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The Fabrication and Characterization of a Honeycomb Ceramic Electric Heater with a Conductive Coating

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***Abstract*—**Porous electric heaters, compared to conventional electric heaters, exhibit excellent heating performance due to their large specific surface area. Porous electric heaters employ porous metallic materials or conductive porous ceramics as the heating element. The former attains a low heating power with a fixed current due to the low electrical resistivity of metal. Although the latter can bypass the inherent challenges of porous metallic materials, the fabrication process of the conductive porous ceramics is complicated and high cost. This work proposed a porous ceramic electric heater with dielectric honeycomb ceramic as a substrate and surface conductive coating as a heating element. The conductive coating was prepared by the sol-gel method using silica sol and methyl trimethoxysilane as raw materials and graphite powder as conductive fillers. The conductive mechanism and degradation reason of the conductive coating was studied by electrical resistivity and thermal stability analysis. The heating performance of the proposed heater was experimentally investigated by heating air and deionized water. The results indicate that the electron transfer is achieved by forming the conductive network through the contact of the graphite flakes. With 30 wt% of graphite, the electrical resistivity of the conductive coating can be as low as 0.88 Ω·cm. The conductive coating exhibits good electrical stability up to 500 °C but degrades beyond 600 °C due to the formation of many cracks in the coating caused by the weight loss and thermal expansion. The results also show that the working medium has a great influence on the volume power density of the heater. With air under natural convection as the working medium, the volume power density attains 640.85 kW/m^3, which can be increased by 5 times when using deionized water as the working medium. The proposed honeycomb ceramic electric heater has the advantages of the simple fabrication method, low cost, and high-volume power density, demonstrating great potential in the fluid heating field.

***Keywords*—**Conductive coating, honeycomb ceramic electric heater, high specific surface area, high volume power density.

I. INTRODUCTION

ELECTRIC heaters, which convert electrical energy into heat, have been used for fluid heating in petrochemical, pharmaceutical, air conditioning, power systems, and other industrial fields [1]-[4]. Conventional electric heaters usually generate heat with coil resistance wires surrounded with magnesium oxide powder which are placed inside heating tube elements with protective sheath [5]. Conventional electric heaters have the advantages of low cost, easy manufacture, and adaptability for different shapes. However, they have high thermal resistance and thus low volume power density due to the small specific surface area.

It has been demonstrated that applying porous materials can improve the heating performance of the electric heater through the large specific surface [6]-[10]. Inserting porous materials into the heating tube element of conventional electric heaters and using porous materials as the heating element are the two main ways for the application of porous materials. Amori and Laibi [11] theoretically and experimentally studied the effect of inserting porous aluminum foam into an air heater pipe. Their results showed that convection heat transfer could be enhanced by porous metallic foam due to its high conductivity and specific surface area and further enhancement can be achieved by increasing the thickness of the porous aluminum foam. Wang et al. [12] obtained a similar result by experimentally investigating the heat transfer characteristics of air flowing through an electric air heating furnace with SiC ceramic foam inserted. Naji and Al-Nimr [13] theoretically analyzed the heating performance of a porous electric heater. They replaced the conventional heater with a porous heater and found that the porous heater achieves lower temperature under the same electric heating power, indicating that the porous heater can conduct higher heating power without the concern of material burnout. Cookson et al. [14] presented using porous Fe-Cr-Al metal foam disk as the heating element in an air heater. The experimental results revealed that the heating power reaches 6.32 W, which corresponds to a volume power density of 240.54 kW/m^3 with an electrical current of 50 A. Zahmatkesh and Yaghoubi [15] investigated the electric heaters with porous material inserted and that with porous material as the heating element. The heating performances of these two heaters were compared to that of the conventional heater. The results indicated that an electric heater with a porous heating element attains the lowest temperature and pressure drop at fixed heating power, airflow rate, and heater mass. Furthermore, employing porous material as the heating element simplifies the heat transfer process, resulting in smaller thermal resistance and improved temperature uniformity. By now most reported porous heating elements are made of porous metallic foam, which has low electrical resistance due to the low resistivity of metal. As a result, there is a high electrical current flowing through the heater with a fixed applied voltage, causing high loss from the lead wires. Besides porous metallic materials, porous ceramics can also be used as heating elements due to their high electrical resistivity and thermal shock resistance [16]-[18]. Fan et al. [18] fabricated a porous SiC ceramic with an electrical resistivity of 0.049 Ω·cm and experimentally evaluated its electrical heating performance by heating water. The results revealed that the heating power reaches 12.3 kW with applied 40 V voltage. Although the porous ceramic heater

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exhibits good electrical performance, the fabrication process is complicated and high cost.

