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Rheological behavior of semi-solid TiB_2 reinforced Al composite

S. THADELA[1], B. MANDAL[1], P. DAS[1], H. ROY[2], A. K. LOHAR[1], S. K. SAMANTA[1]

1. Near Net Shape Manufacturing Technology Group,
CSIR-Central Mechanical Engineering Research Institute, Durgapur 713209, India;
2. NDT & Metallurgy Group, CSIR-Central Mechanical Engineering Research Institute, Durgapur 713209, India

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Abstract: The rheological behavior of the semi-solid TiB_2 reinforced Al−Mg alloy composite slurry was investigated, which is required for the mould filling simulations during the semi-solid processing. TiB_2 reinforced in-situ Al−Mg alloy composite samples were remelted and subsequently brought to the semi-solid temperature regime within the heating chamber of a Searle type rheometer. In order to understand the rheological behavior of composites, three different types of experiment were carried out, namely, steady state test, continuous cooling test and isothermal test. Apart from that, the thixotropic nature of the slurry was confirmed from the obtained hysteresis loops during the experimentation. The results indicate that when isothermally held, the composite slurry exhibits pseudo-plasticity and shows shear-thinning behavior up to the shear rate of 1300 s^{-1}, and at higher shear rates (>1300 s^{-1}), it shows a shear thickening tendency, which is probably due to the agglomeration of non-deformable nano-TiB_2 particles. The pseudo-plastic behavior of the slurry was also estimated employing intermittent step changes of shear rate (shear jump test).
Key words: rheology; shear thinning; in-situ nano-TiB_2; thixotropy; shear thickening

1 Introduction

Rheocasting and thixocasting of metallic alloys and composites utilize semi-solid slurries in which non-dendritic (spheroidal) solid particles are dispersed in a liquid melt [1−4]. The rheological characterization of these metallic alloys and composites in semi-solid state is highly important in the die-casting stage for analyzing their distinctive flow behaviour (rheology). The rheological responses under the rapid changes in shear rate, form the basis of modelling the die filling and die design processes. The reduction in viscosity of semi-solid slurry under application of shear (Thixotropy) leads to complete filling of the mould sections during the casting process. Usually semi-solid slurries exhibit the following complex rheological behaviours, pseudo-plasticity and thixotropy. Owing to the ubiquity of Al alloys in mechanical construction and automotive industries, the rheological characterization of Al alloys (A356, A380) have already been carried out [2,5,6]. However, there is limited information related to the rheological behaviour of Al composites [7−9]. In this study, the rheological characterisation of Al-based in-situ composite reinforced with TiB_2 particles (Al−4Mg−0.15Sc−0.075Zr−2TiB_2) was performed to investigate its capability to fill the die cavity of engineering components under the application of shear at semi-solid state. The present class of composites are popular due to their excellent mechanical and tribological properties, and high stiffness and hardness of submicron size particles. The particles produced by the reaction are rapidly dispersed in the liquid Al alloy and refine the grain structure during the solidification. A uniform dispersion of reinforcement particle in the matrix in a composite is one of the factors for getting improved mechanical properties [10]. Sc and Zr are added within the melt during the composite development to obtain modified microstructural features in the cast parts [11]. Earlier investigation has shown marked improvement in mechanical properties of the present class of composite owing to the presence of nano-TiB_2 particles at the grain boundaries. The formation of such nano-TiB_2 was attributed to the presence of Sc in the melt [10].

In the present study, the remelting and subsequent cooling of the composite was performed within the heating chamber of high temperature rheometer to perform rheological investigations at semi-solid state. At

Corresponding author: P. DAS; Tel: +91-343-6452373; Fax: +91-343-2546745; E-mail: prosenjit.sct.cmeri@gmail.com; prosenjit@cmeri.res.in
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semi-solid state, the whole system becomes one having three components: primary solid phase of the matrix (α(Al)), liquid alloy matrix and reinforced particles (TiB_2). It is therefore interesting to consider the effect of the presence of small reinforced particles (TiB_2) on the rheological behavior of the total system. The understanding of the rheological behavior would therefore be beneficial for semi-solid metal processing of this class of composites.

2 Experimental

The Al−4Mg−0.15Sc−0.075Zr−2TiB_2 composite was cast by diluting Al−2Sc, Al−10Zr and 99.9% Mg master alloys along with commercially pure Al in a resistance heating furnace. TiB_2 was allowed to form by in-situ reaction of K_2TiF_6 and KBF_4 salt mixture at 800 °C for 60 min. The TiB_2 particles produced by the reaction were rapidly dispersed in the liquid Al alloy and refined the grain structure during the solidification. The composite melt was stirred for 2 min with a graphite blade followed by the addition of Mg to form the required Al−Mg−Sc−Zr composite reinforced with TiB_2 particles. Finally, the casting was made by pouring the molten composite into the permanent mould. The chemical composition of the alloy obtained is shown in Table 1.

