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ABSTRACT

High-performance and small-sized heat exchangers have been demanded due to the miniaturization and higher output of electronic devices, lasers, and energy harvesting/storage systems. Graphene nanosheet suspension has attracted attention as a next-generation nanofluid because of its high thermal conductivity and low pressure drop, while being dispersed stably without any additives. Graphene-based nanofluids have been mostly investigated using graphene oxide, and there are a few studies on pure graphene because of the limitation in mass production and stabilization at high concentrations of graphene. In this study, we prepared a 10 wt. % high-concentration few-layer graphene suspension by pulverizing graphite particles. Scanning electron microscopy, atomic force microscopy, and Raman spectra confirmed the few-layer graphene is formed in the suspension. The thermal conductivity of the suspension increased with concentration and suddenly jumped at a specific concentration. Furthermore, a significant improvement in thermal conductivity of >40% compared to base liquid was confirmed at 10 wt. % graphene content. A similar trend was observed for electrical resistance; 10 wt. % graphene suspension showed 62% lower resistance than that of 1 wt. %. These results suggest the percolation of graphene in a liquid, which has not been observed for graphene-based materials in previous research.

The heat generation density of small electronic devices, lasers, energy harvesting/storage systems, and other devices is increasing as they become smaller and more powerful. In other words, the high efficiency of cooling technology is a key factor in improving the performance of various types of devices. Water cooling systems, in which liquid flows through cooling blocks and pipings, are one of the most effective heat removal methods, and there are many attempts to improve their performances.[1] Generally, it is known that the heat transfer coefficient in a heat exchanger tube is proportional to the inverse of the cross-sectional dimension of the tube, and a high heat transfer coefficient can be obtained when the heat exchanger is microchannelized.[2] Because the flow in a microchannel is laminar with a low Reynolds number, the main mechanism of heat transfer is heat conduction. In addition, a high fluid flow in the narrow channel makes the boundary layer thinner, while the temperature gradient in the tube becomes even larger, improving the heat exchange rate through the tube wall. In both cases, the heat transfer performance is governed by the thermal conductivity of the working fluid. For this reason, previous studies have attempted to increase the thermal conductivity of the fluid by suspending various substances with high thermal conductivities. Typically, nanoparticles of metals, metal oxides, and carbides are added to fluids, and thermal conductivities are increased from 10% to >100%, depending on the materials and procedures.[3–5] Previous studies have shown that the size and shape of nanoparticles also affect thermal conductivity. Smaller particles or specific shapes (e.g., nanowires and nanosheets) increase the interfacial area and contribute to the enhancement of thermal conductivity. Jang and Choi discuss in detail the impact of the shape of nanowires on thermal conductivity.[6] The

improvement in thermal conductivity is mainly attributed to the high thermal conductivity of nanoparticles, the reduction of interfacial thermal resistance between particles, and the Brownian motion of the particles. Yang *et al.* stated that these mechanisms are not yet fully understood.[7] However, nanoparticle suspensions are not practically used for thermal fluids because of the increased pressure drop in laminar and turbulent flows, the use of rare metals, and the non-negligible precipitation during long-term use.[8] In addition to these drawbacks, flow resistance increases in a microchannel heat exchanger, increasing pressure drop and requiring more pump power to drive the fluid. The trade-off between heat transfer and pressure drop demands the development of new types of additives.[9]

In turbulent regimes, adding dilute polymer to a fluid significantly reduces the flow resistance compared to a Newtonian fluid, known as the Toms effect.[10] Because of its simplicity and low cost, there have been many studies on reducing flow resistance by using the Toms effect; typical examples include water-soluble polymers,[11] surfactants,[12] and fibers.[13] However, these additives have low thermal conductivity, reducing the heat transfer coefficient and resistance.[14]

Recent studies on thermal manipulation include control using metamaterials. Zhang *et al.* demonstrated that nanoscale structures increase phonon scattering, resulting in reduced thermal conductivity.[15] Such distinctive nanostructures can induce interesting changes in overall thermal properties. However, there are no reports of metamaterials utilized in liquid dispersion. Liquids with deformability could bring new possibilities to the functionality of materials.

