

Microstructure and Thermal Characterization of Multilayer Insulation Materials Based on Silica Aerogels

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Abstract: A new kind of MLIMs are prepared, whose spacer is made from Al_2O_3 fiber mat, and silica aerogels is implanted on the surface of Al_2O_3 fiber through Sol-Gel process. In high vacuum (10^{-3} Pa), the effective thermal conductivity of the new MLIMs could be reduced by 21.8%, at the same time, the weight could be reduced by 67.2%. Furthermore, this MLIMs shows good insulation performance in high temperature of 1200 K and low vacuum (100 Pa).

Key words: multilayer insulations; silica aerogels; thermal conductivity

The multilayer insulation materials (MLIMs) have been widely used in the spacecrafts, polar environments, volcano exploration and so on^[1]. It is composed of alternately stacked low emissivity heat shield and low thermal conductivity spacer. Traditional MLIMs used in the high vacuum environment has good insulation characteristics, which called “super heat insulation material”. However, if the MLIMs are used in the low vacuum environment, the heat barrier performance of MLIMs is relatively not so satisfying^[2]. As is widely known, spaceships will experience about 5 min of low vacuum environment when they return to the earth. Because of the drastic aerodynamic heating effect in this period, it is very necessary to develop new MLIMs used in low vacuum. This study was targeted at reducing the thermal conductivity of the MLIMs used in low vacuum.

Silica aerogel with more than 90% pores has excellent low thermal conductivity, and it is a potential candidate of spacer material for MLIMs^[3]. Granular silica aerogel has been used in some insulation application. However, there is seldom related report about silica aerogels application for the MLIMs. In this paper, a new kind of spacer material implanted by silica aerogels for MLIMs was fabricated; the thermal conductivity in low vacuum as well as the barrier performance in 1200 K was studied.

1 Experimental

1.1 Preparation of silica aerogel- Al_2O_3 fiber composite

Silica aerogels were fabricated using a polyethoxydisiloxanes (E-40) based recipe which is a solution with ingredient of $n(\text{ethanol}):n(\text{E-40}):n(\text{H}_2\text{O}):n(\text{HCl})=3:1:0.2:0.1$. After mixing, the mixed solution was stirred for 30 min at room temperature and placed in the water bath at 60°C for

2 h. Silica hydrogels was obtained after completion of hydrolysis and polycondensation reactions. Thereafter, Al_2O_3 fiber mat was impregnated by the hydrogels. Here, the Al_2O_3 fiber mat was prepared *via* Sol-Gel process. The chemical composition of Al_2O_3 fiber mat is 95% Al_2O_3 +5% SiO_2 . The fiber diameter is about 3 μm to 7 μm , and the length is about 100 mm. To strengthen the gel network, the hydrogel filled mat was immersed into 50vol% ethanol solution at 50°C to exchange part of water in the hydrogels^[4].

After aging, the composite was immersed in hexamethyldisiloxane (HMDSO) for several days to complete solvent exchange, and then proper trimethylchlorosilane (TMSCl) was added for surface modification. Finally, the mat filled by the modified hydrogel was dried at 200°C with an infrared lamp for 2 h and the silica hydrogel in mat changed into silica aerogels^[5].

1.2 Design and assemble of MLIMs samples

The structure of traditional MLIMs is shown in Fig. 1. MLIMs composes of repetitive unit, and every unit is consisted of a heat shield with low emissivity and a spacer with low thermal conductivity, the heat shield is made from Polyimide(PI) coated with Al thin film, and spacer is made from Al_2O_3 fiber mat. The traditional MLIMs are marked as Sample I. In this paper, the new MLIMs are marked as Sample II, in which the spacer is made from Al_2O_3 fiber mat and silica aerogels is implanted on the surface of Al_2O_3 fiber. In order to make a comparison between the two samples, we made them into a similar thickness.

The micro morphology of silica aerogels is analyzed by SEM, and the thermal conductivity is tested by calorimetric emissometer designed according to the steady-statecalorimetric method^[6].

