

Effects of xonotlite additive and forming pressure on the properties of the SiO₂-based nanoporous thermal insulation materials

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Nanoporous thermal insulation materials prepared with fumed SiO₂ possess excellent thermal insulation property, and have been attracted much attention, but their mechanical properties are poor. Adding a small amount of fiber materials as a toughening material can improve the mechanical properties of the nanoporous thermal insulation materials. However, too much fiber materials additives will seriously affect the thermal insulation property. In this study, SiO₂-based nanoporous thermal insulation materials were prepared, and the effects of addition amounts of xonotlite and forming pressure on the mechanical performance and thermal insulation performance of as-prepared thermal insulation materials were investigated. It was found that addition of 10% xonotlite and forming pressure of 2 MPa were proper for the preparation of SiO₂-based nanoporous thermal insulation materials. The compressive strength and thermal conductivity of as-prepared materials were 0.34 MPa and 0.031 W·m⁻¹·K⁻¹, respectively.

Key words: Xonotlite; Nanoporous Insulation Materials; Fumed SiO₂; Compressive Strength; Thermal Insulation Property.

Introduction

Recently, SiO₂ aerogels and fumed SiO₂ nanopowders are mainly used to produce SiO₂-based nanoporous thermal insulation materials [1, 2]. Both SiO₂ materials has excellent insulation performance for preparing nanoporous thermal insulation materials, but the preparation process of SiO₂ aerogels is complex and the supercritical drying process is characterized by high energy consumption and low efficiency. The use of atmospheric drying process greatly limits the large-scale production and application of SiO₂ aerogel-based nanoporous thermal insulation materials [3]. Therefore, fumed SiO₂ nanopowder is generally used as the matrix material for preparing SiO₂-based nanoporous thermal insulation plates in industrial applications.

Commercial SiO₂-based nanoporous thermal insulation materials have a low bulk density and an ultra-low thermal conductivity of less than 0.05 W·m⁻¹·K⁻¹, which means that they display excellent thermal insulation property. Although the fumed SiO₂ has good adiabatic performance as the SiO₂-based nanoporous, it is very brittle and easily broken for the low strength shown in the practical application.

In addition to the good thermal insulation property, high mechanical properties are required for thermal

insulation materials in engineering applications. The maximum external force applied to industrial furnaces is generally the compressive strength, rather than the bending strength [4]. For example, when the fumed SiO₂-based nanoporous thermal insulation materials is used as a thermal insulation layer in a steel ladle, the thermal insulation materials should have a certain compressive strength.

Smith et al. [5] was the first one who added fiber as a reinforcing material into the fumed SiO₂ in 1989. The results show that the mechanical properties of the fumed SiO₂-based thermal insulation materials can be effectively improved by this method. Abe et al. [6] used a mechanical mixing device called Mechanofusion System. When the system is rolling at a high speed, various forces are applied on fumed silica and reinforcing fiber bundle. The fibers are well dispersed into the matrix of fumed silica. The average thickness of the SiO₂ particles adhering to the single fiber was 3 μm, the porosity of the products was 80.1% and the fracture stress can reach 1.58 MPa. At present, the method of adding fibrous materials is still an effective means to improve the mechanical properties of the SiO₂-based nano-porous thermal insulation materials.

Adding a small amount of fiber material as a toughening phase can improve the mechanical properties of the SiO₂-based nanoporous thermal insulation materials, but the addition of more than 5% fiber material will greatly affect its thermal insulation property [7]. Xonotlite (6CaO·6SiO₂·H₂O)

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has been regarded as an ideal toughening agent for the preparation of nanoporous thermal insulation materials, because xonotlite powder has a small bulk density, low thermal conductivity, high compressive strength, better bending strength, and high temperature resistance (up to 1000 °C) [8]. Some scholars try to combine the SiO₂ aerogel and the xonotlite into the super thermal insulation materials which was low thermal conductivity and good mechanical performance. Zeng et al [9] prepared the xonotlite-silica aerogel nanoporous super thermal insulation materials from fiber xonotlite and SiO₂ aerogel. It was found that SiO₂ aerogel were evenly distributed in the calcium silicate substrate, pore sizes were between 10 and 50 nm, and the average particle size was less than 20 nm. Yang et al [10, 11] also used above similar method to investigate the effect of different preparation condition on the thermal conductivity of xonotlite-silica aerogel nanoporous super insulation materials and the optimum conditions of adiabatic performance were obtained.

