IOP Conference Series: Materials Science and Engineering

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IOP Conf. Series: Materials Science and Engineering **394** (2018) 032133 doi:10.1088/1757-899X/394/3/032133

Study on Rheological Properties of SiC Slurry

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Abstract. Two kinds of sic powders with different diameters (d50=1.0um and d50=2.0um) were selected as raw material gradation, SIC ceramic slurry was prepared by adding Tetramethylammonium hydroxide (TMAH) and deionized water mixed ball mill. By testing the viscosity of slurry, settling height and the Zeta potential on the surface of SIC particles, the effects of dosage, gradation ratio and pH value of dispersant Tetramethylammonium hydroxide on rheological properties and stability of slurry were studied. The research results show that: Fine powder (d50=1.0um) and coarse powder (d50=2.0um) gradation ratio is 3:7, The Rheological property and stability of the slurry are the best by adding 0.4wt%TMAH. When the pH value is 10, Zeta potential of the SiC powder surface reaches a maximum of -46.8 mV.

1. Introduction

SiC products are typical covalent bonding materials, with high high-temperature strength, good antioxidant, high thermal conductivity and low heat expansion coefficient, resistance to shock and chemical corrosion and other excellent characteristics, widely used in machinery, metallurgy, chemical and aerospace and other fields of key ceramic parts.

In order to produce a high strength and even defect-free sic ceramic body, it is required that the ceramic slurry should have high solid content, low viscosity, system uniformity, stability and good rheological properties. Among them, low viscosity is advantageous to the elimination of bubbles in slurry and the pouring of complex shape parts. However, the viscosity of ceramic slurry is closely related to the type and amount of dispersant, ph value and so on, only by mastering the mechanism of these factors can we produce high-performance ceramic components. In this experiment, the effect mechanism of dispersant dosage and ph value on the viscosity of SiC slurry was studied [1], and the optimum technological conditions of SiC ceramic slurry with high solid concentration volume fraction, low viscosity and good stability were investigated.

2. The Experimental Setion

2.1. Experimental Raw Materials

First, using SIC powder (d50=1.0um) and coarse powder (d50=2.0um) as raw materials, according to the fine particles than the coarse particles of 1:9, 2:8, 3:7, 4:6 of the mass than the homogeneous mixture made of powder standby, The dispersant Tetramethylammonium hydroxide is weighed proportionally and placed in deionized water, Using constant temperature magnetic stirrer to stir to disperse agent to dissolve fully in water, the mixed powder is added into the ball mill 3h with 160r/min, and the SIC ceramic slurry with a mass fraction of 50% is prepared.

2.2. Characterization and Testing

The viscosity of slurry was measured by Brookfield RST-CC touch screen rheometer, and the Rheological property of slurry was characterized. The stability test is used to pour the prepared ceramic slurry into the 10ml measuring cylinder, at room temperature, the height of the upper supernatant of the suspended liquid is measured with H, i.e. the height of settling, which is used to characterize the stability of slurry. The ph value of the slurry is determined by a Lei-ci ph meter, the zeta potential size of slurry suspension particles was determined by Malvern Zetasizer Nano potentiometer.

3. Results and Discussions

3.1. Effect of Dispersant Dosage on Viscosity of SiC Slurry

By adjusting the content of dispersant TMAH in different gradation sizing slurry, the influence of TMAH content in slurry on the viscosity of slurry was probed. The amount of TMAH added is the mass percentage of SiC powder. Using Rheometer to test slurry viscosity, all slurry test temperature is 25 ℃, slurry to 50s-1 constant shear rate of 120s, and then determine the shear rate in the 50s-1~1000s-1 range of silicon carbide grading slurry viscosity, as shown in Figure 1 as the slurry of the mass fraction 50%, The relationship between the viscosity of different gradation ratio and the dosage of dispersant.

The trend of the curve shows that with the increase of TMAH dosage, the viscosity of slurry decreases. The dispersing mechanism of dispersant TMAH is electrostatic stability mechanism, and the electrostatic stability Mechanism is also called diffusion Electric double layer theory or DLVO theory [2]. Electrostatic stability means that by adjusting the pH value of the solution or the addition of electrolyte, the particle surface charge is increased and the thickness of the double layer is increased, so that the electrostatic repulsion force between the particles can be increased by increasing the Zeta potential and the homogeneous and stable dispersion of the particles is achieved. The powder particles are dispersed in the water, the charge between the water and the surface of the particles is not equal to the positive and negative charge of the two-way transfer, resulting in the particles with positive or negative charge. Because the solid particles and liquid medium are composed of the whole is electrically neutral, so the solid particles adsorbed on the surface of the charge close layer and the surrounding medium of the reverse ion diffusion layer constitutes a double electric layer. TMAH belong to strong electrolytes, the ionized OH- provides a strong alkaline environment for the solution, greatly increasing the surface negative charge of SiC powders, increasing the thickness of the double layer, and the adsorption of organic ions (CH3)4N+ on the surface of SiC particles, which leads to the increase of the thickness of the surface double layer. Thus, the electrostatic repulsion between particles is improved, and the agglomeration behavior of powders is hindered.

