



HAL
open science

Microstructural analysis of the reinforced Al-Cu5mgTi/TiB₂ 5 wt% Alloy for Investment casting applications

Pedro Egizabal, Maider García de Cortázar, Joël Douin, Amaia Torregaray,
Michel Lahaye, Cécil Vincent, Jean-François Silvain

► **To cite this version:**

Pedro Egizabal, Maider García de Cortázar, Joël Douin, Amaia Torregaray, Michel Lahaye, et al.. Microstructural analysis of the reinforced Al-Cu5mgTi/TiB₂ 5 wt% Alloy for Investment casting applications. *Advanced Engineering Materials*, 2010, 12 (6), pp.504-508. 10.1002/adem.201000031 . hal-00505784

HAL Id: hal-00505784

<https://hal.science/hal-00505784v1>

Submitted on 7 Mar 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Microstructural Analysis of the Reinforced Al-Cu5MgTi/TiB₂ 5 wt % Alloy for Investment Casting Applications**

By Pedro Egizabal, Maider García de Cortazar, Joël Douin, Amaia Torregaray, Michel Lahaye, Cécil Vincent* and Jean-François Silvain

The paper describes the influence of 5 wt % titanium diboride (TiB₂) particles on the microstructure of an Al-Cu alloy produced by plaster casting process. The elaboration route leads to a composite material with 1% of in situ TiB₂ particles and 4% ex situ of TiB₂ particles. The comparison of the reinforced alloy with the corresponding non-reinforced counterpart makes clear that the presence of TiB₂ particles has a large influence in the observed microstructure. The presence of TiB₂ particles decreases the grain sizes and the porosity level. It is also found that TiB₂ particles play an important role in the precipitation events of Al₂Cu precipitates that are formed during solidification at the TiB₂/aluminum matrix interfaces.

The Al-Cu5MgTi alloy is a heat treatable alloy that presents good machining properties. It is mainly used in aircraft parts when better mechanical properties than those provided by Al-Si alloys such as Al-Si7Mg0.3 are required. Nevertheless it is not so extensively applied as the latter due to its lower castability. Its solidification range is very large and this makes

it prone to hot tearing related problems. The Al-Cu5MgTi alloy is mainly applied when high mechanical properties are needed at high temperatures. Magnesium alloying increases the strength and hardness of this alloy through thermal treatment and titanium is used as a grain refiner so that the insoluble constituents can be better dispersed as well as the porosity and defect distribution decrease.

The use of grain refiners in these alloys is highly recommended due to the clear benefits they provide. The hot tearing related problems become reduced, mechanical properties get improved, and surface quality and the response to thermal treatments is better. Al-Ti-B type grain refiners have been used with this alloy in different foundry processes. Their use is even more essential in the case of low cooling rate processes like sand casting or lost wax processes as the large solidification intervals of the alloy (450–650 °C)^[1] tends to create problems related to hot tearing and poor mechanical properties.

TiB₂ particles incorporated in Al-Ti-B grain refiners are extensively used by aluminum foundries to promote heterogeneous nucleation and formation of equiaxed microstructure thus to improve the mechanical properties of the castings. The addition of only 0.15 wt% TiB₂ is enough to get a significant refinement of the grains.^[2,3] Even though the main effect of the addition of TiB₂ particles into aluminum alloys is the refinement of the aluminum grains, there are other additional microstructural changes such as an increase in the modulus and ductility of the base alloy as well as a change in the thermal properties that can also be explained by the presence of these particles. The alloy studied in this work contains up to 5 wt% of TiB₂ particles. This amount largely exceeds the level

[*] Dr. C. Vincent, Dr. M. Lahaye, Dr. J. F. Silvain
ICMBC-CNRS, Université Bordeaux 1
33608 Pessac, France

Dr. J. F. Silvain
ICMBC-CNRS, 87 Avenue du Docteur Albert Schweitzer
F-33608 Pessac Cedex – France
E-mail: silvain@icmcb-bordeaux.cnrs.fr

Dr. P. Egizabal, Dr. M. García de Cortazar
Fundación Inasmet
E-20009 Donostia-San Sebastián, Spain

Dr. J. Douin
CEMES – CNRS
UPR 8011, Group: Matériaux cristallins sous contrainte
(crystalline materials under strain) 31055 Toulouse, France
Dr. A. Torregaray
University of Basque Country UPV/EHU
Material Science department, E-48012, Bilbo-Bilbao

[**] Acknowledgements, This work has been partially funded by the EC FP6 project IMINCAST (project number CRAF-1999-72448) under contract number G1ST-CT-2002-50268.

necessary for grain refining with the supplementary aim to elaborate a particle reinforced aluminum matrix composites (AMC).

