ESRF	Experiment title: **Holotomographic analysis of the SiC reinforcement distribution in an Al matrix functionally graded composite fabricated by centrifugal casting.**	**Experiment number**: ME-461
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Report:

Al-matrix composites reinforced with SiC particles have attracted a growing interest from the automotive, aeronautical and aerospace industry, due to their advantageous toughness to weight ratios, combined with the possibility of employing conventional casting technologies. Furthermore, through an adequate control of the ceramic particles distribution from the part surface down to its core, the conventional MMC may become a functionally graded metal matrix composite (FGMMC) in which the wear resistance is expected to be improved at the surface whilst a high global toughness is preserved throughout the bulk of the component. In consequence, the applicants research has long been concerned with processing and characterization of centrifugally-cast SiC particle-reinforced functionally graded aluminium-matrix composites; studies concerning identification of key processing parameters and resulting reinforcement profiles [1-4], wear [5] and tribocorrosion [6] behaviour have been published.

Our first campaign (ME216, 3 shifts, ID19, April 2001) of X-ray microtomography experiments aimed at the elucidation of some aspects regarding particle distribution in Al/SiC_p FGMMCs) The X-ray microtomographic images were obtained in absorption-contrast mode. It could be observed that as a consequence of centrifugal casting SiC particles are partially clustered, with some pores gathered with them, this being associated to imperfect wetting of ceramic particles by the molten aluminium alloy. The results from the ME216 campaign were the object of two publications in journals indexed in the Science Citation Index [7, 8], and also received a broader, more integrated treatment on the context of a PhD thesis [9].

The experiments carried out within the frame of the present (ME461) campaign were designed in order to gain further knowledge about the spatial distribution of SiC reinforcing particles within a centrifugally-cast Al/SiC_p FGMMC, and the influence of metal/ceramic wetting characteristics on that same spatial distribution, by examining interparticle connectivity relationships. For any Al/SiC_p composite, the reinforcement distribution has a two-fold relevance. First, the presence of reinforcing particles is directly implied on the local mechanical properties, with reflection on the overall properties of the composite [10]. On the other hand, the presence of reinforcement clusters will bring a higher probability of crack nucleation, i.e. fatigue failure [10]. Furthermore, for FGMMCs, the existence of both reinforcement-rich and reinforcement-poor regions is inherent to the very nature of the material; in such a case, a smooth transition between those regions must be assured, while at the same time avoiding particle clustering or total depletion.

Precursor composites, prepared from an Al 10 Si 2 Mg alloy and SiC particles (with two different sizes, namely 12 μm in one case and 37 μm in the other), were produced by rheocasting in CENIMAT (Universidade Nova de Lisboa, Faculdade de Ciências e Tecnologia). The material thus produced was then molten and centrifugally cast in CIICS

(Universidade do Minho, Escola de Engenharia), in order to obtain the functionally graded composites. Details of both processes are available elsewhere [9].

From these FGMMCs, cylindrical samples, around 1 mm in diameter, were machined by EDM. The axis of each sample was parallel to the direction of the functional gradient, and the original positions of the samples defined a regular grid.

The samples were analysed by X-ray microtomography at the ID 19 beamline of the European Synchrotron Radiation Facility, in Grenoble. Twenty-two Regions of Interest (ROI) were scanned along each sample. For the purposes of the present work, ROI positioning is schematised in Fig. 1.

The microtomographic measurements were performed with a beam energy of 20 keV, and a multilayer was used as monochromator. For each sample, several sample ↔ detector distances were used (100, 200 and 400 mm). The detector consisted of a FRELON 1024×1024 CCD camera. Through superposition of the images obtained with different distances, holotomographic images were obtained, in order to enhance contrast between SiC particles and aluminium matrix. *Voxel* size was 1.203 μm^3.

Image segmentation and 3-D particle analysis were performed at Laboratoire des Composites Thermo-Structuraux (CNRS-SNECMA-CEA-Université de Bordeaux I). Segmentation has been straightforward, since the contrast provided by the holotomographic method was excellent. The particles were identified by a classical image invasion algorithm, with a simultaneous identification of their surface and volume. They have been partially separated by a one-voxel-depth erosion and a new invasion of the eroded particles. The number of connected subsets has been recorded for every particle, thus allowing (i) to perform statistical analysis on cluster number and volume, and (ii) to improve the analysis of particle data. In order to avoid edge effects, this treatment was applied to sub-sets of each ROI, henceforward designated as Volume of Interest (VOI). The average dimension of VOIs A1 to A4 was $655 \times 660 \times 900$ voxels (0.67 μm^3); however, due to calculation time limitations (calculation time is more strongly dependent on the number of reconstructed objects to separate than on VOI volume), VOI B5 had to be restricted to $710 \times 710 \times 135$ voxels (0.12 μm^3).

