Investigation on the rheological behavior of Al-Fe-V-Si/Alumina composite powders

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Abstract

In this paper, the rheological properties of the feedstocks of Al-Fe-V-Si and Al-Fe-V-Si composites reinforced with alumina were investigated. The feedstocks, containing four different types of powder (Al-Fe-V-Si, Al-Fe-V-Si + 5 % Al_2O_3, Al-Fe-V-Si + 10 % Al_2O_3, Al-Fe-V-Si + 15 % Al_2O_3) and a polyethylene glycol based binder, were prepared. The volumetric fraction of the binder in the feedstock was varied between 37 % and 39 %. After the preparation of feedstocks, the rheology experiments were carried out using a capillary rheometer at various temperatures and pressures. According to the experimental results, the lowest viscosity values were found for the feedstock having the metallic powder. In addition, melt flow index was obtained to be within the range of 90–325 g/10 min for the investigated feedstocks and it increased with increasing temperature and pressure.

K e y w o r d s: metal matrix composite, powder metallurgy, rheology, feedstock

1. Introduction

Powder Injection Molding (PIM), generated from the process of plastic molding, is one of powder shaping techniques. Due to its high shape complexity and low cost, PIM has a strong potential in the production of intricate metallic and ceramic parts. This method is suited to the fast production of small parts having a weight of less than 100 g in large quantities. In PIM, the main steps are: forming a feedstock by mixing a binder and powder, pelletizing and granulating, molding, debinding and sintering [1]. All these steps are influential in the final product properties very much so that the selection of components (i.e. powder type, binder type), process parameters (pressure, temperature, flow rate and cooling rate), thermo-physical properties of feedstock and debinding method is very crucial. Prior to pelletizing and granulating step, the rheological properties of feedstock should be determined to obtain the optimum binder formulation and powder/binder ratio for a material to be produced by PIM. Among the rheological properties, viscosity is the most important one since the moldability heavily depends on it [2]. When the viscosity of feedstock increases, the PIM becomes more difficult. Therefore, the main goal in preparing the feedstock composition is to reach low viscosity levels. In order to get a proper flow in molding process, a viscosity of less than 1000 Pa s is proposed [3]. A significant number of researchers investigated the PIM process and/or the rheological properties of feedstock for various metals [4–18], ceramics [19–28] and composites [29–32]. However, there have been no extensive studies conducted on the PIM and rheological behavior of aluminum alloys and its composites up to now. The main motivation point in this study was to determine the rheological properties of Al-Fe-V-Si and Al-Fe-V-Si composites reinforced with alumina to be used in further PIM processes.

The rapidly solidified Al-Fe-V-Si alloy has a good high temperature resistance up to 350 ℃ so that there has been a considerable interest on this aluminum alloy [33–37] and its composites recently [38, 39]. The materials based on Al-Fe-V-Si alloy may provide savings in the weight of structures by replacing titanium alloys or steels. It is well known that the high tem-

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Table 1. Some important properties of the binders [24]

Property	PEG 8000	PP	SA
Density (g cm^{-3})	1.204	0.85	0.94
Melting point (℃)	60–63	189	68–70
Solubility in water	Yes	No	No

perature resistance of the alloy is related to the formation of intermetallic phases and low coarsening rate of precipitates [40–43]. The Al-Fe-V-Si alloy and its composites produced by the PIM could have an important potential to be used in some critical machine parts, working at relatively high temperatures. Therefore, the PIM may be an available and fast method to produce such materials.

2. Experimental study

2.1. Materials

The powders used in this work were supplied from Alfa Aesar. The stock numbers for the alumina, aluminum, iron and vanadium powders are 42572, 011067, 000170 and 012234, successively. The metallic powders of Al, Fe, V and Si with a high purity (≥ 99.5 %) and the ceramic powder of alumina with a purity of 99.6 % were used in the preparation of material compositions. On the other hand, the binder material was made of 65 % Polyethylene glycol (PEG) 8000 as a main component, 30 % Polypropylene (PP) as a backbone binder and 5 % Stearic Acid (SA) as a lubricant. Some important properties of these binder materials are given in Table 1 [24].

