Cupronickel-based micromesh film for use as a high-performance and low-voltage transparent heater

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The fabrication of uniformly interconnected cupronickel (CuNi) micromesh films on glass and polymer substrates with seamless junctions has been achieved using a simple transfer printing method. When used as a transparent heater, these CuNi micromesh films exhibited an effective and rapid heating performance at low input voltages (below DC 9V) that can be attributed to the creation of a high quality network over the whole surface area that offers a relatively high transmittance, strong adhesion to its substrate and good mechanical flexibility. A high thermal stability and reliability was also observed relative to a pure Cu micromesh film. Transparent heaters based on CuNi micromesh are therefore considered suitable for providing anti-fogging or de-icing in optics and optoelectronic devices, as well as for wearable heating systems.

Introduction

High-performance transparent heaters are very useful for the anti-fogging, anti-icing and de-icing of optics and optoelectronic devices such as outdoor displays, light emitting diode (LED) automobile headlamps, windows, mirrors and camera lenses.[1-3] The main requirements of a transparent heater in such applications are that it provide a fast thermal response time, uniformly heated surface, high transmittance (80–90%) in the visible range, low sheet resistance and long-term stability.[4-6] Currently, tin-doped indium oxide (ITO) films are typically used for this purpose due to their suitable optoelectronic properties;[1, 2, 4-6] however, ITO-based film heaters are limited by a slow thermal response time, high brittleness on flexible substrates, poor thermal stability and the increasing price of ITO.[1, 2, 6, 7] It is for these reasons that ITO film is considered unsuitable for use as a high-performance transparent heater in next-generation optoelectronic devices. A suitable replacement using carbon-based nanomaterials such as graphene and nanotubes has been extensively studied to address the limitations of ITO film heater over the past few years.[1-3, 5-10] Such heaters have, however, required either a relatively high voltage (more than 12 V) or additional chemical treatment to achieve high heating performance.[1, 6, 9, 11]

More recently, metal-based transparent heaters based on random networks of metallic nanowires or metal mesh films have emerged as the most promising alternative material to ITO film by virtue of their fast thermal response and low sheet resistance.[2, 7, 10] Noble metals such as silver (Ag) and gold (Au) have been widely used,[2, 4, 7] as such metals offer the highest thermal stability and lowest sheet resistance of the transparent heaters. These metals are, however, also very rare and relatively expensive, and thus although Cu is slightly less electrical and thermal conductivity than that of Ag, its lower cost and greater abundance make it an attractive alternative.[12] Motivated by these intrinsic advantages of Cu, several researchers have made significant progress over the past few decades in the fabrication of Cu-based transparent electrodes.[12-15]

However, the fatal drawback is the Cu-based electrodes are readily oxidize when exposed to oxygen, moist air and high-temperatures.[12, 16] To solve this problem, some of the most compelling studies have focused on the prevention of the performance degradation from oxidation. But, the majority of existing methods require an additional process, which leads to loss of optical or electrical properties, such as Ni (or Graphene) coating to prevent oxidation.[12, 16, 17] Therefore, there is a strong need to develop prevention method of the performance degradation without an additional process.

For this, we suggest that one of the solutions is the application of a Cu-Ni alloy based uniform micromesh films to the transparent heater. The reasons are that there are some benefits in the Cu-Ni alloy based transparent heater such as relatively high heating performance and resistant to oxidation without a serious loss of initial performance, and relatively less temperature dependent of the resistivity during operating.[18]

In addition, the feasibility of the uniform micromesh structure of the Cu-based alloy with Ni has not yet been studied for use as a transparent heater. Here, we report for

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the first time a high-performance and low-voltage (below DC 9V) transparent heater based on CuNi micromesh film with seamless junctions, which are smooth junction without hot spots from the crossbar junctions. The CuNi mesh film heater exhibited not only a fast and stable thermal response, but also the uniform distribution of the heat-generating temperature and high optical transmittance. The CuNi mesh film heater also showed strong adhesion to its substrate and good mechanical flexibility, with a high resistance to thermal oxidation when compared to a pure Cu mesh film heater.

