Design and Validation of Thermal Insulation for Deep-sea Fluid Sampler of Jiaolong Human Occupied Vehicle

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Abstract. In this paper, a thermal insulation structure with silica aerogel felt as filler material was designed for the requirements of deep-sea fluid thermal insulation sampling technology for Jiaolong human occupied vehicle. Simulation analysis of thermal insulation performance was carried out and an experimental prototype was developed for the thermal insulation structure. Experimental study on thermal insulation performance was conducted with the variation characteristics of the operation environment for Jiaolong human occupied vehicle being taken into account. Results show that the silica aerogel felt with a thickness of 30 mm filled in the radial space between the inner and outer cylinders can achieve the expected thermal insulation effect during the diving-sampling-transferring process, with maximum temperature rise of 8.5 ℃, and can meet the requirements of deep-sea fluid thermal insulation sampling technology.

Keywords: Sampling; Thermal insulation; Silica aerogel felt; Simulation.

1. Introduction
Investigation on submarine cold-seep ecological communities has become a hot spot in geoscience and life science research since microbial communities were discovered in deep-sea extreme environment. As deep-sea cold-seep fluids contain rich information, collecting and analyzing their information on physical and chemical properties, and microbial composition, etc. is of significance for promoting the exploitation of deep-sea fluid microbial resources and deep-sea mineral resources [1–6].

To obtain fluid samples from deep-sea extreme environment, deep-sea samplers of many types have been developed in China and other countries. Deep diving equipment carries such a sampler and dives to a specified depth so that the sampler conducts sampling. However, at present, most samplers for deep-sea cold seeps have only realized pressure holding function, and the influences of temperature variation on the physical and chemical properties and microbial activities of sample during sampling and transferring have not been taken into account. Because of the activity and later cultivation value of the sampled organisms, the temperature under thermal insulation is generally controlled in the range of 2–8 ℃, with maximum of no more than 10 ℃. The passive thermal insulation mode of filling thermal insulation material is preferred [7–9].

In this paper, a high-fidelity sampler with sampling, withdrawing and transferring functions integrated together and with maximum operation water depth of 5000 m, which could realize thermal insulation, pressure holding, and sample dynamic characteristics monitoring, was developed for the requirements of deep-sea cold-seep sampling thermal insulation technology. It could be carried by Jiaolong human occupied vehicle and operated after the vehicle dived. The thermal insulation performance of the
sampler was studied and validated, and results show that the sampler can provide technical support for high-fidelity sampling of deep-sea biological and genetic resources, and for research efforts on biodiversity, and resource exploration, etc.

2. Analysis of Conditions for Deep-sea Fluid Sampling
Jiaolong human occupied vehicle is a 7000-m-level human occupied deep-diving submersible independently designed by China, with high flexibility and rich operation functions, and is an absolutely good platform for deep-sea fluid sampling, as shown in Figure 1.

![Figure 1. Jiaolong human occupied vehicle.](image)

The sampler designed in this paper was planned to be carried by Jiaolong human occupied vehicle. During installing-diving-sampling-withdrawing-transferring, the external operation environmental conditions changed constantly, and the temperature and operation time varied with the operation environmental conditions. Therefore, the heat exchange process between the sampler and the external environment needed to be studied according to the variations in external temperature and operation time. Figure 2 shows the curves of seawater temperature and salinity versus water depth in a sea area.

![Figure 2. Curves of seawater temperature and salinity versus water depth in a sea area.](image)

The operation processes of Jiaolong human occupied vehicle were as follows (the diving and floating-up speeds of the sampler with Jiaolong were taken as 40-50 m/min):
(1) The sampler was installed and fixed on the deck 30 minutes before diving;
(2) It took 30 minutes to arrange the deep-diving submersible on the sea surface;
(3) The deep-diving submersible started to dive half an hour after the sea surface state was confirmed;
(4) It took ~1.5 hours for the deep-diving submersible to dive to water depth of 4000–5000 m. And then, it operated under the sea for 6 hours. Afterwards, it took ~1.5 hours for the deep-diving
submersible to throw load and float up. Finally, it took 30 minutes to withdraw the sampler back to the
dock and it took 30 minutes to remove the sampler. It took ~10 hours in total for the whole process.

