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Parameter Estimation for Semi-Solid Aluminum Alloys using Transient Experiments

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Abstract

A rotating vane-cup rheometer is used to determine the rheological properties of semi-solid slurries, and a procedure is established for characterizing the rheology with emphasis given to the proper and self-consistent evaluation of the material constants.

Introduction

Semi-solid slurries behave as visco-plastic materials with time-dependent material properties [1]. Since the SSM structure breaks down faster than the rate at which structure builds up, it is important to capture accurately the early behavior of the slurry. The rotating vane-cup rheometer is used to determine the properties of semi-solid slurries. Emphasis is given to proper and self-consistent evaluation of the material constants

Two semi-solid 356 aluminum alloys, one prepared by Magneto Hydrodynamic Stirring (MHD), and the other by the Semi-Solid Rheocasting (SSR™) process are used. The chemical composition was measured using spark emission spectrometry. The tests were performed at temperatures ranging from 595 to 585°C, which correspond to solid volume fraction (f) ranging from 0.2 to 0.5 (using Pandat®). The measuring system is a modified Couette system with the inner cylinder replaced by a 4-bladed Anviloy 1150™ alloy vane. Two vanes with length of 43 mm and radii of 23.5 and 26.0 mm and a cup with a radius of 33.75 mm are used to form a coaxial cylindrical system with radius ratio (cup/vane) of 1.43 and 1.29, respectively. The results presented here were obtained using the geometry with cup/vane ratio of 1.43. The cup surface was roughened to reduce wall effects. The high temperature rheometer was equipped with a data recorder, which collected data for on-line analysis with a maximum sampling rate of 1 kHz. The stress was calculated using the measured torque. The sample was loaded into the cup and heated in a rich argon environment at the desired temperature for an additional one hour to ensure homogeneous temperature. A clean sample was used for each test and the tests were repeated at least 3 times in order to insure repeatability. For the geometry of the experimental setup the maximum rotational speed was restricted to 95.66 Rad/s, as the sample spills from the cup at higher speeds. More details of the experimental setup and the calibration of the rheometer can be found in [2].

Results and Discussion

Figure 1 shows the shear stress as a function of time for a MHD slurry at f=0.2 obtained for rotational speeds ranging from 1.07 to 95.66 Rad/s. At a given rotational speed, the stress decreases rapidly pointing to the continuous changes in the material constants, i.e thixotropic behavior. Eventually, the stress approaches a constant value. It is clear that the time required for the shear stress to reach its equilibrium state decreases with increasing rotational speed (about 40 seconds for 1.07 Rad/s vs 10 seconds for 95.66 Rad/s). In all cases the time required to reach a steady state is much longer than typical processing times under industrial conditions. This point highlights the importance of the transient short-term behavior of SSM slurries to shear.

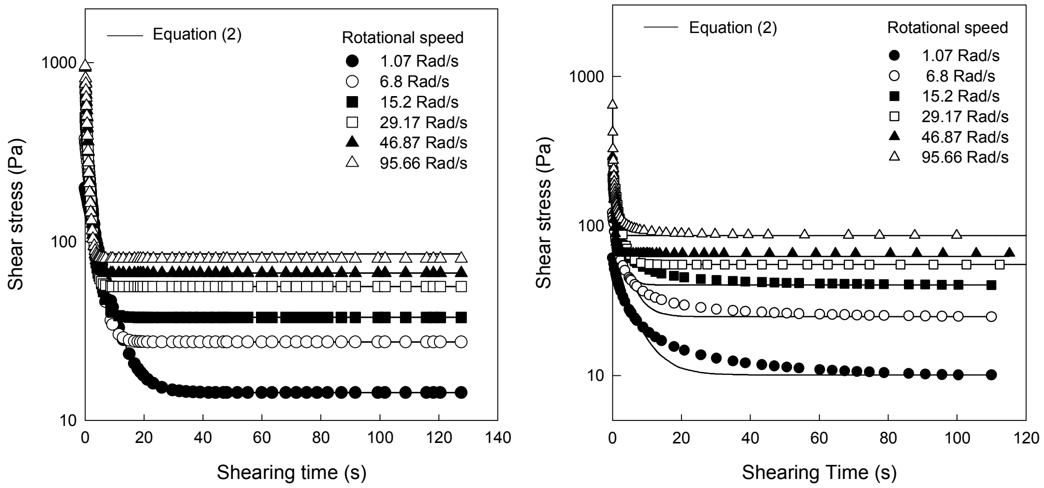

Figure 1: Transient stress data at fixed solid volume fraction of 0.2 under different rotational speeds: (a) MHD; (b) SSR™. The figure shows the effect of the rotational speed on the stress.

