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Experimental Study on Viscosity and Phase Segregation of Al–Si Powders in Microsemisolid Powder Forming

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Abstract
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Semisolid powder forming is a promising approach for near-net shape forming of features in macro-/microscale. In this paper, viscosity and phase segregation behavior of Al–Si powders in the semisolid state were studied with back extrusion experiments. The effects of process parameters including shear rate, extrusion ratio, heating time, and precompaction pressure were analyzed using the design of experiments method. The results showed that the effects of shear rate, extrusion, ratio and heating time were statistically significant factors influencing the viscosity. The semisolid state powders showed a shear thinning behavior. Moreover, microstructure analysis of extruded parts indicated severe phase segregation during the forming process. As the extrusion opening became small (~400 μm), the phase segregation increased. This study expanded the semisolid processing technology by exploring the use of powdered materials instead of typical bulk materials for applications in micro-/mesomanufacturing. Replacing bulk materials with powdered materials may add a new dimension to the technique by allowing tailoring of material properties. [DOI: 10.1115/1.4000636]

Keywords: semisolid forming, powder processing, microforming, phase segregation, viscosity, back extrusion

1 Introduction

There has been increasing demand for 3D microparts for applications in integrated electronic devices, sensors, micro-actuation systems and energy devices [1]. Current micromanufacturing methods, however, are not able to satisfy the needs arising from various industrial sectors, especially when mass production is considered. Recently, techniques involving processing of metallic alloys in the semisolid state [2–7] have been studied due to their advantages over conventional forging [8] and casting [9] processes. The unique behavior of the material having both the liquid and solid phases enables forming of complex shapes at reduced loads. The feasibility of applying the technology to micro-/mesoscale part fabrication has also been reported [2–4]. This study further expands the semisolid processing technology by exploring the use of powdered materials for manufacturing of microparts instead of using typical bulk materials. The semisolid bulk forming and semisolid powder forming (SPF) are schematically compared in Fig. 1. There are several advantages of SPF over semisolid bulk forming. First, the semisolid bulk forming requires the break-down of the dendritic microstructure in the feedstock alloy materials while fine microstructures are readily available for the SPF. Second, replacing the bulk materials with powders enables mixing of various powders for improved properties and fabrication of locally tailored structures.

In this paper, two fundamental aspects of the SPF have been investigated using back extrusion tests. The size of extrusion openings were between 400 μm and 1200 μm to study the flow behavior and phase segregation of the semisolid state powders in micro-/mesoscale ranges. A design of experiment analysis was performed to understand the effects of process parameters, which includes precompaction pressure, shear rate, heating time, and extrusion ratio on the viscosity and phase segregation. In addition, microstructures of the back extruded parts have been examined to gain insights into SPF.

2 Background

Processing of powder materials in the semisolid state has a rather short history when compared with commonly practiced bulk material processing, which started in the 1970s [6]. A summary of various processing routes of SPF is shown in Fig. 2 [10–19]. In general, four basic steps are required: powder preparation, powder compaction, preheating, and semisolid forming. Powders may be mixed either from at elemental or prealloyed state. The powder compaction can take place at room temperature (cold pressing [11,12,19]) or at elevated temperatures (hot pressing [10,16,17]). The heating may be achieved by induction heating [16] or direct furnace heating [10–12,17]. Finally, at a designated temperature, the semisolid state powders are formed into near-net shape parts. Homogeneous and well-densified structures were observed in macroscale parts [10–12,19]. Also, the mechanical properties of the parts produced by SPF were comparable to conventional forming methods [11,13,14,16].

Researchers have investigated flow characteristics of bulk materials in the semisolid state by measuring viscosities. Various techniques such as concentric cylinder rheometer [20,21], capillary viscometer [22], compression tests [23–25], and back extrusion [26–28] have been used. The back extrusion technique for viscosity measurement of semisolid materials is a newer development. It can be used for actual part fabrication, and therefore, the test conditions are similar to the actual fabrication process. The experimental results indicated shear thinning behaviors of the bulk semisolid materials, i.e., the apparent viscosity decreased as the shear rate increased [27,29–32]. As expected, apparent viscosity increased as the solid fraction of semisolid material increased [32–35].