Based on the operation flow of all stages of diving of Jiaolong and the external temperature variation,
different operation states under varied external environmental conditions were delimited, as shown in
Table 1.

**Table 1. Different operation states under varied external environmental conditions.**

| Operation state                                      | Temperature (℃) | Duration (min) |
|------------------------------------------------------|----------------|----------------|
| Fix sampler on deck →                               | 50             | 60             |
| Arrange on sea surface                               |                |                |
| Confirm sea surface state →                          | 25             | 30 + 15        |
| 500 m → 1 000 m                                      | 10             | 15             |
| 1 000 m → 5 000 m                                   | 5              | 90             |
| Operate at 5 000 m                                  | 5              | 360            |
| 5 000 m → 1 000 m                                   | 5              | 90             |
| 1 000 m → 500 m                                     | 10             | 15             |
| 500 m → Sea surface                                 | 25             | 15             |
| Withdraw from sea surface →                          |                |                |
| → Transfer to cultivation kettle                     | 50             | 30 + 30 + 30   |
| Total time                                           |                | 780            |

Difference operation states were delimited according to the operation processes of the deep-diving
submersible and the external environmental temperature variation. The temperature variation was not
even under different operation states, so the maximum temperature value under each state was taken as
the temperature in corresponding time under this state.

3. Design of Thermal Insulation for Fluid Sampler

Due to long period and high thermal insulation performance requirement for deep-sea sampling, the
novel deep-sea cold-seep thermal insulation and pressure-holding sampler designed in this paper was
mainly composed of four parts: pressure-resistant structure, thermal insulation filler material, nitrogen-
filling pressure-holding device, and data acquisition system. The schematic diagram of assembly of
the sampler is shown in Figure 3.

The pressure-resistant structure mainly included inner and outer pressure-bearing cylinder bodies and
sample-entering needle valve body. The radial thickness of the inner and outer cylinder bodies was 30
mm, and the axial thicknesses between inner and outer cylinder bodies were 122 mm, and 100 mm,
respectively, on both sides [10–14]. The thermal insulation filler material was primarily silica aerogel
felt, which had such advantages as small density (146 kg m⁻³), low thermal conductivity (0.022 W m⁻¹
K⁻¹), and small specific heat capacity (502.42 J kg⁻¹ K⁻¹)) [15]. It was filled between inner and outer
cylinders, to reduce the heat transfer between the housing and the inner. In addition, to further improve
the thermal insulation effect, the surfaces of the inner and outer cylinders were coated with thermal
insulation layers.
4. Simulation Study on Thermal Insulation Performance of Fluid Sampler

To analyze the effectiveness of design of the thermal insulation device, simulation study on the thermal insulation performance of the sampler was carried out according to the external temperature variation under actual operation conditions.

4.1. Three Dimensional Model of Thermal Insulation Material Filling

A three dimensional (3D) model of thermal insulation material filled structure was established using 3D design software, as shown in Figure 4. The employed thermal insulation material was silica aerogel felt, and its 3D dimensions are shown in Figure 4.

4.2. Simulation Analysis and Results

The established 3D model of thermal insulation material was imported into the transient thermal analysis module of the Ansys Workbench software for simulation. The temperature variation sub-steps were set at different stages according to the actual external environmental temperature variation, as shown in Figure 5. Stage 1 was that the simulated sampler was at initial temperature of 2℃ in the low temperature chamber. Stage 2 was that the simulated sampler was transferred from the low temperature chamber to room temperature state. Stage 3 was that the simulated sampler was exposed to the air at 50℃ on the deck. Stages 4–10 were that the simulated sampler dived with Jiaolong, operated, and then floated up to the sea surface. Stage 11 was that the simulated sampler was
transferred from the sea surface to the deck. Stage 12 was that the simulated sampler was removed on the deck.