Experimental

Materials

UV-curable polyurethane acrylate (PUA) imprint resin (YNIL-M2) was purchased from Young Chang Chemical Co., Ltd., and ultraviolet (UV)-curable adhesive (NOA 61) was purchased from Norland Products, Inc. A polyethersulfone (PES) film measuring 200 μm in thickness was purchased from i-components Co., Ltd. Finally, Cu (99.99 %) and CuNi (80:20 wt%) for e-beam deposition were purchased from Taewon Scientific Co., Ltd. and Materion Co., Ltd., respectively.

Optical, electrical, and microscopic characterization

Optical transmittance spectra were obtained using a Neosys-2000 UV-Vis spectrophotometer (SCINCO) with air as a reference. Digital infrared (IR) images of the transparent heaters were obtained using a FLIR-E63900 camera. The sheet resistance of each transparent heater was measured using the four-probe method with a sheet resistivity meter (FPP-2400, DASOL ENG) in contact with the center of the heater. The I-V characteristics of the CuNi micromesh films were measured using a two-probe technique with a semiconductor device analyzer (B1500A, Keysight Tech.). The morphology of the transparent heater was confirmed using optical microscopy (ECLIPSE LV 100D, Nikon Instrument Inc.) and FE-SEM (S-4800, Hitachi). Focused ion beam (FIB) cross-sections were obtained on a Helios NanoLab (FEI) at an acceleration voltage of 2.0 kV.

Preparation of uniform CuNi micromesh film

Uniform CuNi micromesh films with seamless junctions were fabricated on a transparent substrate by a transfer printing method.[13, 19, 20] Recently, a considerable number of studies have been conducted on large-scale transfer printing method for the mass production.[1, 13, 20] As shown in Fig. S1, this entailed first duplicating PUA mold from a micromesh-patterned silicon (Si) master using the replica molding method. A 150 nm thick CuNi layer was evaporated onto PUA mold at a rate of about 0.15 nm/s by using an electron-beam evaporator. To provide selective transfer of the CuNi layer, a transparent UV-curable adhesive (NOA61) with thickness of 1 μm was spin-coated onto the transparent substrate (glass or PES film) and partially-cured by a 50 W UV lamp for 24 s. Once the CuNi-deposited PUA mold and partially-cured surface were brought into contact, the entire assembly was pressed by applying a 0.15 MPa static pressure for 3 min. The resulting uniformly interconnected CuNi mesh film with seamless junctions was then peeled from the PUA mold and completely cured on the transparent substrate for 3 min to ensure strong adhesion.

Preparation of AgNWs and ITO-based transparent heaters

AgNWs-based transparent heaters were prepared through the spin-coating process and were formed on pre-cleaned glass substrates. The optical and electrical performances of the fabricated AgNWs-based heaters exhibited a transmittance of 90% and a sheet resistance of 25 $\Omega \cdot sq^{-1}$. And, transferred AgNWs-based heater was prepared in the same method as the metallic micromesh films. ITO-based transparent heaters were prepared by purchasing the commercial ITO glass. The commercial ITO glass has an average sheet resistance of 15.0 $\Omega \cdot sq^{-1}$

Bending test

A CuNi mesh film on a PES film was affixed onto a cylinder with a curvature radius (*r*) of 4 mm, with the CuNi mesh film facing the convex side. The CuNi mesh film was then repeatedly bent by an automatic bending apparatus and changes in its maximum steady-state temperature at 6 V were measured.