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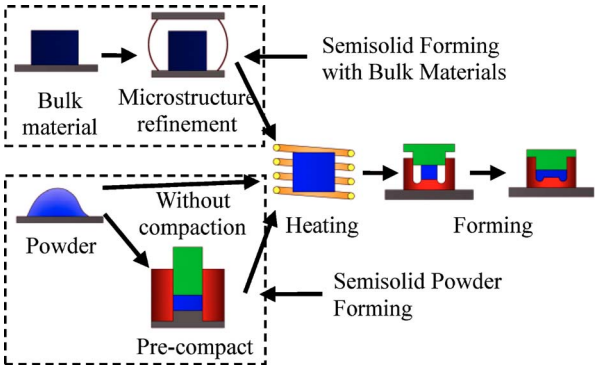

Fig. 1 Comparison between bulk semisolid forming and semisolid powder forming

The phase segregation during forming of semisolid materials is another important phenomenon that can influence the final part quality [36]. In the back extrusion experiments of bulk semisolid materials at macroscale, a higher liquid fraction was typically observed in the extruded region than the remaining region [28,37,38]. The effects of processing parameters including ram speed, extrusion opening, and billet heating time on phase segregation were investigated [16,28,36,39]. The phase segregation increased as the ram speed or the shear rate decreased [16,28,40]. The experiments also showed that phase segregation was eliminated when the shear rate increased to a certain level [36,41]. In addition, it was speculated that the extrusion ratio affected phase segregation significantly [28,41,42]. Xing and Tan [43] studied mold filling behavior of semisolid slurry and also investigated phase segregation. The development of models that can predict the phase segregation of bulk semisolid material are also in progress [36,38,44,45]. The modeling results demonstrated a strong correlation between the segregation behavior with the microstructure, permeability and liquid phase percolation velocity during extrusion of the bulk semisolid materials.

Past and current research on the semisolid processing shows a great potential for the technology to become a viable manufacturing route for micro-/mesosize features. With powders as feedstock materials, the technology can exploit the advantages of powder metallurgy. However, no quantitative work has been performed on the flow behavior and phase segregation of powders in the semisolid state at micro-/mesoscale ranges (10–5000 μm).