2.2. Particle size analysis

The particle size of metallic and ceramic powders was analyzed using the particle size analyzer, Malvern Mastersizer, at Süleyman Sarıtaş Powder Metallurgy Laboratory (SSPML) at the Mechanical Engineering Department of Gazi University. The size distribution of the particles was determined to check the suitability of the average powder size for PIM.

2.3. Mixing and granulating

The matrix composition was considered to be 88.5 % Al, 8.5 % Fe, 1.3 % V and 1.7 % Si (in weight). In order to see the effect of alumina addition to the rheology of Al-Fe-V-Si mixture, 5 %, 10 % and 15 % (in weight) of alumina content were used in the composite samples. Hence, totally four different groups of samples including the Al-Fe-V-Si mixture and the composite samples containing 5 %, 10 % and 15 % alumina in the Al-Fe-V-Si matrix were taken into account. In the meantime, the volumetric fraction of the binder, PEG8000/PP/SA in the feedstock was taken between 37 % and 39 %. These ratios were determined according to the rheology tests and the available literature [24, 44].

First of all, the powders of Al, Fe, V and Si were mixed in Turbula shaker/mixer at the SSPML to get the metallic mixture. In order to get an effective mixing and to prevent an extensive oxidation of aluminum, the powders were mixed with isopropyl alcohol and zirconia balls with a 5 mm diameter for 2 h. And then, the alloy mixture was dried to evaporate alcohol. Second, the binder components were mixed in dry condition in the same mixer. Third, the powders (the alloy or the composite (alloy + alumina)) and binder at suitable proportions were mixed in dry condition for 45 min. Finally, the granulation process was performed utilizing an extruder (Krauss Maffei type) at the SSPML. In order to get a good fluidity, the inlet and outlet cylinder temperatures were adjusted to be 174 ℃ and 180 ℃ for the feedstocks of the metallic mixture and the composite with 5 % alumina, respectively, whereas they were 185 ℃ and 190 ℃ for the composites with 10 % and 15 % alumina, respectively. After the process, the granules were formed to conduct the rheology tests.

2.4. Rheology tests

The rheology tests were made using a capillary rheometer at SSPML according to the standard, ASTM D1238 [45]. The feedstocks were fed into the barrel of rheometer and the viscosity and shear rates at different shear stresses were determined. To do this, a die with 2 mm in diameter and 8 mm in length was used and the rheology experiments were carried out at 180 ℃, 190 ℃ and 200 ℃. In addition to that, two different weights (3 and 5 kg) were used at these temperature levels for comparison. During the experiments, the flowing quantity of the feedstock and the flowing time were measured to be used in shear rate, shear stress and viscosity computations.

3. Experimental results and discussion

The average particle size of Al-Fe-V-Si (mixture) and alumina was found to be 9.9 μm and 3.08 μm, successively. Table 2 gives the particle size distribution results for these two types of powder. The average particle size suggested for PIM is smaller than 20 μm [24, 44], but preferably it is better to use the particles < 10 μm in size [24]. Hence, the particle size used in this study satisfies this criterion. Figure 1 shows the typical granules produced in this work. In order to

Table 2. Powder size analysis results for Al-Fe-V-Si and alumina

	d_{50} (μm)	d_{10} (μm)	d_{90} (μm)
Alumina	3.08	0.61	9.99
Al-Fe-V-Si	9.90	4.05	19.66

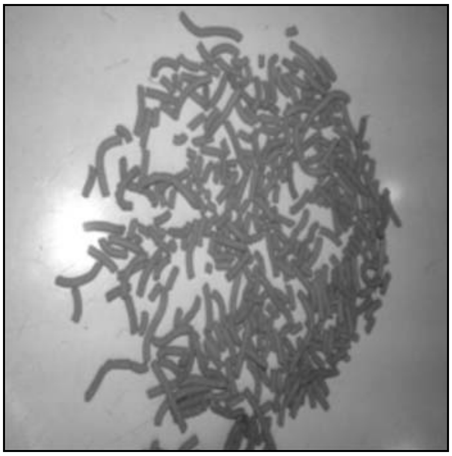

Fig. 1. A typical view of the feedstock granules produced in this work.

