IJE TRANSACTIONS A: Basics Vol. 28, No. 10, (October 2015) 1493-1499

International Journal of Engineering

Journal Homepage: www.ije.ir

# Effects of Micro and Nano Sized SiC Powder on the Rheological Properties of Al Based Feedstocks for Low Pressure Injection Molding

A. Ghanbari[a], M. Alizadeh[a*], R. Yazdani Rad[a], E. Ghasemi[b]

*[a] Department of Ceramics, Materials and Energy Research Center, Iran*
*[b] Institute for Color Science and Technology, Iran*

*PAPER INFO*

*Paper history:*
Received 03 May 2014
Received in revised form 06 October 2014
Accepted 03 September 2015

*Keywords*:
Injection Molding
Al-SiC
SiC Nanoparticles
Rheology

*ABSTRACT*

This study investigates the effects of micro- and nano-sized SiC powder on the rheological behavior of Al based feedstocks for powder injection molding (PIM). Different compositions of Al feedstocks with additions of micro and nano-SiC powder were prepared and their rheological properties were measured with a rotational rheometer. The effects of SiC content and shear rate were investigated and activation energies were compared amongst the feedstocks. The results showed that nano-SiC powder has a prominent effect on the viscosity and yield stress whereas micro-SiC does not have an effective role and the base micro-Al powder determines the overall rheological behavior of the feedstock. It was found that the feedstocks reveal pseudoplastic behavior at low shear rates and dilatancy at high shear rates. The critical shear rate ($\gamma_{crit}$) was 100 $s^{-1}$ for changing the rheological behavior. The activation energy of a given feedstock was found to increase with micro-SiC content while the reverse trend was observed for nano-SiC.

***doi***: *10.5829/idosi.ije.2015.28.10a.13*

## 1. INTRODUCTION

Powder injection molding (PIM) is an effective process for fabricating intricate and small components for high performance applications. This method is similar to plastic injection molding (IM), the distinguishing characteristic being the feedstock composition and the role of the thermoplastic as a binder instead of a component of the final part [1]. In PIM, addition of either metal or ceramic powder to thermoplastic binder causes main differences in composition [2]. In the case of metal powder addition, this process is named metal injection molding (MIM). The MIM process involves four typical steps including mixing powder and binder, injection molding, debinding and sintering [3, 4]. Powder and binder combine to produce a paste which is called the feedstock [5]. In recent years, low pressure injection molding (LPIM) has attracted much attention because of many advantages such as low tooling costs and simple moulds [6]. The binder provides the powder with the fluidity needed for injection molding [7]. The use of a relatively large fraction of low viscosity binders, mainly waxes, is an important characteristic of LPIM [8]. Cetinel et al. [6] investigated the rheological properties of zirconia-paraffin feedstocks for LPIM, which used only paraffin and dispersant as the binder components. They found that an increased amount of dispersant caused a significant decrease in both yield point and viscosity of the zirconia-paraffin feedstocks. Producing specimens without cracks and distortion is expected from successful injection molding. Studying the rheological behavior of feedstock is an important key to reach this goal [9, 10].

In PIM, the feedstock is subjected to shear flow and is consequently under the control of its inherent rheology. The rheology is a complex function of powder and binder properties, powder volume loading, temperature, and the dynamic particle structure resulting from shear stress and interparticle forces [11]. From this point of view, evaluating the sensitivity of a feedstock's viscosity and shear stress to temperature and shear rate is important in determining the suitabilityof a feedstock to a given PIM process [10].

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*Corresponding Author's Email: m-alizadeh@merc.ac.ir (M. Alizadeh)

Please cite this article as:A. Ghanbari, M. Alizadeh, R. Yazdani Rad,E. Ghasemi,Effects of Micro- and Nano-Sized Sic on The Rheological Properties of Al Based Feedstocks for Low Pressure Injection Molding, International Journal of Engineering (IJE), TRANSACTIONS A: Basics Vol. 28, No. 10, (October 2015) 1493-1499