| Steps | Time [s] | Convection Coefficient [W/m²K] | Temperature [°C] |
|-------|----------|---------------------------------|-----------------|
| 1     | 0.1      | 20                              | 2.0             |
| 2     | 100      | 20                              | 10.0            |
| 3     | 200      | 50                              | 15.0            |
| 4     | 300      | 100                             | 25.0            |
| 5     | 400      | 500                             | 5.0             |
| 6     | 500      | 100                             | 10.0            |
| 7     | 600      | 500                             | 15.0            |
| 8     | 700      | 100                             | 25.0            |
| 9     | 800      | 500                             | 30.0            |
| 10    | 900      | 100                             | 40.0            |
| 11    | 1000     | 500                             | 50.0            |
| 12    | 1100     | 100                             | 55.0            |

**Figure 5.** External temperature variation sub-steps at different stages.

The temperature variation curve diagram shown in Figure 6 was obtained through simulation. The upper curve is the set external environmental temperature variation curve, and the lower one is the internal sample temperature variation curve under the influence of the external environment. After the heating was over, the internal sample temperature reached its maximum. The internal sample temperature distribution contour diagram (Figure 7) after the heating was over was obtained accordingly. The maximum temperature of sample was 7.4971 °C, meeting the design requirement that the maximum temperature rise shall not be more than 10 °C.

**Figure 6.** Sample temperature distribution contour diagram.

**Figure 7.** External temperature variation curve diagram.

5. **Experimental Study on Thermal Insulation for Sampler**

Based on the simulation study results, we further fabricated an experimental prototype of thermal insulation structure of sampler, of which the structural parameters were the same as the model in Figure 3. The pressure-resistant shell of the experimental prototype was made of TC4 titanium alloy, and silica aerogel felt (density 146 kg m⁻³, thermal conductivity 0.022 W m⁻¹ K⁻¹) was used as filler material for thermal insulation, as shown in Figure 8. The inner core of the prototype was equipped with a seawater container with a volume of 150 mL, in which a Pt100 thermal resistor temperature sensor was provided, to acquire the internal seawater temperature in a real time manner. Based on the operation flow of Jiaolong and the seawater temperature variation, the external temperature variation during the experiment was designed rationally, to carry out experimental study on thermal insulation.
5.1. Main Apparatus
The data from temperature sensor was acquired by an M-7018 series analog conversion module, and a B-TH-1000C-S high-low temperature tester was used for simulation of high and low temperature environments.

5.2. Experimental Study on Thermal Insulation under Constant Temperature Condition
Thermal insulation experiments were carried out at 40 °C 4 h, 45 °C 4 h and 50 °C 3 h, respectively, with 150 mL of seawater as sample. Before the heating experiment, the sampler was cooled down to the specified temperature, which was kept constant, to ensure that the temperature was the same in the whole sampler. During the heating experiment, the temperature variation data was extracted once per 5 min. Temperature variation curves were plotted based on the extracted data, as shown in Figures 9–11.

Figure 9. Seawater temperature variation curve at 40 °C, 4 h (rise of 4.44 °C), and variation curve of temperature difference between adjacent time points.

Figure 10. Seawater temperature variation curve at 45 °C, 4 h (rise of 7.01 °C), and variation curve of temperature difference between adjacent time points.
Figure 11. Seawater temperature variation curve at 50 °C, 3 h (rise of 4.92 °C), and variation curve of temperature difference between adjacent time points. It can be discerned from the three groups of temperature variation curves under different experimental conditions that, within 1 h in the early stage of constant temperature heating, the curve tended to flat, and the seawater temperature rise was very small, only ~0.5 °C. With the increase in heating time, the temperature variation curve went up continually and the curve slope increased constantly, indicating increased temperature rise. It can be seen from the three groups of variation curves of temperature difference between adjacent time points under different experimental conditions that, within 30 min in the early stage of heating, the variation in temperature difference between adjacent time points was very small, basically ~0.05 °C. After ~3 h of heating, the variation in temperature difference between adjacent time points tended to stable, ranging from 0.15 to 0.2 °C. From then on, external heat was constantly and rapidly transferred to the sample in the inner cylinder, enhancing the rise in sample temperature. Therefore, the operation time under relatively high external temperature condition should not be too long, and the time of exposure of the sampler to external environment at 50 °C should be decreased as far as possible.

The times for temperature to rise to different degrees were calculated further for three groups of data, as shown in Table 2.

