Viscosity of semi-solid a357 alloy in the transient high shear rate regime

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Abstract

A modified, parallel-plate rheometer is described for measuring the viscosity of semi-solid slurries under isothermal or continuous cooling conditions at shear rates up to 2000 s-1 and under transient conditions. The shear rate is rapidly changed by varying either the rotation speed of the plate or the gap between the plates. Shear rates changes of up to 1500 s-2 are obtainable. The semi-solid slurries studied are prepared by reheating rheocast material of industrial origin into the semi-solid range.
Viscosity measurements under conditions of constant and abruptly changing shear rate will be presented and related to structural studies.

Keywords

Semisolid alloys, viscosity, rheometry.

Riassunto

Viene descritto un reometro a piastre parallele modificato per la misurazione della viscosità degli impasti semisolidi sia in condizioni di raffreddamento isotermiche o continue a fattori di taglio fino ai 2000 s-1, sia in condizioni transitorie. Variando o la velocità di rotazione della piastra o l'intervallo tra le piastre si assiste a cambiamenti rapidi del fattore di taglio fino ai 1500 s-2. Gli impasti studiati vengono preparati per mezzo del post riscaldo fino alla gamma dei semisolidi di materiale reocolato di origine industriale. I dati viscometrici rilevati in condizioni di fattore di taglio costante e bruscamente variante verranno presentati e rapportati agli studi strutturali.

Parole chiave

Leghe semisolide, viscosità, reometria.

INTRODUCTION

Rotational viscometry has been extensively used in studies of flow behavior of semi-solid alloys, including commercially important alloys from the Al-Si-Mg group. [1-7] Several alternative techniques have also been employed to investigate the complex rheology of semi-solid metal slurries in a wide range of high shear rates. These include the parallel plate compression, [8,9] capillary rheometry, [10] and back extrusion. [11] Rapid compression between parallel plates [12,13] is an example of a powerful alternative technique for the study of high solid content metal slurries subjected to rapid deformation rates. The experimental evidence accumulated to date shows that semi-solid metal slurries exhibit a shear thinning thixotropic behavior in steady state deformation. Shear rate thickening behavior has also been reported under conditions of rapid, time-dependent deformation. A thorough understanding of the behavior of metal slurries under the latter conditions is highly desirable, considering the short duration and the highly unsteady flow of metal slurries during mold filling operations.Although discrepancies among the available experimental results exist and are usually attributed to the rheological complexity of metal slurries, this may not be the only or the most important contributing factor. Most frequently, the rheological behavior of metal suspensions is deduced from measurements relying on the assumption of a theoretical (viscometric) flow within a particular type of rheometer. To approach ideal flow conditions, most investigators make use of rotational viscometers with narrow gaps. Smaller tool scales relative to the characteristic particle size may lead to conditions where the continuum hypothesis is not respected, in addition to other problems. Coussot, [14] in a comprehensive discussion of shear rheometry of concentrated suspensions pointed out that for those materials no general rule exists. According to the author, the sheared thickness may be smaller than the gap, and a sufficiently large ratio of gap to particle diameter may not be the only condition necessary to guarantee that the continuum hypothesis is respected. This requires at least a large ratio of sheared thickness to maximum particle size. However, this ratio is not only affected by tool dimensions but also by the possible existence of a finite yield stress and by the imposed flow rate or shear stress. In the case of metal slurries, the highly interacting nature of the solid particles also needs to be considered.

With the above requirements in mind, a modified parallel-

plate rheometer was built for this work that allows testing of metal slurries using different geometries. The parallel plate configuration was selected for being better suited for transient experiments in which both the gap and the angular velocity are varied during a given run. This provides additional degrees of freedom to investigate the aforementioned disturbing effects and deduce the true constitutive behavior of the material from rheometric measurements. For this, it is also important to be able to preserve the structures formed after given flow histories. Here we report on viscosity measurements performed by increasing (or decreasing) the shear rate of alloy A357 slurries in a stepwise manner. The alloy slurries used in the present investigation are formed by reheating industrially rheocast material into the semi-solid state.

