

Microstructure and quality of SiC foam filters for casting

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Maximum compressive strain at high temperature was introduced as a new quality indicator of ceramic foam filters used in metal casting. Maximum compressive strain at high temperature, number of thermal cycles before failure, room temperature compressive strength and bulk density of SiC-based ceramic foam filters with the same pore size produced by four manufacturers were measured. The best quality product exhibited the highest number of thermal cycles before failure and minimum strain at high temperature. Differences in properties were elucidated based on X-ray diffraction and scanning electron microscope microstructural characterization. The best quality filter has less glass phase and more quartz phase, while lower quality filters have more glass and cristobalite phases. The glass phase increases the maximum strain at high temperature, while both glass and cristobalite phases reduce the number of thermal cycles before failure. The maximum compressive strain at high temperature can be utilized as the main indicator for evaluating the quality of ceramic foam filters.

Key words: Engineering ceramics, Sintering, Thermal, Microstructure.

Introduction

Porous ceramic foams contain open cells with up to 85-90% porosity [1]. Ceramic foams are widely utilized as filters for hot gas cleaning and liquid metal purification [2, 3], in heat exchangers [4], acoustic absorbers [5], as catalyst supports [6], etc. SiC-based ceramic foam filters are commonly used for casting, mainly for filtration of molten iron. SiC-based ceramic foam filters can effectively filter out impurities in the molten iron and improve the final product quality [7]. The performance of SiC-based ceramic foam filters used for casting differs greatly based on the manufacturer. Fragments of poor quality filters can mix with molten metal causing contamination, while ceramic body collapse can block the passage for filtration. While the quality of SiC foam filters varies due to their microstructural differences, few papers deal with the relationship between the microstructure of filters and their quality.

The main quality indicators of casting filters include the number of thermal cycles before failure, the compressive strength at room temperature, the bulk density and pore size (pores per inch, abbreviated as ppi). The number of thermal cycles before failure is usually considered to be the most important quality indicator [3]. However, each filter can only be used once where it is heated from room temperature to the temperature of molten metal within a short time, and then endures a continuous flow of molten

metal, rather than multiple heating/cooling cycles. Thus it is hard to imagine a direct relationship between the number of heating cycles before failure and the filter quality.

In this paper, a new quality indicator in terms of the maximum compressive strain at high temperature is proposed based on the working conditions of ceramic foam filters used for casting. The maximum compressive strain at high temperature, the number of thermal cycles before failure, the compressive strength at room temperature and bulk density of SiC-based ceramic foam filters produced by four manufacturers were measured. Corresponding relations among the maximum compressive strain at high temperature, the number of thermal cycles and filter performance were investigated. The quality assessment of filters was based on microstructural analysis.

Experiments

SiC-based ceramic foam casting filters with 15 ppi (0.59 pores per millimeter) nominal pore size and 50 mm × 50 mm × 20 mm in size produced by four different manufacturers labeled A, B, C and D were tested. In iron casting of fixed and moving blades (4.5 kg each) used in an air-conditioning compressor produced by the Technology and Materials Research Institute of China Yituo Group Corporation, the qualified casting rate is 80% without using ceramic foam filters. The qualified casting rate increased by 15%, from the former 80% to 95% after using product A. The casting rate increased by 5% after using filter products C and D. However, the iron qualified casting rate decreased after using product B. Thus product A is the best, products C and D take the second place, and product B is the worst in terms of the iron qualified casting rate.

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Bulk density and compressive strength

The middle part of a filter is subjected to pressure imposed by the molten metal flow. Filter edges are embedded in the sand mold and are not in contact with the molten metal. The density of the middle part of the filter is different than the edges and is commonly referred to as the filter bulk density. To obtain the bulk density, sample edges were cut off and the remainder was wrapped in thin plastic foil to avoid water penetration during volume measurement by a water displacement method. Sample mass was measured using an electronic micro-balance. The mass to volume ratio is the filter bulk density.