Results and discussion

Fig. 1a shows a photograph and optical micrographs of 150 nm thick CuNi mesh films with a 2 μm line-width and line-spacings of 100, 200 and 300 μm that were successfully fabricated on a glass substrate. In addition, Fig. S2 shows surface and cross-sectional FE-SEM images of a CuNi mesh film on NOA-coated glass. Energy-dispersive spectroscopy (EDS) analysis was used to confirm the presence of the both Cu and Ni in the metallic mesh films, the results of which are given in Fig. S3.

The optical transmittance spectra of the CuNi mesh film in Fig. 1b reveals an average transmittance at 550 nm of 85.6, 88.4, and 89.5% with line-spacings of 100, 200, and 300 μm, respectively. This compares well with the results for a commercial ITO film, which exhibited a transmittance of 89.7% at 550 nm and an average transmittance of 83.5% over the entire visible range. Through measurement of the sheet resistance of the CuNi mesh films and commercial ITO glass using the four-point probe method, it was found that commercial ITO glass has an average sheet resistance of 15.0 $\Omega \cdot sq^{-1}$, whereas the CuNi mesh film on glass gave values of 16.2, 38.4, and 50.9 $\Omega \cdot sq^{-1}$ with line-spacings of 100, 200, and 300 μm, respectively. These results confirm that both the transmittance and sheet resistance of a metallic mesh film increase with line-spacing, meaning that the optical and electrical properties of a CuNi mesh film can be tailored to match those of ITO glass.

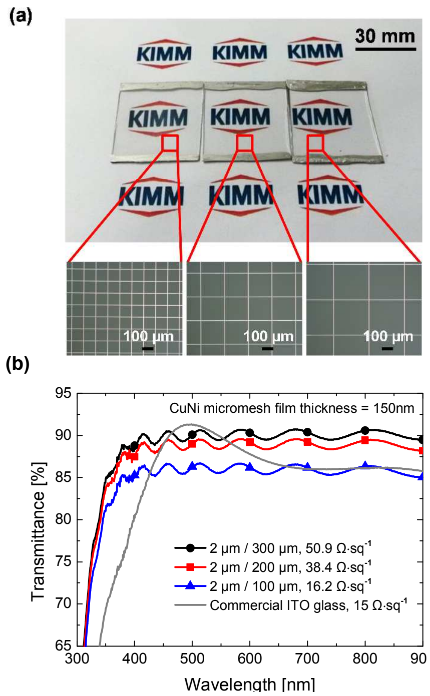

Fig. 1 (a) Optical characteristics of 150 nm thick CuNi micromesh films on a glass substrate with a line-width of 2 μm and line-spacing of 100, 200 and 300 μm. (b) Transmittance of micromesh films and ITO film on glass substrates.

From experimental results obtained, the figure of merit (FoM) for the CuNi mesh films, or the ratio of electrical conductance to optical conductance (σ_{dc}/σ_{opt}), was calculated as follows:[2, 21-23]

$$T = \left(1 + \frac{Z_0}{2\,R_S}\frac{\sigma_{opt}}{\sigma_{dc}}\right)^{-2} \qquad (1)$$

where R_S and T are the measured sheet resistance and transmittance at 550 nm, respectively, and Z_0 is the impedance of free space (377 Ω). Generally speaking, the minimum FoM value necessary for most transparent electrode applications is at least 35,[21, 22] with the FoM values obtained with 100, 200, and 300 μm line-spacings being 145, 77, and 64, respectively. Thus, although the FoM values of the fabricated CuNi mesh film were lower than commercial ITO glass (228), they were still much higher than the minimum value (35) required for most practical applications.

A 150 nm thick CuNi mesh film was also successfully fabricated on a PES film using the same method, with its optical transmittance spectra and sheet resistance indicating that such a film could be used as a flexible transparent heater (Fig. S4).