In recent years, there have been many publications discussing the most suitable rheological behavior of different feedstocks for injection molding. As is known, there are two important rheological behaviors in this regard: Pseudoplastic (shear thining) and Dilatant (shear thickening) behaviors. It should be noticed that the only acceptable behavior for injectable feedstock is pseudoplastic. Because the pseudoplastic behavior results in more favorable filling intothe nozzle. As a general rule, a feedstock exhibiting pseudoplastic (shear thinning) behavior is the most suitable for IM because the pseudoplastic behavior results in more favorable filling into the injection nozzle than dilatant (shear thickening) behavior [12]. Pseudoplastic behavior results mainly from the powder particle ordering and the binder molecule orientation [10]. Separation of powder and binder can be an acceptable reason for dilatant behavior [13]. Abolhasani et.al. [14] studied the rheological behavior of feedstocks containing stainless steel powder and a starch-based binder. They investigated the rheological behavior of feedstocks and found that using tapioca starch as a binder resulted in pseudoplastic behavior, so the feedstock can be injectable. The particle (SiC) reinforced aluminium based composites have been used in many fields, for example as a replacement material in various engineering applications such as for cylinder block liners, vehicle drive shafts, automotive pistons, bicycle frames [15, 16]. There are very few papers to date investigating the PIM of Al and Al-based feedstocks. In this work, the rheological behavior of Al-SiC feedstocks using paraffin as a binder was investigated. The effect of micro- and nano-SiC powders on the rheological behavior of the Al-SiC feedstocks were compared.

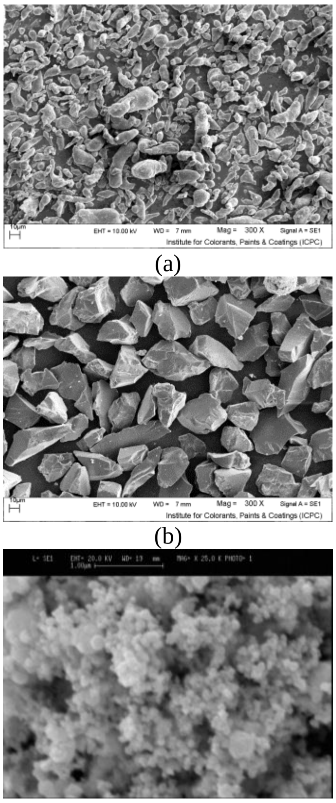
## 2. EXPERIMENTAL

**2. 1. Powder** Industrial grades of Al and SiC powders were selected. The SiC was in two sizes of nano and micro. The average particle sizes of Al, mico-SiC and nano-SiC were 20 μm, 28μm and 40nm, respectively.

**2. 2. Characterization** Morphology of the powders was analyzed using an S-4160 Hitachi scanning electron microscope (SEM). A Fritsch Analysette 22 Laser Particle Size Analyzer was used to measure particle size distribution of the powders. The viscosity and rheological properties of the feedstocks were determined using an Anton Paar Physica MCR 301 rheometer. The rheological behavior of feedstocks was studied at 85˚C and a shear rate rang of 0.1-1000 $s^{-1}$.

Figure 1 shows the SEM determined microstructures of starting powders. Different morphologies were observed for each powder. Al powders were found to have irregular. Micro-SiC particles have relatively coarse shapes while nano-SiC particles are irregular.

**2. 3. Binder** A multi-component binder system containing 89 wt% paraffin wax, 9wt% bees wax and 2wt% stearic acid (SA) was used to prepare the Aluminum based feedstock. The binder composition and characteristics of its components can be seen in Table 1.



(a)

(b)

(c)

**Figure 1.** SEM micrographs of the starting powders: (a) Al powder, (b) micro-SiC and (c) nano-SiC

**TABLE 1.**Characteristics of the binder components

| Binder components | Chemical structure | Melting point (˚C) | Decomposition temp (˚C) | Density (g/$cm^3$) |
|---|---|---|---|---|
| Paraffin wax | $C_{20}H_{42}$ | 75 | 200-400 | 0.90 |
| Bees Wax | $C_{15}H_{31}COOC_{30}H_{61}$ | 66 | 200-400 | 0.96 |
| Stearic acid | $CH_3(CH_2)_{16}COOH$ | 63 | 383 | 0.96 |

**2. 4. Feedstocks** Desired compositions of Al and SiC were ball milled in ethanol for 24h at a rate of 40 rpm. Then, the mixtures were heated in an oven at 95 ˚C to evaporate the ethanol. To prepare feedstocks, binder constituents were premixed together at 80˚C to form the binder. The composite powders were then added to the binder progressively up to the fixed powder loading of 80 wt. % at a mixing temperature of 100 ˚C. Different micro- and nano-powder mixtures were prepared according to Table 2.