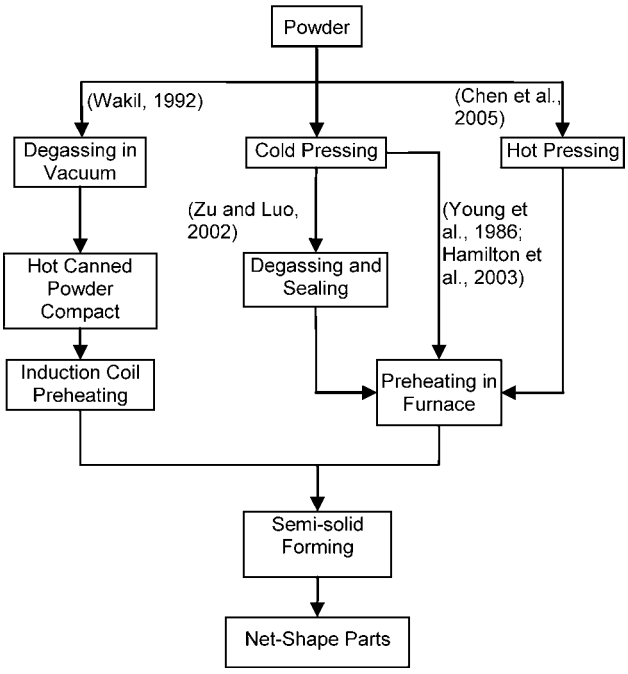

Fig. 2 Process routes of various semisolid powder forming

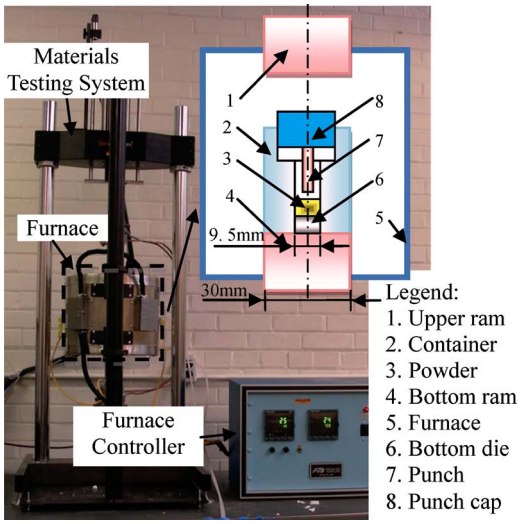

Fig. 3 Setup for back extrusion of semisolid powders

3 Experimental Method

3.1 Experimental Setup and Analysis Method. A back extrusion test has been employed to study the phase segregation of aluminum-silicon (Al–Si) powders in the semisolid state. As shown in Fig. 3, fabricated die set is placed within the furnace (Applied Test System, Inc., Butler, PA, Series 3210) where the load and movement of upper ram are controlled and measured by the materials testing system (TestResources Inc., Shakopee, MN, 800LE). Since the diameter of the container is fixed, the extrusion opening can be changed by varying the punch diameter. The Al–Si powder was poured into the container and was compacted (or kept in a loosed state) before placing in the furnace. After the temperature reached the set point, it was sustained for a required hold period before the upper ram moved down at a fixed velocity. The semisolid state powder (or powder compact) was pushed into the extrusion opening, forming a cup-shaped part. The final pressure applied in each experiment was set to 100 MPa.

Hypereutectic prealloyed Al–50Si powder (supplied by Ames Laboratory of U.S. Department of Energy) was used in this study due to its potential applications for high wear resistant components. The measured mean diameter and particle size distribution are summarized in Table 1. The microstructure of the original powder and phase diagram of Al–Si binary alloy system are shown in Figs. 4 and 5, respectively. The Al–50Si is composed of liquid and pure solid Si phase between 577°C and 1051°C, and therefore, a large window of operating temperature is provided.

The experiments were carried out at 610°C at which temperature the solid fraction was 0.41 (calculated by THERMOCALC version 3.1). At 610°C, all of the Al (50 wt % of the total weight) and a limited amount of Si (9 wt % of the total weight) are

Table 1 Size distribution of the original Al–50Si powder

Mean size (μm)	Powder size (μm)		
	d_{10}	d_{50}	d_{90}
16.96	9.55	15.53	25.69

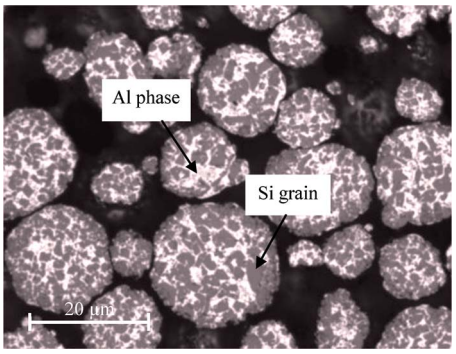

Fig. 4 Original Al–50Si powder used in the experiments

melted. Therefore at 610 °C, the Si volume fraction in the hypoeutectic liquid phase is about 16%.

A design of experiments analysis was performed to understand the effects of process parameters on the viscosity and phase segregation. Four experimental parameters including shear rate, precompaction pressure, extrusion ratio, and heating time were selected. The commercial statistical software, JMP, was utilized to produce the experiment design and to perform the data analysis. The experimental array and results are summarized in Table 2. The samples were molded and then cut into half (Isomet 2000 precision saw, Buehler Ltd., Lake Bluff, IL). They were ground and polished following appropriate procedures. The Si grains were observed using optical microscope without aid of any etchant. The microstructures of the samples were observed with an optical microscope (Zeiss, Axiovert 200 M). The commercial software, IQMATERIAL, was used to analyze the microstructure of the samples.

