Effect of Fiber Content on Properties of Fiber Reinforced Nano-SiO$_2$ Thermal Insulation Material

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Abstract. In this paper, fumed nano-SiO$_2$ as the main raw material, SiO$_2$ insulation materials were prepared by the dry processing. Porous characteristic, insulation performance and mechanical properties have been systematically studied. The dry processing method for the fabrication of fibrous fumed silica boards is used. As a reinforcing fiber, K$_2$O•6TiO$_2$ not only improves the strength of the material, but also enhances the thermal insulation performance of the material at high temperature. The effect of fiber content on the mechanical and personal properties of materials was studied. At 20% fiber content, the compressive strength reaches 3.96 MPa, and the thermal conductivity increases from 0.048 W/m•k at 200°C to 0.094 W/m•k at 600°C.

1. Introduction

At present the insulation materials commonly used on industrial were mainly represented by lightweight shaped insulation materials and various kinds of fiber composite insulation materials. Due to the relatively high thermal conductivity of these traditional insulation materials, the effect of heat preservation is hard to realize the requirements of insulation design. Therefore, increasing the thermal insulation layer thickness to satisfy the requirements of thermal insulation were often used in practical application, which resulting many problems such as the kiln cost increased, the effective space reduced, and so on. So the researching of new insulation materials with light weight and low thermal conductivity has become a hotspot area.

In the study of new type thermal insulation material, the nanoscale pore structure of insulation material with low thermal conductivity, known as super thermal insulation material, become a hot topic of research at home and abroad in recent years. The characteristics of the nano pore size less than the gas molecular mean free path are used by nanopores insulation material, which restraining the collisions between the gas molecules, thus reducing the gas thermal conductivity. Its thermal conductivity of general temperature is lower than air, while the thermal conductivity of high temperature also far less than the average of fiber felt insulation material.

Currently, the most studied nano porous insulation materials are aerogels and nano SiO$_2$ based thermal insulation materials. Among them, SiO$_2$ aerogels are the most aerogel super insulation materials, which have the most extensive research at home and abroad. They have excellent thermal insulation, heat resistance, corrosion resistance and other properties. However, SiO$_2$ aerogel insulation materials have excellent thermal insulation properties, but there are many problems in their preparation process[1]. The preparation process is extremely complex, and the requirements for pressure, sealing and other parameters are extremely high. Because the supercritical drying technology...
is needed in the preparation process, and the price of supercritical drying equipment is also very expensive, the preparation cost of SiO$_2$ aerogel is too high, which is not conducive to the popularization and practical application of aerogel products.

Nano SiO$_2$ powder based thermal insulation material is made of nano SiO$_2$ powder, and through a certain molding process, heat insulation material with nano pore structure similar to aerogel is obtained. This process is mainly a dry process, which is to obtain nano SiO$_2$ powder based thermal insulation materials prepared by dry method by directly pressing the raw materials in the die after mixing uniformly. The preparation process is simple, pollution-free and short cycle. The pore structure of the insulation material is nanometer size, and has the thermal insulation property similar to the aerogel. Therefore, compared with aerogel insulation materials, this process can greatly reduce the preparation cost.

Nano-SiO$_2$ thermal insulation materials were prepared by dry forming in Zhang [2]. The effect of forming pressure on the pore structure and thermal insulation properties of thermal insulation materials was studied. It can be seen that the pore size covers all the range of the third-order pore. (1) The first-order voids: the internal voids in the hard aggregates, i.e. the voids between the primary particles that make up the hard aggregates, with a size range of less than 50 nm; (2) the second-order voids: the voids between the hard aggregates, or the internal voids in the soft aggregates, with a size range of 50-500 nm; (3) the third-order voids: the voids between the soft aggregates, with a size range of more than 500 nm. With the increase of forming pressure, the third and second stage void structures are destroyed successively, while the first stage void structures are not destroyed. When the molding pressure is 4 MPa, the thermal conductivity is the lowest at 200$^\circ$C, 400$^\circ$C, 600$^\circ$C, and 800$^\circ$C, which are 0.023 W/m•k, 0.041 W/m•k, 0.088 W/m•k, 0.197 W/m•k.

