<http://www.diva-portal.org>

Postprint

This is the accepted version of a paper published in *Solid State Phenomena*. This paper has been peer-reviewed but does not include the final publisher proof-corrections or journal pagination.

Citation for the original published paper (version of record):

Payandeh, M., Jarfors, A., Wessen, M. (2013)

Effect of superheat on melting rate of EEM of Al alloys during stirring using the RheoMetal process.

Solid State Phenomena, 192-193: 392-397

<http://dx.doi.org/10.4028/www.scientific.net/SSP.192-193.392>

Access to the published version may require subscription.

N.B. When citing this work, cite the original published paper.

Permanent link to this version:

<http://urn.kb.se/resolve?urn=urn:nbn:se:hj:diva-19514>

Effect of superheat on melting rate of EEM of Al alloys during stirring using the RheoMetal process

Mostafa Payandeh, Anders E. W. Jarfors, and Magnus Wessén

Jönköping University / School of Engineering
Jönköping, Sweden

e-mail: mostafa.payandeh@jth.hj.se

e-mail: anders.jarfors@jth.hj.se

e-mail: magnus.wessen@jth.hj.se

Keywords: Semi-Solid casting, melting, non-dendritic slurry, RheoMetal Process, Experimental Design, Rapid S, RSF

Abstract. The RheoMetal process (previously called the Rapid S- and RSF- process) is a novel method to produce cost effective, high quality, semisolid slurries for component casting. The RheoMetal process uses an Enthalpy Exchange Material (EEM) as cooling agent to absorb heat and produce a slurry. Critical process parameters to create a slurry by robust melting of the EEM are alloy content, stirring speed, EEM to melt ratio, EEM temperature, EEM microstructural characteristics and melt superheat.

In this paper, the melting sequence and melting rate of the EEM was studied experimentally. The effect of EEM composition, as well as superheat, on evolution of shape and dimension of the EEM during stirring was investigated. Initial material freezing onto the EEM was observed, followed by a stationary phase with subsequent gradual melting of the EEM. It was shown that the characteristics of freeze-on layer were strongly correlated to melt superheat, EEM temperature as well as material composition, hence also has significant influence on the melting sequence.

Introduction

Non-dendritic slurry production for semi-solid casting is a key factor to improve quality of finished products[1, 2]. The semisolid material can be produced by reheating a non-dendritic feedstock bar for use in thixocasting or by cooling a melt in a controlled fashion to yield a globular slurry for use in rheocasting. Despite that the raw material must be prepared in advance for thixocasting, in contrast to rheocasting, where a normal melt is used, thixocasting has reached a high degree of industrialization. Thixocasting has now evolved into a process capable of preparing slurries with excellent thixotropic characterization and process controllability [3, 4].

Recently, a new process called the RheoMetal process, that uses a so called Enthalpy Exchange Material (EEM) as an internal cooling agent, offers means to enhance controllability and efficiency of the cooling process compared to traditional direct slurry making methods based on external cooling concepts. In the RheoMetal process enthalpy exchange occurs between a superheated melt and a solid metal alloy piece (hence the EEM). As the EEM is heated and melts, the slurry is generated [5]. In the present study, a general description of melting during stirring of an EEM into the melt of the same composition will be presented. The EEM volume changes during slurry generation are to be described using regression analysis of statistically significant parameters based on experimental data.

Experimental

Two aluminium alloys, Stenal Rheo1 and alloy 6082 were selected. Table 1 shows general properties of these alloys. As independent fixed parameters the EEM to melt ratio, EEM temperature and stirring speed were set to 6-7 wt%, 150 °C and 900 rpm respectively. The melt was prepared in 50 kg batches using a standard resistance furnace. EEM cylinders of typically a diameter of 38 mm and 25 mm height were prepared in batches of 20. The EEM mould was kept between 80 to 120 °C to yield the same solidification conditions for all EEMs. Superheat was

controlled by measuring the temperature of the melt using a K-type thermocouple. The EEM stirring time, as well as stirring speed, was adjusted by a *Siemens Simatic S7-200 PLC*TM. The EEMs were preheated to 180 °C and attached to rotational device a few seconds before slurry formation started.