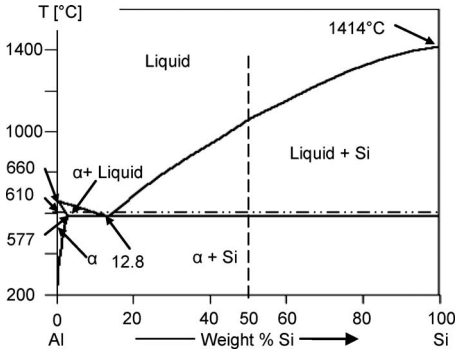

Fig. 5 Phase diagram of Al–Si binary alloy system

The Si phase, Al phase, porosity, and Si grain characteristics were analyzed.

3.2 Calculation of Viscosity and Phase Segregation. In the back extrusion experiment, the apparent viscosity and shear rate can be calculated by a set of equations developed by Loue et al. [26]:

$$\eta_{\text{app}} = \frac{1}{2\pi\lambda C_1 V_p} \cdot \frac{dF}{dt} \tag{1}$$

$$\dot{\gamma}_{\text{av}} = \frac{C_1\left[\ln\left(\frac{-C_1}{2C_2R_cR_p}\right) - 1\right] - C_2(R_c^2 + R_p^2)}{R_c - R_p} \tag{2}$$

where dF/dt is the load change rate, R_c and R_p are the radii of the container and punch, respectively, V_p is the punch velocity, and λ is the extrusion ratio. λ, C_1, and C_2 are given in the following equations:

$$\lambda = \frac{R_c^2}{R_c^2 - R_p^2} \tag{3}$$

$$C_1 = \frac{1}{\ln\left(\frac{R_p}{R_c}\right)} \cdot (C_2 \cdot (R_c^2 - R_p^2) - V_p) \tag{4}$$

$$C_2 = \frac{V_p}{(R_c^2 - R_p^2) - (R_c^2 + R_p^2)\ln\left(\frac{R_p}{R_c}\right)} \tag{5}$$

Apparent viscosity and shear rate can be obtained from geometric dimensions (R_c and R_p), punch velocity (V_p), and rate of force change (dF/dt).

In this paper, a simple mathematical approach to quantify the severity of phase segregation based on 2D images is developed. The deviation of the element content from its original content at a local position i can be defined as

$$ps_i = |c_i - c_0| \tag{6}$$

where ps_i is the measure of phase segregation at local position, c_i is the element fraction (e.g., Si fraction) at a position i, and c_0 is the initial element fraction. Therefore, the phase segregation over the total cross sectional area can be defined as

Table 2 Experiment array and result

	Parameters				Results	
No.	λ	$\dot{\gamma}_{\text{av}}(1/\text{s})$	P (MPa)	t (min)	η_{app}	ps
1	3.27273	4	0	20	44068.9	0.248
2	3.27273	4	0	40	19342.7	0.304
3	6.12268	100	0	20	344.7	0.245
4	6.12268	100	50	20	308.3	0.199
5	6.12268	4	100	40	2881.8	0.291
6	6.12268	20	50	40	1012.6	0.307
7	3.27273	20	100	20	7239.2	0.232
8	2.28571	4	50	20	71235.3	0.230
9	2.28571	20	100	20	8059.3	0.270
10	2.28571	20	0	40	4183.5	0.240
11	3.27273	100	50	40	1156.1	0.210
12	2.28571	100	100	40	1296.4	0.218

Note: λ is extrusion ratio, $\dot{\gamma}_{\text{av}}$ is shear rate, P is precompaction pressure, and t is heating time.