## 3. RESULTS AND DISCUSSION

**3. 1. Homogeneity of Feedstocks** Variation of a feedstock's torque with time at the shear rate of 200 $s^{-1}$ is represented in Figure 2. Variation in the mixing torque over a period of time is an indicator of the homogeneity of a feedstock [15, 17]. The more steadily the torque varies, the more homogenous the feedstock is [17]. As can be seen, the time variation of torque for all samples is relatively smooth and no significant fluctuations can be seen. Therefore, it was concluded that all feedstocks have suitable homogeneity.

**TABLE 2.** Composition of feedstocks investigated

| Feedstock | Al (wt. %) | Micro-SiC(wt. %) | Nano-SiC (wt. %) |
|---|---|---|---|
| Al-100 | 100 | 0 | 0 |
| ASM5 | 95 | 5 | 0 |
| ASM10 | 90 | 10 | 0 |
| ASM15 | 85 | 15 | 0 |
| ASM20 | 80 | 20 | 0 |
| ASN5 | 95 | 0 | 5 |
| ASN10 | 90 | 0 | 10 |
| ASN15 | 85 | 0 | 15 |
| ASN20 | 80 | 0 | 20 |

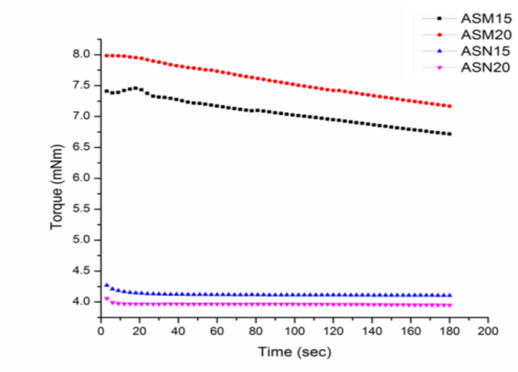


**Figure 2.** Variations of torque versus time for feedstocks

**3. 2. Effect of Shear Rate** Figure 3 shows a log plot viscosity of feedstocks versus shear rate at 85˚C. It can be seen that the viscosity of all Al-SiC feedstocks is notably higher than that of Al-feedstock. This indicates that the presence of SiC particles causes an increase in attractive forces among particles and the binder. The viscosity of all feedstocks exhibited pseudoplastic behavior. ASN (Al-nano-SiC) feedstocks have higher viscosity than ASM (Al-micro-SiC) feedstocks. This can be related to higher particle-binder interactions for feedstocks that contain nano-SiC than for those that contain micro-SiC. Khakbiz et. al has reported similar behavior for stainless steel-TiC feedstocks [9]. They reported that the viscosity of the feedstocks increased as the fraction of fine TiC powder increased, which might have resulted from increased particle-binder interaction [11] due to the increased specific surface area of the powder in the feedstock [18]. Therefore, the increase in viscosity of ASNs compared to that of ASMs can be assigned to the presence of nano-SiC powder with a larger specific surface area.

The rheological behaviors of ASMs are related to the behavior of Al-feedstock. It is clear that the variation of viscosity for Al is the same in all ASMs. Thus, it can be said that the addition of micro-SiC particles did not affect particle interactions, nor did it change rheological behavior of the feedstocks. As mentioned before, Al particles have rod-like shapes with mean aspect ratios higher than 3. Hence, the dilatant behavior of micro-feedstocks can be related to the morphologies of Al particles. However, for ASNs, rheological behaviors are different. It can be seen that at low shear rates the viscosity of ASNs are appreciably higher than that of Al and ASMs. This difference becomes even more significant as the concentration of nano-SiC particles increases. In fact, addition of nano-SiC particles increases the particle-binder interactions. The slight increase in viscosity at shear rates higher than 730 $s^{-1}$ can be related to the tumbling of flow.

