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Tensile creep testing of an Al–Cu alloy above solidus with a dynamic mechanical analyser

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ABSTRACT

A dynamic mechanical analyser (DMA) was used to perform tensile creep tests at constant load and temperature to improve the accuracy in the measurement of mechanical properties above the solidus for an aluminium alloy. The binary Al–5.78 wt%Cu alloy was chosen for the investigation and specimens having columnar dendritic or equiaxed globular microstructures were tested to assess the validity of the technique and to see how the distribution of the liquid phase can affect the mechanical properties above solidus. It was found that the equiaxed globular microstructure was more rigid than the columnar dendritic microstructure with 8–9 vol% liquid phase. This was explained by the small thickness of the dendrite arms, which make the grains behave like sponges. Failure of the specimens occurred when the strain rates exceeded a critical level. Microstructure examinations performed before and after the creep tests gave indications that liquation occurred even at coalesced grain boundaries. Grain boundary sliding was promoted by liquid films at the contact points and generated cavities which accelerated the failure. The DMA was proven to be a very sensitive and reliable tool, able to heat specimens with good temperature uniformity while providing accurate measurements of the displacement and excellent control of the applied load.

1. Introduction

The mechanical behaviour of alloys at high temperature is generally described in terms of creep parameters, where strain rates or stress relaxation rates are the response of the alloy under specific mechanical boundary conditions. Deformation mechanisms at temperatures below solidus involve the activation of defects like grain boundaries, dislocations and vacancies. During solidification there is a point where the microstructure has enough cohesion to sustain some stress while having sufficient liquid phase to promote localized straining and rupture. This phenomenon is called hot tearing. In such a case, the liquid phase can be seen as another kind of defect, acting in a very dynamic way when compared to other defects. The role of the liquid phase is considerable in the development of hot tearing and its fast dynamics increase the difficulty of reliably characterizing the mechanical behaviour of semi-solid materials.

To understand how semi-solid materials fail, it is important to have a good description of the evolution of their microstructure. During the solidification of alloys, a first transition point called the coherency temperature occurs, above which the melt can be considered to be a liquid with solid particles in suspension [1]. Below the coherency temperature, the solid volume fraction is above 40% and the dendrites interact with their neighbours. The melt then has a mushy behaviour and its viscosity increases with the solid volume fraction until the temperature reaches a second transition point called the maximum packing point [2]. At this temperature the grains are mechanically in contact with their neighbours, even if there are very thin liquid films only a few nanometers thick separating the grains at the points of contact [3]. Depending on the solid–liquid interfacial energies involved, coalescence of dendrites and grains will occur after some undercooling [4]. With coalescence, the liquid phase disappears locally and a solid–solid boundary is formed. As the temperature decreases, the proportion of dry grain boundaries increases up to a point where the solid forms one continuous network with isolated liquid pockets. This is the rigidity point where the semi-solid microstructure acquires some strength [5] and is subject to thermal contraction [6]. The coherency temperature and the rigidity temperature are dependent on the composition of the alloy and must be determined experimentally. Once the rigidity temperature is reached, the liquid film between the grains becomes smaller and more isolated as the solid fraction increases. Failure in this regime cannot be healed by liquid flow and hot tearing occurs. Vernède et al. [3,7] developed a 2D granular model simulating the evolution of the equiaxed microstructure based on the work of Mathier et al. [8] and Rap-

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paz et al. [4]. They were able to simulate the different transitions in the mushy state, including grain contact, liquid isolation, percolation by contact, and percolation by coalescence. However, the granular model of Vernède et al. [3,7] assumes that the grains are undeformable and move only by translation. The former assumption limits the capability of this model to predict the mechanical behaviour at high solid fraction considering that the elastic component is not negligible due to the continuity of the solid network. Phillion et al. [9] considered that solid grains behave as an elasto-plastic material in their finite element granular model. The liquid phase was treated like a solid phase, having an elastic modulus and a low yield stress, thus limiting the validity of predictions to low strains. This model is appropriate to simulate the mechanical behaviour of the semi-solid microstructure at high solid fraction; however it cannot describe the phenomena associated with the segregation of the liquid phase. The discrete element model recently proposed by Sistaninia et al. [10] has the same limitations since the liquid phase is modelled by a connector element made of a spring and a damper in parallel. These models were proposed to take into account the strain localization effect occurring under tensile stress, which was confirmed experimentally. Further modelling developments need a better description of how a semi-solid microstructure behaves under stress, and this requires that different experimental approaches and observations be performed.

The difficulty in testing semi-solid material is caused by the presence of liquid in the samples and the rapid response of the microstructure it implies even at low stresses. Some experience has been gained over the years by different researchers on the mechanical testing of semi-solid samples. Testing methods to study hot tearing can be divided in two categories:

- Reheating techniques, where the alloy is taken from the as-cast state and heated to the mushy state before the mechanical test.
- Solidification techniques, where the alloy is tested while solidifying.