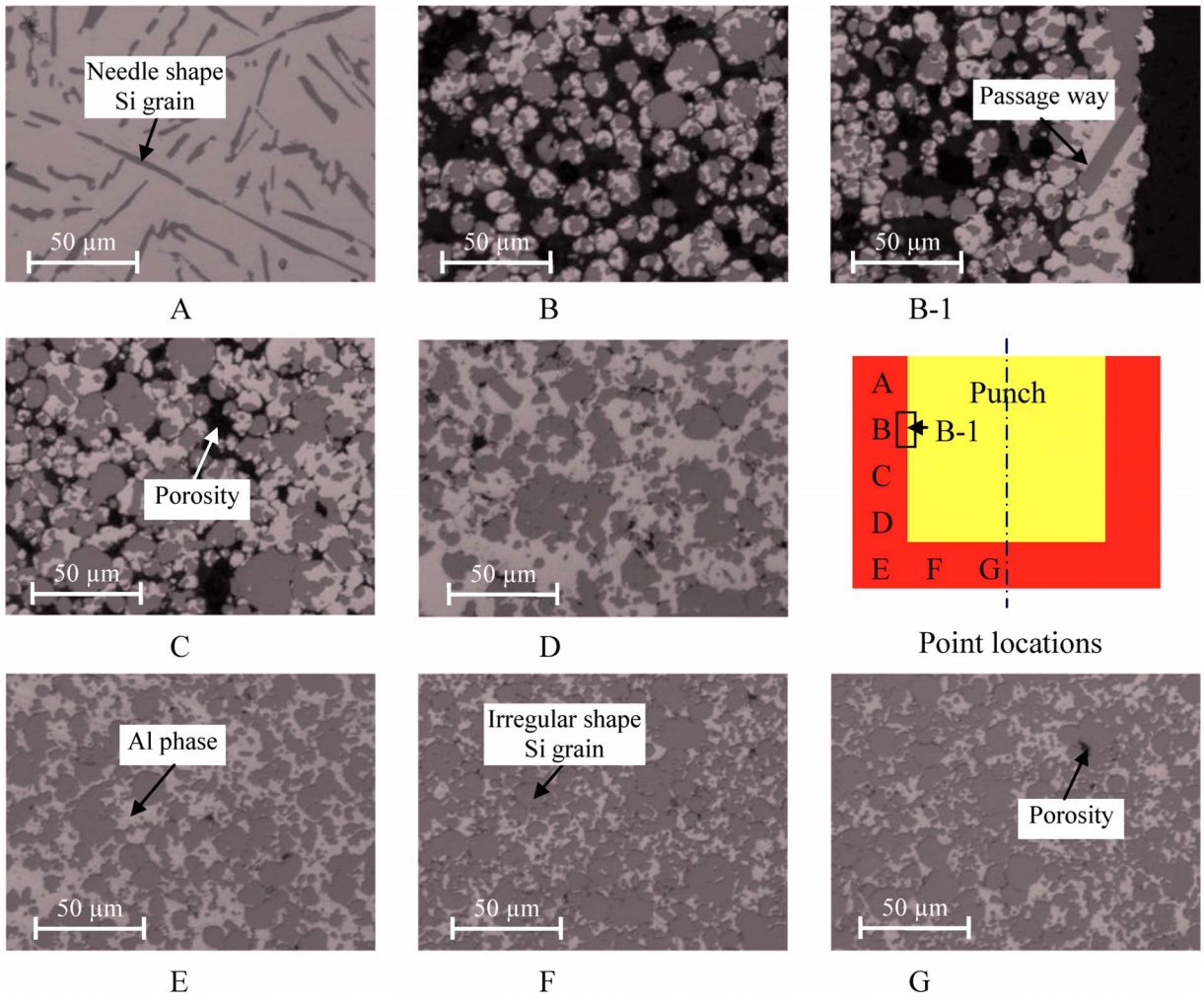

Fig. 6 Microstructures at different locations of a back extruded part (run No. 11 in Table 2)

$$ps = \frac{1}{A}\int_A ps_i dA \tag{7}$$

where A is the total area of interest. Equation (7) can be discretisized to calculate phase segregation over finite number of elements with equal area

$$ps = \frac{1}{A}\sum_{i=1}^{N} ps_i A_i = \frac{1}{N}\sum_{i=1}^{N} |c_i - c_0| \tag{8}$$

where N is the total number of discretisized areas that phase segregation is measured.