This work proposes a porous ceramic electric heater that employs dielectric honeycomb ceramic as the substrate and surface conductive coating as a heating element. The fabrication process of the conductive coating, as well as the heater, is presented. The electrical property and thermal stability of the conductive coating are characterized. The heating performance of the proposed electric heater is experimentally investigated in a custom-made set-up by heating air and deionized water.

II. MATERIALS AND METHODS

A. Materials

Silica sol (acidic, 30 wt%) was purchased from Qingdao Jiyida Silica Gel Reagent Ltd. Methyl trimethoxysilane (MTMS, 99 wt%) was purchased from Xuzhou Yihuiyang New Material Ltd. Ethanol absolute (99.9 wt%) was purchased from Ziyansheng (Shanghai) Fine Chemistry Ltd. Graphite powder (98 wt%) was purchased from Jinan Lixia Yaxuan Chemical Products Operation Department. The dielectric honeycomb ceramic made of cordierite was purchased from Shenlin Honeycomb Activated Carbon Plant. As shown in Fig. 1 (a), the honeycomb ceramic has polygonal columnar pores that form a two-dimensional array, making a porosity of 60%. The diameter and height of the honeycomb ceramic are 95 mm and 40 mm, respectively. The side length and wall thickness of the pores are 3 mm and 0.7 mm, respectively. The glass slides (25.4 mm × 6.2 mm) and ceramic sheets (10 mm × 10 mm) were also used as the substrates of the coating.

B. Fabrication of the Honeycomb Ceramic Electric Heater

The fabrication of the honeycomb ceramic electric heater contains conductive paint preparation, coating, and heater assembly. The conductive paint was prepared with the sol-gel method, which is simple, low-cost with low curing temperature [19]-[22]. The detailed procedures are as follows. First, 56 g MTMS was added into 100 g silica sol at 25 °C for 5 h stirring at a speed of 200 rpm. Then, 26 g ethanol absolute was added for obtaining the modified silica sol after 30 mins stirring. Finally, a certain amount of graphite powder was added into the modified silica sol for obtaining the conductive paint after 2 h stirring at 600 rpm.

The conductive paint can be applied to the ceramic or glass substrate through different techniques, such as dip-coating and spin-coating, which are the two most used coating methods. In this work, the dip-coating method was used since it is simple, low cost, reliable, and applied to the substrate with any shape. Before applying the conductive paint, the honeycomb ceramic substrate was washed with ethanol and dried in a thermostatic oven (Taicang Jinghong Instrument Equipment Ltd, DHG-9076A). Then the honeycomb ceramic substrate was immersed in the conductive paint for 3 s and withdrawn from the paint at a constant speed. The remaining paint on the substrate was removed by the gravitational force and thermal treatment in a thermostatic oven by the following procedures. First, it was heated to 80 °C for 10 mins. Then, it was increased to 220 °C for 15 mins. Finally, it was cooled down to room temperature to obtain honeycomb ceramic with a conductive coating on the surface, as shown in Fig. 1 (b). The same method was used to prepare conductive coatings on the glass substrate and ceramic sheet for further analysis.

Two brass sheets with the same area and pore distribution as the honeycomb ceramic were machined as the electrodes and mounted on the two ends of the honeycomb ceramic with four ceramic screws, as shown in Fig. 1 (c). The four screw holes are distributed in a circle every 90° with the axis of the ceramics as the center. The thickness of each electrode is 2 mm. The electrode should cover the entire end face of the honeycomb ceramic to form an equipotential surface so that the current can be distributed uniformly with the honeycomb structure.