The influence of TiB_2 particulates on the microstructure of the Al-Cu5MgTi alloy has been analyzed and compared to the corresponding non-reinforced alloy. The microstructure of the reinforced alloy has been thoroughly studied through optical microscopy (OM), scanning electronic microscopy (SEM), and transmission electronic microscopy (TEM). The chemical analysis of the different phases has been carried out by electron probe micro analysis (EPMA) and Auger electron spectroscopy (AES). It is shown that apart from the grain refining effect the particulates play an important role in solidification events, selection of precipitating phases, and decrease of defects density.

Technical Approach

The alloy containing 5 wt% TiB_2 particles is not commercially available and has been produced by the company London & Scandinavian Co. Ltd through a proprietary process based on their technology to produce aluminum master alloys.^[4,5] The addition of two salts (K_2TiF_6 and KBF_4) to aluminum that react with each other to produce TiB_2 particles is the basis of this process. This process can directly produce up to 10 wt% particles content. However the viscosity of the produced material is high, degrading its cast properties, and the rests of the salts are very difficult to remove and affects negatively the final properties of the casting. Therefore it was decided that the reaction be adjusted so that only 1 wt% TiB_2 particles are formed in situ. Subsequently 5 wt% of commercially available TiB_2 particles of around 5 μm were added to obtain a material that could keep the sufficient castability and provide the best possible combination of mechanical and thermal properties.

The production of the samples has been carried out through the plaster casting process. The process consists of producing a replica on wax or plastic of the part to be produced. This replica or pattern is assembled with a filling and feeding system of similar material. This assembly is coated or invested with hard-setting plaster based refractory

slurry. After allowing the plaster to set, the mold is placed into a furnace at around 150–180 °C to be dewaxed. The mold is then heated or fired to burn off the residual wax and to obtain the refractory properties needed to withstand the casting step. When the firing cycle is completed molten metal is cast into the preheated mold.

The production route chosen was optimized in order to obtain sound samples with a correct and homogeneous distribution of TiB_2 particles. The casting temperature was established at 730 °C for the Al-Cu5MgTi and Al-Cu5MgTi + TiB_2 5 wt% materials. The plaster mold temperature was kept at 250 °C and the rest of parameters were kept unchanged through all the casting trials (time to fill the molds, vacuum applied during the castings and amount of plaster used to build the molds).

Subsequently both reinforced and non-reinforced samples were submitted to different microstructural and chemical analysis: SEM, TEM, AES, and EDS mapping. TEM thin films have been prepared with the FIB technology. The AES data were acquired on a VG-MICROLAB 310-F Microprobe with a lateral resolution of 20 nm. Analysis conditions were an accelerated voltage and beam current of 10 KeV and 1 nA, respectively. The energy investigated in this multiplexing technique were the following for each element: Al: 1375–1405 eV, Ti: 365–405 eV, Cu: 895–935 eV, and B: 155–195 eV.

Discussion of Results

Microstructural Analysis

Figure 1(a) and (b) present OM images of both alloys. The alloy is known to be prone to hot tearing mainly due to its large solidification interval that is further affected by the low heat dissipation capacity of the plaster mold. Its solidification interval is in the range of 560–645 °C (the freezing range is 85 °C) and the hot tearing index is between 4.3 and 4.6 for the Al-Cu5MgTi alloy.^[1,6] Micro shrinkages appear [see arrows Fig. 1(a)] that tend to reduce in the presence of the TiB_2 particulates. Furthermore the grain size decreases from 315–200 μm in the non-reinforced alloy down to 125–200 μm attesting of the grain refining effect of TiB_2 . It