In the present work, the thermal insulation and mechanical properties of fumed SiO₂-based nanoporous thermal insulation materials under different experimental conditions were analyzed, and the influences of addition amounts of xonotlite and forming pressure on the compressive strength and thermal insulation performance were investigated. Finally, proper addition amounts of fumed SiO₂ and forming pressure for preparing the SiO₂-based nanoporous thermal insulation materials with excellent overall performances including thermal insulation and mechanical properties were determined.

Experimental

Materials

Tables 1 and 2 list the basic properties of SiO₂ nano powder and other raw materials.

Specimen preparation

SiC, fiber materials [12], and a small amount of Al₂O₃ or TiO₂ nano-powder [13, 14] were generally added into the fumed SiO₂ matrix, and they were respectively used as an infrared opacifier, toughening material, and sintering inhibitor, so SiO₂-based nanoporous thermal insulation materials with superior performances can be obtained.

In this experiment, the SiO₂-based nanoporous thermal insulation materials were prepared by the dry mixing and compression molding process, and fumed SiO₂ as the matrix material and some additives are shown in Tables 1 and 2.

Table 3 shows the preparation conditions of the specimens under different addition amounts of xonotlite powder and forming pressure. The specimens are respectively numbered as E₀, E₁, E₂, E₃, E₄ and E₅. For the purpose of the comparison of the influences of different additions, the additions of xonotlite powder and the

Table 1. Basic properties of SiO₂ nano powder.

Si source	Fumed SiO ₂
Model	HL200
Appearance	Fluffy white powder with high adsorption
Refractoriness (°C)	1750
SiO ₂ (%)	99.8
Specific area (m ² ·g ⁻¹)	200 ± 20
Primary particle size (μm)	0.015
Bulk density (g·cm ⁻³)	0.04 ~ 0.06

Table 2. Raw materials for the preparation of SiO₂-based nanoporous thermal insulation materials.

Raw materials	Specifications
SiC powder	W3.5
Glass fiber	Length = 4.5 mm, diameter = 9 μm
xonotlite powder	74 μm
Fumed Al ₂ O ₃	AEROXIDE Alu C

Table 3. Preparation conditions of specimens.

Specimens	Xonotlite powder (wt%, extra)	Forming pressure (MPa)	Bulk density (g·cm ⁻³)
E ₀	0	2	0.307
E ₁	0	5	0.523
E ₂	5	2	0.311
E ₃	5	5	0.543
E ₄	10	2	0.327
E ₅	10	5	0.532

matrix material were the same in all the specimens.

The raw materials and additives were weighed according to the mass ratio of fumed SiO₂ : SiC : glass fiber : fumed Al₂O₃ : xonotlite powder of 70 : 15 : 5 : 10 : (0, 5, 10), and different amounts of xonotlite was added to enhance the compressive strength. The raw materials and additives were mixed, and then pressed into a pie shape with the size of Φ180 mm × (20-25) mm.

Performance testing

Scanning electron microscopy was used to observe the microstructures of the specimens doped with reinforced fibers after spraying gold nanoparticles. X-ray diffraction analysis was performed with Cu target under the conditions of 40 mA, 40 kV, and scanning step of 0.08 °·min⁻¹. The compressive strength was measured with a unidirectional electronic tensile testing machine under the testing speed of 1 mm·min⁻¹. The thermal conductivity of specimens was measured using high-temperature thermal conductivity meter under the heating speed of 10 °C·min⁻¹, and the averaged value measured for three times was determined as the final thermal conductivity.

