Enhanced evaporation of porous materials with micropores and high porosity

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Abstract
Owing to their large surface area and capillary effect, porous materials are highly desired for heat-transfer applications. In this study, we investigated the evaporation behavior of a water droplet on porous epoxy resin with a three-dimensional network structure. As the material existed in the liquid state before sintering, it could be easily converted into a porous body on a heating surface. The material showed a pore size of 0.15 \( \mu \text{m} \) and a porosity of 50%. First, the evaporation behavior of a water droplet placed on the surface of this porous body was investigated. It was found that the evaporation rate increased when the evaporation was carried out on the surface of the porous body. Then, a vapor chamber experiment was carried out with the porous material. The porous body acted as the generating surface and its heat transfer performance was estimated. The cooling performance of the porous body was 1.2 times higher than that of the non-porous material.

Keywords: A three-dimensional network structure, Evaporation rate, A vapor chamber, A pore size, A porosity

1. Introduction
A porous material constitutes a large number of pores and exhibits unique reactions, transportation phenomena, large surface area, and capillary effect. Porous materials are used for various engineering applications (Yuki, 2015). Nanostructured surfaces exhibit peculiar evaporation characteristics. For example, Hara and Suzuki investigated the evaporation of a water droplet at the surface of nanoporous pillars and found that the evaporation occurred via forced convection (Hara and Suzuki, 1998) (Hara, 2000). The surface pattern of unglazed pottery with nano polars was observed using an inter-atomic force microscope (Satake, 2001). It was found that the nano-micro structure of the porous surface showed evaporation characteristics. Porous materials are classified on the basis of their void percentage and pore diameters. Various porous materials that have been extensively investigated are sintered particles (Yuki et al., 2010), fiber-sintered porous materials (Togashi et al., 2005), and bubble-sintered porous materials (Straatman et al., 2006)). Many porous materials have been investigated for heat transfer enhancement (Patil and Kandlikar, 2014) (Liang and Mudawar, 2019).

In our previous study (Satake at al., 2016), we synthesized a porous material with a large number of voids and micropores and investigated the evaporation at its surface. As compared to conventional porous materials, the porous material prepared by us with a pore diameter of 25 \( \mu \text{m} \) and 80% voids showed enhanced evaporation performance. The condition of pore diameter and void is being unknown territory by conventional classification, be also different from
conventional behavior in a material mechanical characteristic and be being watched as a new heat-resistant material (Inoue et al., 2018) and the case that carbonized (Kubota et al., 2018). In this study, we investigated the evaporation behavior of a water droplet placed on the surface of a microporous material with a large percentage of voids. The porous body applied to the heating surface by liquid and it is indurated by curing. Therefore, this porous material can be fabricated on an arbitrary shape face. In this study, the porous material was directly fabricated on a heating surface in a vapor chamber and its heat transfer characteristics were investigated.

2. Materials and experiments

2.1 Fabrication of porous resin with a three-dimensional network structure

Epoxy resin (TETRAD-C, Mitsubishi Gas Chemical Company, Inc, Tokyo, Japan) was used as the raw material. Ethylene glycol (Wako Chemical Corp., Tokyo, Japan) and bis(4-aminocyclohexyl)methane (Tokyo Chemical Corp., Tokyo, Japan) were used as the solvent and curing agent, respectively. The resin, curing agent, and solvent were mixed and the resulting solution was agitated using a vortex mixer (VG 3 S004, IKA cop., Staufenberg, Germany). For vapor chamber experiments, an epoxy was coated on Cu substrate by dip-coating. Surface of copper substrate was simply soaked in epoxy solution. Then, the surface was ground and polished by SiC paper in order to remove surface skin layer. The thickness of the porous layer was 80-150 μm.

The solution was cured at 110 °C for 2 h in a temperature-controlled chamber (DRN320DD, Toyo Roshi Kaisha Ltd., Tokyo, Japan). After curing, the samples were dissolved in water at 85 °C to remove the solvent. The samples were then dried at 85 °C for 24 h. Finally, the samples were ground to remove their skin layer.