Table 1- Composition [wt%] and liquidus temperature [°C] of alloys used for experimental work

Alloy	Si	Fe	Cu	Mn	Mg	Zn	Ni	Cr	T _L
6082	0.95	0.17	0.011	0.49	0.61	.0038	0.012	0.025	651
Stenal Rheo1	5.8	0.6	2.2	0.28	0.03	0.5	0.14	0.025	615

Figure 1 shows the experimental set-up used. Approximately 1 kg of melt was picked up using a cylindrical steel ladle and as temperature of the melt reached target superheat, the EEM was immersed into the melt at a rotation rate of 900 rpm. This was the maximum rotation rate possible without excessive vortex formation in the ladle. Before complete melting, the EEM was extracted at predetermined melt / EEM contact durations.

Figure 1-Experimental set-up for melting study

The screening tests had to be performed to find best windows of experiment for each alloy separately. Subsequently, a D-Optimal experimental plan was generated, using DesignExpertTM, illustrated in Figure 2 [6]. The numbers in this figure correspond to number of replicates for those experimental conditions. Eq. 1 indicates selected response as a ratio of the initial EEM volume to that after the incomplete melting. The volume of EEM was found by inserting the EEM into the water and use $v = m/\rho_w$ formula (Archimedes principle). The volume of the central steel rod was also compensated for. The density of water (ρ_w) was corrected variations in ambient temperature [7].

$$\text{volume ratio} = \frac{(v_{\text{EEM}} - v_{\text{rod}})_{\text{after melting}}}{(v_{\text{EEM}} - v_{\text{rod}})_{\text{before melting}}} \quad (1)$$

Figure 2- Running points base on D-optimal algorithm Stenal Rheo1(left) Alloy 6082(right)

Results

Figure 3 illustrates the actual versus predicted responses for both alloys. The predicted values have been calculated by Eq. 2 and Eq. 3, which are the regression models based on the experimental data. These models describe the EEM volume ratio as a function of stirring time (A) and superheat (B) during melting process for Stenal Rheo1 and alloy 6082 respectively. Table 2 contains analysis of variance results for these models. The p -value less than 0.05 indicates statistical significance of the model, individual parameters and interactions terms as there is only a 5% likelihood that it is due to noise [8]. The four terms A (Stirring time), B (Superheat), AB (Interaction between Stirring time and Superheat, A2 (nonlinear regression term for Stirring time) are significant for Stenal Rheo1. Similarly, the terms A, B, A2 are significant but the interaction term (AB) with the p -value between 0.05 and 0.10 is marginally significant. Due to satisfaction of the model hierarchy, this term is included in the model. No significant lack of fit also shows that the polynomial model is fitting all of the design points well. The response for both cases was transformed using a square root transformation for best possible model fit.

The contour plot of volume ratio of EEM versus superheat and stirring time for both alloys are presented in Figure 4. The time to complete melting for Stenal Rheo1 alloy ranged from 15 to 20 seconds depending on superheat. The time to complete melting was shorter for alloy 6082; needing about 10 to 12 seconds to complete dissolution, depending on superheat. At low superheat, maximum freeze-on layer for Stenal Rheo1 occurred after 8 seconds, while for 6082 the maximum EEM volume occurred instantaneously at immersion.

Figure 3- Predicted value versus. Actual value of volume ratio; Stenal Rheo1(left) Alloy 6082(right)

$$\sqrt{\text{volume ratio}} = 0.13 + 0.26 A + 0.017 B - 3.22 \cdot 10^{-3} AB - 0.014 A^2 \quad (2)$$

$$\sqrt{\text{volume ratio}} = 1.05 + 0.14 A + 0.015 B - 5.76 \cdot 10^{-3} AB - 0.013 A^2 \quad (3)$$