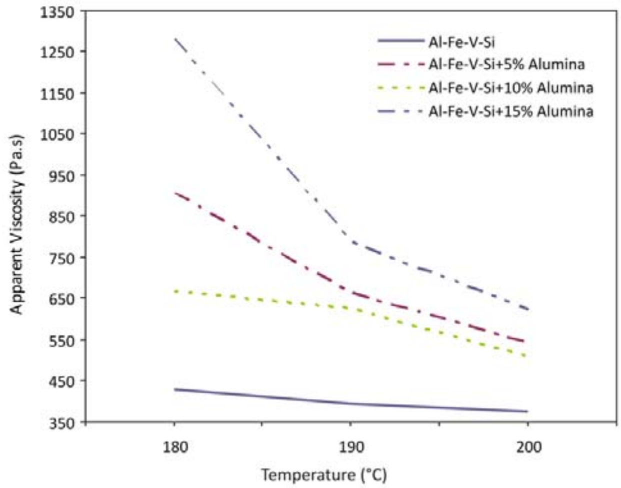

Fig. 2. Apparent viscosity of the feedstocks as a function of temperature under the pressure of 0.6 MPa.

maintain the fluidity of feedstock through the capillary rheometer, various compositions of powder + binder were initially tried in the viscosity experiments. The fraction of binder was considered to be 37 % for the metallic mixture, Al-Fe-V-Si and the composite with 5 % Al_2O_3 feedstocks, whereas it was chosen to be 38 % and 39 % for the composites with 10 % Al_2O_3 and 15 % Al_2O_3 feedstocks, respectively, since the flow did not take place below these binder fractions for the respective feedstocks. The main reasons for the selection of the binder, PEG8000/PP/SA, were the good solubility of PEG based binders in water and no remarkable rise in the oxygen level of the feedstock during processing when they are used [6, 14, 15, 23, 24]. These two properties of the PEG based binders are strongly required to prevent the extensive oxidation of aluminum.

Figure 2 illustrates the change in the apparent viscosity (η) of the investigated feedstocks with respect to temperature under the pressure of 0.6 MPa. It is clear that when the reinforcement content is increased, the viscosity rises rapidly. It is most probably due to the higher hardness and density of ceramic particles. Relatively small and harder ceramic particles cause the locking of metallic particles by entering the interstitial positions. This makes somewhat low or high resistance to the flow of feedstocks. In addition, an increase in the green hardness of feedstock via addition of ceramic reinforcement also retards the flow. In this case, higher operation temperatures or higher binder contents would be needed. Moreover, an increase in temperature lowers the viscosity of feedstocks as expected. The effect of temperature on the four materials is different. Although the viscosity of the feedstock containing metallic powders varies with temperature slightly, the viscosity of feedstock containing composite powders decreases with increasing temperature significantly. The influence of temperature on the viscosity of composite samples rises with reinforcement content. The viscosity level of the feedstocks consisting of Al-Fe-V-Si, Al-Fe-V-Si + 5 % Al_2O_3 and Al-Fe-V-Si + 10 % Al_2O_3 is lower than 1000 Pa s at the investigated temperatures. On the other hand, for the feedstock with Al-Fe-V-Si + 10 % Al_2O_3 powder, it becomes higher than 1000 Pa s at 180 ℃ but drops down to 788 Pa s and 621 Pa s at 190 ℃ and 200 ℃, respectively.

Figure 3 represents the variation of apparent viscosity as a function of temperature for the investigated feedstocks under the pressure of 1 MPa. Again, an increase in temperature makes the flow of feedstocks much easier. The viscosity values are found to be lower than 1000 Pa s for all cases. The addition of reinforcement to the metallic mixture appears to cause an increase in the viscosity of the feedstock. Moreover, an increase in the pressure from 0.6 MPa to 1 MPa leads to the higher viscosities of the metallic feedstock. However, it does not have a significant change in the viscosity of composite feedstocks.