However, the nano-SiO$_2$ powder-based thermal insulation materials prepared by this process also have the defect of poor mechanical strength. Such as in the study of Zhang [2], the Strongest compressive strength is 1.03MPa. The practical nano-SiO$_2$ powder-based thermal insulation materials developed at home and abroad have to adopt various processes to strengthen the matrix. Domestic research only adds a large number of fibers or binders to increase the strength, but at the same time, the strength improves, it also brings thermal insulation performance degradation. Because the preparation process is relatively simple, some domestic manufacturers have also introduced related products, but the products have few types, low strength, easy to drop powder and slag, there are still many problems in practical application. Therefore, how to prepare products with uniform structure, strength and toughness which can meet the market demand has become the focus of current research.

Potassium hexatitanate is a new reinforcing material. The composition of potassium hexatitanate is K$_2$O•6TiO$_2$, which is a chain tunnel structure with K$^+$ ions in the middle of the tunnel. The direct result of this structure is that K$^+$ ions have high stability. Because of this structure, potassium hexatitanate whiskers have excellent mechanical and physical properties, stable chemical properties, low thermal conductivity at high temperature, high heat capacity, excellent corrosion resistance, heat insulation, wear resistance, lubricity, and extremely high infrared reflection and diffuse reflectance.[3] Therefore, potassium hexatitanate is often used in the field of thermal insulation materials, such as space launchers, ordnance, automotive exhaust pipes, dryers, petrochemical pipelines, thermal insulation materials or coatings in specific areas requiring high temperature.

2. Experimental

2.1. Materials.
Firstly, the nano-SiO$_2$ powder was fully dispersed by the strong shear action of the ultra-high speed disperser, and the aggregates of larger particles were smaller to avoid the existence of aggregates as far as possible. At the same time, the fiber bundles were peeled off by a fiber disperser to reduce the cross-linking between single fibers. Then the dispersed fibers and K$_2$O•6TiO$_2$ were fully mixed in the raw materials of nano-SiO$_2$ powders. Because the prepared mixture is extremely light and the particles are fine and easy to float in the air, it needs to be pre-pressed by micro-porous ventilation device to
extrude the air inside the mixture. Subsequently, the pre-pressing body is placed in the die and pressed by a hydraulic press. In the process of pressing, the pressure speed should be strictly controlled, because too fast pressure speed will produce stress concentration and lead to sample cracking. Finally, after a certain heat treatment, fiber reinforced nano-SiO2 composite insulation material can be obtained.

The dry processing method for the fabrication of fibrous fumed silica boards are shown in figure 1 [4]. Silica nanoparticles were first loosely coated on the glass fibers to make a composite with a porous coating layer. Then, the composites were compacted to form a board [5]. Its uniqueness was that the fiber composites were mechanically produced by processing fumed silica and glass fibers without collapsing the nano pores associated with the fumed silica. This was based on the fact that appropriate mechanical processing could produce core/shell-type composite particles with metal oxide powders without using any binders in the dry phase.

Figure 1. Schematic illustration of the dry processing method for the fabrication of fibrous fumed silica boards: (a) mixing of raw materials, (b) mechanical processing to coat glass fiber with fumed silica and (c) dry pressing of the composite (b) to produce bulk body.[4]

2.2. Testing.

The thermal conductivity of the samples at high, medium and low temperatures was measured by PDB-15-7 (high temperature) plate thermal conductivity tester of Luoyang Antelier Instruments Co., Ltd. and HC-074-200 thermal conductivity tester of EKO in the United States.

The pore size distribution of various samples prepared in the study was determined by using the OrMaster 60 mercury porosimeter of Quantachrome.

The compressive strength of the samples before and after reinforcement was measured by UMT53050 000 Capability Testing Machine of Shenzhen Sansi New Material Company.

3. Results and discussion

Fiber reinforcement is the most common reinforcement method at present. Its reinforcement mechanism mainly includes the following three kinds[6]:

1. Crack bending and deflection: The fibers cause the crack to bend and rotate by interfering with the stress field, which lengthens the crack propagation path and consumes more energy, which hinders the development of cracks and plays a toughening role.

2. Fiber bridging: When the fibers are directionally distributed, a bridging zone will be formed at both ends of the crack. A compressive stress can offset the external tensile stress, which makes the crack propagation difficult and thus plays a toughening role.

3. Fiber pull-out: The pull-out of the fibers requires the crack to separate along the interface between the matrix and it under the external force, that is to say, the pull-out work is needed to overcome the friction, so it can play a toughening role.
For fiber reinforced nano-SiO₂ thermal insulation materials, nanoparticles bear most of the loads at the initial stage of the matrix loading. When the load gradually increases to a certain extent, the matrix begins to produce micro-cracks and begin to expand. When the crack propagation meets the fibers, it will be hindered and the crack propagation path will be deflected. With the further expansion of the crack, the fiber pulls out and the material breaks. In this process, the fiber increases the pull-out work, thus increasing the energy required for the material to break, thus playing a reinforcing role.