Compressive strength at room temperature was measured using a SHIMADZU AG-1250KN tensile testing tester. 2 mm thick rubber sheets were placed underneath and on top of the sample compressed with a 25 mm diameter steel cylinder head. The filter was compressed until it was crushed with the maximum load recorded, yielding its compressive strength. While there are standards for testing ceramics in compression [8, 9], highly porous ceramics are challenging to test, thus rubber sheets were used to prevent the local porous structure crushing underneath the compression tester steel cylinder head. Calculated bulk density and compressive strength at room temperature are shown in Table 1.

It is obvious that there are differences in bulk density of ceramic foam filters with the same pore size. The bulk density of product D is the highest, and the bulk density of product B is the lowest, with the bulk density of products A and C in between. There was no pore-clogging observed for these products. Bulk density reflects the quantity of ceramic slurry adhered to the polyurethane foam during preparation as well as whether the pores get clogged. A lower bulk density indicates that there is a low quantity of ceramic slurry adhered to the polyurethane foam, which would influence the filter strength both at room and high temperatures. Too high a bulk density causes pores clogging. Optimal bulk density in terms of strength and pores clogging is between 0.25 and 0.65 g·cm⁻³ [7].

A filter has to survive transportation and installation and thus must have adequate strength at room temperature. Table 1 lists room temperature compressive strength of the four products tested. The compressive strength of product B is the lowest (0.99 MPa), although it is almost twice the minimum strength of 0.5 MPa required for construction ceramic products by the JC/T895-2001 Building Materials Professional Standard of the People's Republic of China [10], thus all four products have adequate mechanical strength at room temperature for transportation and installation.

Table 1. Bulk density and room temperature compressive strength

Physical property	Product A	Product B	Product C	Product D
Bulk density (g·cm ⁻³)	0.52	0.50	0.54	0.62
Compressive strength (MPa)	1.21	0.99	1.53	1.31

While bulk density and compressive strength of ceramic foam filters satisfy the basic requirements including no pores clogging and adequate compressive strength, these two properties are not directly related to a filter's performance in application.

Thermal shock resistance and high temperature strain

The number of thermal cycles before failure was obtained by heating the samples to 1100 °C for 15 minutes and immediately quenching them in tap water. These steps were repeated until the sample was destroyed yielding the number of thermal cycles before failure. First small cracks appeared at the sample edges and corners, and then some pieces dropped from the edges and corners, followed by large cracks penetrating the rest of the sample until it became loose, and finally the whole sample chipped. The number of corresponding thermal cycles for the four mentioned conditions was recorded, and specified in Table 2.

The maximum strain at high temperature is the ratio of the indentation depth and the initial thickness of the filter under high temperature and a load. The loading temperatures were 1500 °C, 1550 °C and 1600 °C, and the load application time was one hour. The compressive force imposed on the sample by a 25 mm diameter alumina cylinder was 15 N, which corresponds to a 0.03 MPa compressive stress. Experimental results are summarized in Table 3.

Thermal shock resistance can be expressed in terms of the number of repeated abrupt thermal cycles before failure. In service a filter only experiences a one-time abrupt temperature change. Although there is no direct correlation between the number of thermal cycles before failure and filter performance in service, it is obvious that materials with better thermal shock resistance are of higher quality.

The high temperature maximum compressive strain (creep resistance) is often used to evaluate refractory quality [11], although it has not been utilized as a ceramic foam filters quality indicator. The measured maximum com-

Table 2. Number of thermal cycles before failure

Failure conditions	Product A	Product B	Product C	Product D
Cracks at edges and corners	29	5	10	16
Pieces dropping from edges and corners	-	-	12	17
Whole sample becoming loose	38	7	13	19
Chipping completely	43	8	15	22

Table 3. Maximum strain at high temperature

Heating process	Maximum strain under 15 N load, 0.03 MPa stress (%)			
	Product A	Product B	Product C	Product D
1500 °C × 1 h	0	10	0	0
1550 °C × 1 h	0	-	15	0
1600 °C × 1 h	0	-	-	18

pressive strain at high temperature has a good correspondence with filter quality, i.e. the smaller the maximum strain, the better the quality. A ceramic foam is subjected to pressure from the molten metal flow under a high temperature. If the filter does not deform much under a continuously applied load at high temperature, it is less likely to soften and break when molten metal flows through the pores. Thus the maximum strain at high temperature reflects the application characteristics of filters and can be utilized as the key quality indicator for evaluating ceramic foam filters. Based on this approach, product A has the best quality, product C and product D take the secondary place, and product B has the worst quality.