Results and Discussion

Figs. 1(a) and 1(b) respectively show SEM images of xonotlite powder and specimen E_2 doped with 5% xonotlite powder and pressed at 2 MPa. It can be clearly seen that the particles of xonotlite are in a micron scale and show the lamellar structure. The energy spectra of the specimen E_2 with a doping mass fraction of 5% showed that the xonotlite powder had been well dispersed in the matrix material (Figs. 1(c) and 1(d)). The complete dispersion is a precondition for toughening fumed SiO_2 -based nanoporous thermal insulation materials.

Fig. 2 shows XRD pattern of specimen E_2 doped with 5% xonotlite powder and pressed at 2 MPa. It was found that various additives had been well dispersed in the powder matrix.

Fig. 3 shows the compressive stress-deformation rate curves of the specimens E_0 - E_5 . Under the forming pressure of 2 MPa and the compression ratio of less than 15%, the doped specimens E_2 and E_4 showed the higher compressive strength than undoped specimen

E_0 . Under the same compression ratio of 10%, the required compressive strengths of E_2 and E_4 were respectively increased to 0.34 MPa and 0.31 MPa because the sheet-like xonotlite particles additive acted as the supporting skeleton in the matrix material to a certain degree. However, when the forming pressure increased to 5 MPa, compared with E_1 , the specimens E_3 and E_5 did not show the high compressive strength under a low compression ratio. The doped specimen E_3 did not show the significantly higher compressive strength than specimen E_1 until the compression ratio was increased above 8% and the doped specimen E_5 did not display the significantly higher compressive strength than specimen E_1 until the compression ratio was increased above 13%. The phenomenon might be interpreted as follows. The specimen E_1 was prepared under higher forming pressure and the dopant did not play the role of the supporting skeleton under the low compression ratio. The comparison results of the specimens E_1 , E_3 , and E_5 indicated that when the forming pressure increased to 5 MPa, the addition of xonotlite (5%)(E_3) allowed the significantly increased compressive

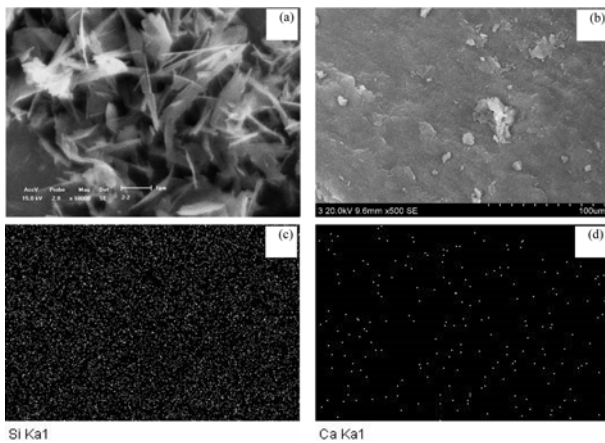


Fig. 1. SEM images of xonotlite powder (a) and specimen E_2 doped with 5% xonotlite powder (b) as well as Si (c) and Ca (d) element distributions of specimen E_2 measured by energy dispersive spectrometer.

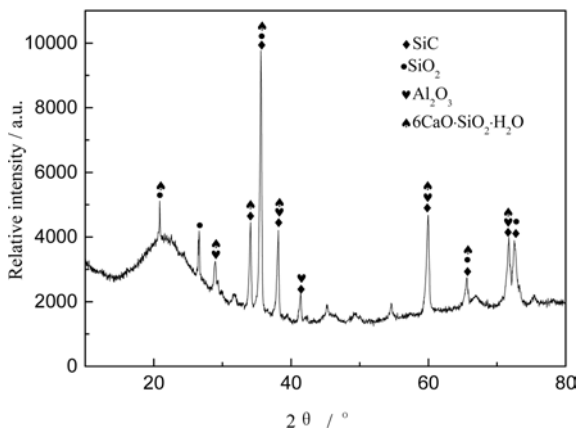


Fig. 2. XRD pattern of specimen E_2 doped with 5% xonotlite powder and pressed at 2 MPa.