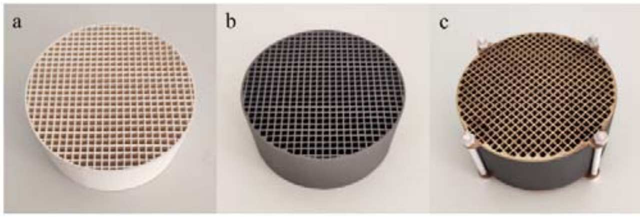

Fig. 1 Honeycomb ceramic electric heater: (a) Dielectric honeycomb ceramic substrate, (b) Honeycomb ceramic substrate with a conductive coating, (c) Assembly honeycomb ceramic electric heater

C. Characterization Methods

The surface morphology and cross-section morphology of the conductive coating was observed by scanning electron microscope (SEM, GeminiSEM 500). The electrical resistivity of the conductive coating was tested by a Four-probe tester (Suzhou Jingge Electronics Ltd, M-3). In addition, the conductive coating was scratched off from the glass substrate and grounded into powder, and a thermal gravimetric (TG) analyzer (TA Instruments, SDT Q600) was used to conduct the TG analysis of the conductive coating as well as the graphite powder and modified silica sol. The heating performance of the honeycomb ceramic electric heater was characterized by heating air and deionized water. The temperature distribution on the surface of the heater was measured by a thermal infrared imager (FLUKE, TiX660).

III. RESULTS AND DISCUSSION

A. Electrical Property of the Conductive Coating

To analyze the effect of the content of the conductive fillers on the electrical resistivity of the conductive coating, several coating samples with varying contents of graphite powder were prepared on the glass substrate, and their electrical resistivities were tested. As shown in Fig. 2, the electrical resistivity of the coating decreases with the increase of the content of graphite powder. The coating is not conductive with 10 wt% graphite powder. When the graphite powder increases to 15 wt%, the electrical resistivity of the coating is 20.48 Ω·cm. As the graphite powder further increases to 25 wt% and 30 wt%, the electrical resistivity of the coating decreases to 1.99 Ω·cm and

0.88 Ω·cm, respectively. However, the electrical resistivity barely changes with the further increase of the graphite powder content. To understand the conduction mechanism of the coating, the surface morphology and cross-section morphology of the conductive coating on ceramic sheets were observed by SEM (see insets in Fig. 2). It shows that the coating has good contact with the ceramic substrate at the interface. The graphite is distributed in the modified silica sol with flaky morphology, and the contact between graphite flakes is formed as a graphite conductive network for the electron transfer. The content of the conductive fillers has a percolation threshold above which the contact between the graphite flakes can be formed for the electron transfer. As the content of the conductive fillers increases, the contact between graphite flakes becomes closer for improved electrical conductivity. However, the continuous increase of the content of the conductive fillers will not have a continuously improved effect on the electrical resistivity when the graphite conductive network is widely and completely formed in the coating. Therefore, the conductive coating with 30 wt% graphite powder is used for the following analysis. It is also observed that a lot of pores and cracks are formed in the coating, which could be caused by the volatilization of the organic compounds and the thermal expansion during the thermal treatment.

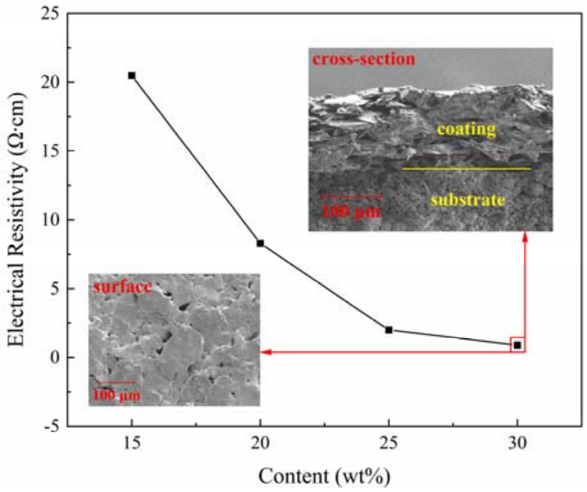

Fig. 2 The effect of the content of the graphite powder on the electrical resistivity of the conductive coating. Insets are the SEM images of the surface and cross-section of the conductive coating on a ceramic substrate with 30 wt% graphite powder