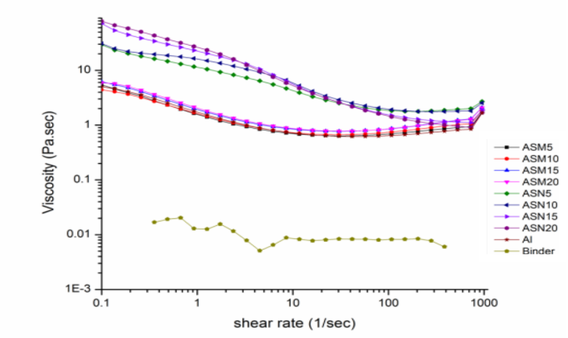


**Figure 3.** Variation of viscosity with shear rate for all evaluated feedstocks and components.

It can be observed that the difference of viscosity decreases when the shear rate increases. For ASMs, the shear thinning behavior continues up to a shear rate of 40 $s^{-1}$ and then the viscosity increases continuously till reaching the shear rate of 1000 $s^{-1}$ which is the shear thickening or dilatant behavior. For nano-feedstocks, the shear thickening behavior starts from 730 $s^{-1}$. This reveals that the attractive interactions between particles are stronger for nano-feedstocks than for micro-feedstocks. It has been mentioned that the separation of the powder from the binder can be a possible reasons for the dilatant behavior [19]. Nevertheless, this is why that in nano-feedstocks the dilatant behavior is being seen at higher shear rates than for micro-feedstocks. This can be related to higher attractive interaction in nanoparticles and their higher surface area which help to better homogeneity of mixture than for microparticles at high shera rates.

The shear rate at which the rheological behavior is changed from shear thinning to shear thickening is considered as critical shear rate and has been compared for all feedstocks in Figure 4. It is clear that for ASM samples this parameter does not change with micro-SiC content. However, in ASNs the critical shear rate increases with the amount of nano-SiC powder. A drastic increase can be seen from 10 to 15 wt % of nano-SiC and it reaches a constant value for higher contents of nano-SiC powder.

For all the micro-feedstocks, the critical shear rate is approximately the same (30.4 $s^{-1}$) while for nano-feedstocks this is at least 6.8 time larger and increases with the nano-SiC powder content. From 10 to 15wt. %, an intense increase is observed and remains unchanged afterward. As was mentioned, the attractive interactions are greater for ASNs than with ASMs. For ASMs, the increase of SiC content within the range studied did not affect the attractive interactions, and the separation of binder was observed at a relatively low shear rate of about 40 $s^{-1}$.

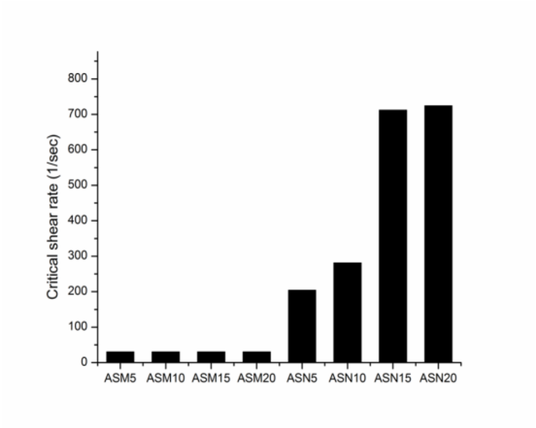


**Figure4.** The critical shear rates of feedstocks

### 3. 3. Correlating the Rheological Behavior with Rheological Models

The rheological behavior of feedstocks was analyzed with Power Law and Bingham models. The Power law model is given by the following equation:

$$\tau=k(\mathring{\gamma})n \tag{1}$$

where, k is a constant, $\tau$ is the shear stress, $\mathring{\gamma}$ is the shear rate and n is the flow behavior index, which is an indicator of shear sensitivity. If $n<1$, the behavior is pseudoplastic and if $n>1$ the feedstock behavior is dilatant [20]. Newtonian behavior is characterized by $n=1$. The Bingham model is represented by:

$$\tau=\tau_0+\eta\mathring{\gamma} \tag{2}$$

where, $\tau_0$ is the yield stress, which is the minimum stress required for feedstocks to start flowing.

### 3. 3. 1. Pseudoplastic Behavior Range

The correspondence of rheological behavior in the pseudoplastic regime for each sample with the power law and Bingham models is given in Table 3. The regression coefficient, $R^2$, is considered as the degree of correspondence. It was found that for the ASM samples the Bingham model more accurately fit the rheological data while the power law model had better compliance for ASNs. for ASNs, the compliance increases when nano SiC content increases while for ASMs no distinct variations can be seen.