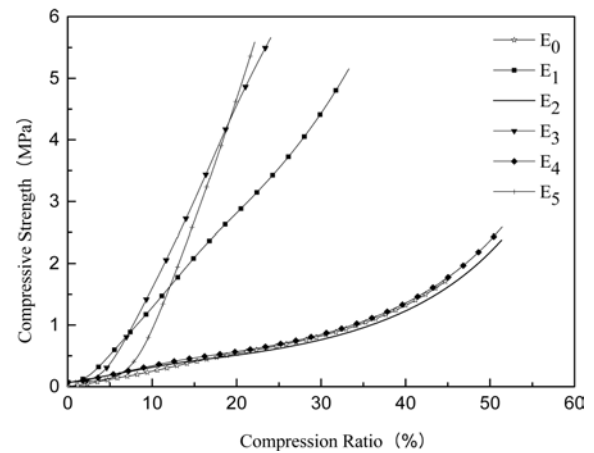


Fig. 3. Curves of compressive strength versus the deformations of specimens E_0 - E_5 .

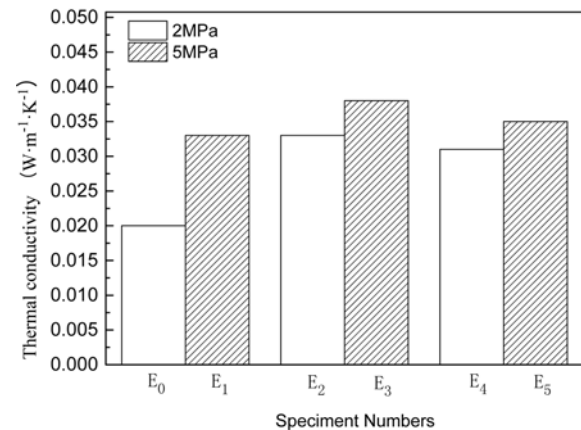


Fig. 4. Comparison of the thermal conductivity of specimens E_0 - E_5 at 1000 °C.

strength (1.60 MPa) under the compression ratio of 10% compared to the undoped specimen (E_1). However, the addition of xonotlite (10%) (E_5) did not show the significantly increased compressive strength compared to the addition of 5% until the compression ratio increased to 20%.

Fig. 4 shows the thermal conductivity of the specimens E_0 to E_5 tested at 1000 °C. The thermal insulation mechanism of fumed SiO₂-based nanoporous materials is that the microholes among raw SiO₂ particles and multi-stage composite nanoporous structures within and among aggregates limits the internal flow of gas molecules and collisions among gas molecules, thus suppressing the convective heat exchange. In other words, the effect of suppressing heat transfer stems from the multi-stage composite nanoporous structure.

When the forming pressure of the specimen was gradually increased (from 2 MPa to 5 MPa), the thermal conductivity of the specimen E_1 increased to 0.033 W·m⁻¹·K⁻¹ because the nanoporous structure for suppressing heat conduction and convection was weakened. Since the particle sizes of the xonotlite powder and the matrix material were in two orders of magnitude, the doping process might be considered as the process that introducing a certain number of defects inside the aggregates destroyed the nanoporous structure, thus increasing the solid-phase heat transfer to a certain degree. Therefore, the thermal conductivity of the specimens E_3 and E_5 was increased to different degrees.