4 Results and Discussion

4.1 Microstructure Analysis. Typical microstructures at various locations of an extruded part are shown in Fig. 6. Representative microstructures of positions A–G were selected from run No. 11 in Table 2. In general, connected solid structures were observed under the punch. The wall section (B–D) showed disconnected original particles. Completely different Si grain shapes were observed at location A, where the Si grains were mostly needle shape. The Si grains in other areas were spherically shaped or interconnected, irregularly shaped. The distinct difference in the shapes of the Si phase indicated different formation processes at respective locations. The microstructure of Al–Si shown at position A seemed to have resulted from the divorced eutectic structure of Al–Si. The fibrous (needle shape) Si phase is a typical eutectic Al–Si microstructure. The Si volume fraction measured at location A varied from 15% to 18%, which is consistent with the calculated value of 16%. At other locations, the size of Si grains grew larger during the heating process. Depending on the pressure applied during the forming process, either interconnected irregular Si grains (E, F, and G) or Si trapped in disconnected particles were formed.

The highest Si concentrations (a mean volume fraction of 0.73) were found in locations F and G. Since the majority of Si is in solid state at 610°C, the liquid phase was squeezed into the extrusion opening during the forming process. Therefore, locations F and G were left with high remnant Si concentrations. From the microstructural observations, it was speculated that liquid phase separated from the solid phase during the extrusion process and traveled although the wall section along the punch edge. As shown in Fig. 6(B-1), liquid phase was observed along the edges of the punch indicating a passage way for the liquid phase. Thus, the liquid phase does not homogeneously flow into the wall section. Rather, it finds a minimal resistant passage along the punch edge, and ends up at the top of the wall (location A).

4.2 Viscosity. The viscosity calculation requires force change rate measurement (dF/dt). A typical force-displacement curve is shown in Fig. 7. During the extrusion process, the structure of the semisolid material also evolves. Initially, the material is at relatively loose state. As the material is squeezed, the powders from interconnecting structure. Therefore, overall structure becomes

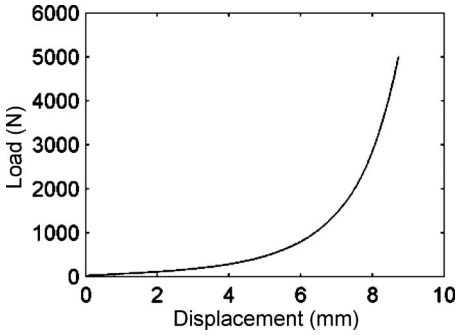

Fig. 7 Typical force-displacement during back extrusion of semisolid Al–50Si powders (run No. 1 in Table 2)

more rigid during the extrusion, as indicated by the steep increase in the slope of the force-displacement curve. For the calculation of viscosity, the initial dF/dt response was used.

A design of experiments approach was employed to determine the statistically significant factors. Analysis of variance result is summarized in Table 3. The p-value indicates the significance of each parameter. The factor is significant when p-value is less than 0.05. The statistical analysis shows that the shear rate, extrusion ratio and heating time are statistically significant factors influencing the viscosity of powder in the semisolid state. The precompaction pressure was not a significant factor for the range covered in this study.

The effects of each parameter on the apparent viscosity are reported in Fig. 8. The viscosity decreased as shear rate increased, which showed that the powder material in the semisolid state behaved like the shear thinning material. Furthermore, the viscosity decreased with the increase in extrusion ratio. When the size of extrusion opening decreased (i.e., as the extrusion ratio increased),

Table 3 Effect test result for viscosity

Source	*DF*	Sum of squares	*F* ratio	*P*-value
Heating time	1	8.56×10^8	11.29	0.0283
Extrusion ratio	2	1.07×10^9	7.03	0.0490
Precompaction pressure	2	7.71×10^8	5.08	0.0769
Shear rate	2	2.51×10^9	16.57	0.0116

Note: *DF* is degree of freedom and *F* is distribution ratio.