Table 2-Analysis of variance (ANOVA) table

	Stenal Rheo1		6082	
Source	p -value		p -value	
Model	< 0.0001	significant	< 0.0001	significant
A-Stirring Time	< 0.0001	significant	< 0.0001	significant
B-Superheat	0.0003	significant	0.0144	significant
AB	0.0022	significant	0.0983	Marginally significant
A ²	< 0.0001	significant	0.0265	significant
Lack of Fit	0.2617	not significant	0.1178	not significant
R Squared	0.824		0.743

Figure 4- The contour plot of the EEM volume ratio; Stenal Rheo1(left) Alloy 6082 (right)

As a preliminary study of the freeze-on layer, the chemical composition of one distinctive part from each alloy was determined using optical emission spectrometry. Based on the chemical composition, the solidification curve of each freeze-on layer as well as EEM were calculated with the help of ThermoCalc Software [9, 10] and illustrated in Figure 5. The temperature sensitivity of fraction of solid phase can be described as $S^* = df_s/dT$, where f_s is the fraction of solid phase in the melt and T is temperature [11]. The average S^* value of 0.034 for 6082 alloy as compared to 0.013 for Stenal Rheo1 from liquidus temperature (T_L) down to the temperature corresponding to 80% in solid fraction (T_{80}), results in a temperature interval of 20°C and 80°C respectively. Therefore, rapid formation of freeze-on layer in 6082 alloy can be described by higher temperature sensitivity of solid fraction at temperature interval between T_L and T_{80} .

Table 3- Composition [wt%]and liquidus temperature [°C] of the freeze-on layer around EEM

Freeze-on layer	Si	Fe	Cu	Mn	Mg	Zn	Ni	Cr	T_L
6082	0.61	0.03	0.011	0.4	0.45	.0038	0.0002	0.01	653
Stenal Rheo1	3.6	0.3	1.3	0.28	0.05	0.5	0.01	0.02	638

On other hand in the EEM melting process, the value of 0.001 for temperature sensitivity of 6082 alloy near solidus temperature is followed by very rapid increasing of S^* to 0.031 after temperature corresponding to solid fraction of 80%. This behaviour results in longer stationary period of EEM and melting off in shorter time for EEM which is made 6082 alloy. In Stenal Rheo1 alloy, by increasing the Si-content, the α -Si eutectic is formed in the grain boundaries. The lower melting temperature of the eutectic makes this a preferred site for melting as compared to the primary phase [12, 13]. Therefore, the melting of eutectic phase reduces the local strength of the EEM significantly and leads to separation of α -phase particles by the external shear forces and consequently gradual melting process.

From the process duration perspective, the longer melting procedure for Stenal Rheo1 arises from the significant difference between composition of EEM and freeze-on layer which leads to higher liquidus temperature for freeze-on layer. By means of K-type thermocouple the final slurry temperature for Stenal alloy and 6082 alloy were measured at 607°C and 646°C respectively. These temperatures are corresponded to 18% and 50% solid fraction for EEM and freeze-on layer for Stenal Rheo1 based on the evolution of the fraction solid versus temperature (Figure 5). For alloy 6082 at slurry temperature, solid fraction will be 38% for EEM and 61% for freeze-on layer. The difference between solid fraction of EEM and freeze-on layer due to micro segregation can be also a possible description about higher solid fraction in the final slurry compare to predicted solid fraction by assuming Scheil segregation which had been discussed by Granath *et al* [14, 15].

Figure 5- Solidification curve for Stenal Rheo1 (left) and 6082 (right)

Figure 6 shows dissolution of EEM over stirring time for low and high superheat. Besides, the confidence interval (CI band) of each model is indicated by two dashed lines. Confidence intervals indicates the range of possible effect sizes compatible with the data. These non-parallel curves in interaction plot specify that the effect of time factor depends on the degree of the superheat. Also, it indicates that the delay in the melting procedure at low superheat for both alloys be governed by formation and dissolution of freeze-on layer.