Strictly speaking, filters would rather break in tension before they break in compression because bulk ceramics are 10-15 times stronger in compression than in tension. This ratio is even higher for highly porous ceramics. The reason samples were tested in compression rather than in tension is associated with the difficulty of conducting tensile tests of ceramics at high temperature and the stochastic nature of the fracture of porous ceramics in tension. Also the fact that the alumina cylinder head diameter was smaller than the sample in-plane dimensions induced tensile stresses in the porous brittle sample [12].

Results and Discussion

Fractography analysis

The fracture surface morphology of the four products was analyzed using a JOEL JSM-5610LV scanning electron microscope (SEM). Corresponding fracture surfaces are shown in Fig. 1. Product A has large amounts of crystalline phases. Product B, C and D have a substantial amount of the glass phase, which covers the crystalline phases. There is less glass phase in product D than in products B and C.

The glass phase originates from the super-cooled liquid phase. The liquid phase is formed by the high temperature reaction of some substances in the ceramic clay body of

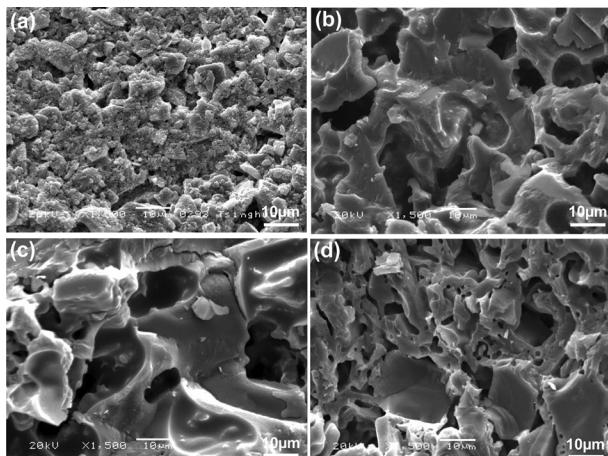
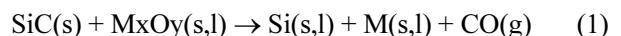


Fig. 1. Fracture surfaces of four samples: (a) Product A, (b) Product B, (c) Product C, and (d) Product D.

the filter. This wets crystal grain boundaries and fills spaces among grains. As a result the glass phase plays an adhesive role for crystalline grains and enhances the room temperature compressive strength. Product C has a lower bulk density than product D, and a higher room temperature compressive strength than product D. The bulk density of product C is approximately equal to that of product A, but the room temperature compressive strength of product C is obviously higher than that of product A. This must be due to the effect of the glass phase.

The glass phase will soften and even become liquid at a high temperature. Crystalline grains can easily slip because of the glass phase softening at high temperature. The glass phase increases the maximum strain at high temperature. The load-bearing capacity of the filter with more glass phase is lower than that with less glass phase at a high temperature. Micro-cracks tend to form in the glass phase during thermal shock. Therefore, the number of thermal cycles before failure is less for the products with more glass phase. The quantity of the glass phase obviously influences the number of thermal cycles and the maximum compressive strain at a high temperature. This is the reason why product A has a higher number of thermal cycles before failure and a lower maximum strain at a high temperature, which both signify its better performance.

Impurities and additives in raw materials promote glass phase formation, thus impurities must be controlled, especially Fe_2O_3 impurities in raw SiC powders [13]. If the raw materials contain more impurity metal oxides (metal oxides usually act as sintering aids), the quantity of sintering aids should be decreased to prevent more glass phase formation. At high temperature impurity metal oxide M_xO_y may react with SiC [14]:



where s, l and g in parenthesis denote solid, liquid and gas phases, respectively. The liquid phase can transform into a glass phase during cooling.