The morphologies of the samples were observed using scanning electron microscopy (SEM) (JCM-6000 Plus, JEOL, Tokyo, Japan). The mean pore diameters and pore size distributions of the samples were investigated by mercury intrusion porosimetry (MIP, AUTOPORE IV9520, Shimadzu Corp., Kyoto, Japan). The initial pressure was set at 3 kPa. The measurements were carried out at ~414 MPa. Over this pressure range, the pore diameter, \( D_p \), ranges from 3 nm to 50 μm, is calculated using Washburn’s equation:

\[
D_p = \frac{4\gamma \cos \theta}{P}
\]

where \( P \), \( \gamma \), and \( \theta \) are the mercury pressure, surface tension of mercury (485 dynes/cm), and contact angle (130°), respectively.

Figure 1 shows the typical SEM image of the as-fabricated porous material. The material showed a continuous network structure (monolith). Figure 2 also shows the cumulative pore diameter distribution measured by MIP. The porosity (\( p \)), median pore diameter (\( d_m \)) and average diameter (\( d_a \)) were calculated. Here, \( d_m \) is defined as a diameter that corresponds to the 50th percentile of pore volume. \( d_a \) is also given by,

\[
d_a = \frac{4V}{A}
\]

where \( V \) is total intrusion volume, and \( A \) is total pore area. \( p \), \( d_m \), and \( d_a \) of the material were 49.61%, 197.06 nm, and 150.65 nm, respectively.

![Fig. 1 SEM images of the as-fabricated porous material. (a) cross-section view and (b) the surface of porous body.](image-url)
2.2 Evaporation and vapor chamber experiments

Figure 3 shows the experimental setup for the investigation of the evaporation behavior of a water droplet with or without the porous material. This experiment was carried out in a clean booth whose air temperature and relative humidity were 18 °C and 40%, respectively. A high-precision electronic balance (BM-20, A&D Company Ltd., Tokyo, Japan) was used to measure the mass of the water droplet on the sample. First, zero-point adjustment was carried out once the sample was placed on the electronic balance. To generate a water droplet, an electronic pipette (MPA-10, A&D Company Ltd., Tokyo, Japan) was used. The accuracy and repeatability (CV) of the pipette are ±2.0% and 0.8% at 5.0 μL. The volume of the water droplet was 5.0 μL. The initial temperature of water was the same as that of air (18 °C). Mass measurement was carried out when the water droplet was placed on the sample. The mass of the water droplet on the sample was recorded after every 5 s until the water droplet evaporated completely.