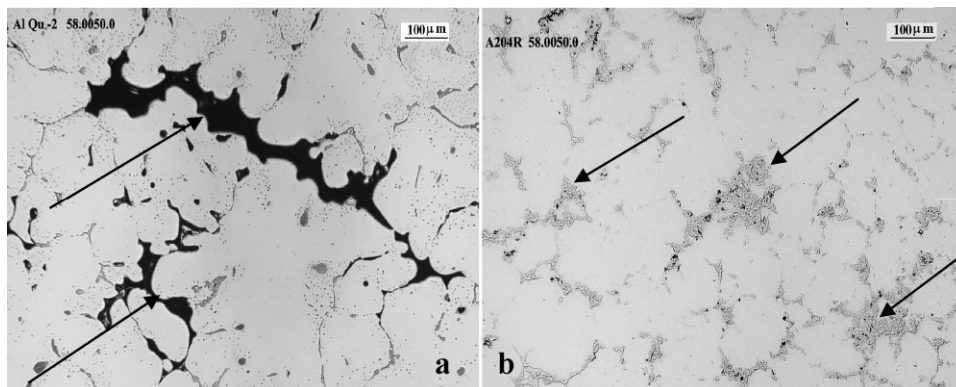


Fig. 1. Representative optical microscope of (a) the non-reinforced Al-Cu5MgTi alloy showing a region with micro shrinkages and (b) the equivalent reinforced material (Al-Cu5MgTi/ TiB_2).

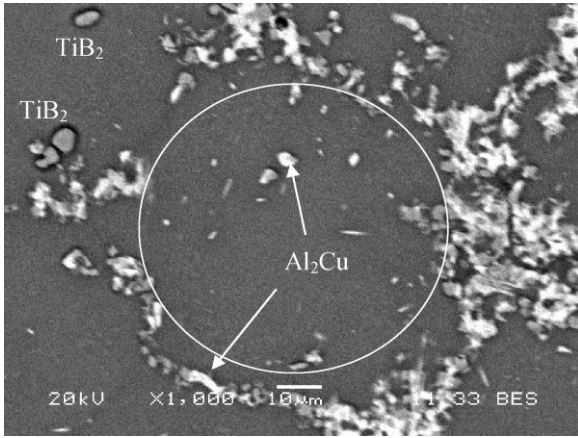


Fig. 2. Back scattered electrons (BSE) micrograph of the reinforced alloy showing an isolated α -Al grain surrounded by TiB_2 and Al_2Cu particles. Al_2Cu phases: white contrast; TiB_2 particles: grey contrast.

can also be seen that the TiB_2 particles are mainly located in the grain boundary region pushed by the solidification front [see arrows Fig. 1(b)].

Figure 2 shows a general aspect of the microstructure of the reinforced alloy. Most of the TiB_2 is found in the boundary regions together with different phases identified as $\text{Al}_7\text{Cu}_2\text{Fe}$ and Al_2CuMg needles. Al_2Cu phases (white contrast) appear sometime connected to the TiB_2 particles (grey contrast). This phenomenon can be more clearly seen in the detailed micrographs of the regions where there is an agglomeration of TiB_2 particles (See Fig. 3).

The TEM analysis of samples prepared with the focused ion beam (FIB) technology and the EDS analysis provided additional information on the interaction of the TiB_2 particles and the Al-Cu matrix.

Figure 4 shows a TEM image of an isolated TiB_2 particle embedded in the Al matrix. When observed in a $\{1213\}$ plane of the particle, the TiB_2 hexagonal particle is faceted. The facets are mainly parallel to the $\{10\bar{1}0\}$ and $\{11\bar{2}1\}$ family planes of TiB_2 . Rough interfaces indicate that some kind of diffusion between TiB_2 and the Al-matrix have also taken place during

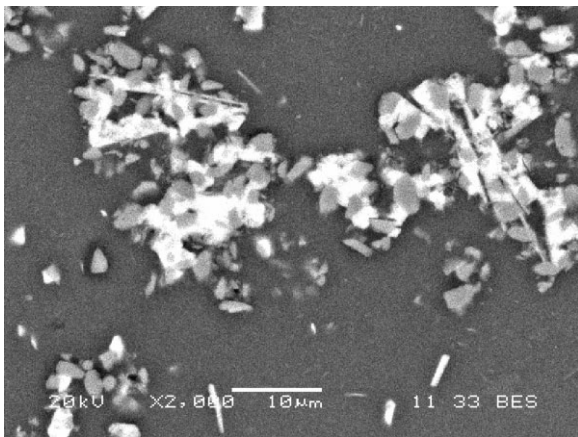


Fig. 3. BSE micrograph of a grain boundary region showing a high concentration of particles (TiB_2 particles have a grey contrast and Al_2Cu particles a white contrast).