Table 2. Times for temperature to rise to different degrees.

| Temperature | Temperature rise of 0.1 °C (min) | 5% of temperature growth (min) | 10% of temperature growth (min) | 50% of temperature growth (min) | Time for 50% of temperature growth/Total experimental time |
|-------------|---------------------------------|------------------------------|---------------------------------|---------------------------------|--------------------------------------------------------|
| 40 °C 4 h, seawater | 30 | 55 | 75 | 155 | 0.64 |
| 45 °C 4 h, seawater | 30 | 55 | 70 | 155 | 0.64 |
| 50 °C 3 h, seawater | 40 | 50 | 65 | 125 | 0.69 |

It can be seen from Table 2 that, in the first ~30 min, the temperature rise was 0.1 °C; therefore, it could be thought that the temperature did not change in this stage. When the heating reached ~50 min, the temperature reached 5% of total temperature rise in the experiment. When the heating reached ~70 min, the temperature reached 10% of total temperature rise in the experiment. When the heating reached ~155 min, the temperature reached 50% of total temperature rise in the experiment. The time for 50% of temperature growth accounted for 60%–70% of total time, so the other 50% of temperature rise consumed a shorter time during later temperature growth. Analysis showed that the main causes were as follows: (1) it took some time for the heated chamber to be heated to a specified temperature; (2) it took some time for the heat to be transferred to the liquid through the thermal insulation layer; (3) it took some time for the temperature to achieve even distribution in the liquid; (4) with the increase in heating time, the curve of variation in temperature difference between adjacent time points went up constantly till tending to stable.
The experiment of sample temperature rise in a certain time under constant external temperature showed that, the temperature rise was not evident in the early stage of heating. With the increase in heating time, the temperature difference between adjacent time points increased continually, and the sample temperature rose constantly. When the temperature difference between adjacent time points tended to stable, the sample temperature rose constantly at a nearly fixed temperature difference, till the heating was over, achieving the maximum temperature rise.

5.3. Experimental Study on Thermal Insulation Performance under Actual Operation Conditions
To fully understand the influence of external temperature variation on the temperature rise of sample in the inner cylinder under actual operation conditions, experimental study on thermal insulation was carried out with a high-low temperature tester used to simulate the external temperature variation under actual operation conditions.

The temperature rise or drop rate of the high-low temperature tester could be adjusted by setting the temperature slope; however, in the actual operation, the variation in temperature rise or drop rate in unit time was inaccurate. Through repeated no load experiments, the high-low temperature tester was set as shown in Table 3 for simulation of temperature variation under actual external environment.

Table 3. Settings of high-low temperature tester for simulation of temperature variation under actual external environment.

| Operation environment                                                                 | Temperature (℃) | Duration (min) |
|--------------------------------------------------------------------------------------|-----------------|----------------|
| Temperature of high-low temperature tester rose from 2 °C to deck temperature         | 2 → 50          | 10             |
| Sampler was installed on deck and deep-diving submersible was arranged on sea surface | 50              | 30+30          |
| Temperature of high-low temperature tester became the same as that of sea surface     | 50 → 25         | 30             |
| Deep-diving submersible dived from sea surface to underwater 1,000 m                 | 25 → 5          | 30             |
| Deep-diving submersible lowered from underwater 1,000 m to 5,000 m, operated at 5,000 m for 6 h, and rose from 5,000 m to underwater 1,000 m | 5               | 532            |
| Temperature of high-low temperature tester became the same as temperature at underwater 500 m | 5 → 10          | 5              |
| Deep-diving submersible rose from underwater 1,000 m to 500 m                       | 10              | 10             |
| Temperature of high-low temperature tester became the same as that of sea surface     | 10 → 25         | 6              |
| Deep-diving submersible rose from underwater 500 m to sea surface                    | 25              | 10             |
| Temperature of high-low temperature tester became the same as that of deck           | 25 → 50         | 8              |
| Sampler was transferred from sea surface to deck, removed on deck, and then transferred to laboratory | 50              | 30+30+30       |
| Total time consumed                                                                 | 792             |                |

The high-low temperature tester heating temperature was set according to the actual external environmental temperature variation, and the total time consumed was 13.2 h. Figure 12 shows the temperature variation curve of high-low temperature tester under ideal state. Figure 12 shows the actual temperature variation curve of high-low temperature tester.