Figure 2 shows the compressive strength and density curve of the sample with different K₂O₆TiO₂ fiber content. As can be seen from the figure, the compressive strength of the sample increases with the increase of fiber content. When the fiber content was 10%, the compressive strength of the sample almost doubled to 3.53 MPa. When the fiber content increases from 10% to 20%, the compressive strength increases to 3.96 MPa, and the compressive strength increases slowly with little increase. At the same time, with the increase of fiber content, the density of the sample increases, which has a certain effect on the strength of the sample. However, in general, the compressive strength of samples can be significantly increased with the increase of fiber content.

![Figure 2. Compressive strength and density curve of the sample with different fiber content.](image)

Figure 3 shows the distribution of fibers in the matrix. It can be seen that the fibers bind well with the nano-SiO₂ matrix. Most of the fibers are oriented parallel to the compression surface and distributed evenly without obvious agglomeration.

![Figure 3. Distribution of fiber in matrix.](image)
Figure 4 shows the variation curve of thermal conductivity at different temperatures with different fiber content in the sample. The thermal conductivity increases obviously with the increase of fiber content at 200°C. The obvious decrease in thermal insulation performance is due to the fact that when a large number of fibers are added to the matrix of the material, as shown in figure 5, fibers and fibers no longer exist in the matrix like a single steel bar, but interlace with each other to form a cobweb-like structure. In this way, the solid-state heat transfer mode of the material changes from the contact heat transfer of the path and its long nanoparticles to the straight-line heat transfer, that is to say, the thermal bridge effect is formed in the interior of the material. The thermal conductivity of quartz fibers is much larger than that of nano-powder particles, so the heat transfer rate is greatly increased in the process of heat transfer, which affects the overall thermal insulation performance.

![Figure 4](image.jpg)

**Figure 4.** Variation curve of thermal conductivity at different temperatures with different fiber content.

![Figure 5](image.jpg)

**Figure 5.** SEM of samples with a large number of fibers.

It is noteworthy that the influence of fiber content on thermal conductivity decreases with the increase of test temperature. At 600°C, the thermal conductivity is maintained at about 0.09 W/m•K, which may be the result of many factors. As mentioned above, K₂O•6TiO₂ has a negative temperature coefficient, that is, it has a low thermal conductivity at high temperature. At the same time, it has high reflectivity or refractive index, which can change the transmission path of infrared radiation and play a role in preventing infrared radiation heat transfer.
Figure 6 is a comparison curve of pore size distribution between samples with 0%, 5%, 10%, 15% and 20% K₂O•6TiO₂ fibers respectively. From the figure, it can be seen that the pore size distribution of the sample before and after adding fibers has not changed much, and the peak size of the most concentrated pore size is about 10 nm, which is the inner pore of the hard aggregate. In addition, there is also a large peak near 100-300 nm, which is the pore between hard aggregates. Only in the sample with fibers added, some smaller peaks can be seen in the large size region. These peaks are concentrated in the range of 5-10 μm, but the peak area is very small, which indicates that the proportion of these macropores is very small. It can be seen that the addition of fibers less than 20% will introduce some macropore, but it does not change the main pore size structure of the sample. Most of the pore size is in the nano-scale range.

4. Summary

(1) Fumed nano-SiO₂ as the main raw material, SiO₂ insulation materials were prepared by the dry processing. The dry processing method for the fabrication of fibrous fumed silica boards are used. As a reinforcing fiber, K₂O•6TiO₂ not only improves the strength of the material, but also enhances the thermal insulation performance of the material at high temperature.

(2) When the fiber content was 10%, the compressive strength of the sample almost doubled to 3.53 MPa. When the fiber content increases from 10% to 20%, the compressive strength increases to 3.96 MPa, and the compressive strength increases slowly with little increase.

(3) The thermal conductivity increases obviously with the increase of fiber content at 200°C. At 600°C, the thermal conductivity is maintained at about 0.09 W/m·K.

(4) The pore size distribution of the sample before and after adding fibers has not changed much, and the peak size of the most concentrated pore size is about 10 nm, which is the inner pore of the hard aggregate.

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