Figure 2 shows the effect of the solid volume fraction (temperature) on the stress. The data show that f has a significant effect on the initial shear stress τ_o, (the shear stress at $t \rightarrow 0$) and equilibrium shear stress (τ_e, the shear stress at $t \rightarrow \infty$) . Typically the magnitude of τ_o and τ_e increase with increasing f.

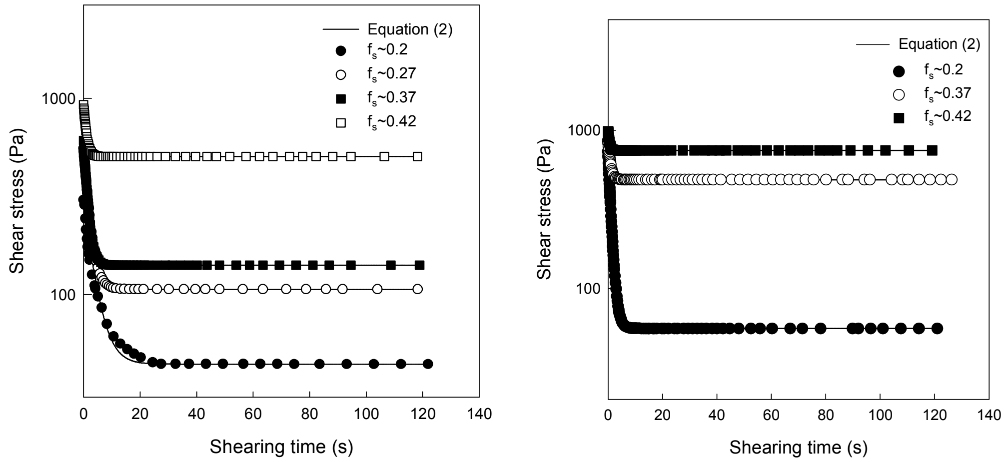

Figure 2: Transient shear stress data at fixed rotational speed of 29.17 Rad/s: (a) MHD; (b) SSR™ . The figure shows the effect of the solid volume fraction (temperature) on the stress.

The data can be quantified to some extent by using a simple exponential rate equation [20]:

$$\frac{d\tau}{dt} = -\frac{1}{k}(\tau - \tau_e) \qquad (1)$$

The parameter k represents the characteristic time that controls the rate at which the stress decays. This parameter is a function of the degree of agglomeration of particles, local f etc., and the conditions of the experiment, i.e. the applied rotational speed and the implied level of rate of strain $k(\dot{\gamma}, f, \ldots)$ [3,4] . The exact functional form of k is crucial in the description of the thixotropy of SSM slurries. Many works use Eq. (1) to describe the time-dependent behavior of semi-solid metal slurries. For simplicity, a constant value for k is assumed for each run. Unfortunately while this simplifies the analysis and it is attractive to use its validity is limited. Actually k is a strong function of $\dot{\gamma}$, and since during each experiment $\dot{\gamma}$ changes with the deformation and breakdown of the material (and, hence, with time), k cannot be considered as a constant. Nevertheless analysis of the data (not shown here due to lack of space) by assuming a constant k shows that τ_o and τ_e for the SSR™ samples are lower than those of the MHD samples. The rate of structure breakdown (k) for the SSR™ samples is higher than the rate for the MHD samples. This may indicate that SSR™ samples have a less developed solid network than the MHD samples

The data can also give information on the initial "strength" of the slurry (τ_y) in the limit $\omega \to 0$. The finite yield stress is responsible for many flow phenomena and "apparent irregularities" observed in SSM flows and is the key for understanding such flows. Table I shows the yield stress obtained by linear extrapolation of the constants. In general the data confirm the lower values for SSR material compared to the MHD materials. For f_s~0.42 the results show a decrease in the yield stress contrary to an expected higher value. This discrepancy may be due to slippage, or other wall-slurry interactions due to the high solid volume fraction. This issue needs a more detail investigation.