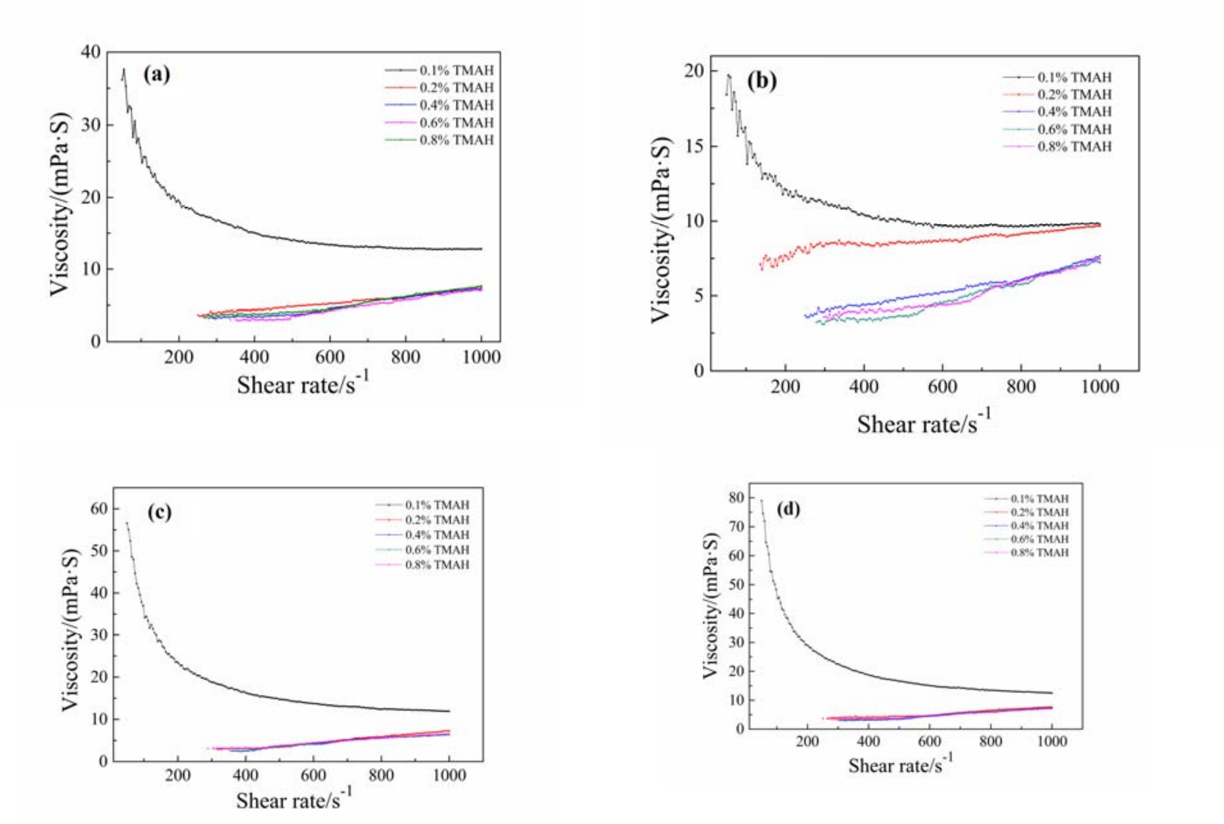

FIG.1 The relation between slurry viscosity and dispersant dosage under different proportions: 1:9(a), 2:8(b), 3:7(c), 4:6(d)

When the TMAH content is from 0.1% to 0.2%, the viscosity of the slurry decreases rapidly. Continue to add TMAH, viscosity almost unchanged. This is because when the TMAH content is less than 0.2%, the surface potential of SiC particles changed greatly, so that the repulsion force of the double layer increased quickly, so the viscosity of the slurry decreased rapidly. And when the amount of TMAH added to 0.2%, continue to add TMAH, the surface potential of SiC particles changed little, the double layer repulsion force no longer increase, so the slurry viscosity almost unchanged. Table 1 shows that the viscosity of slurry can be minimized by adding the optimum TMAH in different gradation ratios.

Table 1. The optimum amount of TMAH added under different gradation ratio

Gradation ratio	1:9	2:8	3:7	4:6
TMAH Optimal Quantity	0.4%	0.6%	0.4%	0.4%

3.2. Rheological Properties of SiC Slurry with Different Gradation Ratios

Figure 2 is a comparison of the viscosities in the case where the optimum amount of TMAH is added at the four grading ratios.

