Influence of components on rheological behavior of SiC-based slurries

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Abstract. Rheological behavior including of SiC-based slurries containing Si, Y₂O₃, cornstarch and dispersant was investigated in order to provide theoretical guidance for the preparation of SiC-based porous ceramics. Shear stress and viscosity of SiC-based slurries with the shear rate were measured. Thixotropy of slurries without and with foams was characterized by thixotropic hysteresis loops. The results showed that the viscosity of slurries rapidly declined with the increasing of the shear rate when the shear rate was less than 0.02 s⁻¹. While the viscosity of slurries basically kept constant at higher shear rate. Both of shear stress and viscosity of SiC-based slurries at a certain shear rate increased with the addition of components. The variation on rheological behavior of SiC-based slurries depended on each component.

1. Introduction
Due to excellent mechanical properties, good chemical resistance, high thermal conductivity, low coefficient of expansion, high thermal shock resistance and other unique properties, SiC-based porous ceramics are widely used in petroleum, chemical, metallurgical, automotive, aerospace and other industries. SiC-based porous ceramics could be prepared by many routes such as adding pore forming agent, gel-casting, organic foam and impregnation foaming and so on. The porosity of porous ceramics with the stable structure can reach more than 90% by foaming method in which non-toxic harmless protein was mostly used as a foaming agent. Generally, the solid content of the slurry played a decisive role in the porosity of porous ceramics. The degree of foaming depended on the viscosity of the slurry, which determines the characters of the microstructure. With the increase of the solid content, the viscosity of the slurry increased, which led to the reduction of the foaming degree, the porosity and the pore size [1]. In some cases, porous ceramics need a certain strength and high porosity. Alumina foam ceramic filter with the open pore of 80% prepared by through the direct foaming method possessed the compressive strength of 3.2-4.8MPa, which is higher than the foam ceramic filter industry standards [2]. In this paper, rheology and thixotropy of SiC-based slurry with different components were investigated in order to provide a certain basis for the preparation of SiC-based porous ceramics.

2. Experimental procedure

2.1 Raw materials
The slurries were made up of SiC powders (d₅₀=0.69 µm), Si (d₅₀=64.22 µm), water reducer FS60, additive composition of yttria, starch, foam (proteins as Vesicant) and pure water. Raw materials by
the components were weighted as showed in Table 1. Powders were pre-mixed before adding distilled water, and then slowly stirred well in a glass cup.

2.2 Rheological experiment
Rheology and thixotropy of the configured SiC paste slurry were measured by a rheometer produced by Anton Paar GmbH (MCR301). In the rheological experiment, the shear rate was kept constant at 100s\(^{-1}\) for 30s and stood 30s. Then, the shear rate was increased from 0.001 to 0.1s\(^{-1}\) in 120s. Meanwhile, the viscosity and shear stress were recorded per 4s. After that, the shear rate was similarly increased from 0.1 to 100s\(^{-1}\) in 120s, the data were recorded per 2s differently.

2.3 Thixotropic experiment
In thixotropic experiment, the shear rate was firstly kept constant at 100s\(^{-1}\) in 30s and subsequently stood for 30s. Then, the shear rate was increased from 0.01 to 1s\(^{-1}\) in 75s. Meanwhile, viscosity and shear stress are recorded per 5s. The shear rate was secondly increased from 1 to 100s\(^{-1}\) in 150s. The data were recorded per 1s. Finally, the shear rate was decreased on the contrary.

Table 1. The components of slurry (%)

| No. | SiC | FS60 | Si | Y\(_2\)O\(_3\) | Starch | Foam |
|-----|-----|------|----|------------|--------|------|
| 1   | 40  | -    | -  | -          | -      | -    |
| 2   | 40  | 0.05 | -  | -          | -      | -    |
| 3   | 29  | 0.05 | 11 | -          | -      | -    |
| 4   | 27.4| 0.05 | 10.5 | 2.1   | -      | -    |
| 5   | 26  | 0.05 | 10  | 2.0       | 2.0    | -    |
| 6   | 24.8| 0.05 | 9.5 | 1.9       | 1.9    | 1.9  |

3. Results and discussion

3.1 Rheological behavior
Figure 1 shows the variation of viscosity and shear stress with the shear rates. It can be seen that the viscosity and shear stress of the slurry presented obvious changes with the change of the shear rates. The viscosity of the slurry decreased sharply with the increase of shear stress, which was in consistent with the characteristics of shear thinning. When the slurry was stationary, the particles were arranged in disorder. While the shear stress was applied, the large micro aggregates as well as the flow resistance became smaller. Therefore, the viscosity of the slurry decreased with the increase of the shear stress. When the shear rate was greater than 0.02 s\(^{-1}\), the viscosity of slurries basically kept constant. The viscosity and shear stress both increased with the increase of the components. As the solid content increasing, the distance between the particles became less. Thus the larger interaction force hindered the relative motion of the particles, which led to the increase of the viscosity and shear stress. The reduction extent of the viscosity for slurries with the water-reducing agent was greater at the shear rate of 0.003s\(^{-1}\). There was a peak in the shear stress-shear rate curve as shown in Fig. 1(b). The value peak located at the shear rate of 0.006s\(^{-1}\) in the curve of sample without the water-reducing agent. This could be accounted for the influence of the water-reducing agent. Because the water-reducing agent FS60 is a kind of surfactant, the surface tension of the liquid could be reduced. Therefore, the flocculation structure of the slurry was destroyed under the lower shear rate.
Figure 1. The rheological behavior of slurries: (a) the variation of viscosity with shear rate; (b) the variation of shear stress with shear rate

3.2 The fitting of rheological results

Casson model and Herschel-Bulkley model were used to calculate the rheological behavior (shear rate form 1 s\(^{-1}\) to 100 s\(^{-1}\)) of the slurries. The fitting results are shown in Figs. 2 and 3.