The adhesion of a CuNi mesh film to its underlying substrate is a very important consideration when it comes to practical application in optoelectronic devices. For this reason, the fabricated CuNi mesh film was subjected to 30 min of ultra-sonication in deionized (DI) water to estimate its adhesion to its substrate and measure any change in its sheet resistance. The spin-coated AgNWs and transferred AgNWs networks on glass were also tested under the same conditions for comparison. The sheet resistance of spin-coated AgNWs increased significantly due to the poor adhesion between AgNWs network and substrate during the test (Fig. S5). In contrast, the sheet resistance of the transferred AgNWs and CuNi mesh films remained unchanged under the same conditions (Fig. S5). This suggests that the adhesion between the metallic films and substrate was very strong.

To assess the potential of CuNi mesh as a transparent heater, the metal alloy films were prepared with dimensions of 25 mm × 25 mm and a two-terminal side-contact configuration. As shown in Fig. 2a, a direct current (DC) voltage of 3, 6, or 9 V was applied to this CuNi mesh film heater through an Ag side-contact, and the resulting variation in temperature was measured using an IR camera. The temperature profile obtained by the IR-imaging was confirmed by a direct measurement with a thermocouple mounted on the back side of the heater.

Heating of the metallic mesh film occurs first by Joule heating, after which the heat generated dissipated through conduction in the substrate, convection to the air, and radiation.[24] Thus, although the measured temperature is mainly associated with the heat of convection,[24] the amount of heat generated is determined with the input power, sheet resistance, and heating surface area of the heater.[6] The total power consumption (P) of the CuNi mesh film heater can be calculated by solving the following equation:[1, 6]

$$P = I^2 R \qquad (2)$$

where I is the current passing through the heater, and R is its sheet resistance. Using this, the total power consumption of heaters with 100, 200, and 300 μm line-spacings at 9 V were determined to be 2.72, 1.69, and 1.15 W, respectively.

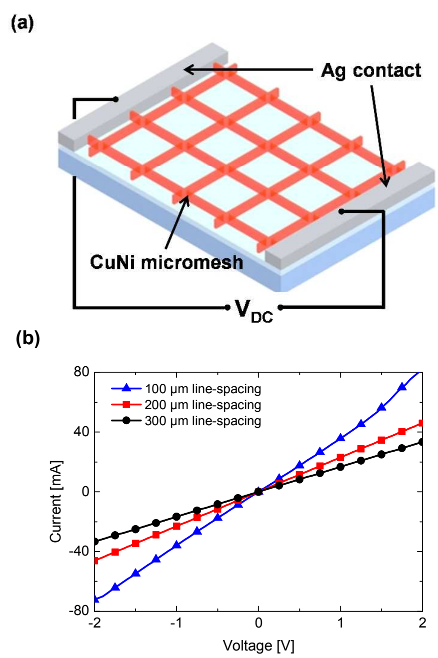

Fig. 2 (a) Schematic structure and (b) electrical properties of a CuNi transparent heater.

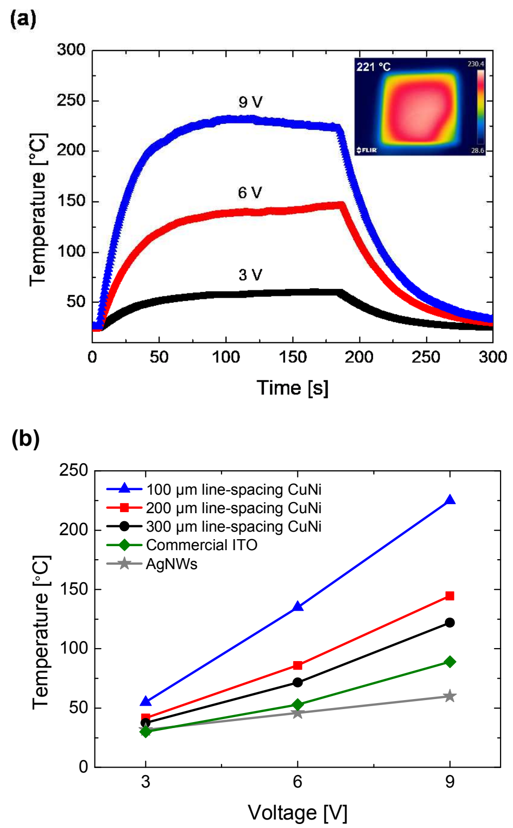

Fig. 3. (a) Temperature profile of a transparent heater with a 100 µm line-spacing as a function of time at applied voltages of 3, 6 and 9 V. (b) Temperature variation in various transparent heaters (CuNi micromesh films, commercial ITO film, and AgNWs network) as a function of applied voltage.