Comparing Figures 12 with 13, temperature fluctuation occurred in the heated chamber during temperature change, but the temperature variation basically agreed with the set variation state.
Study on thermal insulation performance of thermal insulation material was conducted under the above set external temperature conditions, and three groups of experiments were carried out respectively. The experiment data was acquired once per 5 min, and the sample temperature variation curves are shown in Figures 14–16.

It can be discerned from the three groups of experimental data that, in the initial stage of heating, the sample temperature rose slowly, fundamentally maintained at ~2 °C, and the temperature difference between adjacent temperature points was basically 0. As the sampler was exposed to external operation environment of 50 °C for a long time, the sample temperature rose evidently, and the
The temperature difference between adjacent temperature points started to increase. When the sampler was arranged on the sea surface and dived with the deep-diving submersible, the sample temperature and the temperature difference between adjacent temperature points remained rising. As the deep-diving submersible dived, the surrounding seawater temperature dropped constantly, the temperature difference between adjacent temperature points started to decrease, and the sample temperature rise started to slow down, with the “second highest peak 1” reached. As the deep-diving submersible dived to water depth of more than 3,500 m and conducted underground operation, the sample temperature dropped constantly and the temperature difference between adjacent temperature points became negative value and tended to stable. During the rising of the deep-diving submersible after completing the operation, the sample temperature remained dropping, with the “second lowest valley 2” reached. At this moment, the temperature difference between adjacent temperature points started to increase. In the process that the deep-diving submersible rose to the sea surface and was withdrawn to the deck and the sampler was removed from the deep-diving submersible and transferred to a cultivation kettle, the sample temperature remained rising constantly. After the sampler was transferred to the cultivation kettle, the temperature difference between adjacent temperature points started to decrease. After the sampler was transferred to the cultivation kettle at constant temperature of 2 °C, the temperature remained rising for a time period, with “the highest peak 3” reached. In the three groups of experimental data, the values of the “second highest peak 1” of sample temperature were 8.19 °C, 8 °C, and 7.65 °C, respectively, those of the “second lowest valley 2” were 5.84 °C, 5.59 °C, and 5.52 °C, respectively, and those of the “highest peak 3” were 8.4 °C, 8.2 °C, and 8.5 °C, respectively. All of these values did not exceed the design indicator required maximum temperature rise of 10 °C.

It can be discerned from the above experiments that, in the experiment of thermal insulation (temperature variation) under the simulated actual operation conditions, the total time consumed was 13.2 h, and the maximum temperature variation was 8.5 °C. The temperature variations basically met the requirements that the mean temperature variation shall be within 8 °C and the maximum temperature rise shall not be more than 10 °C.

6. Comparative Analysis between Simulation and Experiment of Thermal Insulation Performance

Comparative analysis was further conducted between the experimental results and the simulation results in this section. Figure 17 shows the temperature variation curves of the sample under simulation and experimental conditions.

![Temperature variation curve under external temperature change conditions (rise of 7.8 °C)](image)

**Figure 17.** Sample temperature variation curves. (a) Temperature variation curve under external temperature change conditions (rise of 7.8 °C). (b) Simulation result curve.

It can be seen from the sample temperature variation curve obtained from experiment (Figure 17a) that, with the change in external temperature, the sample temperature rose slowly first. With the rise in external heating temperature, the slope of the sample temperature variation curve increased constantly, and the sample temperature rise was sped up. When the external heating temperature dropped gradually and was maintained at 5 °C, the slope of the sample temperature variation curve became negative value, and the sample temperature started to continually drop. With the external heating
temperature rising to the sea surface temperature and the deck temperature, the sample temperature decreased to a certain extent and then started to rise, till the heating time was over. At that moment, the sample temperature was 8.4 °C.