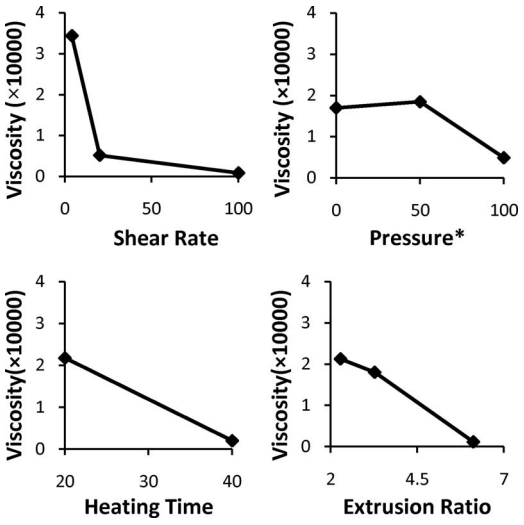

Fig. 8 Mean values of viscosity at different parameter levels *: precompaction pressure

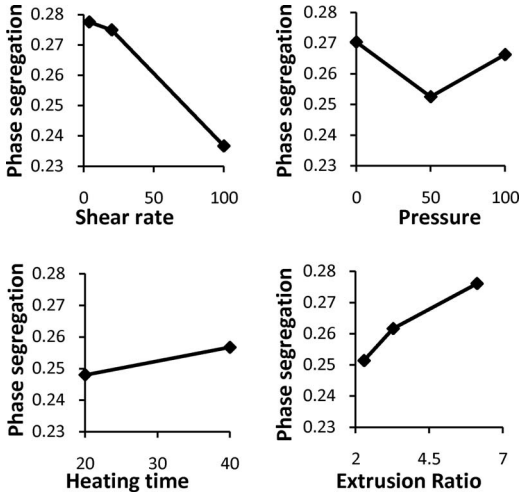

Fig. 9 Mean value of phase segregation at different parameter levels

it became more difficult for the semisolid state particles to pass through the opening. Consequently, more liquid phase passed through the extrusion opening, which resulted in lower viscosity for smaller openings. Higher phase segregation observed at smaller opening supports the speculation (see Fig. 9). The apparent viscosity also decreased as the heating time increased. As shown in Fig. 10, large amount of small Si grains were found in the images took from part heated for 20 min while most of small Si grains grew to larger ones after 40 min of heating. The mean size of the Si grains grew by 140% as the heating time increased from 20 min to 40 min. For a given extrusion opening, liquid phase separation is more likely to occur for the structures with larger Si grains causing the viscosity to drop.

4.3 Phase Segregation. The analysis of variance results for the phase segregation are summarized in Table 4. None of the

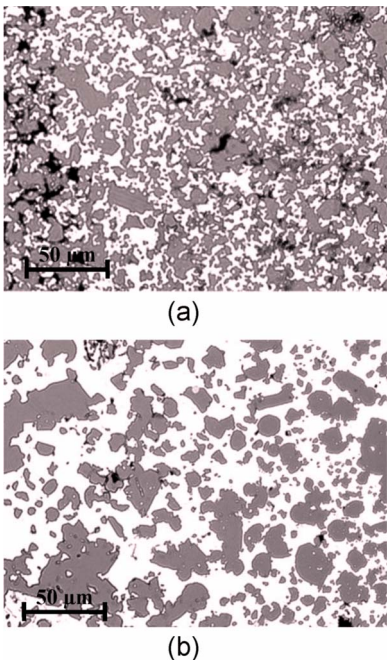

Fig. 10 Optical images showing the growth of Si grains with increasing heating time: (*a*) 20 min heating (run No. 1) and (*b*) 40 min heating (run No. 11) at location D

Table 4 Effect test result for phase segregation

Source	*DF*	Sum of squares	*F* ratio	*P*-value
Heating time	1	0.0018	2.01	0.2295
Extrusion ratio	2	0.0027	1.53	0.3199
Precompaction pressure	2	0.0005	0.31	0.7506
Shear rate	2	0.0065	3.62	0.1267

parameters had statistically significant effect on the phase segregation for the parameter range selected in this work. The absolute phase segregation values, however, were quite large with an average value of 0.25. The distribution of Si fraction is graphically illustrated in Fig. 11, which shows the severity of phase segregation. Figures 11(*a*) and 11(*b*) compare the effect of shear rate on the phase segregation—at higher shear rate, the magnitude of phase segregation is smaller.