One explanation for these observations is that the average particle size significantly decreases upon addition of nano-SiC and orientation and regular flow of the particles become easier; Hence n decreases. Besides, increasing the amount of silicon carbide particles increases the interactions between particles in paste, So the viscosity increases and n decreases as a result.

**TABLE 3.** The compliance of ASMs feedstocks to Power law model

| | Power law | | Bingham | |
|---|---|---|---|---|
| **Feedstock** | **$R^2$** | **n** | **$R^2$** | **$\tau_0$ (Pa)** |
| Al | 0.984 | 0.614 | 0.998 | 1.017 |
| ASM5 | 0.98 | 0.600 | 0.998 | 0.853 |
| ASM10 | 0.988 | 0.632 | 0.998 | 0.843 |
| ASM15 | 0.982 | 0.683 | 0.998 | 1.052 |
| ASM20 | 0.983 | 0.598 | 0.998 | 1.142 |
| ASN5 | 0.996 | 0.609 | 0.986 | 14.369 |
| ASN10 | 0.998 | 0.599 | 0.982 | 22.602 |
| ASN15 | 0.997 | 0.454 | 0.971 | 29.294 |
| ASN20 | 0.998 | 0.409 | 0.964 | 31.472 |

**3. 3. 2. Dilatant Behavior Range** The rheological behavior of all feedstocks in the dilatant regime corresponded to the power law model. The analyses are shown in Table 4. It should be noted that the value of n increases with the SiC powder fraction, indicating that the dilatant behavior becomes more pronounced (from 15 to 20 wt% of ASM the variation of n is negligible). The magnitude of n versus SiC content is plotted in Figure 5. It can be seen that no effective difference for n is observed below 5 wt% SiC between ASMs and ASNs with increasing shear rate; but above 5 wt%, different variation can be seen. For shear rates lower than 100 $s^{-1}$, the magnitude of n is less than 1, which corresponds to pseudoplastic behavior for both micro and nano feedstocks. It is clear that n decreases continuously for nano-feedstocks when the SiC content increases whereas for micro-SiC an increase is observed from 5 to 15 wt% and then it decreases. This initial increase of n can be related to size distribution of mico-SiC which helps to decrease the interactions of particles. In other words, SiC microparticles distribute between Al particles and decrease their interactions, but the interactions increase between SiC particles for SiC content more than 15wt%. For shear rates higher than $100 s^{-1}$, the magnitudes of n is >1 which presents the dilatant behavior for both micro and nano-feedstocks. For micro-feedstocks a small increase can be observed from 0 to 25 wt% SiC. In nano-feedstocks, on the other hand, n decreases slightly from 0 to 5 wt% SiC, but increases significantly at higher contents of SiC powder.

**3. 3. 3. Yield Stress** Figure 6 shows the yield stress variations of feedstocks versus SiC contents for both micro and nano-feedstocks. As can be seen, significant differences between the yield stress of ASMs and ASNs were observed in this figure. For ASMs, the SiC content has a minor effect on the yield stress variations but in ASNs, the yield stress increases notably with SiC content. This indicates that the interactions between particles is influenced by SiC nanoparticles more significantly.

**TABLE 4.** The compliance of ASMs feedstocks to Power law model in dilatant behavior range

| | Power law | |
|---|---|---|
| **Feedstock** | **$R^2$** | **n** |
| AL | 0.999 | 0.118 |
| ASM5 | 0.999 | 1.133 |
| ASM10 | 0.999 | 1.159 |
| ASM15 | 0.999 | 1.176 |
| ASM20 | 0.999 | 1.172 |
| ASN5 | 1.000 | 1.095 |
| ASN10 | 1.000 | 1.430 |
| ASN15 | 1.000 | 1.450 |
| ASN20 | 1.000 | 1.480 |