B. Thermal Stability of the Conductive Coating

To determine the operating temperature limit of the honeycomb ceramic electric heater, the thermal stability of the conductive coating needs to be analyzed. Three conductive coating samples on the glass substrate (A, B, and C) were heated at different temperatures in a furnace for 1 h. After each heating, the samples were cooled down to room temperature and the electrical resistivities were tested. As shown in Fig. 3 (a), the three conductive coating samples follow the same trend. The electrical resistivity of the conducting coating increases slightly after being heated at temperatures below 500 °C for 1 h but increases rapidly after being heated at 600 °C for 1 h, indicating that the conductive coating has good electrical stability up to 500 °C but degrades beyond 600 °C. To study the reason for the degradation of the conductive coating, the coating samples after being heated at various temperatures were observed by SEM. It is found that heating causes the formation of cracks in the coating, and more cracks are formed with the increase of the heating temperature. As discussed before, the electron transfer is achieved by forming a conductive network through the contact of the graphite flakes. The formation of cracks in the coating reduces the contact between graphite flakes, thus increasing the electrical resistivity of the coating. Figs. 2 (b) and (c) are the SEM images of the conductive coating samples after being heated for 1 h at 500 °C and 600 °C, respectively. Much more cracks are observed in the conductive coating after being heated at 600 °C for 1 h than that after being heated at 500 °C for 1 h, which results in a significant increase in the electrical resistivity and degradation of the coating.

To further investigate the physical mechanism of the electrical resistivity evolution, the conductive coating was scraped off from the glass substrate and grounded into powder for the TG analysis, as also shown in Fig. 3 (a) (black line). For comparison, the TG analysis was also performed for graphite powder (green line) and modified silica sol (blue line). According to the TG curve, weight loss occurs in the conductive coating during the heating process. The weight loss rate is approximately 3% at 500 °C but rapidly increases to 15% at 800 °C. The weight loss rate in silica sol is less than 2% at 500 °C and reaches a maximum of 6% at 600 °C due to the volatilization of organic compounds. For graphite powder, the weight loss rate is also less than 2% at 500 °C but increases to 15% at 800 °C due to the graphite oxidation. Therefore, the weight loss of the conductive coating is mainly caused by the volatilization of organic compounds below 500 °C while by the oxidation of graphite powder beyond 600 °C. Since the weight loss is negligible below 500 °C, the slight increase of the electrical resistivity is mainly attributed to the formation of a few cracks caused by thermal expansion during the heating process. When the temperature exceeds 600 °C, the high weight loss causes the formation of a lot of cracks in the coating, resulting in the rapid increase of the electrical resistivity and thereby the degradation of the coating. Based on the above discussions, the conductive coating demonstrates good electrical stability up to 500 °C, indicating the operating temperature limit of the honeycomb ceramic electric heater of 500 °C.