Casson model:

\[
\tau^{1/2} = \tau_c^{1/2} + (\eta_c D)^{1/2}
\]

where \(\tau\) is shear stress (Pa), \(D\) is the shear rate, \(\tau_c\) is yield stress (Pa) of Casson model, \(\eta_c\) means the limit viscosity.
Figure. 2 Fitting curves based on Casson model

Table 2 The parameters of Casson model

| Parameter | $\tau_c$ | $\eta_c$ | $R^2$ |
|-----------|----------|----------|-------|
| 1         | 2.38     | 0.003    | 0.998 |
| 2         | 2.35     | 0.004    | 0.998 |
| 3         | 6.73     | 0.005    | 0.995 |
| 4         | 7.42     | 0.006    | 0.995 |
| 5         | 11.13    | 0.004    | 0.997 |

Herschel-Bulkley (H-B) model:

$$\tau = \tau_b + KD^n$$

$\tau_b$ is the yield stress (Pa) of H-B model, $K$ is the fluid consistency index which is a measured of the viscosity (Pa·s) in H-B model, $n$ is the flow characteristic index, when $n>1$, the slurry is shear thickening type; when $n<1$, slurry is shear thinning type.
Fig. 3 Fitting curves based on Herschel-Bulkley model

Table 3 The parameters of Herschel-Bulkley model

| Parameter | τb  | K   | n   | R²  |
|-----------|-----|-----|-----|-----|
| 1         | 2.80| 0.044| 0.792| 0.999|
| 2         | 2.86| 0.037| 0.846| 0.999|
| 3         | 3.95| 1.909| 0.280| 0.999|
| 4         | 8.24| 0.144| 0.725| 0.996|
| 5         | 12.18| 0.100| 0.784| 0.999|

Tables 2 and 3 respectively show the parameters of Casson and H-B model. According to the correlation coefficients, both of Casson and H-B model could be used to describe the rheological behavior of slurries. The yield stress fitting by the two equations increased with the increase of the components of the slurry. The spatial network structure between the particles in slurries was gradually destroyed under the shear force because the magnitude of the yield stress reflected the strength of the spatial structure.

3.3 Thixotropic experiment

The relation curves of shear stress and shear rate in the thixotropic experiments are showed in Fig. 4. The flocculation structure formed in slurries as a consequence of intermolecular forces was gradually broken by the action of shear stress. When the shear rate decreased again, the broken structure was not ready to recombination. So the thixotropic ring formed with the shear stress varied. The area of thixotropic loop represented the strength of the thixotropy. The higher the shear rate corresponded the more serious the damage of the structure after the high speed stirring. On the contrary, the slower the combination, the bigger the thixotropic loop. From the statistics of areas as shown in Table 2, the thixotropy of slurries with foams was relatively smaller. With the adding of foam, the interaction force between particles decreased. The structure was easier to recover after being destroyed, so the thixotropic loop smaller. In Fig. 4(a), the emerging of negative thixotropy was due to the poor dispersion of the slurry in the study, which may cause coagulation. Thus, the measured shear stress with the shear rate increasing was smaller than that during the reduction of the shear rate.

4 Conclusions

(1) With the increase of shear stress, the spatial network structure between the particles in slurries was gradually destroyed, so the viscosity of slurries decreased.

(2) The degree of asymmetry between particles increased with the increase of components. The viscosity and shear stress of slurries rose as a consequence of the interaction force between particles.

(3) The rheological behavior of slurries conformed both of Casson and Herschel-Bulkley model according to the correlation coefficients of fitting.

(4) With the adding of foam, the interaction force between particles decreased. The thixotropic
loop for slurries containing foams was smaller because the structure was easier to recover after being destroyed.

![Shear Stress vs Shear Rate](image)

**Figure. 4** The relation curves of shear stress and shear rate in the thixotropic experiments: (a) 100s\(^{-1}\); (b) 200s\(^{-1}\); (c) 300s\(^{-1}\)

| No. | Shear rate /s\(^{-1}\) | Thixotropic ring’s area (Pa/s) |
|-----|----------------------|-------------------------------|
| 5   | 100                  | 0.030                         |
| 6   | 200                  | 0.525                         |
| 6   | 300                  | 1.544                         |

Percentage of area reduction (%)

| No. | Percentage of area reduction (%) |
|-----|----------------------------------|
| 5   | 99.6                             |
| 6   | 0.766                            |

**Table 2** Thixotropic ring’s area (Pa/s)

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