Fig. 3 Experimental setup for the measurement of the natural evaporation rate of the water droplet on the porous material.
Figure 4 shows the experimental setup used for investigating the cooling performance of the porous material in a vapor chamber. The chamber consisted of an upper plate (oxygen-free copper, thickness of 2 mm), a lower plate (stainless-steel, thickness of 1 mm), and a silicone rubber spacer (thickness of 3 mm). At the center of the bottom plate, a copper square block made up of oxygen-free copper was installed. The height of the copper block was 15 mm and the area of the heating surface was 4 cm² (20 mm × 20 mm). For this experiment, the porous material was fabricated on the copper block by dip coating and the skin layer of the porous material was removed using emery paper #500. The gap between the bottom plate and the copper block was sealed with a room-temperature vulcanizing silicone. The size of the vapor chamber was 60 mm × 60 mm × 3 mm (volume of 10.8 mL). This height is referred to commercial vapor chambers. Pure water was used as the working fluid and the filling ratio of the vapor chamber was 1:1. The vapor chamber was not evacuated and the remaining space (50%) was filled with air. All components of the vapor chamber were fastened using 12 bolts. To condense the water vapor generated in the chamber, a water jacket was attached to the upper plate using a thermal grease. The temperature of the water circulating in the water jacket was maintained at 25 °C using a chiller. To ignore the temperature distribution on the copper upper plate, a water jacket with a high cooling performance was used. A heating block with cartridge heaters was attached to the copper block using the same thermal grease. To insert K-type sheathed thermocouple (0.5 mm in diameter, Class 1, non-ground type) between the upper plate and the water jacket, a groove was fabricated on the upper plate with 0.6 mm of depth. The temperature of heating block was measured to prevent over-heating of the heating block above 200 °C. In addition, the temperature distribution was measured using three sheathed thermocouples (K-type, 0.5 mm in diameter, Class 1, non-ground type), which were installed in the copper block. The distances between the heating surface and the thermocouples were 3, 6, and 9 mm. These positions were carefully measured by using a height gage. The insertion depth of the thermocouples into the copper block was 10 mm. Therefore, all thermocouples were aligned on the center axis of the vapor chamber. The copper block and the thermocouples in the copper block were covered with a thermal insulator. In this study, the steady-state temperature distribution in the copper block was assumed to be one-dimensional. Therefore, the temperature of the heating surface (hereafter, called as the wall temperature $T_w$ (°C)) could be extrapolated by linear approximation, as shown in Fig. 5. The calculated uncertainty of $T_w$ was less than 5% in this study.

![Fig. 4 Experimental set-up for the vapor chamber with the porous material (a) top view and (b) cross-section view.](image)
The input power $P$ (W) from the cartridge heaters to the copper block was calculated using the following equation:

$$P = V \times I.$$  \hspace{1cm} (2)

where, $V$ (V) is the voltage applied to the cartridge heaters and $I$ (A) is its total current. The heat loss from the side wall of the copper block was estimated as below 5%. In this study, the applied voltage was increased in steps of 10 V. The steady-state temperatures of the thermocouples were recorded by varying the input power of the cartridge heaters. The experiment was stopped when the temperatures of the copper block increased rapidly and became equal because the vapor chamber no longer worked at this point. Figure 6 shows the photograph of the vapor chamber with the porous material after the experiment.

Furthermore, the thermal resistance $R$ (K/W) was calculated as follows:

$$\Delta T = T_w - T_c,$$  \hspace{1cm} (3)

$$R = \Delta T / P.$$  \hspace{1cm} (4)

where, $T_c$ (°C) is the temperature between the upper plate and the water jacket. In this study, a copper block, which was polished using emery paper #500 along one direction, was also used to investigate the thermal resistance of the vapor chamber. Finally, the thermal resistances of the vapor chamber with and without the porous material were evaluated.
3. Results and discussion

Figure 7 shows the mass vs. time curves of the water droplets on the surfaces of various epoxy resin samples, as obtained from the evaporation test. The evaporation behaviors of the droplets on the surfaces of the materials with and without the three-dimensional network structure were different. In the stability region (500–1000 s), the evaporation rates of the non-porous and porous surfaces were 0.00239 and 0.00302 mg/s, respectively. The evaporation rate increased by approximately 1.2 times in the case of the porous material. In addition, the evaporation rate at the dip-coated epoxy porous material was 0.00338 mg/s, which is larger than that at the bulk material. The increase in the evaporation rate in the case of the porous material can be attributed to the dependence of the evaporation rate of the water droplet on the wettability of the surface (Unno et al., 2014) (Unno et al., 2018).

![Graph showing mass vs. time for water droplets on various samples.](image1)

The evaporation rate of the water droplet on various samples. (a) all data and (b) enlarged figure (500-1000 s).