In summary, Under the forming pressure of 2 MPa, although the thermal conductivity of the specimen E_2 was increased to 0.033 W·m⁻¹·K⁻¹, its compressive strength was increased to 0.31 MPa (at the compression ratio of 10%), whereas the compressive strength of undoped specimen E_0 was 0.25 MPa. After the addition of xonotlite increased to 10%, the thermal conductivity of the specimen E_4 was only 0.031 W·m⁻¹·K⁻¹, its compressive strength was further increased to 0.34 MPa. When the forming pressure of the materials was increased from 2 MPa to 5 MPa, the bulk density of the specimen was significantly increased from 0.307 g·cm⁻³ (E_0) to 0.523 g·cm⁻³ (E_1) and the compressive strength of the specimen was significantly increased from 0.25 MPa (E_0) to 1.29 MPa (E_1) at the compression ratio of 10%. The thermal conductivity of the specimen was increased from 0.023 W·m⁻¹·K⁻¹ (E_0) to 0.033 W·m⁻¹·K⁻¹ (E_1). Under the addition of xonotlite of 5%, the compressive strength was significantly increased to 1.60 MPa (E_3) and the thermal conductivity increased to 0.038 W·m⁻¹·K⁻¹ (E_3). In addition, this toughening effect becomes more significant as the compression ratio increased. The results showed when the addition of xonotlite powder was increased to 10%, the compressive strength decreased and the decrease of the specimen E_5 was more significant compared to the specimen E_3 at the compression ratio lower than 20%,

Table 4. Properties comparison of specimens.

Materials	Compressive strength (MPa)	Thermal conductivity (W·m ⁻¹ ·K ⁻¹)	Bulk density (g·cm ⁻³)
E_6 [10,15]	0.35	0.025 (25 °C) 0.048 (480 °C)	0.220
E_2	0.31	0.033 (1000 °C)	0.311
E_4	0.34	0.031 (1000 °C)	0.327

as shown in Fig. 3. Therefore, the proper addition of xonotlite powder was 5%.

Table 4 compared the properties of specimens prepared in this work and some data from literatures. E_6 was the xonotlite-silica aerogel nanoporous super insulation materials and the data of E_6 comes from the literatures [10] and [15]. The compressive strength of the E_6 can be improved to about 0.35 MPa, which is a little higher than the compression strength of E_2 and E_4 . But the thermal conductivity of E_6 is 0.048 W·m⁻¹·K⁻¹ tested at 480 °C, which is far higher than those of the specimens E_2 and E_4 tested at 1000 °C of 0.033 W·m⁻¹·K⁻¹ and 0.031 W·m⁻¹·K⁻¹, respectively. Thus, the SiO₂-based thermal insulation materials by addition small amount of xonotlite display better comprehensive properties.

Conclusions

In this study, SiO₂-based nanoporous thermal insulation materials were successfully fabricated and the influences of the addition amounts of xonotlite and forming pressure on the mechanical and high-temperature thermal insulation properties of as-prepared materials were explored. The conclusions were drawn as follows:

(1) Proper addition of xonotlite could improve the compressive strength of SiO₂-based nanoporous thermal insulation materials while basically maintaining the thermal conductivity. Under the forming pressure of 2 MPa and the addition of xonotlite (10%), the compression strength of the specimen was increased from 0.25 MPa to 0.34 MPa at the compression ratio of 10%, whereas the thermal conductivity was only increased from 0.023 W·m⁻¹·K⁻¹ to 0.031 W·m⁻¹·K⁻¹.

(2) Under the forming pressure of 5 MPa and the addition of xonotlite (5%), its compressive strength was significantly increased to 1.60 MPa at the compression ratio of 10%. However, the addition of xonotlite (10%) did not show the significantly increased compressive strength compared to the addition of 5% until the compression ratio increased to 20%. Considering the effects of xonotlite on the performance of SiO₂-based nanoporous insulation materials, the addition amount of 5% xonotlite was recommended.

Acknowledgments

The authors would like to express their gratitude for

the financial support from the National Natural Science Foundation of China (Grant Nos. 51704223 and 51504133).

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