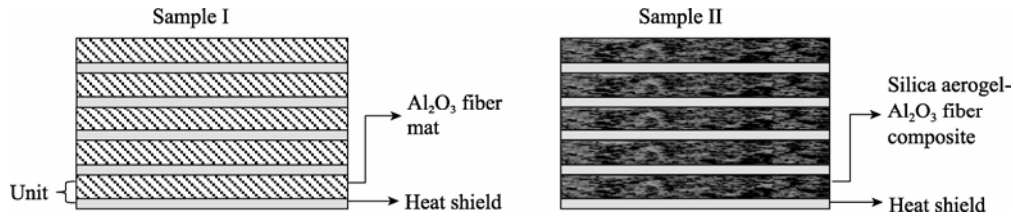


Fig. 1 The structure of sample I and II

1.3 Effective thermal conductivity measurement

The effective thermal conductivity (λ_e) of MLIMs was measured based on the steady state calorimeter method^[7]. For a MLIM, its effective thermal conductivity, λ_e is the most critical performance factor. In this work, λ_e is determined according to the cylinder steady-state conduction model, λ_e could be expressed as formula (1)^[8]:

$$\lambda_e = \frac{Q \ln(r_2 / r_1)}{2\pi L(T_1 - T_2)} \quad (1)$$

Where, r_1 , r_2 is the radius of the sample inside and outside (m), L is the length of the state-temperature area (m), T_1 , T_2 is the inner and outer temperature of the sample (K), Q is the power of heat consumption (W).

In this work, the testing equipment has a heat pipe with coatings and heater associated with thermostat. The sample was mounted in the vacuum chamber. To reduce errors, we fetch about 40 mm length located the middle of the sample as measuring area, which will match the requirement of cylinder steady-state conduction model.

2 Results and discussions

2.1 Microstructure of silica aerogel- Al_2O_3 fiber composite

Figure 2(a) shows the microstructure of silica aerogel- Al_2O_3 fiber composite. It can be seen that the silica aerogels is a porous structure material. It is proposed that the silica aerogels has 80%–99% open porosity and a three-dimensional solid network like silica framework which changes the heat transfer mode in MLIM. The heat conduction path in the porous material is extended. The mean free path of air molecules is about 70–100 nm (300 K), which is close to the particle size of silica aerogels^[9], hence, air molecules could be bound by pores in aerogels. As a result, the thermal conductivity of this new material has a significant reduction.

From Fig. 2(b), it could be seen that a special structure was formed with some silica aerogels clung to Al_2O_3 fibers (point A) and parts of silica aerogels filled in the space of Al_2O_3 fibers (point B). This particular steric configuration could reduce the heat transfer between fibers. It also could make the material has a certain strength, and other special properties.

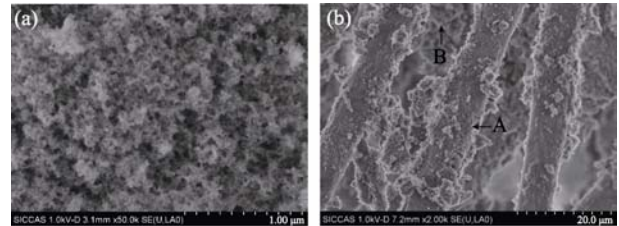


Fig. 2 SEM images of silica aerogels granules (a) and silica aerogels-fiber composites (b)

2.2 Effective thermal conductivity of MLIM

The effective thermal conductivity of Sample I and II at different temperatures, was tested, and the air pressure is 10^{-3} Pa. Figure 3(a) shows. the λ_e of sample II is always lower than that of sample I. From 300 K to 550 K, the thermal conductivity of sample II changes from 1.27×10^{-3} W/(m·K) to 2.24×10^{-3} W/(m·K). Such as, when