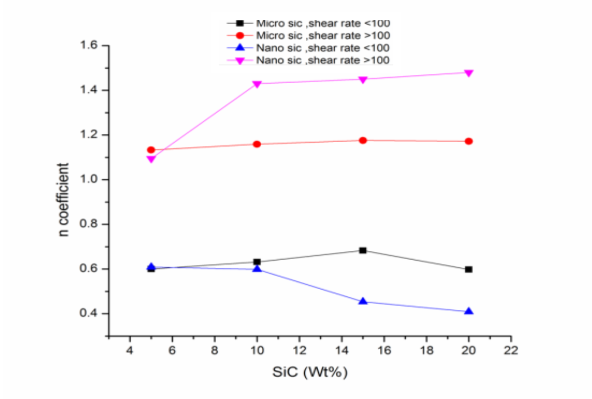


**Figure5.** Magnitude of n versus SiC content

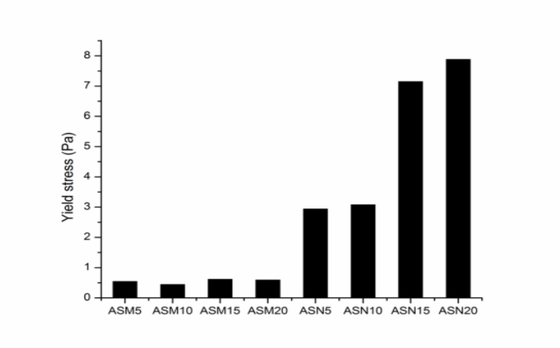


**Figure 6.** Yield stress for micro and nano-feedstocksversus SiC contents.

## 3. 4. Temperature Dependence of Viscosity

Viscosity of the feedstocks varied with termperature according to the Arrhenius equation [10, 21]:

$$\eta=\eta_0 \exp(E/RT) \quad (3)$$

where, $\eta_0$ is viscosity at the reference temperature, E is flow activation energy, R is the universal gas constant and T is the temperature.

The E value determines the sensitivity of viscosity to temperature. If the value of E is low, the viscosity is less sensitive to temperature variations, which is desirable in the fabrication of defect-free parts [10].

Flow activation energy (E) can be determined from the slope of a plot of ln (η) versus 1/T.

Figures 7 and 8 illustrate the graph viscosity versus temperature for micro and nano-feedstocks respectively. According to the above graphs, temperature dependence of viscosity was found to be in good agreement with straight line. The slope is approximately the same for all variations excluding the Al sample.

Figure 9 shows the variations of calculated E for all feedstocks. It can be seen that the magnitude of E increases versus SiC concentration for ASM feedstocks till 15 wt.% of SiC (ASM15) and then decreases for 20 wt. % (ASM20) although it decreases for ASNs. This can be related to the variations of thermal conduction which changes with SiC size and distribution [8]. In Figure 2, it can be seen that the morphology of micro-SiC particles is irregular and angular; therefore, a perfect contact between SiC- SiC particles or SiC- Al particles is impossible. This results in the reduction of heat transfer which in turn increases the flow activation energy. However, a further increase of SiC content from 15 to 20 wt% leads to a decline in the value of E.This is mainly because addition of SiC beyond 15 wt % helps particles lock together and increase thermal conductivity and decrease the E value as a result. On the other hand, increasing the nano-SiC fraction leads to increased contact between particles due to their small size and more spherical morphology. The subsequent increase in thermal conductivity decreases the value of *E*.