Base on the solidification curves (Figure 5), 6082 alloy has approximately three times larger value of the temperature sensitivity at liquidus temperature S_L^* than Stenal Rheo1 which due to relation between the formation of a freeze-on layer and the temperature sensitivity, leads to higher standard deviation in 6082 alloy at the beginning of process. As well, at low superheat for both alloys constant deviation from the norm in contrasted with decrease of this deviation at high superheat, states the significant relation between freeze-on layer and magnitude of standard deviation. In spite of the sources of variation are not fully apparent, some confounding variables such as EEM symmetry and EEM to melt ratio can be varied due to random error in EEM preparation step and become sources of inaccuracy. EEM non-symmetry can change the freeze-on pattern and increase the vibration in the system, which also can lead to uncontrolled disintegration of the EEM because of crack propagation and thereby a low slurry quality. The role of EEM as a heat absorber and relation of volume of EEM with superheat [14] makes the ratio of EEM to melt as an important variable.

Figure 6- The interaction plot for superheat and stirring time on the volume ratio; Stenal Rheo1(left) Alloy 6082(right)

Conclusion

The evolution of the EEM shape at the initial stage of melting has strong effect on the slurry formation procedure by increasing process duration. Relation between the formation of a freeze-on layer and the temperature sensitivity, S^* leads to different freezing route for Stenal Rheo1 as a high Si content alloy and 6082 as a low Si content alloy. Moreover, the composition of the freeze-on layer shows reduction of alloy elements in this part for both alloys. Additionally, the larger difference of composition for Stenal Rheo1 leading to a higher liquidus temperature of freeze-on layer than the base EEM. Moreover by investigating of interaction plots, large deviation of experimental result from statistical models, shows considerable influence of confounding variables. Furthermore, reduction of standard deviation by increasing superheat indicates the significant effect of freeze-on layer on instability of the process. Consequently, for industrial operations a high superheat is preferred due to best possible robustness.

Acknowledgement

This research work was supported by the KK-foundation (RheoCom project 20100203) which is gratefully acknowledged. The authors would like to thank Stena Aluminium AB and Sapa AB for the supply of materials. Huawei Technologies' Sweden AB and COMPtech AB are acknowledged for help and technical support.

Reference

- [1] Flemings M, Mehrabian R. Casting semi-solid metals. AFS Trans. 81.(1973) 81-8.
- [2] Ilegbusi O, Brown S. Mold filling of semisolid metal slurries. Journal of materials engineering and performance. 44.(1995) 486-93.
- [3] Fan Z. Semisolid metal processing. International Materials Reviews. 472.(2002) 49-85.
- [4] Kirkwood DH, Suéry M, Kapranos P, Atkinson HV, Young KP, Semi-solid Processing of Alloys: Springer 2009.
- [5] Wessén M, Cao H, The RSF technology; A possible breakthrough for semi-solid casting processes.Proc. Int Conf of High Tech Die Casting; 2006; Vicenza, Italy.
- [6] Stat-Ease, Inc. Design-Expert® Version 8, 2011
- [7] Weast RC, Astle MJ, Beyer WH, CRC handbook of chemistry and physics: CRC press Boca Raton, FL, 1988.
- [8] Montgomery DC, Design and analysis of experiments: John Wiley & Sons Inc, 2008.
- [9] Sundman B, Jansson B, Andersson JO. The thermo-calc databank system. Calphad. 92.(1985) 153-90.

- [10] Saunders N. The application of calculated phase equilibria to multi-component aluminum alloys. *Keikinzoku/Journal of Japan Institute of Light Metals*. 513.(2001) 141-50.
- [11] Hirt G, Kopp R, Thixoforming: Semi-solid metal processing: Wiley, 2009.
- [12] Wang Q, Davidson C. Solidification and precipitation behaviour of Al-Si-Mg casting alloys. *Journal of materials science*. 363.(2001) 739-50.
- [13] Rettenmayr M. Melting and remelting phenomena. *International Materials Reviews*. 541.(2009) 1-17.
- [14] Granath O, Wessén M, Cao H. Determining effect of slurry process parameters on semisolid A356 alloy microstructures produced by RheoMetal process. *International Journal of Cast Metals Research*. 215.(2008) 349-56.
- [15] Granath O, Wessén M, Cao H. Influence of holding time on particle size of an A356 alloy using the New Rapid Slurry Forming process. *Int Conf of High Tech Die Casting; Vicenza, Italy* (2006).