EXPERIMENTAL APPARATUS

Figure 1 shows a schematic view of the parallel plate rheometer built for this work. The metal slurry is contained in a high-density graphite crucible of 83-mm inner diameter, which is housed in a vacuum chamber and heated by induction coils powered by a 20-kW source. A 200-g sample can be heated into the semi-solid range in about ten minutes. Temperatures are measured and controlled by means of calibrated thermocouples protected by quartz sheathing and attached to the crucible's inner wall. The thermocouples remain immersed in the semi-solid metal during the whole experiment. The induction source is provided with a closed-loop temperature controller, which limits temperature variations to within ±0.5¡C over the entire duration of an experiment. Spatial temperature variations in the melt do not exceed 1¡C along both the vertical and radial directions. Oxidation of the melt is minimized by maintaining the chamber pressure in the 10-1 torr range.

Parallel plates are also made out of high density graphite and are of 30-mm diameter. The geometry of the plates is shown schematically in Figure 1.The lower plate is positioned in the tapered crucible as an insert and the top plate can be moved up and down while rotating. This arrangement allows for easy sample recovery after completion of an experiment. To help prevent slip, a serrated design previously used in work with greases, [15] was chosen for the rotating plate. The rough surface of the bottom plate also contributes to minimize slip. The serrated plate is rotated by a variable speed DC motor capable of a maximum angular velocity of 2500 revolutions per minute. A dynamic torque sensor is mounted in between the driving motor and the rotating steel shaft by means of flexible couplings. The motor controller is capable of accelerating the motor at variable rates. Acceleration times to reach maximum speeds can be set between 2 and 10s. A tachometer is used to measure angular velocities. A motorized linear slide capable of moving the motor and connected components along the vertical direction is used to position the rotating plate. An LVDT is used to measure the plate separation. The experimental data consist of temperature, torque, plate separation and angular velocity DC signals and can be acquired at rates of up to 1000 Hz using a data acquisition system from Omega Engineering.