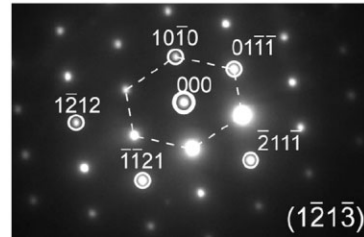
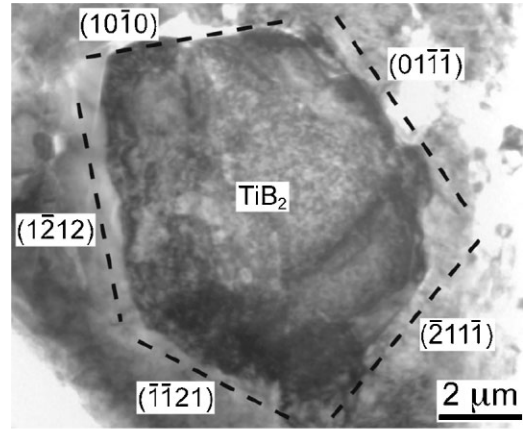


Fig. 4. TEM image of an isolated TiB_2 particle within the Al matrix of the Al-Cu5MgTi alloy.

the production process. Nevertheless the surrounding aluminum grains do not present any particular orientation relationship with the particle. This, in conjunction with the fact that the diameter of the particle is large (about $8\mu\text{m}$) indicates that it is one of the ex situ particles.

It is worth emphasizing that the density of dislocations observed by TEM in the matrix around the particle is small in spite of the CTE mismatch between TiB_2 and the matrix ($8.1 \cdot 10^{-6}$ and $25.3 \cdot 10^{-6} \text{ } ^\circ\text{C}^{-1}$, respectively). Thus the stress relaxation related to the presence of such particle within the aluminum matrix has been probably accommodated by the formation of small $\sim 1\mu\text{m}$ in diameter – Al grains in the close vicinity of the particle.

Chemical Analysis

The analysis was completed with the EDS mapping of the main elements (Al, Cu, Ti, and B). The results are shown in Figure 5. Two large TiB_2 particles, noted (1) and (2), can be clearly seen in the titanium and corresponding boron maps. It has to be noticed that the weak contrast of the boron map can be associated with the low EDS sensibility of this element. Cu segregation can be observed all around the TiB_2 particles (Cu map) and is attributed to the presence of Al_2Cu phase. Taking into account these results we can postulate that TiB_2 particles may act as nucleation sites for Al_2Cu precipitates and therefore induce the segregation of the copper element at the matrix/reinforcement interface.

In order to obtain more precise information at nanometric scale, AES line profile has been performed at the Aluminum matrix/ TiB_2 interface. The peak indentation was determined by reference to an EAS data base^[7] or from reference

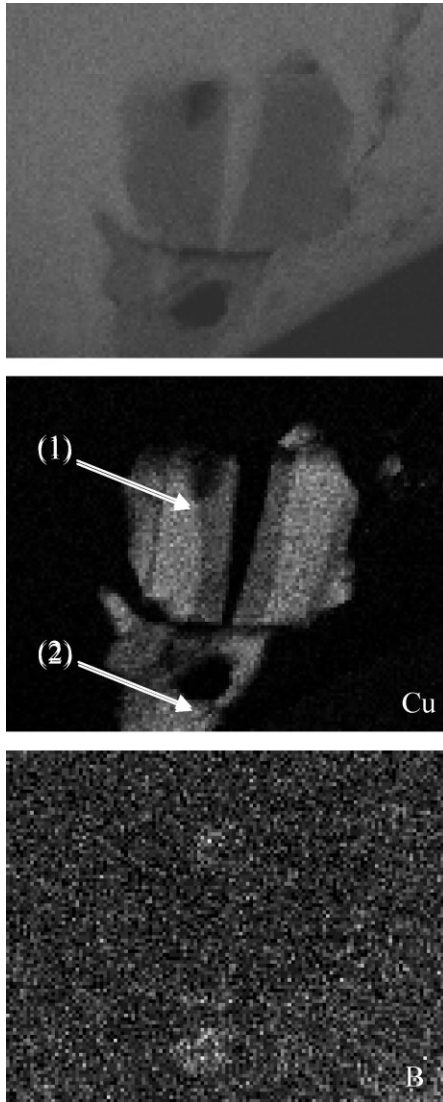


Fig. 5. EDS maps of Al, Cu, Ti, and B.

compounds analyzed in the same apparatus. The profile shows that some Al rich phases are formed in the interphase (IP) zones between the Al alloy matrix and the TiB_2 particles. It is difficult to define their composition as they may also contain some Fe, Mg, or Si (Table 1) and because of the semi quantitative level of this technique. In order to try to resolve small changes in chemical states, a special procedure was followed to analyze the Al line profile data and to plot the