Table 1 Chemical composition of TiB_2 reinforced Al composite (mass fraction, %)

Mg	Sc	Fe	Si	Zr	Al
0.34	0.16	0.20	0.40	0.065	Bal.

The rheological experiments were performed with a high-temperature Searle type rheometer. Searle type rheometer has a rotating inner bob and a stationary outer cup, as shown in Fig. 1. The detailed working principle of Searle type rheometer was explained elsewhere [6]. Though the solidus and liquidus temperatures of the base Al−Mg alloy are available in Ref. [12], however, in order to carry out the rheological study, the digital scanning calorimetry (DSC) analysis of the in-situ composite was performed and the temperature regimes of rheological investigations were selected based on the DSC findings. The DSC findings reveal the solidus and liquidus temperatures of the composite which are approximately 580 and 650 °C, respectively (Fig. 2).

The steady state test was performed to determine the change in the viscosity of semi-solid slurry with fixed shear rate. The time dependent nature of slurry was generally observed from the steady state test. During an isothermal holding (615 °C) and at a shear rate of 600 s^{-1}, the apparent viscosity of slurry was recorded for a period of 60 min. The test was terminated at 60 min to nullify

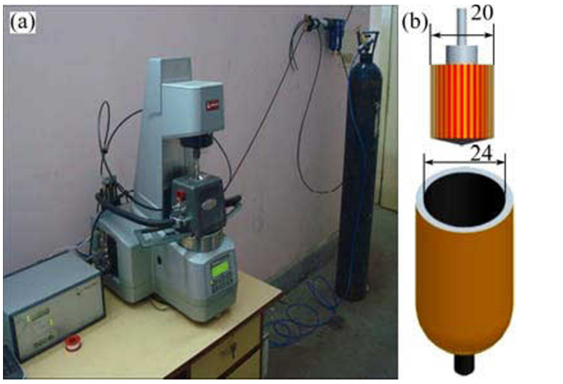

Fig. 1 Searle type rheometer (a) and schematic view of bob and cup arrangement (b) (unit: mm)

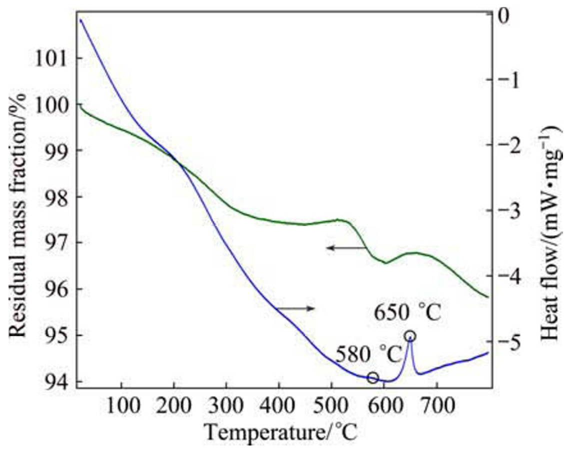

Fig. 2 DSC curve of composite

the dominance of Ostwald ripening over the globularization of primary Al grains [13].

During the continuous cooling test, the superheated melt was cooled from 660 °C at a cooling rate of 5 °C/min down to the semi-solid temperature of 605 °C while the shear rate is fixed at 700 s^{-1}. Whereas for the isothermal test, the shear rate varied from 10 to 1500 s^{-1} at the selected temperatures. The shear rate jump test was also performed following an earlier investigation on A356 Al alloy [6,14] in order to simulate the real life situations, where sudden increase and decrease in shear rate were inevitable. It is well known that semi-solid slurries exhibit time-dependence, non-Newtonian flow known as thixotropy, therefore, the thixotropic nature of the slurry is confirmed from the hysteresis loops obtained during the experimentation. The thixotropic test for the Al−4Mg−0.15Sc−0.075Zr−2TiB_2 composite slurry was performed at a temperature of 615 °C. Firstly, a steady state viscosity value of the slurry was obtained at a shear rate of 400 s^{-1}. The shear rate was then ramped down to zero and kept at this level for a rest period of 10 min. The shear rate was then immediately ramped back to 400 s^{-1} and immediately ramped back to zero by setting the ramp up and ramp down period of 5 s each.