The holographic modification of the X-ray CMT method allowed to obtain neatly contrasted images, as opposed to classical CMT [11]; this aspect is emphasised in Fig. 2. The image quality, coupled with the processing algorithm employed, led to a good agreement between the particle size evaluated by holotomography and by laser interferometry.

Particle clustering has been evaluated in number and volume, showing that a lower mean particle size is related to more clustering, as shown in Fig. 3. Since molten aluminium poorly wets SiC, dispersion of smaller SiC particles is harder to achieve, thus explaining the results. Also, the mean particle size has been found to increase as a function of the number of particles within a cluster, which must be related to a higher probability for larger particles to get in contact with others. Tomographic results also supported previous observations regarding the existence of radial and dimensional segregation of the reinforcing particles in the FGMMC, as a result of the balance between the phenomena governing their distribution during centrifugal casting.

Up to the present date, the ME461 experiment gave rise to a paper published by an international journal [12], resulting from a communication made to the 8th International Symposium on Multifuntional and Functionally Graded Materials, Leuven – Belgium, 2004 July 11-14. Also, the inherent data treatment has motivated the formal establishment of a luso-french cooperation scheme, encompassing Universidade do Minho, Universidade Nova de Lisboa and Université de Bordeaux 1. As a result of this cooperation, the data processing software package initially created by G. Vignoles at LCTS in the subject of on-going further development at Universidade Nova de Lisboa, in order to suit it to parallel-computing techniques; when completed, such work should provide the portuguese research teams with a suitable facility for the treatment of the large datasets originated by the synchrotron radiation microtomography techniques, and should allow a faster turnover time for the results of future experiments aiming to elucidade the microstructural aspects of innovative types of FGMMC materials currently under development, such as *in-situ* FGMMCs [13] and syntactic FGMMCs [14-16].

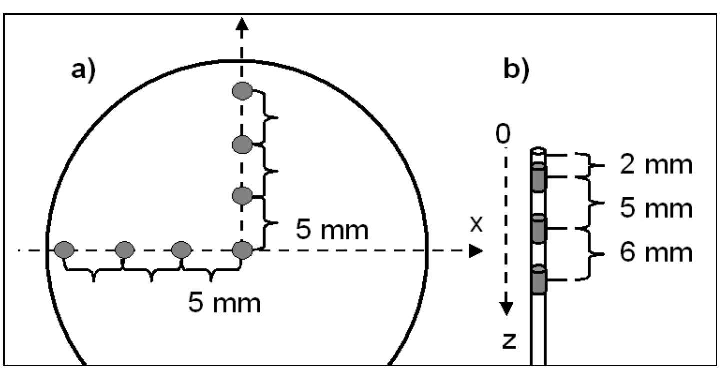

Figure 1 – ROI schematic positioning: a) planform view, showing the cylindrical samples positioning within the FGMMC dowel; samples were extracted at positions echeloned along two perpendicular axes $\overline{Ox}$ and $\overline{Oy}$; the longitudinal axis of each sample was parallel to the longitudinal axis of the corresponding dowel; b) ROI positioning along the longitudinal axis of each cylindrical sample; scanned ROIs were located at 2, 7 and 13 mm from the SiC-rich end of each sample.