Table 1. Composition of the reinforced and non-reinforced Al-Cu5MgTi alloy

Al-Cu5MgTi + TiB_2 5 wt%								
	Al	Si	Fe	Cu	Mg	Zn	Ti	B
wt%	Balance	0.08	0.11	5	0.2	<0.01	3.4	1.7
Al-Cu5MgTi								
wt%	Balance	0.10	0.10	4.94	0.3	0.01	0.15	0

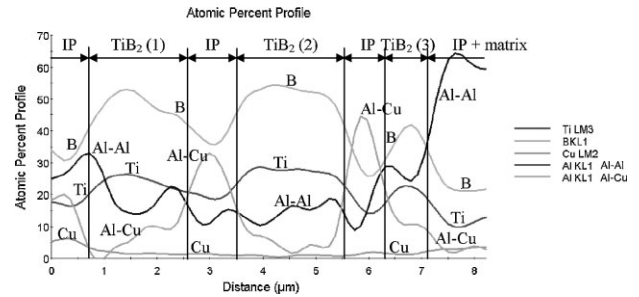


Fig. 6. Line profile obtained by applying the NLLSF software. Two different aluminum species can be appreciated. The Al linked to Cu appears to be segregated on the interface of the TiB_2 particulate.

atomic concentration of the Al element versus the point position in line, in respect to its chemical state. Data acquired at different position across the sample are computer-analyzed using a numerical method (Non linear least square fitting) applied to the matrix data (see Fig. 6).

The results presented in Figure 6 show that there are two different aluminum species related to two different chemical environments. The analysis of the resulting profile indicates that one Al KL1 peak corresponds to a simple environment (aluminum–aluminum) and a second Al KL1 peak corresponds to a more complex environment: Al–non Al (for example Al–Cu) that segregates on the interface of the TiB_2 particles. These results are in agreement with the observations of the X-ray diffraction where Al_2Cu crystals were observed in the Al-Cu7MgTi + TiB_2 material as well as the results obtained with OM and the image analysis of SEM specimens.

It has to be noticed that the absence of sharp interfaces and the presence of Al inside the TiB_2 particles is not attributed to chemical reaction but is rather associated to polishing artifact and the fact that the particles are spherical and buried into the matrix.

Conclusions

The comparison of the reinforced alloy with the corresponding non-reinforced counterpart makes clear that the presence of TiB_2 particles has a large influence in the final microstructure. This influence is not limited to the grain refining phenomena that were previously known to take place when small amounts of TiB_2 particles were incorporated as part of Al-Ti-B grain refiners.^[8,9] It has been seen that TiB_2 particles play an important role in the precipitation events of the different phases that are formed during solidification. Furthermore they have a beneficial influence on the decrease of porosity and solidification shrinkage related defects. TiB_2 particles also play a role in the selection and amount of precipitations that are formed. The Al_2Cu phase precipitates on the surface of the TiB_2 particles. The mapping of the elements in the samples has demonstrated that Al_2Cu precipitates appear associated with the TiB_2 particles. This has a direct influence on the mechanical properties as it is

known that the size and distribution of this phase (Al_2Cu) partly control the mechanical properties as they impinge dislocations avoiding their movement and increase in the strength of the material.

-
- [1] P. Grandier Vazeille, J. Morice, *Rev. Aluminum Février* **1971**, 177.
- [2] A. Hardman, F. H. Hayes, *Mater. Sci. Forum* **1996**, 217, 247.
- [3] P. Schumacher, A. L. Greer, J. Worth, P. V. Evans, M. A. Kearns, P. Fisher, A. H. Green, *Mater. Sci. Technol.* **1998**, 14, 394.
- [4] S. C. Tjong, Z. Y. Ma, *Mater. Sci. Eng.* **2000**, 3, 15.
- [5] R. Taghiabadi, M. Mahmoudi, M. Emamy Ghomy, J. Campbell, *Mater. Sci. Technol.* **2003**, 19, 45.
- [6] P. R. Beeley, R. F. Smart, *Investment Casting, 1st ed.*, The Institute of Materials, London **1995**, ISBN 09 0171666 9,262.
- [7] *Handbook of Auger Electron Spectroscopy*, Physical Electronic Publisher, Eden Prairie, USA **1978**.
- [8] L. Greer, P. S. Cooper, M. W. Meredith, W. Schneider, P. Schumacher, J. A. Spittle, A. Tronche, *Adv. Eng. Mater.* **2003**, 5, 81.
- [9] M. Braccini, M. Suery, C. Laguerre, M. Stucky, *Rev. Métall.-Cah. Inf. Tech.* **2003**, 100, 157.
-