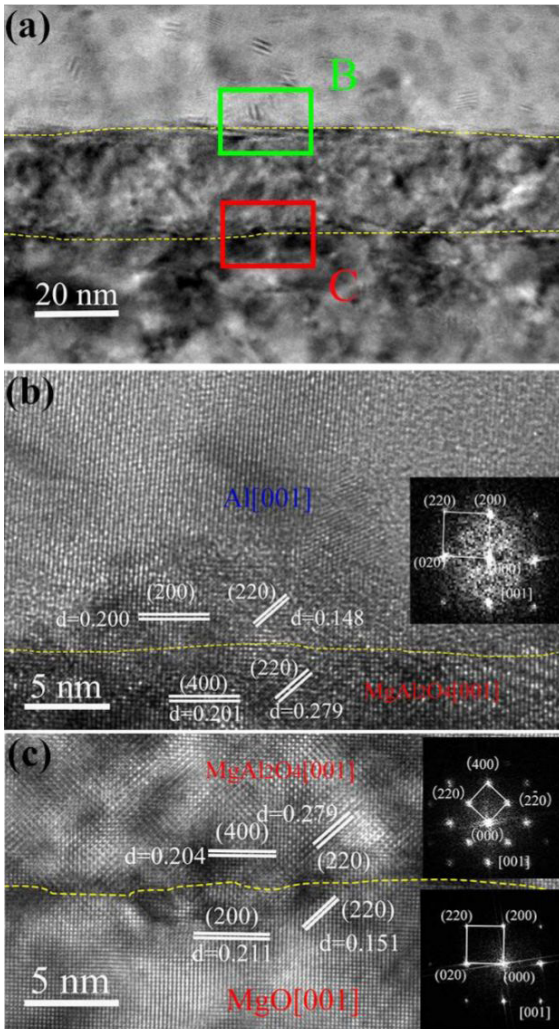


Figure 10. (a) TEM image of an Al(up)/MgO(down) composite with an MgAl₂O₄ interfacial layer. (b) HRTEM image of the Al/MgAl₂O₄ interface, corresponding to frame (B). (c) HRTEM image of the MgAl₂O₄/MgO interface, corresponding to frame (C). FFTs are included for each phase. (Reproduced with permissions from¹⁹⁴).

intermetallics between MgO and Cu or the effect of this element was not found for these composites.

As can be seen for composites of the system MgO-Al alloys the most important and studied alloying element is Mg, in this case due to its influence avoiding interfacial reactions.

4. Summary

After the analysis of the interactions between oxide reinforcements and Al matrices with Si, Cu and/or Mg as alloying elements, it can be concluded that research in literature about this topic is insufficient. It is generally accepted that the increase in the reinforcement volume fraction modifies the microstructure and decreases the grain size. Nevertheless, it is essential an adequate manufacturing process in order to avoid reinforcement agglomerations that could avoid grain refinement. Related to interfacial reactions, the conditions for their occurrence are different and highly dependent on

temperature and composition of the matrix. It is necessary an individual study of each combination reinforcement-matrix, mainly including the alloying elements content. For some oxides used as reinforcements such as Al₂O₃ and SiO₂ the reactions and interactions are well studied, but for others the information is limited or inexistent, as the case of Ce₂O₃ and Y₂O₃. Interfacial reactions could lead to the diffusion of elements, which may decrease the precipitation in the region near the interface if the alloying element segregates to the interface; or generate new precipitates if an element diffuses from the reinforcement to the matrix. In general, Mg is the most studied element, being also who has the highest effect on interfacial reactions. This could be attributed to the high reactivity of this element and to its role as wettability improver when is added to Al matrices. Among the reactions products, spinel MgAl₂O₄ phase was the most reported because it is present for all the oxides except ZrO₂ and Y₂O₃. The presence of Si in interfacial products was lower, but this element was found in all the oxides forming interfacial compounds or precipitates. On the other hand, the effect of Cu was barely found in literature, with few reaction products, only for Al₂O₃ and TiO₂. Besides, it was found that an effective way for minimizing the reduction reaction of an oxide reinforcement is adding to the alloy the same element of the oxide.

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