Figures 4 and 5 depict the variation in the apparent viscosity with respect to the apparent shear rate ($\dot{\gamma}$) at four cases. Shear rate is very important parameter affecting the viscosity. As well known, the higher the shear rate occurs, the lower the viscosity is

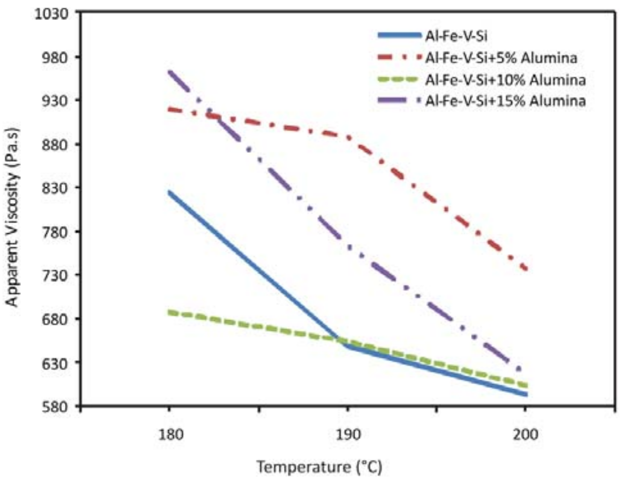

Fig. 3. The graph of apparent viscosity versus temperature of the feedstocks under the pressure of 1 MPa.

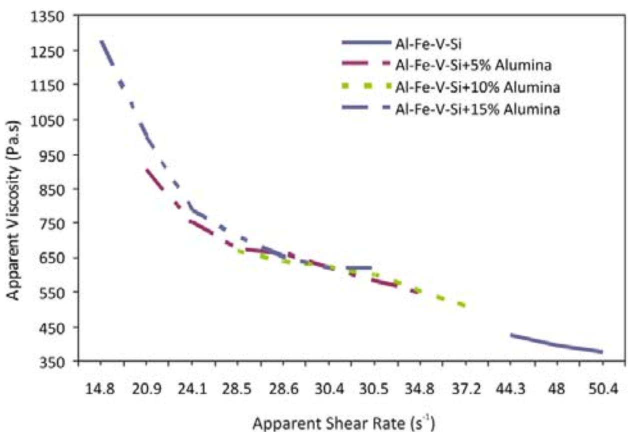

Fig. 4. Change in the apparent viscosity of feedstocks with apparent shear rate at 0.6 MPa.

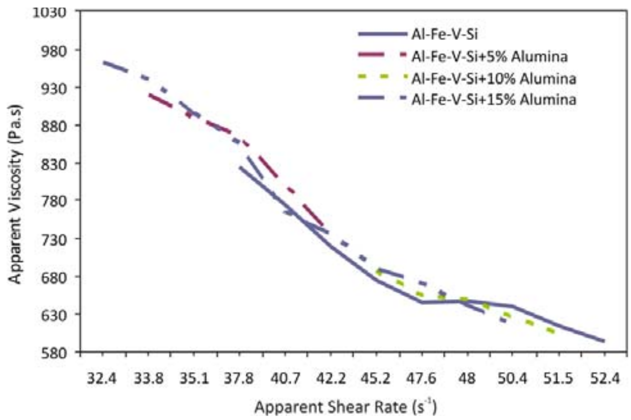

Fig. 5. Variation in the apparent viscosity of feedstocks with apparent shear rate at 1 MPa.

obtained for a material. The general tendency is such that adding alumina powders to the metallic powders decreases the shear rate and so increases the viscosity for the feedstock. At the pressure of 0.6 MPa, the viscosity decreases sharply up to the shear rate of 26 s^{-1} for the feedstock with 5 % or 15 % alumina. At higher shear rates, it drops linearly with increasing shear rate but with lower slopes for all investigated feedstocks. At the pressure of 1 MPa, a similar behavior can be seen for the feedstocks.