XRD phase analysis

Crystal phases were analyzed using a Philips X'pert Pro MPD X-ray diffractometer (XRD). Corresponding XRD patterns are shown in Fig. 2. All four products have large amount of the SiC cubic phase; products C and A have some $\alpha\text{-Al}_2\text{O}_3$ phase and a certain amount of quartz phase; product B has some mullite phase and a certain amount of cristobalite and quartz phases; product D has some $\alpha\text{-Al}_2\text{O}_3$ and cristobalite phases but also has a certain amount of the mullite phase. The mullite phase provides many advantages such as a uniform thermal expansion, a high thermal shock resistance, a high softening temperature under load and a small creep strain at a high temperature [15]. The mullite phase plays a small part in improving thermal shock resistance and in reducing the maximum strain at a high temperature for products B and D. The cristobalite undergoes phase transitions during heating and cooling, i.e.

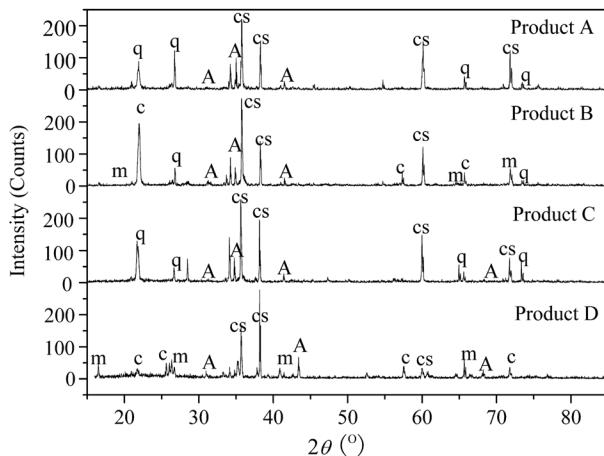
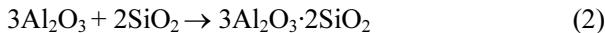


Fig. 2. XRD patterns of four products. Here, m is mullite, A is α - Al_2O_3 , cs is cubic SiC, c is cristobalite, and q is quartz.

α -cristobalite and β -cristobalite interchange during heating and cooling. There is 2.8% volume change during this phase transition (expansion or shrinkage), which degrades the thermal shock resistance. There is 0.82% volume change associated with the quartz phase transition [16].

Differences in crystal phases between products C and A are not distinct, although product C has a large amount of the glass phase. Therefore, the thermal shock resistance of product C is lower than that of product A.

Al_2O_3 as a sintering aid and silica sol as a high temperature adhesive are usually added in the ceramic slurry of SiC-based ceramic foam filters [13], thus mullite in products C and D can be synthesized from Al_2O_3 and SiO_2 in the silica sol:



The cristobalite in products B and D, and quartz in products A, B and C can be transformed from SiO_2 in the silica sol. Some minerals such as ferric oxide and calcium oxide are added in the ceramic slurry in order for α -cristobalite to be transformed into tridymite. The volume change is 0.2% during the tridymite transition [16], which is far less than that of cristobalite (2.8%). However, the quantity of mineral additives must be strictly controlled to prevent larger amounts of the glass phase from forming.

Conclusions

(1) The maximum strain at a high temperature was introduced for SiC-based foam as a quality indicator for casting filter applications. Better SiC filters have a

lower maximum strain at a high temperature and a higher number of thermal cycles before failure due to a proper phase content and microstructure.

(2) Higher quality filters have less glass and more quartz phases, while lower quality filters have more glass and cristobalite phases.

Acknowledgements

This study was supported by the Henan Province Program for Science and Technology Innovation Talents (104100510005 and 2009HASTIT023), by the Luoyang Applied Technology Study and Development Foundation (0901011A) and Henan Province Key Problem Project (092102210012). Alex Volinsky would like to acknowledge support from the National Science Foundation.

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