C. Arrangement Mode of the Lead Wire

To assemble the honeycomb ceramic electric heater, two lead wires are needed for providing an electrical connection between the heater and the power through the electrical pad at the end of the wires. One of the lead wires is connected to the top electrode on the screw hole while the other one is connected to the bottom electrode on the screw hole with the radial angle of 0°, 90°, or 180°, as illustrated in Figs. 4 (a1), (b1) and (c1), respectively. To study the effect of the arrangement mode of the lead wire, the assembly heater was connected to a DC power and tested by heating air with applied 10 V voltage. The voltage and current were recorded for obtaining the resistance. The results, as shown in Table I, reveal that the electrical resistances of the heater with three lead wire arrangement modes are 2.44 Ω, 2.40 Ω, and 2.46 Ω, respectively, demonstrating that the arrangement mode of the lead wire has little effect on the resistance of the heater. Fluke thermal imager was used to observe the temperature distribution on the surface of the heater at a steady state with varying lead wire arrangement modes, as shown in Figs. 4 (a2), (b2), and (c2), respectively. It is obvious that the lead wire arrangement mode has little effect on the surface temperature distribution. When the heater reaches a steady state, the region near the ceramic screws attains a higher temperature than the central region. As the heater is connected to the circuit, the electrical current flows into the top electrode from one lead wire, then flows into the bottom electrode through the conductive coating on the inner and outer walls of the honeycomb ceramic, and finally flows out from the other lead wire. Note that the measured resistance of the assembly heater contains the resistance of the honeycomb ceramic and electrical contact resistance at the electrode/ceramic interface, both of which can be equivalent to a parallel connection of multiple resistances. Theoretically, changing the lead wire arrangement mode does not affect the electrical contact resistance. However, due to the experimental error, electrical contact resistance may vary for different lead wire arrangement modes, resulting in a certain discrepancy in several resistance measurements. In addition, the contact between the electrode and honeycomb ceramic is closer in the region near the ceramic screws than in the central region, which results in reduced electrical contact resistance and increased heating power, thus leading to a higher temperature. In the following discussion, the two lead wires are connected to the heater with a radial angle of 180°.

TABLE I
THE MEASURED VOLTAGE, CURRENT, AND RESISTANCE OF THE HONEYCOMB CERAMIC ELECTRIC HEATER WITH VARYING LEAD WIRE ARRANGEMENT MODES

The radial angle of the two lead wires	0°	90°	180°
Voltage/V	10.0	10.1	10.1
Current/A	4.1	4.2	4.1
Resistance/Ω	2.44	2.40	2.46

D.Heating Performance of the Honeycomb Ceramic Electric Heater

According to the thermal stability test of the conductive coating, the honeycomb ceramic electric heater is supposed to operate below 500 °C. The heating performance of the heater was investigated by heating air in natural convection. This air heating process was divided into three stages with applied voltages of 20 V, 24 V, and 28 V, respectively. For each stage, the heater was kept heated until it reaches a steady state. The electrical voltage and current were recorded every 3 mins. The K-type thermocouple was inserted into the pore of the heater near the screw hole at a depth of 2 cm for measuring the temperature of the honeycomb ceramic electric heater. Fig. 5 (a) depicts the temperature and electrical resistance of the heater during the heating process. The electrical resistance of the heater increases with the temperature, which is due to the formation of cracks in the conductive coating mainly caused by the thermal expansion as discussed before.