The current and voltage (I-V) curves for each CuNi mesh film were measured using a two-probe technique with a semiconductor device analyzer, and the results of which are given in Fig. 2b, are comparable to the values obtained using the four-point probe method. The CuNi mesh films created Ohmic contact and an increase in the electrical resistance with increasing the line-spacing.

The response time of a heater, or the time taken to reach a steady-state temperature from room temperature, is also an important factor in evaluating its performance. To this end, Fig. 3a depicts the time-dependent temperature of a CuNi mesh film with a 100 µm line-spacing, revealing that a steady-state temperature is reached quite rapidly (within 60 s) across a range of input voltages. The IR image (inset) of the Fig. 3a reveals the relatively more uniform heat-generation on the CuNi mesh film than the spin-coated AgNWs on glass (inset of the Fig. S6). In the case of the AgNWs-based heaters, even at an input voltage of 15V, hot spots and non-uniform heat-generating performance could be observed even at relatively low temperature of ~90 °C. These experimental results are in agreement with previous studies.[10] Here, the reason for the low temperature at the edges of the transparent heater in the IR image is additional heat loss at the edges.[24] Additionally, in the case of the spin-coated AgNWs, relatively high voltage is required to reach a steady-state temperature in comparison with the CuNi mesh film, and the maximum heat-generating temperature of the AgNWs-based heater fails to reach 200 °C due to the breakage at the networks between AgNWs (Fig. S6 and Fig. S7).

The temperature variation in the CuNi mesh films, commercial ITO films, and spin-coated AgNWs film as function of applied voltage is shown in Fig. 3b, and indicates that the greater power consumption with a 100 µm line-spacing correlates to a higher maximum temperature at steady-state (225 °C at 9 V). Moreover, the increase in heating surface area of metallic mesh also ensures the maximum temperature increase for the lowest sheet resistance. This higher output power for a given input voltage means that electrical energy is more efficiently converted into Joule heating due to the high degree of connectivity created by the seamless junctions spread uniformly over the substrate by the mesh structure. Although the power of ITO film (6.4 W) was higher than that of CuNi mesh film (2.72 W) with a 100 um line-spacing, the mesh film showed the higher maximum temperature. The better heating performance of the CuNi mesh film was attributed to the higher power density (0.1 W/mm^2) than that of ITO films (0.01 W/mm^2) due to the mesh structure.[1, 7] ITO films did not attain a higher temperature than the metal mesh, despite equilibrium for ITO due to larger heat transfer coefficient of oxides than that of the metal mesh (Fig. S8).[25, 26] More importantly, this heating performance with respect to applied voltage is quite sufficient for use as a transparent conductive heater rather than the ITO film and AgNWs film heaters (Fig. S6 and Fig. S8).

The thermal stability and reliability of a CuNi mesh transparent heater (23 mm × 19 mm) with a line-spacing of 100 µm was further tested by switching the applied voltage from 0 to 9 V for 20 cycles. As shown in Fig. 4a, the temperature of the pure Cu mesh film heater with the same line-spacing as the CuNi mesh film heater decreased sharply at around 240 °C during the first cycle and failed to generate heat after the first cycle due to the rapidly thermal oxidation of the pure Cu (Fig. S9).[12, 16, 27-29] Meanwhile, the CuNi mesh provided thermally stable and reliable switch performance even after 20 switching cycles, as the improvement in oxidation resistance and less temperature dependent of resistivity provided by alloying Cu with Ni helps to ensure a stable electrical conductivity.[12, 16-18] It can therefore be safely concluded that a CuNi mesh transparent heater should easily satisfy the reliability requirements of a transparent heater in most, if not all, applications.