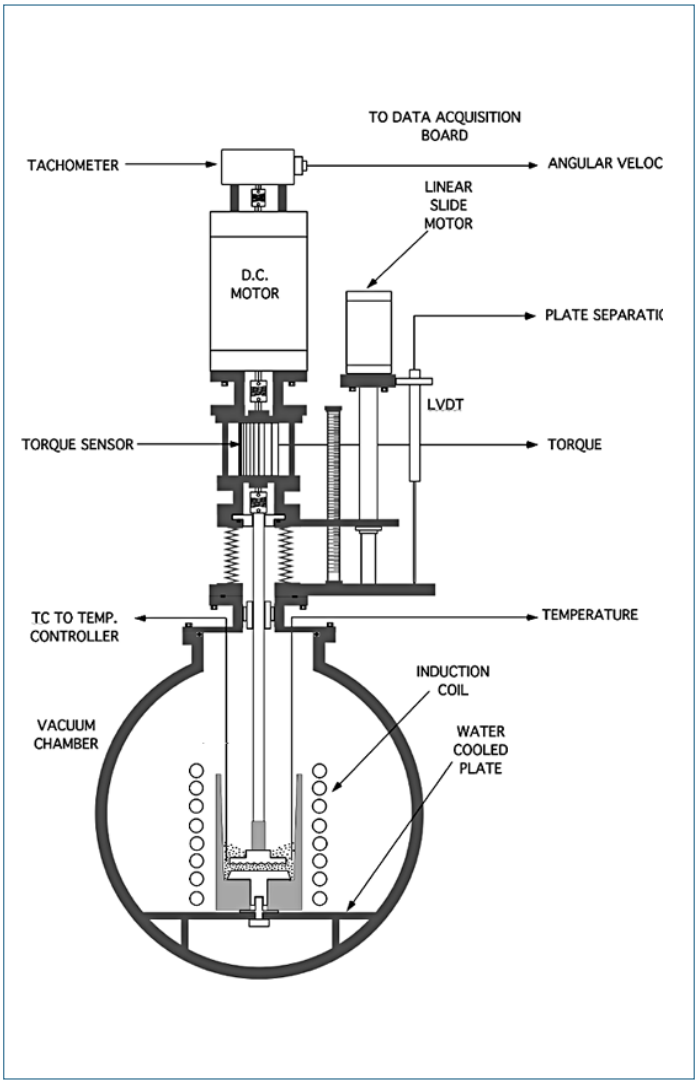

Fig. 1: Schematic view of the parallel plate rheometer built for this work

In the typical parallel plate geometry, the shear rate ($\dot{\gamma}$) is proportional to the radius (r) at which it is measured and its maximum value, at the perimeter of the plate, is given by

$$\gamma R = \frac{\Omega R}{h} \qquad (1)$$

where Ω, R, h are the angular velocity, plate radius and plate spacing, respectively. In this geometry, the shear stress for a

non-Newtonian fluid is given by the following expression: [16]

$$\tau(R) = \frac{M}{2\pi R^3}\left[3+\frac{dlnM}{dln\dot{\gamma}_R}\right] \quad (2)$$

With Equation 2 and the relation $\tau(R)=\eta\dot{\gamma}_R$, an expression for the apparent viscosity can be written as:

$$\eta_{app} = \frac{M}{2\pi R^3\dot{\gamma}_R}\left[3+\frac{dlnM}{dln\dot{\gamma}_R}\right] \quad (3)$$

MATERIAL AND EXPERIMENTAL PROCEDURES

Alloy

The material used in this work is of industrial origin and consists of alloy A357 in the form of rheocast billets fabricated by an electromagnetic stirring route. As-received cylindrical billets are 75 mm in diameter and 100 mm long. Cylinders or discs for the experiments are cut from the as-received billets as described below. Typical ranges of chemical composition (wt.%), measured on a few billets, are as follows: 6.74-7.47%Si, 0.47-0.51%Mg, 0.13-0.15%Fe, 0.01%Mn, Zn and Ti, <0.01%Cr and Ni, and <0.15% other elements. The solid content reported in this work is calculated with the help of Thermocalc® using the SGTE database and assuming non-equilibrium solidification. Discrepancies between calculated and measured solid fractions have been reported and discussed elsewhere. [17,18]

Experimental Procedures

Two different methods have been employed for viscosity measurements that differ somewhat from the standard approach usually followed with parallel plates. In the first method, measurements are taken with the rotating plate completely immersed in the semi-solid slurry. The second method is a combination of the standard parallel plate approach and the plate-in-a-cup approach. The two methods are illustrated schematically in Figure 2. In method 1, the crucible is loaded with a 250-cm3 cylindrical billet section. The rotating plate is then lowered to a position just above the surface of the billet, the chamber evacuated, and the temperature of the crucible rapidly raised to a value in the semi-solid range of the alloy. For volume fractions below 0.5, completely semi-solid and homogeneous materials are obtained after a holding time of 45 min. At that point, the plate is slowly immersed into the semi-solid mass while rotating at a few rpm, until a gap of 12 mm is established between the plates. After an additional waiting time of 5 min to ensure proper temperature uniformity throughout the slurry, both the data acquisition sequence and the plate rotation are initiated. The slurry is then sheared at a constant rate of 400 s-1 for 100 s. This procedure leads to a near constant torque reading for all volume fractions examined and is the reference state adopted for this type of experiment. After the 100-s shearing time, a stepwise increase in shear rate is imposed ton the slurry by means of a stepwise decrease in plate gap, from the initial value of 12 mm to values near 4 mm. The typical duration of a step is between 20 and 30s. Figure 3(a) illustrates the output of such an experiment.

