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# Microtomographic study of the evolution of microstructure during creep of an AlSi12CuMgNi alloy reinforced with Al<sub>2</sub>O<sub>3</sub> short fibres

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#### Abstract

The evolution of the microstructure during creep of an AlSi12CuMgNi alloy with 15 vol% of  $Al_2O_3$  short fibres is investigated by means of synchrotron microtomography. The results reveal a change of the morphology of the hybrid reinforcement composed of eutectic-Si, short fibres and intermetallic particles. This change takes place due to the diffusion induced coarsening of Si and a coarsening of the intermetallic particles. The interconnectivity of the hybrid reinforcement increases during long-term creep exposure reaching a degree of almost full interconnectivity after 6400 h of creep. The analysis of creep damage during secondary creep stage shows an increase of void volume fraction by a factor 2, while the number of voids per volume remains practically constant (void sizes larger than 8 voxels were analysed). An analysis of the voids' location indicates that pores generated during processing of the composite grow, but no new pores are produced during the primary and secondary creep stages.

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# 1. Introduction

Short fibre reinforced metals (SFRMs) [1] are directly strengthened by means of an effective transfer of the applied load from the matrix to the short fibres (SFs) [2] which is more effective at elevated temperatures under creep conditions. This transfer of load results in a redistribution of stresses within the matrix, which leads to a reduction of stress, and henceforth the creep rate in the matrix [3,4]. The ceramic reinforcement does not creep at the test conditions considered in the present investigation.

In the case of Al–Si based SFRMs with Si content >7 wt%, the eutectic Si and the SFs form a three-dimensional hybrid network [5] in which the SFs are connected by Si bridges. The strength and stability of this structure is sensitive to the size and amount of the Si bridges. Furthermore, the morphology of the eutectic Si varies during high temperature exposure

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due to the diffusion driven coarsening [5] resulting in a subsequent change of the interconnectivity of the hybrid Si–SFs structure.

Results of the long-term creep behaviour at 300 °C of an AlSi12CuMgNi-based SFRM produced by squeeze casting have been reported recently [4]. During the creep tests, the SFRMs showed a decrease in steady state strain rate after a period of moderate load increasing (<20% of the yield stress) as well as an increase of the creep exponent, n. Such a "training" effect reduces, for example, the steady state creep rate of  $3.5 \times 10^{-10}$  s<sup>-1</sup> achieved at 30 MPa for the SFRM by about 50% after a load increase of 10 MPa for about 400 h [4]. It has been proposed [4] that one of the reasons for the creep strengthening of short fibre reinforced Al-Si alloys during long-term creep exposure is that the diffusion of Si increases the interconnectivity of the Si-SF hybrid structure. Hardness tests carried out on the extracted hybrid Si-SF-intermetallics structure showed an increase of the rigidity of this network with increasing creep exposure times [4]. However, no data are available on the real architecture of the rigid phases and its evolution during longterm creep of this kind of composites.

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Table 1	
$Chemical\ composition\ of\ the\ AlSi12CuMgNi\ alloy$	

Element	Si	Cu	Mg	Ni	Fe	Mn	Zn	Ti
In wt%	11-13	0.8–1.3	0.8–1.3	1.3	0.7	0.3	0.3	0.2

In the present investigations, micro-computed tomography  $(\mu CT)$  was carried out on AlSi12CuMgNi-based SFRM before and after long-term creep exposure with the following objectives:

- (1) To study the Si–SFs hybrid network to reveal its evolution and the change of interconnectivity during long-term creep exposure.
- (2) To study the damage process in SFRMs, focusing on the fracture of the SFs and the determination of the sites of formation and growth of voids both during the fabrication of the composite as well as during long-term creep exposure.

## 2. Experimental

### 2.1. Description of materials

The SRFM investigated in this work is an AlSi12CuMgNi alloy reinforced with 15 vol% of Al<sub>2</sub>O<sub>3</sub> SFs. This particular volume fraction of the reinforcement was selected based on the consideration that 15 vol% of SFs gives the highest creep resistance compared to 10 and 20 vol% [4]. The matrix alloy is typically used for combustion engines [6]. Its chemical composition is shown in Table 1 [7]. The SFRM was produced at the Institute of Materials Science and Technology at the TU Clausthal in Germany using a squeeze casting process [8]. Preforms of randomly planar distributed Saffil® SFs consisting of 96–97% Al<sub>2</sub>O<sub>3</sub> and 3–4% SiO<sub>2</sub> [9] were used as reinforcement. The SFs have lengths ranging from 10 to 200  $\mu$ m with diameters between 1 and 10  $\mu$ m.

Fig. 1 shows the microstructure of the SFRM in T6S condition looking onto the plane of random orientation. Besides the SFs and the matrix, the eutectic Si (grey), primary aluminides



Fig. 1. SEM micrograph showing the constituents of the composite:  $\alpha$ -Al (background), eutectic Si (bright grey), primary aluminides formed by Ni, Cu, Mg, Si or Fe, Cu, Mg, Si (white), Al<sub>2</sub>O<sub>3</sub>–SFs (dark grey) and pores (black) at the interface of the SFs. The plane shown corresponds to the plane of random planar fibre orientation in which the creep stress was applied.



Fig. 2. Si bridges connecting the  $Al_2O_3$  SFs for the SFRM in T6S condition revealed by chemical dissolution of the  $\alpha$ -Al matrix.

with Ni, Cu, Si, Mg or Fe,Cu, Si, Mg (white) and some pores (black) at the interface between the matrix and SFs and between adjacent fibres are also observed. These pores show up during the processing of the composite because the squeeze casting process may not be able to fill all the shrinkage pores between fibres during solidification [10]. Fig. 2 shows a scanning electron micrograph of the SFRM in T6S condition wherein the  $\alpha$ -Al was leached out using a 25 vol% solution of HCl. The remaining structure reveals the presence of Si bridges connecting the Al<sub>2</sub>O<sub>3</sub> SFs forming thus a mechanically stable reinforcing hybrid 3D network.

The SFRM was investigated in 2 different conditions:

- (1) T6S condition: solution treated at  $480 \circ C/4 h + oil$ quenched + aged at  $190 \circ C/4 h + overaging$  at  $300 \circ C/3 h$ . This represents the initial condition of the composite.
- (2) After long-term creep exposure at 300 °C. For this, the samples investigated in the present work were taken from creep samples tested for the longest period in an earlier investigation [4]. This period was 6400 h (approximately 9 months). Fig. 3 shows the strain versus time (a) and strain rate versus time (b) curves obtained from the creep test. This condition will be called 6400G hereafter, where 6400 states for the 6400 h of creep exposure and G indicates that the samples were taken from the gauge length of crept samples. A detailed description of the methodology employed for the creep investigations has been reported elsewhere [4].

### 2.2. Microtomography

 $\mu$ CT was carried out at the ID19 beamline at the European Synchrotron Radiation (ESRF) in Grenoble [11], France. The energy used was 21.2 keV. The  $\mu$ CT projections were recorded using a 2D-CCD camera developed at ESRF using an effective pixel size of 0.7  $\mu$ m. Eight hundred projections were acquired. The phase contrast mode [12] was used for the acquisition of the  $\mu$ CT projections by placing the camera at a distance of 20 mm behind the sample. The edge enhancement resulting from the phase contrast mode produces the necessary con-

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