The best fluidity values of the powder and binder mixtures are given in Table 3. The fluidity values are found to be between 1.6×10^{-3} $Pa^{-1} s^{-1}$ and 2.7×10^{-3} $Pa^{-1} s^{-1}$ for the investigated materials. The fluidity level of the mixture having the metallic powders is remarkably higher than that of others. Melt Flow Index (MFI) values were also calculated for different temperatures and pressures. These values are given in Table 4. The MFI is obtained to be in the range of 90–325 g/10 min. It increases with increas-

Table 3. The best fluidity values for the investigated mixtures

Mixture	Temperature (°C)	Shear rate, $\dot{\gamma}$ (s^{-1})	Fluidity, $1/\eta$ (10^{-3} $Pa^{-1} s^{-1}$)
63 % (Al-Fe-V-Si) + 37 % PEG8000/PP/SA	190–200	50.5–48.0 (at 0.6 MPa)	2.53–2.66
63 % (Al-Fe-V-Si + 5 % Alumina) + 37 % PEG8000/PP/SA	200	34.77 (at 0.6 MPa)	1.84
62 % (Al-Fe-V-Si + 10 % Alumina) + 38 % PEG8000/PP/SA	200	37.16 (at 0.6 MPa)	1.96
61 % (Al-Fe-V-Si + 15 % Alumina) + 39 % PEG8000/PP/SA	200	50.40 (at 1 MPa)	1.62

T a b l e 4. Melt Flow Index for the feedstocks

Feedstock	Temperature (℃)	Pressure (MPa)	Melt Flow Index (MFI) (g/10 min)
63 % (Al-Fe-V-Si) + 37 % PEG8000/PP/SA	180	0.6	276
		1	235
	190	0.6	298
		1	298
	200	0.6	313
		1	325
63 % (Al-Fe-V-Si + 5 % Alumina) + 37 % PEG8000/PP/SA	180	0.6	130
		1	210
	190	0.6	178
		1	218
	200	0.6	216
		1	262
62 % (Al-Fe-V-Si + 10 % Alumina) + 38 % PEG8000/PP/SA	180	0.6	176
		1	280
	190	0.6	188
		1	294
	200	0.6	230
		1	318
61 % (Al-Fe-V-Si + 15 % Alumina) + 39 % PEG8000/PP/SA	180	0.6	91
		1	199
	190	0.6	148
		1	250
	200	0.6	188
		1	310

ing temperature and load for the investigated samples. These results are coherent with the previous studies conducted on the different types of feedstocks [19, 22, 24, 25, 27]. Flow activation energy (E_a) values, calculated by using Arrhenius type equation, are presented in Table 5. The reduction in the E_a is recorded for the metallic feedstock with increasing the applied pressure. This is consistent with its viscosity results. In the meantime, the flow activation energy increases with an increase in the loading pressure. E_a values are found to be within the range of 4.2–26.6 kJ mol^{-1} depending on the feedstock composition and pressure. The lower E_a is strongly needed to make PIM effectively [2]. Another important parameter in PIM is the moldability parameter (α) which is defined by Weir et al. [46]. The moldability equation [46] is given as

Table 5. Flow activation energy results for the mixtures

Mixture	Pressure (MPa)	E_a (kJ mol^{-1})
63 % (Al-Fe-V-Si) + 37 % PEG8000/PP/SA	0.6 1	4.2 12.5
63 % (Al-Fe-V-Si + 5 % Alumina) + 37 % PEG8000/PP/SA	0.6 1	18.3 7.5
62 % (Al-Fe-V-Si + 10 % Alumina) + 38 % PEG8000/PP/SA	0.6 1	9.1 5.0
61 % (Al-Fe-V-Si + 15 % Alumina) + 39 % PEG8000/PP/SA	0.6 1	26.6 15.8