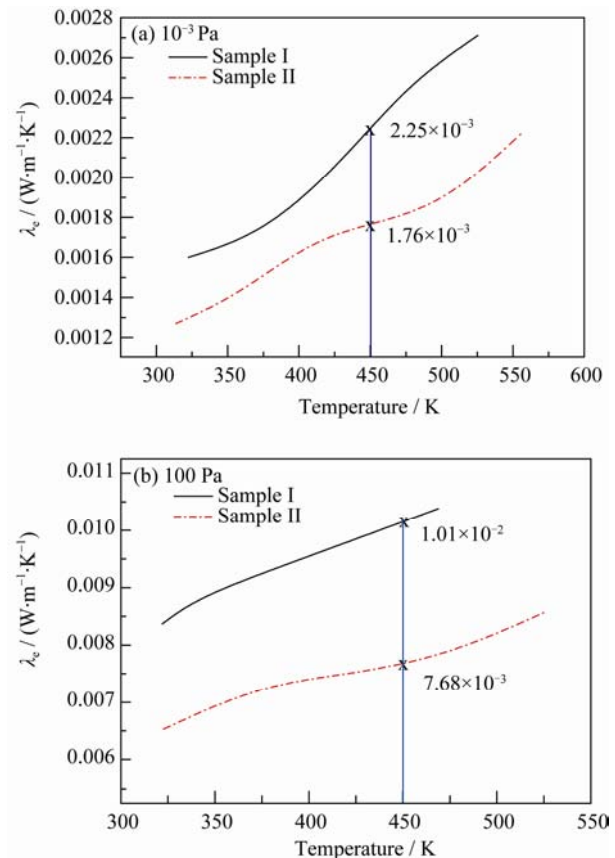


Fig. 3 Thermal conductivity with temperature changes of Sample I and II

the temperature is 450 K, λ_e of sample I and sample II are 2.25×10^{-3} and 1.76×10^{-3} W/(m·K), respectively. It has 21.8% reduction amplitude.

Because 100 Pa is a typical air pressure in Near space (20–100 km from the earth surface)^[10], thermal conductivities of two samples were tested at this pressure. As shown in Fig. 3(b), Sample II has obviously better performance than Sample I. From 300 K to 550 K, the thermal conductivity of sample II changes from 6.53×10^{-3} W/(m·K) to 8.57×10^{-3} W/(m·K). At 450 K, λ_e of Sample I is 1.02×10^{-2} W/(m·K), and it is 7.68×10^{-3} W/(m·K) for sample II which is reduced by 24.7%. Sample II still shows good insulation performance at low vacuum.

Sample II has better thermal barrier performance than sample I in all the temperature and air pressure because of the silica aerogels. In the experiment, we also found that compared with sample I, the weight of the Sample II is lighter. Sample II has 67.2% reduction amplitude, which is very important for the space science and technologies. It can be clearly seen from above results that silica aerogels pacer has great potential in multilayer insulation field. Under the condition of quantity production, the cost of new MLIMs system can be reduced to about 40 €/m².

2.3 Simulation of high-temp test by ANSYS

At high temperature (>1000 K), PI heat shield is easily melted down, and the structure of silica aerogels is also destroyed. For high temperature test, we prepared a new Sample II with 30 units as shown in Fig. 4 and Table 1. In the sample, the MLIM was divided into two parts, which were high-temp section and low-temp section.

In order to predict the thermal performance of the MLIM at high temperature, we developed a method to simulate the temperature response by using the software ANSYS. ANSYS is a kind engineering simulation software based on the Finite Element Method^[11].

To simplify the problem, the MLIMs were considered as a uniform thermal conductivity at both two sections. The heating boundary kept constant temperature of 1200 K for 240 s, and the cold border was adiabatic. The background temperature was 293 K. Table 2 shows thickness, density, and specific heat capacity of two samples.

As shown in Fig. 5, the cold boundary of traditional sample rose to 300 K while cold boundary of new sample only rose to 294 K. The temperature difference became larger with prolonging the time.

From the simulation, it can be seen that the new MLIMs shows better thermal barrier properties than traditional MLIMs at high temperature. At 240 s, the cold boundary of new MLIMs keeps at room temperature, which is very important to precision machines normal operation and astronaut's life safety.

2.4 New MLIMs in high-temp test

In order to verify the accuracy of the simulation results, we make an actual test of the new MLIMs. The high-temp section of the sample is contacted a heat source whose temperature is 1200 K, the low-temp section face the black environment and the air pressure is high vacuum (10^{-4} Pa) in high-temp experiment. To monitor the temperature variation of two section of sample, three thermal couples were bonded on the surface of unit in heating surface, low-temp section and the cold boundary.