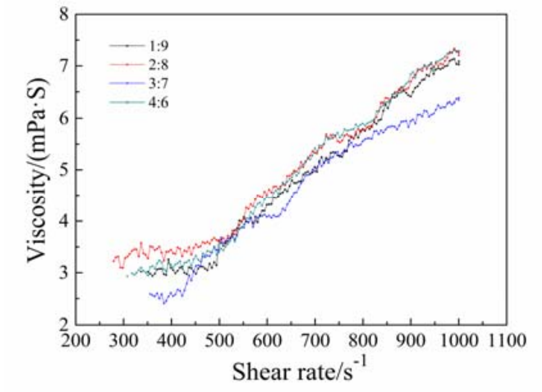

FIG.2 Viscosity Comparison of different graded sic slurry

In order to further study the rheological properties of the slurry under the 4 gradation ratios, the stability of the slurry was determined. Observe and record the height H of the supernatant. In the same settling time, suspended particles sink through their own gravity or produce flocculation phenomena. The large H value indicates that the settlement is relatively fast, the stability is poor, and the small H value indicates that the slurry has better stability. Table 2 is a comparison of the height of slurry settlement under the condition of adding optimal TMAH to 4 kinds of gradation ratio.

Table 2. Settlement height of different graded proportions of slurry

Gradation Ratio	1:9	2:8	3:7	4:6
Settlement Height(ml)	2.2	3.0	1.8	2.8

The combination of FIG. 3 and Table 2 shows that when the mass fraction of SiC slurry is 50%, the ratio of fine particles (d50=1.0um) to coarse particles (d50=2.0um) is 3:7. The Rheological property and stability of the slurry were best when the amount of TMAH was 0.4%.

3.3. Effect of pH Value on the Zeta Potential of Slurry

A certain amount of silicon carbide slurry with a grading ratio of 3:7 and TMAH addition amount of 0.4% was taken and diluted to a mass fraction of about 0.1%wt. The Zeta potential of the slurry under different pH conditions was measured with a potentiometer. , as shown in Figure 3.

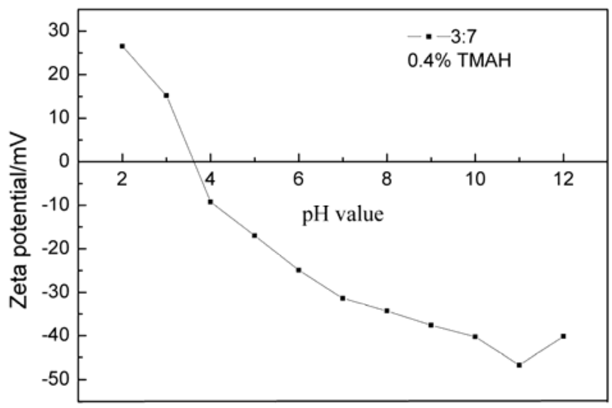

FIG.3 Zeta potential of SiC particles

As can be seen from Figure 3, the gradation ratio is 3:7, the isoelectric point of silicon carbide powder with 0.4% TMAH added was around pH 3.5. As the pH value increases, the electronegativity of the particles increases. At a pH of around 11, the absolute value of the Zeta potential is a maximum of -46.8 mV. When the pH value continues to increase, the potential absolute value is reduced due to the excessive ion-compressing double layer in the solution.

Studies have shown that the SiC surface is often covered with a thin layer of highly active amorphous SiO2. When the surface of the SiO2 covered with silicon carbide powder is dispersed in water, the SiO2 layer will be hydrolyzed to generate a stable net charge of zero. Silicon alcohol (Si-OH) layer, when the solution corresponds to the pH is called the isoelectric point. Since Silicon alcohol layer are both acidic and alkaline, they are acidic. When the pH value of the solution is lower than the isoelectric point, the hydrolysis reaction between the silanol and the H+ in the solution produces a cationic group Si-OH2+, so that the surface of the silicon carbide particles is positive. Electricity, Zeta potential is positive. The smaller the pH value of the solution (the stronger the acidity), the higher the H+ concentration, the greater the degree of reaction, the more positive charges on the surface of the silicon carbide particles, and the more positive the Zeta potential. When the pH of the solution is greater than the isoelectric point, the silanol reacts with OH- in the solution to form an anionic group Si-O-, so that the surface of the silicon carbide particles is negatively charged and the Zeta potential is negative. Similarly, the higher the pH of the solution (the stronger the alkalinity), the higher the OH- concentration, the greater the degree of reaction, and the more negative charges on the surface of the silicon carbide particles, the more negative the Zeta potential[3]. Experiments show that the absolute value of the Zeta potential of the SiC powder near the pH value of 11 is the largest,so it is beneficial to the stability and dispersion of SIC powders under alkaline conditions.

4. Conclusion

(1) The ratio of fine powder (d50=1.0um) and coarse powder (d50=2.0um) is 3:7, and 0.4wt% TMAH is added, and the rheology and stability of the slurry are best.

(2) Adjust the pH value of the slurry. When the pH value is 10, the Zeta potential of the SiC powder surface reaches a maximum of -46.8 mV.

5. Acknowledgments

This work was financially supported by the 13th Wuhan Institute of Technology Student Rector Foundation fund (2018057) and Project of Technology Innovationin Hubei Province (Grant No.2016ACA161).

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