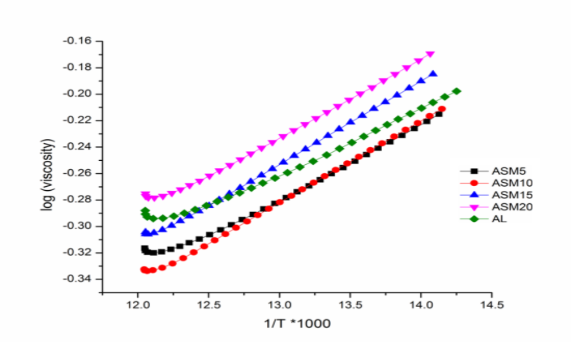


**Figure 7.** Variation of ASMs viscosity with temperature

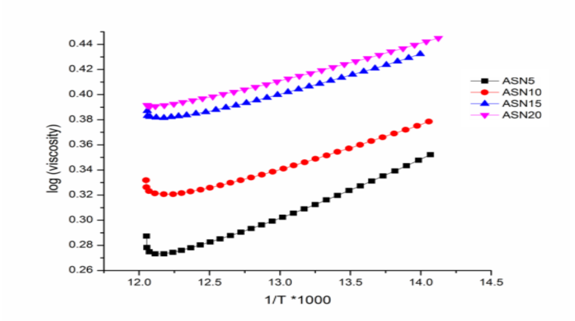


**Figure 8.** Variation of ASNs viscosity with temperature

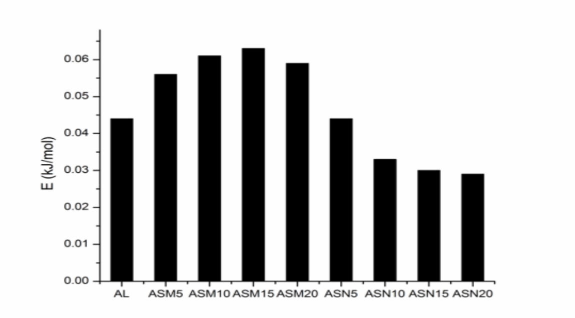


**Figure 9.** Variation of ASNs viscosity with temperature

## 4. CONCLUSIONS

The effects of micro and nano-SiC content on the rheological behavior of the Al-based feedstocks were studied. Rheological behavior of the feedtocks was compared with rheological models, and the dependence of the yield stress and flow behavior index to shear rate were analyzed. To study thermal behavior of the feedstocks, flow activation energy of feedstocks was determined. The results indicated that the effect of micro-SiC on rheological properties is different from that of nano-SiC powder. The micro-SiC does not affect viscosity while nano-SiC increased viscosity noticeably. This was attributed to the impact of SiC particle size on interactions between particles. In ASMs, Al powder determines the overall rheological behavior whereas in ASNs the behavior of nano-SiC particles is dominant. All the micro and nano-feedstocks reveal good conformity to the Arrhenius equation. Results indicated that the activation energy is dependent upon particle size and morphology of the added SiC

## 5. ACKNOWLEDGEMENTS

This work was supported by “MATERIALS AND ENERGY RESEARCH CENTER” (Ministry of Science, research and Technology), of Iran. The authors wish to express their thanks and appreciation to the editor of this article, Brian N. Turner. Technical Specialist of METTLER TOLEDO.

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A. Ghanbari[a], M. Alizadeh[a], R. Yazdani Rad[a] ,E. Ghasemi[b]

*[a] Department of Ceramics, Materials and Energy Research Center, Iran*
*[b] Institute for Color Science and Technology, Iran*

*PAPER INFO*

*Paper history:*
Received 03 May 2014
Received in revised form 06October 2014
Accepted 03September 2015

*Keywords*:
Injection Molding
Al-Sic
Sic Nanoparticles
Rheology

**چکیده**

هدف از این تحقیق، بررسی تاثیر پودر سیلیکون کارباید (درابعادمیکروونانو) بر رفتار رئولوژی خمیرهای کامپوزیت زمینه آلومینیومی (خمیرهای مورد استفاده در فرایند قالبگیری تزریقی) می‌باشد. از این رو ترکیبات متفاوتی از خمیرهای آلومینیوم با افزودن پودر سیلیکون کارباید میکرومتری و نانومتری تهیه شدند و رفتار رئولوژی آنها توسط دستگاه رئومتر چرخشی مورد بررسی قرار گرفت. مقدار سیلیکون کارباید، نرخ برشی و انرژی های فعالسازی خمیرها پارامترهای در نظر گرفته شده در این مطالعه می باشند. نتایج نشان داد که پودر سیلیکون کارباید نانومتری تاثیر حایز اهمیتی بر ویسکوزیته و تنش تسلیم دارد در حالی که سیلیکون کارباید میکرومتری نقش مهمی نداشته و پودر آلومینیوم میکرومتری تعیین کننده رفتار رئولوژی خمیرهای حاوی سیلیکون کارباید میکرومتری است. همچنین خمیرها دارای رفتار شبه پلاستیک در نرخ برشی های پایین و رفتار دیلاتانت در نرخ برشی های بالا هستند. نرخ برشی بحرانی ($\gamma_{crit}$) ( نرخ برشی که در آن رفتار رئولوژی تغییر می کند) برابر با ۱۰۰ دور بر ثانیه است. انرژی فعالسازی خمیرهای حاوی سیلیکون کارباید میکرومتری با افزایش مقدار سیلیکون کارباید افزایش یافت در حالی که در خمیرهای حاوی سیلیکون کارباید نانومتری رفتار عکس مشاهده شد.

***doi****: 10.5829/idosi.ije.2015.28.10a.13*