As can be seen from the sample temperature variation curve obtained from simulation (Figure 17b), the upper curve shows the temperature variation values at different time periods set according to the external operation environment, that is, a sub-step of simulation analysis. The time points on the abscissa axis were delimited on average based on the total time consumed for the sub-steps in all stages, and this was different from the sample temperature variation curve obtained from experiment. It can be obtained from the lower curve in the figure that, in the initial stage of heating, the sample temperature rise trend was not evident. In the time period 4 000–10 000 s, as the analysis sub-step (the upper curve) conducted, the external heating temperature rose and the sample temperature rise was sped up. In the time period 10 000–35 000 s, the heating temperature in the sub-step dropped gradually, the sample temperature rise rate started to slow down, and the highest temperature value since the start of heating was reached. In the time period 35 000–40 000 s, the sample temperature droppe from the interval-based maximum to the interval-based minimum. In the time period 40 000–47 460 s, the heating temperature in the sub-step rose gradually, corresponding to the external environmental temperature variation during sampler rising and withdrawal, and the sample temperature rose from the interval-based minimum to the whole heating process-based maximum, that is, the temperature 7.4971 °C shown in the sample temperature distribution contour diagram.

As can be discerned from comparison between the experimental and simulation results, both sample temperature variation curves have similar change trends. In the initial stage, the sample temperature rose slowly. With the rise in external heating temperature, the sample temperature rise was sped up and the sample temperature reached the interval-based maximum (that is, the “second highest peak” in the sample temperature curve from experiment). With the drop in external heating temperature the internal sample temperature started to drop, reaching the interval-based minimum. In the late stage, when the sampler rose and was withdrawn together with the deep-diving submersible, the external heating temperature rose constantly, causing rise in sample temperature, from the interval-based minimum (that is, the “second lowest valley” in the sample temperature curve from experiment) to the whole heating process-based maximum. The difference was that the three maximum temperatures from experiment during heating were 8.4 °C, 8.2 °C, and 8.5 °C, respectively, and the maximum temperature from simulation was 7.4971 °C. The main causes for the difference are as follows: (1) The experimental material filling mode is greatly influenced by human factors, and the material filling evenness is poorer in the experiment than in the insulation; (2) The temperature changing rate of the high-low temperature tester is not stable. When the external temperature changes, there should be response time reserved sufficiently, causing different external heating temperature settings between experiment and simulation; (3) The simulation model is a simplified model, with some difference from the real structure. Comparison between the experimental and simulation results shows that simulation can reflect the experimental results, and can be used as an analysis method for study on thermal insulation performance of material to validate the feasibility of thermal insulation performance of one material or combination of multiple materials through simulation analysis.

7. Conclusions
In this paper, the thermal insulation performance of thermal insulation filler material for sampler was studied based on the requirement of deep-sea fluid thermal-insulation and pressure-holding sampling technology for Jiaolong human occupied vehicle, and the following conclusions were made:
(1) Silica aerogel felt was selected as the main filler material, and was filled between the inner and outer cylinders (thickness 30 mm). Different external temperature variation experiments showed that the material could meet the requirements that the thermal insulation temperature shall be controlled in the range of 2–8 °C with maximum of no more than 10 °C. The feasibility that silica aerogel felt was used as thermal insulation material for cold-seep fluid sampler was validated.
(2) The heating experiment was carried out using the high and low temperature testing machine to simulate the influence of the external temperature on the temperature rise of the inner cylinder sample, and the maximum temperature rise of the heating experiment was 8.5°C, which verified the reliability of the insulation performance of the silica material.

(3) Using ANSYS Workbench software to simulate the sampling process, the highest temperature rise obtained is 7.5°C, which can better simulate the experimental results. Combined with the heating experiment of the high and low temperature testing machine, it can be used as an analysis and verification method for the research of material insulation performance.

Acknowledgments

This work was supported by the NSFC-Shandong Joint Fund for Marine Science Research Centers (Grant No. U1606401), the Public Service Platform Project in Collaboratively Industrial Chain Innovation of Qingdao Marine Economy Innovation and Development Demonstration City (Test and Inspection Service Platform for Deep-sea Technology and Equipment), and the Taishan Scholar Project Funding (Grant No. tspd20161007). The authors would also like to express gratitude to the editors and the anonymous reviewers for their constructive comments and suggestions.

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