We, therefore, turn to graphene-based suspensions as the working fluid. Graphene is noted for its attractive physical and chemical properties, is composed of an abundant earth element, and is environmentally safe.[16] Graphene has high mechanical strength and is recognized to have a high thermal conductivity of 3000 W/mK in the planar direction.[17] These remarkable thermophysical properties make graphene an important candidate for next-generation fluid additives. Previously, graphene oxide has been applied as the additive to reduce flow resistance and improve heat transfer efficiency; the results showed that the tube friction coefficient was reduced by up to 36.5%, and the heat transfer efficiency was increased by 10%–20% compared to distilled water.[18] Thus, graphene-based material has the potential to simultaneously achieve resistance reduction and heat transfer efficiency, which have traditionally been in a trade-off relationship. The cause of the resistance reduction effect is not clearly understood, but it is known that high concentrations of two-dimensional materials can affect rheological properties. It is known that graphene oxide solutions form liquid crystal phases, and this orientation of two-dimensional materials in the flow is considered to be a possible reason.[19,20]

The well-known method to produce graphene is to exfoliate graphite with adhesive tapes.[21] However, it is difficult to mass-produce and suspend in a solution at high concentrations. In this study, we prepared a highly concentrated (10 wt. %) few-layer graphene suspension by pulverizing graphite particles by ultrasonic treatment. Graphene nanofluids have been studied mostly with graphene oxide, and there are a few studies on pure graphene.[22] Pure graphene is free of functional groups and is expected to have higher thermal conductivity than graphene oxide suspensions, but it was difficult to disperse in a fluid and to achieve concentrations high enough to improve heat transfer. So far, even 5 wt. % graphene has been called "ultra-high concentration graphene," and its thermal conductivity has not been studied.[23]

Focusing on the thermal conductivity of graphene suspensions, Kole and Dey reported a 15% increase in thermal conductivity for a 0.394 vol. % liquid suspension of graphene in ethylene glycol solution.[24] Mehrali *et al.* reported a 27.64% increase in thermal conductivity for graphene suspensions up to 0.1 wt. %.[25] In this study, we prepare significantly concentrated (up to 10 wt. %) few-layer graphene dispersions and report their structural analysis and various thermophysical properties.

First, we prepared highly concentrated few-layer graphene suspension. The maximum concentration of graphene in the previous studies is about 1 wt. %.[26] Graphite with a particle size of approximately 1 μm was mixed with aqueous ammonia to make a 5 wt. % graphite mixture, which was then subjected to ultrasound (Hielscher, UP400St) for 1 h.[27] Undispersed graphite was removed by centrifugation (2000 rpm, 10 min), and solid contents were collected by filtration. Powdered few-layer graphene was obtained by lyophilization. Water was added to obtain a concentration of 10 wt. % and dispersed by a mixer.

The thermal conductivities of the prepared few-layer graphene suspensions were measured by the hot disk method using a thermophysical property measurement device (Hot Disk, TPS 500). In the hot-disk method, the sensor is energized to generate a known amount of heat, and the time-series change in the temperature rise of the sensor is measured. From the relationship between time, temperature, and heat quantity, Eq. (1) is used to obtain thermal conductivity,

$$\lambda = \frac{Q \cdot \ln(t_2/t_1)}{4\pi \cdot (T_2 - T_1)}, \tag{1}$$

where λ is the thermal conductivity, Q is the heating value per unit time and unit length of the heater (W/m K), T is the temperature, and t is the measurement time. The base temperature of the samples at the beginning of the measurement was controlled by a thermostatic water bath. The base temperature was varied from 25, 30, 40, 50, and 60 °C to verify the temperature dependence of the physical properties.

Solid disks of dry powder samples pressed in a φ14 cylindrical container at 20 kN were prepared, and thermal conductivity was obtained.

Specific Heat: The specific heat capacity of the prepared suspension was measured using a differential scanning calorimeter (Hitachi High-Tech Science, DSC7000X). Distilled water was used as the standard material. Three measurements were performed to calculate one specific heat capacity. The empty container, standard material, and target sample were maintained at 20 °C for 10 min, and then, the temperature was increased to 80 °C at 10 °C/min. The specific heat was calculated from the difference in the obtained differential scanning calorimetry (DSC) curves using the following equation:

$$Cp_s = \frac{H}{h} \cdot \frac{m_r}{m_s} \cdot Cp_r, \tag{2}$$

where Cp_s is the specific heat capacity of the sample, H is the difference between the DSC curves of the empty container and the sample, h is the difference between the DSC curves of the empty container and the reference material, m_r is the weight of the reference material, m_s is the weight of the sample, and Cp_r is the specific heat capacity of the reference material.