Table 6. Moldability parameters for the feedstocks

Feedstock	Shear stress (kPa)	Moldability parameter α
63 % (Al-Fe-V-Si) + 37 % PEG8000/PP/SA	18.65 30.63	272 512
63 % (Al-Fe-V-Si + 5 % Alumina) + 37 % PEG8000/PP/SA	18.65 30.63	399 775
62 % (Al-Fe-V-Si + 10 % Alumina) + 38 % PEG8000/PP/SA	18.65 30.63	609 129
61 % (Al-Fe-V-Si + 15 % Alumina) + 39 % PEG8000/PP/SA	18.65 30.63	190 350

below:

$$\alpha = \frac{10^9}{\eta_0} \frac{\partial \ln \eta / \partial \ln \dot{\gamma}}{\partial \ln \eta / \partial \left(\frac{1}{T}\right)} \approx \frac{10^9}{\eta_0} \frac{|m-1|}{E_a/R}, \quad (1)$$

where η is the apparent viscosity, η_0 is the apparent viscosity at the reference shear rate ($10\,s^{-1}$), $\dot{\gamma}$ is the apparent shear rate, m is the flow behavior index, E_a is the flow activation energy, and R is the universal gas constant. The results for moldability parameter of the feedstocks are presented in Table 6. Nevertheless, there is no proper correlation recorded between moldability parameter and viscosity for the investigated feedstocks. The case in the feedstocks containing composite powder seems to be different and more complicated in comparison to plastic materials [46]. This equation needs to be arranged and developed for metallic based composites to get meaningful relations between viscosity and moldability.

Table 7. Flow behavior index results for the feedstocks

Feedstock	Load (kg)	m
63 % (Al-Fe-V-Si) + 37 % PEG8000/PP/SA	3 5	0.99 0.90
63 % (Al-Fe-V-Si + 5 % Alumina) + 37 % PEG8000/PP/SA	3 5	0.89 0.91
62 % (Al-Fe-V-Si + 10 % Alumina) + 38 % PEG8000/PP/SA	3 5	0.93 0.99
61 % (Al-Fe-V-Si + 15 % Alumina) + 39 % PEG8000/PP/SA	3 5	0.91 0.90

Flow behavior is also an important parameter which should be determined before PIM. It is defined by using flow behavior index (m). It is computed by the Eq. (2) given below [47, 48]:

$$m = \frac{d\,(\log \tau_w)}{d\,(\log \dot{\gamma}_a)}, \quad (2)$$

where τ_w is shear stress at wall and $\dot{\gamma}_a$ is apparent shear rate. The value of m determines the flow type of feedstock. When m is equal to 1, the flow is of Newtonian type. For the fluids not obeying the Newtonian rule, viscosity decreases with increasing shear rate and $m < 1$. In this case, the flow is termed pseudo-plastic. If m is greater than 1, the flow is called as dilatant [47, 48]. The results for the m values of investigated feedstocks are reflected in Table 7. It is clearly seen that m is found to be lower than m for all cases and the flow for the feedstocks exhibits a pseudo-plastic behavior. The m values are within the range of 0.89–0.99 which is suitable for PIM [19].

4. Conclusions

In this study, the effect of alumina addition, temperature and loading pressure on the rheological properties of feedstocks having Al-Fe-V-Si powder and PEG8000/PP/SA binder was examined. According to the experimental results, the main conclusions can be drawn as below:

– Good rheological properties were attained when 37 % binder was used in the feedstocks containing Al-Fe-V-Si and Al-Fe-V-Si + 5 % Al_2O_3. The binder ratios should be 38 % and 39 % for the feedstock made of Al-Fe-V-Si + 10 % Al_2O_3 and Al-Fe-V-Si + 15 % Al_2O_3 to keep the viscosity level lower than 1000 Pa s.

– Alumina addition to the powder in the feedstock caused an increase in the viscosity.

– MFI was determined to be in the range of 90–325 g/10 min for the investigated feedstocks. It increased with temperature and pressure.

– For all mixtures, the melt behavior index (m) was obtained in between 0.89–0.99 and so the flow showed a pseudo-plastic character.

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