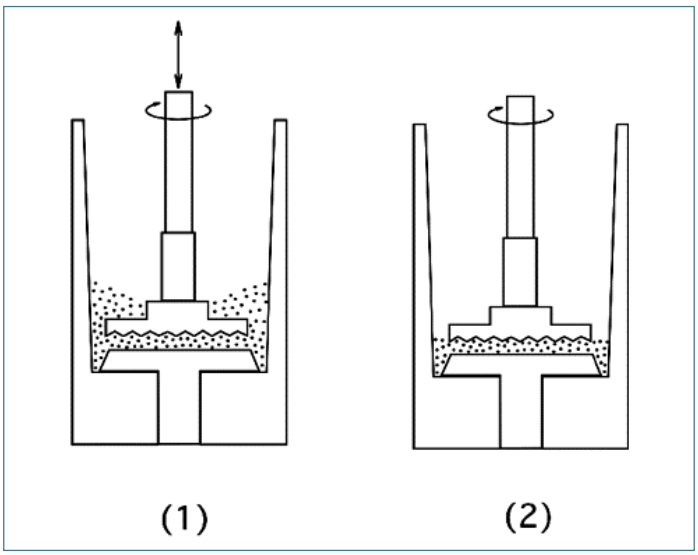

Fig. 2: Methods used for viscosity measurements. In (1) the rotating plate is completely immersed in the slurry and calibration is used to convert torque readings to viscosity. In (2) the viscosity is calculated from torque measurements by means of Equation 3

Equations 2 and 3 are not valid when the rotating plate is immersed in the fluid. In this case, for a given shear rate, the conversion of torque readings to viscosity values has to rely on instrument calibration. This was accomplished by measuring the ratio of torque-to-shear ratio for equivalent volumes of standard silicone oils and correlating the measurements to their viscosity. In method 2, the crucible is loaded with a smaller quantity of alloy in the form of a 75-cm3 disk. Similarly to method 1, the rotating plate is positioned just above the disk, the chamber is evacuated and the crucible heated to the temperature of interest. Fully semi-solid and homogeneous materials are obtained after holding the sample in the semi-solid range for 15 min. At this point, the plate is lowered further until it makes contact with the semi-solid. At this time, the data acquisition sequence and the plate rotation are initiated in this order and the material is sheared for 100 s at a constant shear rate (constant plate gap) to obtain a nearly constant torque reading. Again, a stepwise increase (or decrease) in shear rate is imposed on the slurry by a stepwise increase (or decrease) in the angular velocity at a

fixed gap value of 6mm. The typical response obtained in this case is shown in Figure 3(b) for the case of a multiple step decrease in shear rate, which is imposed by reducing the angular velocity. Increasing the shear rate by a number of steps leads to the opposite behavior, i.e., for every step increase in shear rate, a step decrease in viscosity is observed. For the experiments employing method 2, viscosity values were calculated directly from the torque measurements using Equation 3. Correction factors have been discussed in the literature to account for reservoir effects. [19] These correction factors, however, are small when the diameter of the crucible is much larger than that of the plate, and in the present case are below 30%.

Microstrutures

Figure 4(a) shows the microstructure of the rheocast alloy billets, which is typical of electromagnetically stirred materials. Figure 4(b) is representative of the structures found after the experiments. That structure was produced by remelting the as-received rheocast material to a volume
fraction of 0.35, soaking at the temperature for 10 min, and then imposing a shear rate of 785 s-1 for 200 s. The parallel-plate geometry utilized in this case was that shown in Figure 2-2. Rapid gas cooling contributed to the preservation of most of the structural features. The qualitative and quantitative observation of microstructures formed at different shear histories may contribute important information about the flow resistance of these materials, particularly on its dependence on globule size, globule morphology, amount of entrapped liquid, and the nature of the bonds between the primary phase particles.