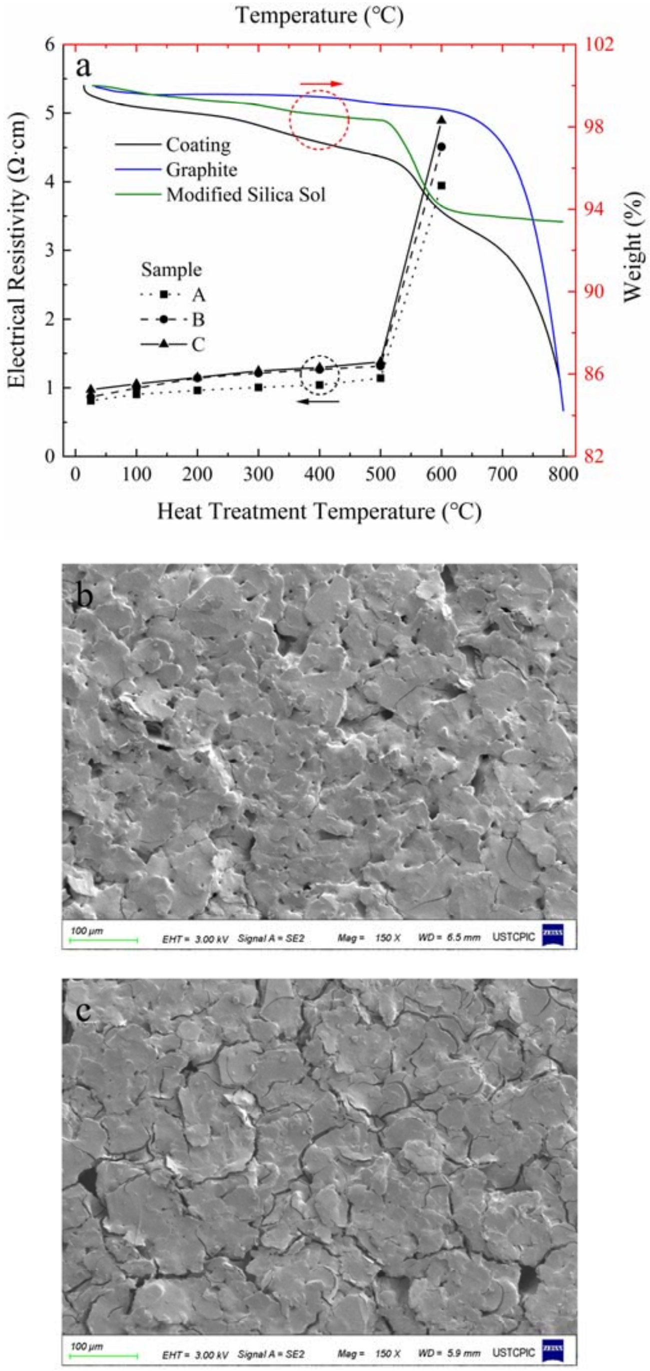

Fig. 3 Thermal stability of the conductive coating: (a) The electrical resistivity of the conductive coating after being heated at varying temperatures as well as the TG analysis curves. SEM images of the conductive coating samples after being heated at (b) 500 °C and (c) 600 °C for 1 h

At the first stage, the heater attains 200 °C and a resistance of 3.48 Ω at a steady state, conducting a heating power of 115 W, corresponding to the volume power density of 404.93

kW/m^3. At the second stage, the temperature and the resistance of the heater increase to 330 °C and 3.90 Ω, attaining a heating power of 148 W at a steady state and a volume power density of 521.13 kW/m^3. At the third stage, the heater reaches the operating temperature limit of 500 °C at a steady state. Meanwhile, the resistance of the heater increases to 4.30 Ω, which is 34% higher than the initial value of 3.22 Ω. The maximum heating power of the heater is 182 W, which corresponds to a volume power density of 640.85 kW/m^3.