Figure 8 shows the schematic of water evaporation with the porous material. The wettability improvement also affected the evaporation rate of the water droplet. In addition, in the case of the porous material, the water droplet penetrated the surface. As can be observed from Fig. 8, many liquid-vapor interfaces were generated when the water droplet penetrated the porous material. The porous material showed high porosity. The surface area of the liquid-vapor interface with micro liquid layers in the case of the porous material was higher than that in the case of the non-porous material. Therefore, in the case of the porous material, the water droplet could be readily evaporated and water vapor was emitted to air through the path in porous, shown as red arrows in Fig. 8. Hence, the porous surface showed a higher...
evaporation rate than the non-porous surface. The penetration speed of water into a porous material depends on the capillary force of the material. Materials with smaller pores show stronger capillary force. The total surface area of the liquid-vapor interface of a porous material is determined by its capillary force. However, small pores decrease the vapor emission speed from porous materials to air. Hence, the evaporation speed of a water droplet at the surface of a porous material depends on the capillary force and vapor emission speed of the material. The water evaporation rate, vapor emission speed, and capillary force of a porous material show a trade-off relation. Therefore, in order to increase the evaporation rate of a liquid from the surface of porous materials it is imperitive to optimize their pore diameters by taking into consideration the material properties of the liquid.

The cooling performance of the porous material was examined in a vapor chamber. In order to create a contact between the copper heating surface and the obtained porous material, the material was directly fabricated on the heating surface by dip-coating. Figure 9 shows the steady-state temperature distribution in the copper block measured by the three sheathed thermocouples with or without porous at 27 W. As a result, it seemed that the temperature distribution in the copper block was almost one-dimensional. Figure 10 shows the relationship between the input power and the wall temperature of the vapor chamber. Figure 11 shows the calculated thermal resistance of the vapor chamber with and without the porous material as a function of the input power. The thermal resistance of the vapor chamber with the porous material was lower than that of the chamber without the porous material when the input power was less than 40 W. The maximum input power of the porous material was almost double that of the raw copper heating surface. The decrease in the thermal resistance of the vapor chamber with the porous material can be attributed to the penetration of water (which evaporated subsequently from the pores) because of the capillary action. The porous material absorbed the heat of the heating surface. Moreover, the vapors in the pores evaporated in the chamber and condensed to form water. The condensed water penetrated the porous material again. The temperature of the heating surface decreased with the repetition of this cycle. In addition, the capillary force of the pores increased the maximum input power of the chamber by pulling the liquid back to the heating surface. The wall temperature was less than 150 °C until the dry out was observed. This temperature is lower than the heat resistance temperature of the porous material. The results showed that the porous material enhanced the evaporation of the water droplet not only during natural evaporation but also under the heated condition.
Fig. 9 Typical temperature distribution in the copper block measured by the three sheathed thermocouples.

Fig. 10 Relationship between the wall temperature and the input power of the vapor chamber with and without the porous material.

Fig. 11 Thermal resistance $R$ of the vapor chamber as a function of the input power with and without the porous material.
4. Conclusions

The natural evaporation behavior of a water droplet was investigated by placing it on an epoxy monolith with a three-dimensional network structure. The cooling performance of the porous material was investigated by fabricating it on a heating surface in a vapor chamber. The evaporation rate of the water droplet enhanced by approximately 1.2 times when the porous material with a three-dimensional network structure was used. This structure was easily obtained by baking the solution coated on the heating surface. As a result, the epoxy monolith with a three-dimensional network structure was directly formed on the surface of the oxygen-free copper block. The copper heating surface with the porous material was used for the vapor chamber test. The thermal resistance and maximum input power of the vapor chamber could be increased simultaneously by synthesizing the porous material on the heating surface. The heat transfer of the porous material was enhanced by the latent heat of evaporation of water in the pores. Even though the evaporation was not the main phenomenon occurring at the surface of the porous material, the heat transfer from the heating surface could be enhanced by the large surface area of the porous material. Consequently, the experimental result found that the epoxy monolith can be a promising method to enhance heat transfer, especially with a low water level. We believe that the technique used in this study is an efficient energy conservation approach for cooling next-generation devices because it requires only the coating and baking processes to fabricate porous materials with a three-dimensional network structure.

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