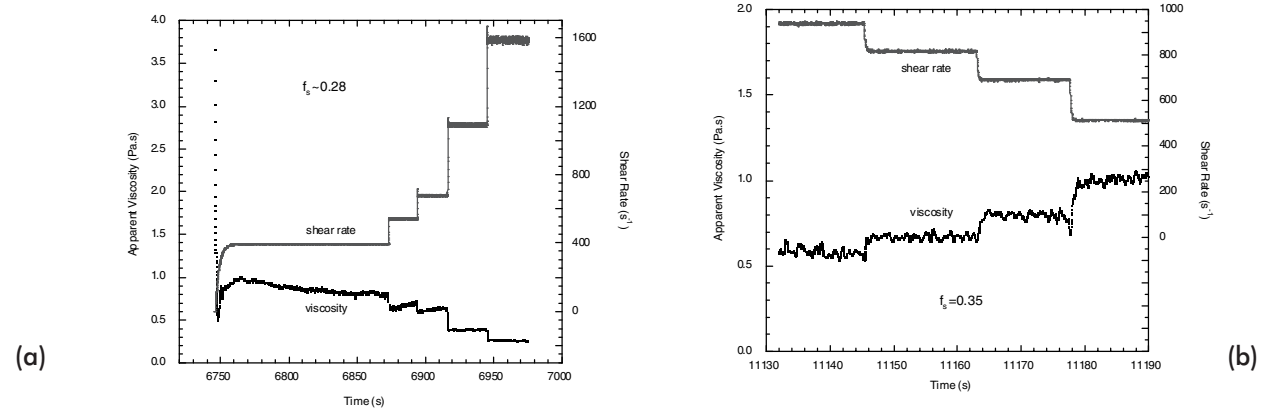

Fig. 3: (a) Viscosity response to a stepwise increase in shear rate imposed by decreasing the plate gap with plates immersed in the semi-solid metal (method 1), and (b) Viscosity response to a stepwise decrease in shear rate imposed by reducing the angular velocity of the plate (method 2).

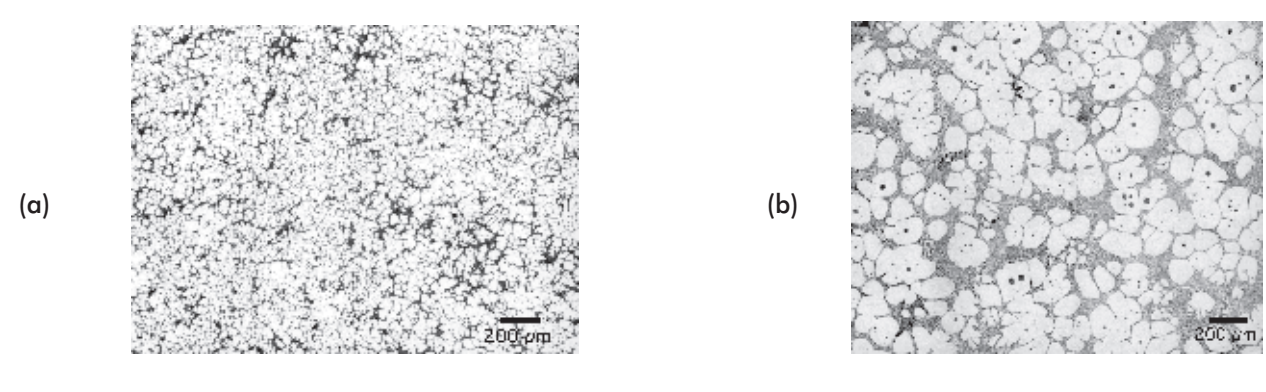

Fig. 4: (a) Typical microstructures of the rheocast A357 billets used in the experiments prior to re-heating into the semi-solid range; (b) Structure obtained by reheating the material shown in (a) to a fraction solid of 0.35 (10 min soaking time), imposing a shear rate of 785 s-1 for 200 s and rapidly cooling the resulting slurry.