In order to further demonstrate the potential of a CuNi mesh film as an efficient defroster, defrost testing was performed by placing on it a clean slide-glass that had been kept in a refrigerator for 15 min to allow frost to form over its entire surface. As shown in Fig. 4b, the frost on the slide-glass was completely removed within 60 s with the applied voltage of 9 V to the heater. A water droplet evaporation test was also performed in which water droplets with red color were applied to the back side of the heater while operating at 12 V, having the result of the droplets evaporated within 90 s as shown in

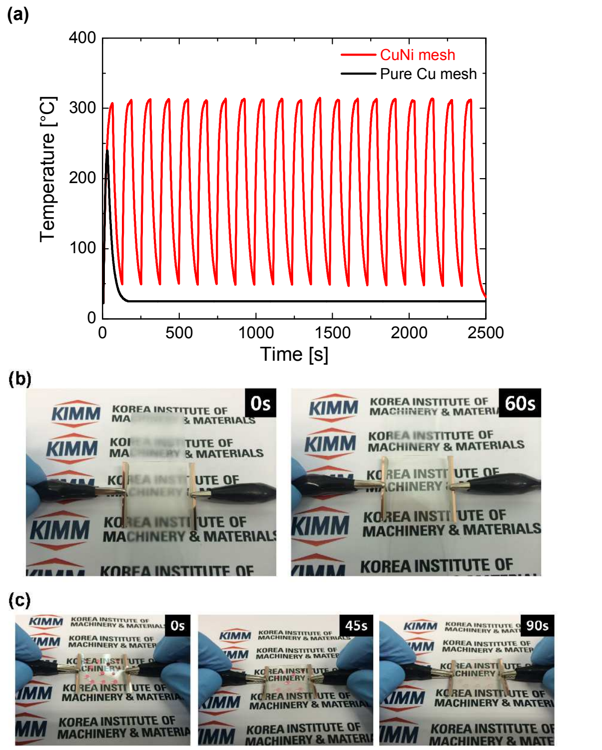

Fig. 4 (a) Thermal stability of pure Cu and CuNi transparent heaters with 100 µm line-spacing under switching cycles at an applied voltage 0 to 9 V. (b) Defrosting results before and after operation of the CuNi transparent heater at 9 V. (c) Evaporation of water droplets on the back side of the heater within 90 s when operated at 12 V.