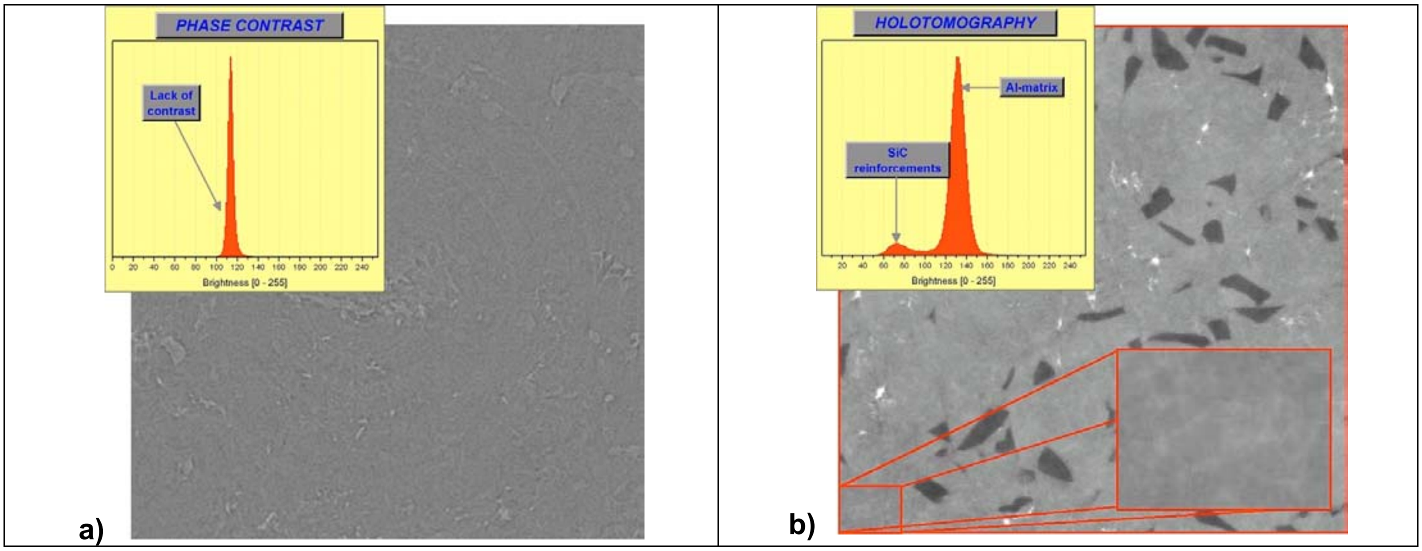

Figure 2 – a) Untreated slice image obtained in the absorption-contrast mode; the SiC particles are virtually undistinguishable. The weak Al/SiC_p contrast requires the use of a purposely developed segmentation technique, exploiting the interference fringes occurring near the matrix/reinforcement interfaces. b) Untreated slice image obtained in the holotomographic mode, clearly showing the location of the SiC reinforcing particles. Given the strong matrix/reinforcement contrast, no special segmentation procedure is expected to be needed, allowing for a more accurate morphology of the particle 3-D reconstruction. Furthermore, some matrix microstructural details become distinguishable (inset) opening new prospects for the study of particle pushing/engulfment phenomena.

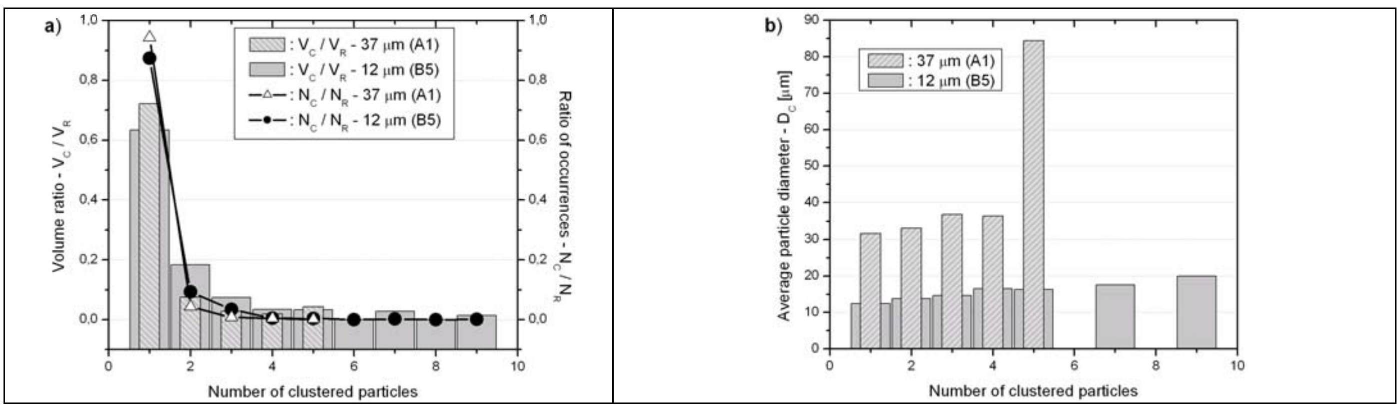

Figure 3 – Particle size effects on: a) volume ratio of clustered particles *vs.* overall particle volume – V_C / V_R – and ratio of occurrences of clustered particles *vs.* overall particle number – N_C / N_R; b) average particle diameter, D_C. In both graphs, the mention to "single-particle" clusters constitutes an expedient way to compute the number of isolated, non-contacting particles (at least up to the precision of the separation algorithm). [12]

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