To verify the exfoliation by ultrasonic pulverization, samples were measured with a Raman spectrometer (RENISHAW, inVia
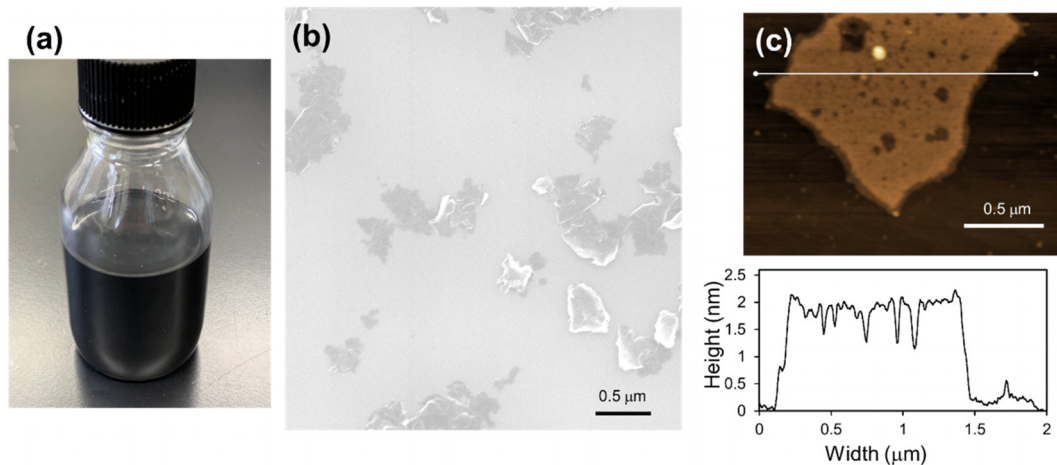
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FIG. 1. (a) Appearance of few-layer graphene suspension, (b) SEM image, and (c) AFM image and cross section analysis.

Reflex and JASCO Corp., NRS-3100) using a $\lambda = 532$ nm laser beam, laser intensity of 10 mW, and integration frequency and time to 10 times and 10 s, respectively. 2D band, which is a graphene-specific spectrum around 2700 cm^{-1}, was observed for few-layer graphene deposited on a silicon substrate.[28] Since the shape of 2D band Raman spectrum shows the ideal shape of the Lorentz function, the obtained 2D band full width at half maximum W (cm^{-1}) and the Raman spectrum of the 2D band x_0 (cm^{-1}) were fitted to the Lorentz function using the following equation:

$$W \frac{\omega^2}{4(x - x_0)^2 + \omega^2}. \tag{3}$$

The dispersion of few-layer graphene was stable for more than one month and did not settle [Fig. 1(a)]. The dispersion was diluted and dropped onto a silicon substrate and observed by scanning electron microscopy (SEM) [Fig. 1(b)] and atomic force microscopy (AFM) [Fig. 1(c)]. Although the thickness of the particles could not be determined by SEM, particles smaller than 1 μm were observed. The exfoliation process has little effect on the size of the product because the raw graphite is about 1 μm in diameter. AFM confirmed that the few-layer graphene is a two-dimensional sheet with a thickness of 2 nm. The thicknesses of ideal single-layer graphene and defective graphene oxide are 0.345 and 1 nm, respectively. Therefore, our few-layer graphene ranges from six to two layers. Raman spectra were measured to confirm the defects introduced during the exfoliation process (Fig. 2); as a result, the D band of few-layer graphene became larger than graphite, indicating that defects were introduced. In addition, the 2D band shifted to a lower wavenumber, and the ratio of the 2D band and the G band (I_{2D}/I_G) became more significant after the exfoliation process, suggesting successful exfoliation.[29] Although the intensity of the 2D band is much larger than that of graphene oxide (supplementary material), it is still smaller than that of pure graphene; therefore, the physical and chemical properties of our few-layer graphene might lie between graphene and graphene oxide.