The detailed plots for each factor are shown in Fig. 9. As the shear rate increased, the phase segregation decreased. The observation is also in agreement with results for bulk semisolid forming [28]. At higher shear rates, it is more likely that both the liquid and solid phases flow simultaneously resulting in more homogeneous microstructure. At extremely low shear rate, severe liquid phase separation was observed in prior experiments conducted by the authors. The liquid phase traveled through the particles, leaving the solid phase under the punch. The phase segregation increased with the increase in extrusion ratio. With thinner wall sections (i.e., higher extrusion ratio), the flow of the solid phase will be severely restricted, and therefore, greater phase segregation will occur.

5 Conclusions

The flow behavior and phase segregation of Al–Si alloy powders in the semisolid state were studied for the development of novel SPF for micropart fabrication. The effects of shear rate, extrusion ratio, heating time, and precompaction pressure on microstructures, viscosity, and phase segregation were investigated using back extrusion experiments.

Overall, a shear thinning behavior was observed for powders flowing in the semisolid state. The shear rate, extrusion ratio and heating time were found to be statistically significant factors affecting the viscosity. As the size of the extrusion opening decreased to 400 μm, the flow of semisolid powders was severely affected. The resulting microstructures showed significant phase segregation. Typical phase segregation amount observed for this study was *ps* value of 25% (from Eq. (8)). At smaller extrusion openings, entrapped liquid phase within the particles separated from the original structure and traveled through the thin wall section, leaving behind the solid phase contents. This resulted in lowering of apparent viscosity and increasing of phase segregation at smaller extrusion openings. The phase segregation also decreased with increasing shear rate. To minimize phase segregation at micro-/mesolength scales, higher punch speed is recommended. It was also observed that phase segregation and viscosity were not strongly influenced by the precompaction pressure range (0–100 MPa) covered in this study. Compared with the semisolid bulk forming, the flow behavior of semisolid powders showed similar characteristics. However, the semisolid powder flow is more complex due to the existence of high porosity during the initial stage. The porosity will continually evolve during the process and eventually close-out at the final stage.

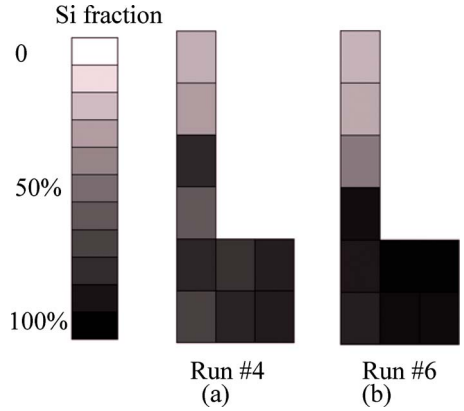

Fig. 11 Silicon fraction at different positions in the samples: part (*a*) is fabricated with $\lambda=6.12$ and $\dot{\gamma}_{av}=100$ and (*b*) $\lambda=6.12$, and $\dot{\gamma}_{av}=20$; darker area and bright area mean either high Si concentration or Al concentration, respectively

The potential of processing materials in the semisolid state is very promising. Replacing bulk materials with powdered materials may add a new dimension to the technique by allowing tailoring of material properties. In this study, two fundamental aspects, viscosity, and phase segregation behavior of the SPF was investigated for application in micro-/mesomanufacturing. To fully develop the technique, more in depth understanding of the process parameters and underlying physics is needed.

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