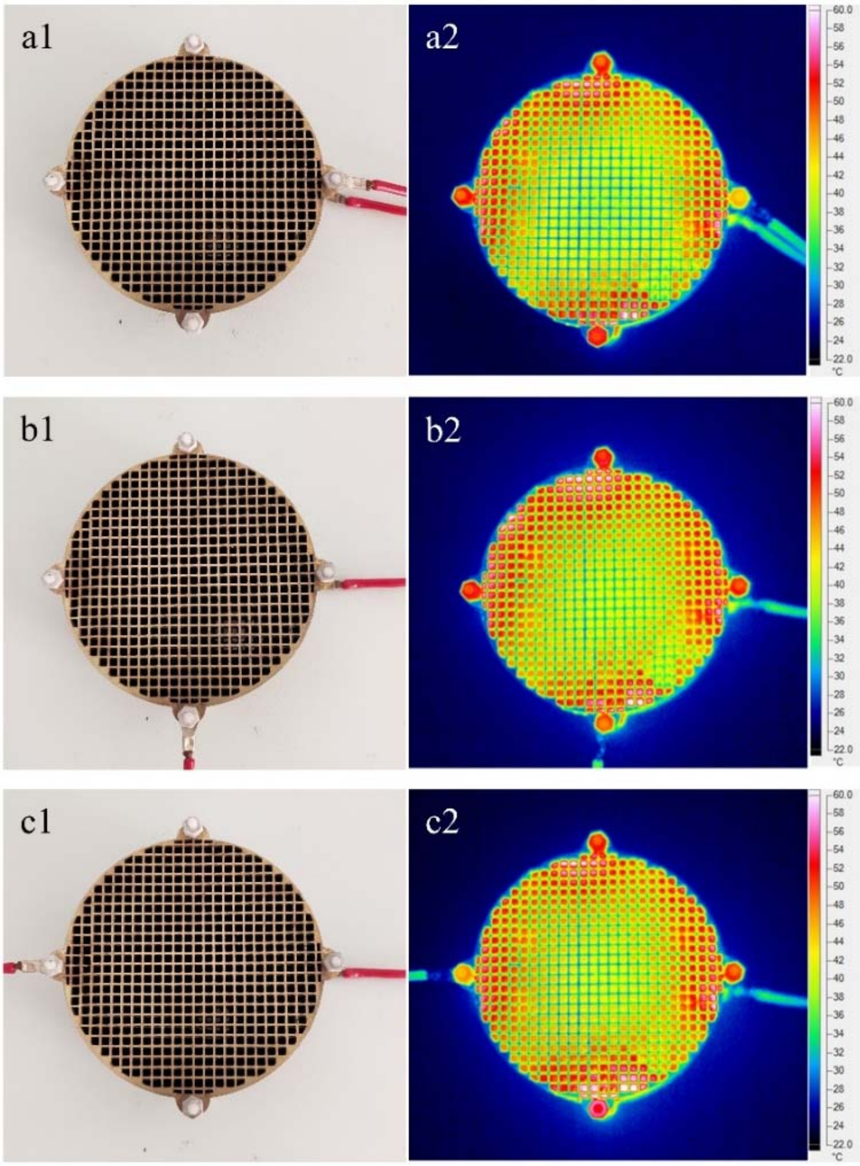

Fig. 4 The lead wire arrangement mode and surface temperature distribution of the heater. Two lead wires with the radial angle of (a) 0°, (b) 90°, (c) 180°

Although the conductive coating has been demonstrated to exhibit good electrical stability up to 500 °C, it remains a concern whether it maintains electrical stability in the long term. Therefore, a thermal cycle test was conducted for this honeycomb ceramic electric heater. The heater went through 5 heating/cooling cycles and each cycle consists of a heating time of 30 mins with an applied voltage of 28 V followed by a cooling down process in the air. The maximum temperature of the heater was around 500 °C during the thermal cycle test. The voltage and current were recorded every 3 mins. Fig. 5 (b) displays the electrical resistance revolution with the heating time. It is observed that the electrical resistance increases the most with time during the first thermal cycle and then changes slightly with time afterward. Also, the electrical resistance of the heater at steady state increases with the thermal cycle times and barely changes after 3 heating/cooling cycles. This is because the influence of the cracks formed in the coating concentrates in the first thermal cycle and weakens in the subsequent thermal cycles. The results demonstrate the good long-term electrical stability of the heater.

To study the effect of the working medium on the heating performance of the honeycomb ceramic electric heater, the heater was tested by heating deionized water. The reason we used deionized water is for safety concerns since there is an electrical current flowing through the heater surface during the heating process. The experiment was carried out in a wooden box containing 5 L of deionized water with the honeycomb ceramic heater completely immersed in it. The temperature of the heater was measured via a K-type thermocouple inserted into the pore of the honeycomb ceramic heater near the screw hole at a depth of 2 cm. The temperature of the deionized water was measured via another K-type thermocouple immersed in the deionized water. The initial temperature of the deionized water and the environment were both 6 °C. The applied voltage was fixed at 60 V, and the current was recorded every 1 min. Fig. 5 (c) shows the electrical resistance and volume power density of the heater with time. The electrical resistance of the heater changes within 4% during the heating process, indicating that the heater has reached a steady state. The heating power of the heater at steady state is 1107 W, corresponding to a volume power density of 3897.89 kW/m^3, which is 16 times higher than the previously reported result [12]. The experimental results display that the volume power density of the heater can be enhanced by 5 times by changing the working medium from air to deionized water with a larger convection heater transfer coefficient, demonstrating a significant effect of the working medium on the volume power density. In addition, considering that the heater reaches 70 °C at a steady state, which is far below the operating temperature limit, we thus believe that there is still room for improvement of the volume power density of this honeycomb ceramic electric heater. In general, the proposed honeycomb ceramic electric heater is more applicable to the field with strong heat dissipation capacity in practical use.