Figure 3 summarizes the thermal conductivity of few-layer graphene at different volume concentrations and temperatures using water as a reference. A positive correlation between the concentration

of few-layer graphene and thermal conductivity was observed. The trend of increasing thermal conductivity with concentration was not linear, and a significant increase in thermal conductivity was observed at 10 wt. %. This phenomenon can be explained by the percolation theory, where adjacent graphene sheets come into contact with each other, forming a path through the entire system. The hopping conduction mechanism includes "nearest neighbor hopping (NNH)," where charges jump between nearby available states, and "variable range hopping (VRH)," where charges jump between wider ranges with stronger temperature dependences. This study demonstrates a strong temperature dependence, indicating that VRH is likely the mechanism at play. >40% thermal conductivity increase compared to water was observed at 50 °C, which is larger than that of carbon-based nanofluids in previous studies ($\sim$30%).[30–33] The temperature dependency of the thermal conductivity was not observed; the slight increase in the thermal conductivity by temperature relies on the property of the base fluid.

Figure 4 shows the relationship between concentration and thermal conductivity or electrical resistance at 25 °C for few-layer graphene and graphene oxide suspension. Due to the high viscosity,
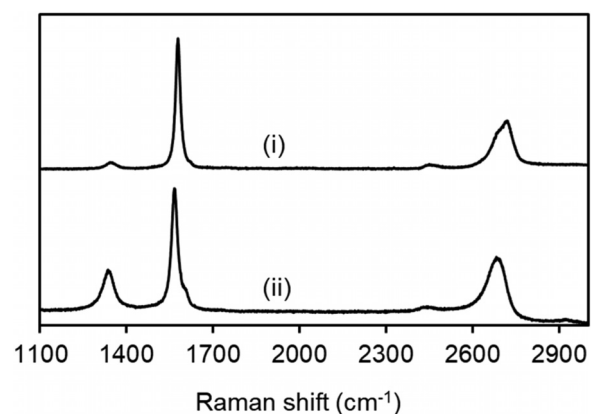

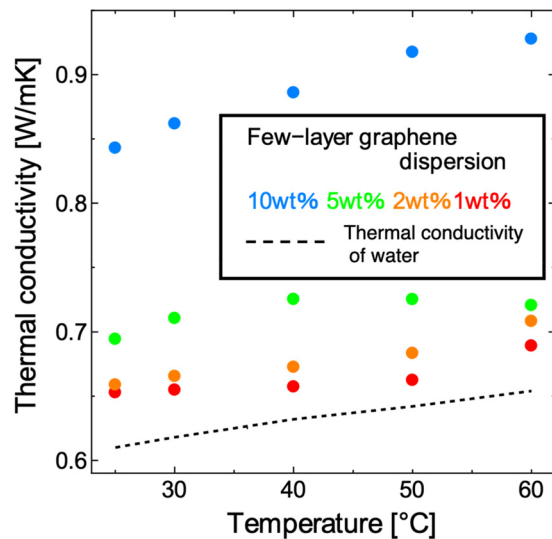**FIG. 2.** Raman spectra of (i) graphite and (ii) few-layer graphene.

FIG. 3. Relationship between concentration and temperature of few-layer graphene and thermal conductivity.

FIG. 5. Relationship between temperature and specific heat and concentration of few-layer graphene.

increasing the concentration >1 wt. % for graphene oxide was impossible. Few-layer graphene showed higher thermal conductivity than graphene oxide at 1 wt. %; only a 3.9% improvement in thermal conductivity was observed for graphene oxide. The thermal conductivity of few-layer graphene was significantly improved at 10 wt. %. A similar trend was observed for electrical resistance, with higher concentration decreasing the resistance, which is 62% lower at 10 wt. % than at 1 wt. %. The electrical percolation effect of graphene has been reported only for solid polymer composites, where graphene changes polymer character from an insulator to a conductor at a specific concentration.[34–36] Our results suggest that graphene also exhibits percolation in a liquid phase, paving the potential for innovative and dynamic fluid functions by taking advantage of graphene.
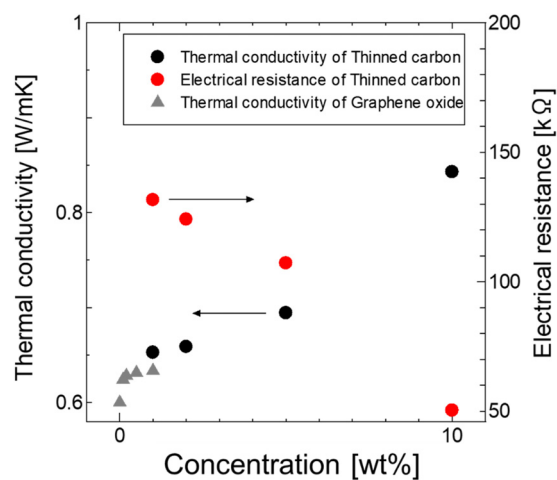
Figure 5 shows the specific heat ratio of the few-layer graphene suspension to water for different concentrations and temperatures. The concentration of few-layer graphene showed an inverse correlation with specific heat, because the specific heat of few-layer graphene is lower than the specific heat capacity of the base fluid, water. Finally, infrared camera imaging was performed to demonstrate the high thermal conduction of graphene-based suspension. The temperature distribution in a bottle filled with few-layer graphene and water is shown in Fig. 6. To observe only the effect of heat transfer, both samples were solidified with agar [1 wt. %, thermal conductivity of 0.58 W/(m K)], and then, the bottom of the bottles was heated on a hot plate. The few-layer graphene conducts heat to the top of the bottle, clearly visualizing a higher thermal conductivity. In previous studies on suspensions of metal nanoparticles, it has been observed that higher concentrations of nanoparticles result in more significant improvements in thermal conductivity. However, excessive concentrations can impair dispersion stability, making the optimal concentration setting crucial, which is generally below 1%. In this study, however, high stability was achieved at a high concentration of 10 wt. %.[37]