Table I. The initial finite yield stress as a function of the solid volume fraction

MHD	Yield stress ($\omega \to 0$)	SSR™	Yield stress ($\omega \to 0$)
f_s~0.2	60.77 Pa		
f_s~0.27	195.69 Pa	f_s~0.27	166.84 Pa
f_s~0.37	583.52 Pa	f_s~0.37	552.10 Pa
f_s~0.42	537.43 Pa	f_s~0.42	514.1 Pa

The Herschel-Bulkley model (H-B) has been shown to represent well the steady-sate behavior of SSM slurries [1]. According to the model, the material will not flow unless the local stress τ exceeds τ_y. Formally, the model is expressed by: $\tau = \tau_y + K\dot{\gamma}^n$ when $\tau > \tau_y$ and $\dot{\gamma} = 0$ when $\tau < \tau_y$. K is known as the consistency index and n is the power-law exponent. In the case of the H-B fluid, the power-law expression for $\dot{\gamma}$ is no longer valid. Depending on the value of τ_y, the error in estimating $\dot{\gamma}$ can be orders of magnitude off from its true value, since τ_y determines the effective gap of the rheometer (the gap where yielding takes place). Depending on τ_y, the gap can be arbitrarily small and the resulting rate of strain arbitrarily large. Unfortunately, it is common practice in the literature to evaluate $\dot{\gamma}$ using power-law or Newtonian expressions.

The velocity for the H-B fluid can be shown to be given by

$$u(r)=r\left[-\int_{R_1}^{r}\frac{1}{r'}\left(\frac{C}{Kr'^2}-\frac{\tau_y}{K}\right)^{1/n}dr' + C_1\right] \qquad \text{where} \qquad \omega=\int_{R_1}^{R_2} I(r',C)\,dr' = \mathbf{C}_1 \tag{2}$$

C is evaluated using Simpson's integration rule for the integral coupled with a Newton-Raphson iteration procedure. A special non-linear least square fit procedure is developed to evaluate the material constants. The least square is adjusted to produce only positive values for τ_y. The steady-state flow properties are shown in Fig. 3 and the H-B constants are given in Table II. Table II indicates that τ_y^{H-B} and K increase with increasing solid volume fraction, while n decreases with increasing solid volume fraction (f). The decrease in n with increasing f suggests that the viscosity of semi-solid slurries becomes less sensitive to the shear rate as f increases (temperature decreases). In general, for the same values of f and τ_y^{H-B}, the values of K in the MHD samples are consistently higher than those in the SSR™ samples. On the other hand, the power-law exponent is consistently higher in the SSR™ samples.

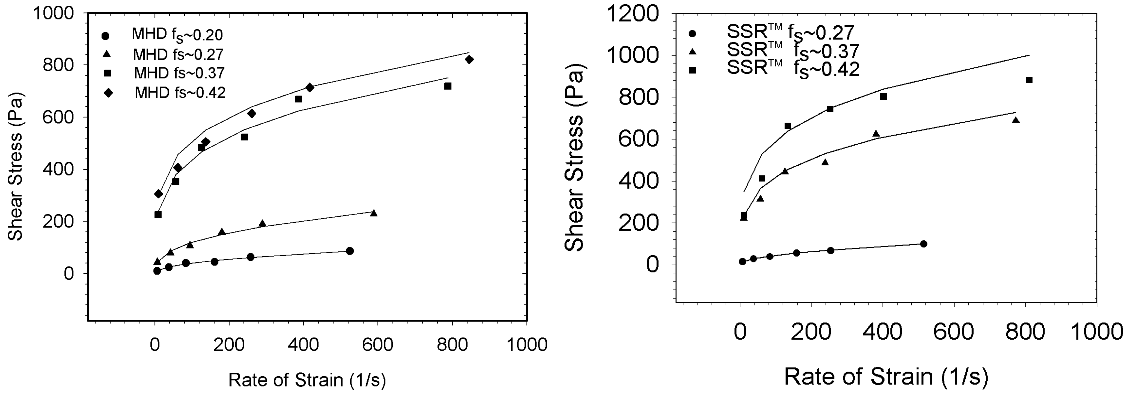

Figure 3: Steady-state flow curves: (a) MHD; (b) SSR™.

Table II: The steady-state Herschel-Bulkley constants (τ_y^{H-B}, K and n)

	f_s~0.2	f_s~0.27	f_s~0.37	f_s~0.42
MHD				
τ_y^{H-B} (Pa)	0.475	1.868	5.425	25.03
K (Pa.s^n)	4.596	19.717	130.341	156.213
N	0.468	0.389	0.262	0.247
	f_s~0.2	f_s~0.27	f_s~0.37	f_s~0.42
SSR™				
τ_y^{H-B} (Pa)	-	1.675	24.86	54.286
K (Pa.s^n)	-	4.575	112.408	156.278
N	-	0.489	0.276	0.269

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