IV. Conclusions

In summary, we proposed and fabricated a porous ceramic electric heater with dielectric honeycomb ceramic as the substrate and surface conducting coating as the heating element. The surface conductive coating was prepared with the sol-gel method. The electrical property and thermal stability of the conductive coating were analyzed, and the results demonstrated the electrical stability of the conductive coating up to 500 °C, which should be the operating temperature limit of the proposed porous ceramic heater. The conductive coating degrades after 600 °C, which is because a lot of cracks formed in the coating due to the graphite oxidation and thermal expansion destroying the graphite conductive network, significantly reducing the electrical resistivity. The heating performance of the heater was experimentally investigated by heating air and deionized water. It is found that the heater exhibits long-term electrical stability. The volume power density of the heater is tested to be 640.85 kW/m^3 using air as the working medium, which can be enhanced by 5 times when using deionized water as the working medium. The proposed porous ceramic electric heater has a great potential in the field of fluid heating with high volume power density.

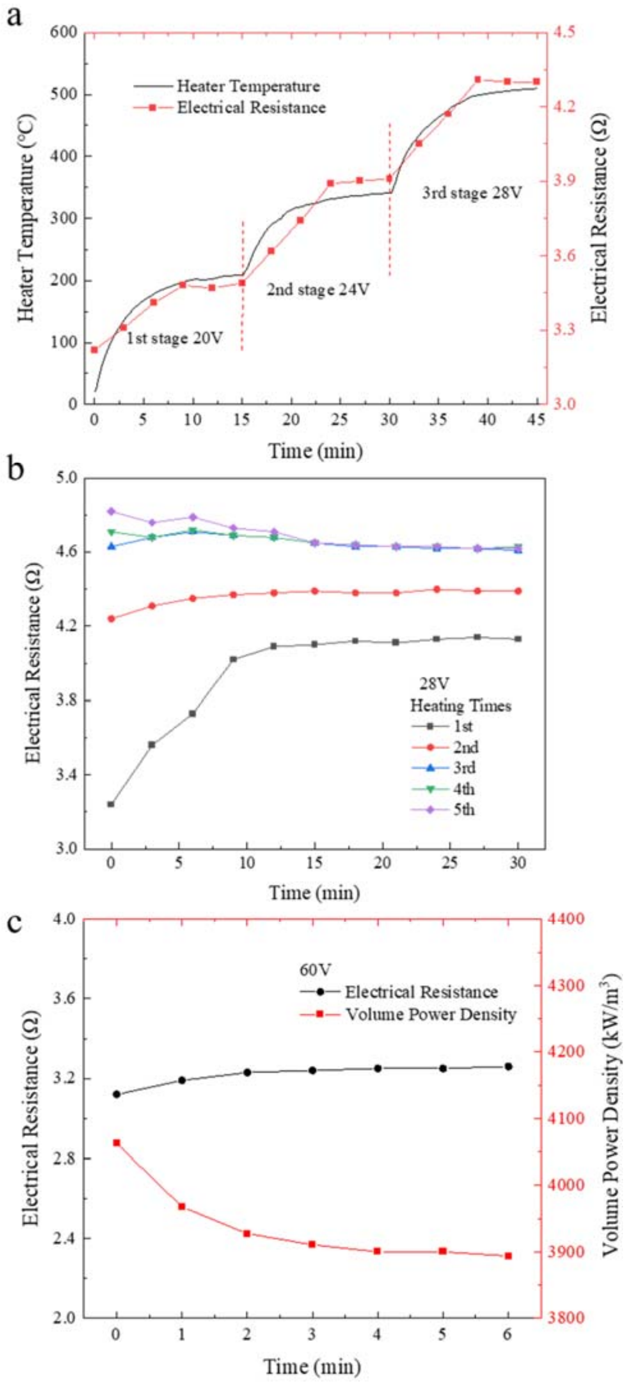

Fig. 5 The heating performance of the honeycomb ceramic electric heater:(a) The temperature and electrical resistance of the heater using air as the working medium, (b) The electrical resistance with time for the thermal cycle tests, (c) The electrical resistance and volume power density of the heater using deionized water as the working medium

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