Both techniques yield different microstructures and different results. The solidification technique produces results that are closer to real castings with the drawback that it is harder to get stress–strain data because of the non-uniformity of the temperature field. The interaction of the melt with the mould is also one of the issues with this technique. The reheating method is easier to use but the as-cast microstructure of the samples is modified in reheating. Han et al. [11] proposed heating to a higher temperature for reheating tests before letting the sample cool to the target temperature. In this way, they claim to achieve results closer to the solidification method without the technical difficulties. Recently, the Gleeble 1500 and 3500 thermomechanical testing units were used by van Haaften et al. [12] and Colley et al. [13] to characterize the mechanical properties of aluminium alloys in the semi-solid state. This apparatus uses the Joule effect to quickly heat the sample to the desired temperature. It has a heating capacity of 50 K/s and load cells ranging from 10 N to 10 kN. Ludwig et al. [14] used another type of tensile test apparatus equipped with an induction coil and an alumina mould around the sample to investigate the tensile behaviour of semi-solid aluminium alloys. Both techniques have the advantage of very high heating rates, minimizing microstructure changes before tensile testing. However, the samples used for mechanical testing are large and temperature gradients can be a problem. Since the deformation mechanisms suspected of leading to hot tearing generally occurs in localized areas, it could be worthwhile to use much smaller samples in order to have a more uniform distribution of temperature in the specimen. In this study, a dynamic mechanical analyser (DMA) was used to characterize the creep behaviour of a semi-solid aluminium alloy. The main advantage of the DMA lies in its accuracy for measuring small forces and displacements. Using small samples with uniform cross sections, one can easily determine the stress and strain evolutions with time and temperature. The small size of the specimens also helps to get adequate heating time while maintaining a good temperature uniformity. Finally, the samples can be polished prior to the creep test to facilitate the observation and comparison of the microstructure before and after the test.

Table 1
Cast alloy composition (wt%) determined by atomic absorption spectroscopy.

	Cu	Fe	Mg	Si	Al
Alloy composition	5.75%	0.1%	0.0025%	0.085%	Bal.

2. Experimental procedure

2.1. Material

The alloy investigated was a binary Al–5.78 wt% Cu prepared in a SiC crucible at 750 °C with aluminium of commercial purity and pure copper. The composition of the melt was determined by atomic absorption spectroscopy with a PerkinElmer Analyst 800 instrument, and the weight contents of the main elements are presented in Table 1.

Two casting procedures were conducted. The first one was followed to obtain a fine equiaxed globular microstructure, giving the possibility to measure mechanical properties with a minimum of deviations caused by the microstructure itself. This was made to validate the repeatability of the technique. The second one was designed to obtain a unidirectional solidification microstructure and was used to compare the properties obtained by imposing different temperatures and stresses.

In the first procedure, the alloy was poured into a permanent mould mounted on a vertical beam equipped with a compressed air vibrator device. The latter was used to generate mechanical vibrations promoting equiaxed globular solidification. The geometry of the mould cavity is presented in Fig. 1. The mould cavity was coated with boron nitride prior to casting. The runner had a bottom fill pattern to help reduce turbulence and the melt entered the cavity through the center of the 6.32 mm thick section. The mould was first preheated with a gas burner to 400 °C while the melt was kept at 700 °C. After casting, the piece was allowed to solidify in the cavity and was air cooled after extraction. Thin rectangular plates of 0.5 mm thickness were extracted from the 19.05 mm thick section as shown in Fig. 1. Three 45 mm × 8 mm rectangular plates were machined to conduct creep testing with specimens having a uniform cross section. This simple geometry was adopted to evaluate the applied strain with good accuracy considering that the deformation of the steel grips is negligible because of their large cross section and high stiffness. Other thin rectangular plates were machined to obtain flat tensile specimens (Fig. 2). The latter were dedicated to the observation of the microstructure evolution in the reduced zone before and after creep testing. They were mechanically polished on their larger faces prior to creep testing and the microstructure was observed in a scanning electron microscope (SEM).

In the second procedure, the liquid metal was poured in a cast iron mould having a 20 mm deep cylindrical cavity of 138 mm diameter and was previously heated in a furnace at 800 °C. Once the mould cavity was filled, a water cooled aluminium plate was fixed on top of the mould with clamps and the assembly was quickly flipped 180° to ensure the alloy was cooled from the bottom. The casting was ejected after 5 min of cooling and quenched in water in order to preserve as much of the solidification microstructure as possible. The resulting Al–Cu disk was cut down into rectangular blocks 45 mm long, 9 mm wide with the thickness being that