FIG. 4. Relationship between the concentration of few-layer graphene and thermal conductivity and electrical resistance.

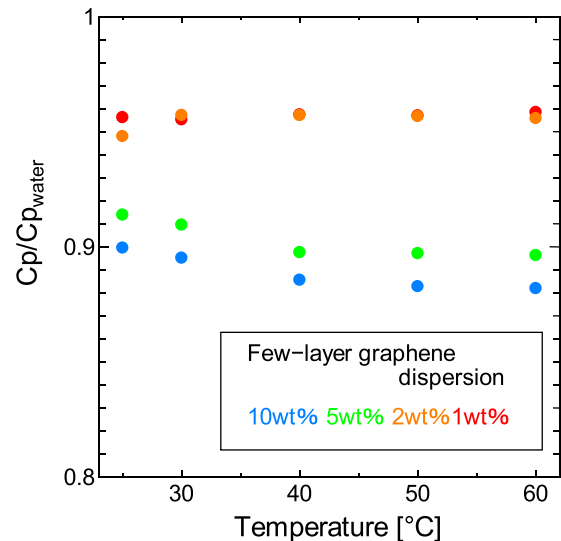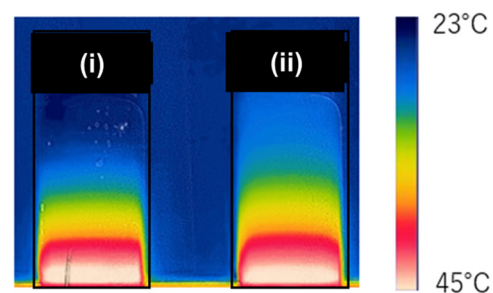**FIG. 6.** Temperature distribution of (i) water [thermal conductivity of 0.59 W/(m K)] and (ii) 10 wt. % few-layer graphene suspension by heating the bottom surface.

An unprecedentedly high concentration of 10 wt. % few-layer graphene suspension was prepared. SEM, AFM, and Raman spectra confirmed that the few-layer graphene suspensions consisting of several layers of graphene were observed. Thermal conductivities increased with concentration and tended to increase sharply at a certain concentration. An increase in thermal conductivity of up to 42.9% was observed at 50 °C compared to the base liquid water. Simultaneously, the electrical conductivity decreased sharply at a specific concentration. Percolation in the liquid by a graphene-like material was observed, paving the potential for innovative and dynamic fluid functions.

See the supplementary material for structural analysis, thermal conductivity, and specific heat of graphene oxide.

AUTHOR DECLARATIONS
Conflict of Interest

The authors have no conflicts to disclose.

Ethics Approval

All study participants provided informed consent, and the study design was approved by an ethics review board.

Author Contributions

Keiko Ishii: Conceptualization (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Visualization (equal); Writing – original draft (equal). **Takahiro Ogiyama:** Formal analysis (equal); Investigation (equal); Methodology (equal). **Koji Fumoto:** Project administration (equal); Supervision (equal); Writing – review & editing (equal). **Yuta Nishina:** Conceptualization (equal); Funding acquisition (equal); Methodology (equal); Writing – review & editing (equal).

DATA AVAILABILITY

Raw data were generated at Okayama University. Derived data supporting the findings of this study are available from the corresponding authors upon reasonable request.

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