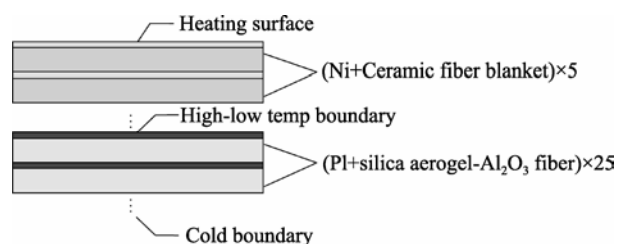


Fig. 4 The structure of new MLIMs in high-temp experiment

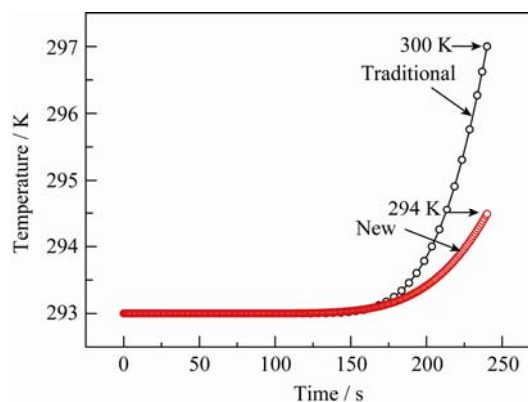


Fig. 5 Simulated result of two samples in high-temp test

Table 1 Two parts of MLIMs used for different temperatures

Section	Temperature range/K	Low-emissivity shield	Spacer material
Low-temp	300–600	Aluminized polyimide film (PI) ($T=30 \mu\text{m}$)	Silica aerogel- Al_2O_3 fiber mat
High-temp	600–1300	Ni foil ($T=15 \mu\text{m}$)	Ceramic fiber blanket

Table 2 Spacers parameters of two samples

Spacers	Thickness/mm	Density/($\text{kg}\cdot\text{m}^{-3}$)	Specific heat capacity/($\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$)
Al_2O_3 fiber mat	0.5	210	1.04
silica aerogel- Al_2O_3 fiber composite	0.8	180	0.53

Figure 6 shows the time dependence of temperature in sample II. Line (1) is the temperature of heating surface, Line (2) is the temperature of high-low temp boundary, and Line (3) is the temperature of cold boundary. It can be found that although the sample is contacted with high temperature heat source, the temperature of the low temp-section keep at room temperature 295 K, which demonstrates the excellent thermal barrier properties of the Sample II. And this experimental data is in accordance with the simulation result above.

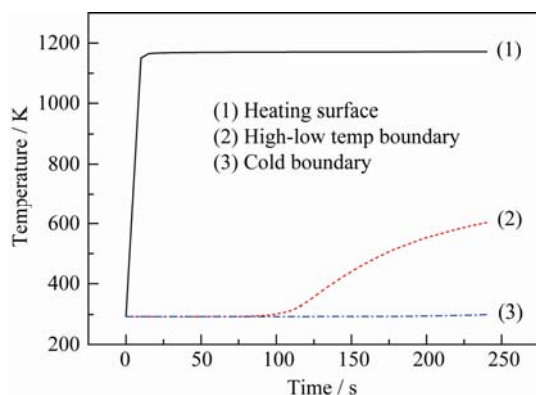


Fig. 6 The high-temp experiment results of new MLIMs

3 Conclusion

In this paper, a new kind of MLIMs was systemly reported, and the following conclusions can be drawn:

1) By Sol-Gel method, silica aerogels could be implanted on the surface of Al_2O_3 fiber and filled in the space of fibers.

2) Compared with the traditional MLIMs, the thermal conductivity (λ_e) and the weight of the new MLIMs are reduced by about 21.8% and 67.2%, respectively. Even in low vacuum (100 Pa), the new MLIMs still has great heat insulation performance.

3) According to the high-temp experiment and ANSYS simulation, the new MLIMs with 30 units shows good thermal barrier properties at high temperature. It is a potential candidate of thermal insulation material for space environment application.

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基于二氧化硅气凝胶制备的多层隔热材料微观结构及热性能研究

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摘 要: 成功制备了一种新型多层隔热材料, 其间隔层基于氧化铝纤维制备, 并由溶胶-凝胶法植入二氧化硅气凝胶颗粒。在高真空环境下(10^{-3} Pa), 新型多层隔热材料当量热导率比传统材料低 21.8%, 同时重量比传统材料降低了 67.2%。此外, 这种新型多层隔热材料在 1200 K 的高温以及低真空条件下(100 Pa)也表现出良好的隔热性能。

关 键 词: 多层隔热材料; 二氧化硅气凝胶; 热导率

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