SLURRY VISCOSITY

The shear rate dependence of the viscosity of the A357 slurries studied is presented in Figure 5(a) for low to moderate volume fractions (up to 48 vol%). The curves of Figure 5 show the viscosity
evolution for shear rate histories similar to that depicted in Figure 3(a). In this case, viscosity values
measured in between two consecutive jumps decrease monotonically with shear rate and the slurry behavior is shear thinning for all volume fractions examined. Moreover, Fig-

ures 3(a) and 3(b) shows that step changes in shear rate did not lead to stress (torque) overshoots or undershoots, and transient shear thickening behavior was not detected for transition times as low as ~200 ms. For the most part, the viscosity curves level off immediately after a rapid increase or decrease in shear rate. This behavior was observed for both the experimental methods employed, as shown in Figure 3(b) and 5(b), and is in disagreement with results from previous investigations on Sn-Pb alloys. [20] For the same shear rate and fraction solid ranges, the present results are higher than those reported in the
literature for slurries prepared on cooling, [3-5] and lower than the values deduced from rapid compression or back-extrusion. [12,13]

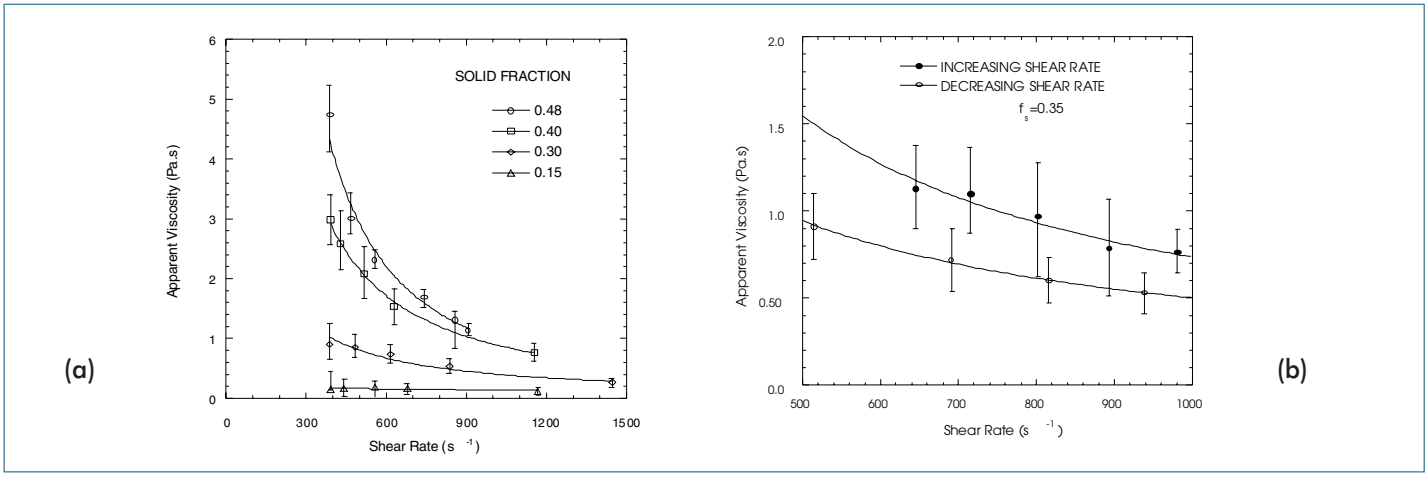

Fig. 5: (a) Dependence of the viscosity of A357 slurries on shear rate and volume fraction. (b) Viscosity evolution for an A357 alloy slurry containing 35 vol.% solids following a series of steplike decreases (bottom curve) and increases (top curve) in shear rate. Lower viscosities are measured when the shear rate is decreased in a steplike fashion, as shown in Figure 3(b).

CONCLUSIONS AND FUTURE WORK

A modified parallel plate rheometer is being used to investigate the flow behavior of alloy A357 slurries. The slurries are produced by re-heating rheocast material of industrial origin into the semi-solid temperature range by means of experiments of relatively short duration (~200 s). Viscosity measurements taken during a stepwise increase (or decrease) in shear rate show that, for all volume fractions examined, the slurry behavior is shear thinning. Fairly constant viscosity values are measured between two consecutive transients. Transient shear thickening behavior was not observed for transition times as lows as ~200 ms. Experiments are under way to examine the viscosity response of the slurry to shear rate transients of duration shorter than 200 ms and larger shear rate amplitudes. Slurry structures after given shear rate histories will be analyzed and correlated to viscosity mesurements.

ACKNOWLEDGEMENTS

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