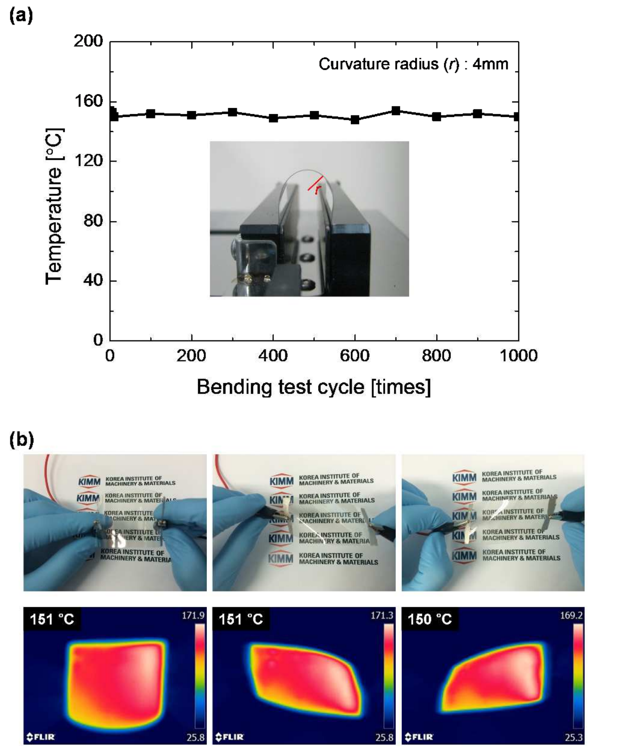

Fig. 5 Mechanical flexibility and stability of a CuNi transparent heater on a polymer substrate. (a) Variation in temperature at 6 V under repeated bending cycles (inset shows a photo image of the cyclic bending test). (b) Photographs and IR images of the heater at 9 V under bending and twisting conditions.

Fig. 4c. These experimental results demonstrate that heat is uniformly distributed over the whole surface area of the CuNi mesh film heater due to its uniformly interconnected mesh structure and suggests that it could be used for either anti-fogg or water-removal in optoelectronic devices.

Since the flexibility of a heater is very important consideration for use in wearable devices, a CuNi mesh film was fabricated on a flexible substrate (PES film) and evaluated by bending-fatigue cycling around a curvature radius (r) of 4 mm at a speed of 1.0 cycle/s. The temperature of the CuNi mesh film heater at 6 V was measured during this test and compared with its initial value, as shown in Fig. 5a, revealed that the change was within ± 5 °C. Fig. 5b shows photo and IR images of the bended and twisted CuNi mesh film heater at 6 V, which clearly demonstrate the high mechanical flexibility of the CuNi mesh film.

Conclusions

This study has demonstrated the feasibility of fabricating a transparent heater based on a uniformly interconnected CuNi mesh film with seamless junctions on glass or polymer substrates using a simple transfer printing method that eliminates the need for a lift-off or etching process. The resulting CuNi mesh film heater exhibited excellent electrical and mechanical connectivity over its whole surface area; accordingly, it exhibited not only a rapid and stable thermal response, but also the uniform distribution of the heat-generating and good optical transmittance (83–90 %). Specifically, the entirety of the heater surface reached ~225 °C within 60 s of applying 9 V DC, and the heating performance remained stable during switching testing. The CuNi mesh film also showed strong adhesion to its substrate and good mechanical flexibility, with a high resistance to thermal oxidation when compared to a pure Cu mesh film heater. On the basis of these results, we believe that CuNi mesh transparent heaters could be widely used in fields that require excellent electrical and thermal properties, and mechanical flexibility.

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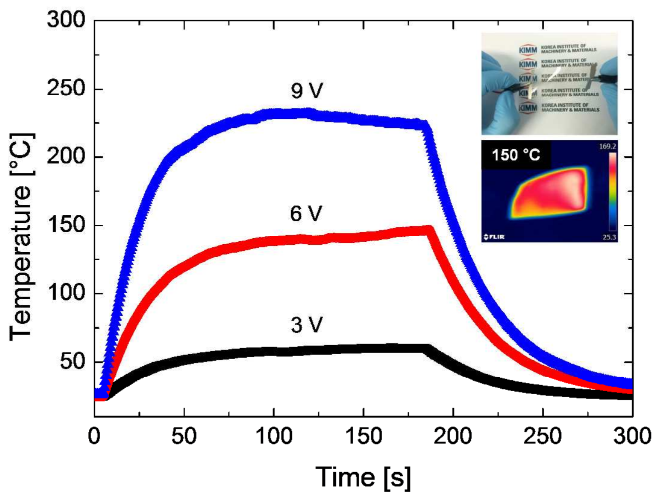
Graphical Abstract

Cupronickel-based micromesh film for use as a high-performance and low-voltage transparent heater

Han-Jung Kim, Yoonkap Kim, Jun-Ho Jeong, Jun-Hyuk Choi, Jihye Lee and Dae-Geun Choi*



Uniformly interconnected CuNi mesh film with seamless junctions was prepared without the lift-off or etching process by using simple transfer printing method. The CuNi mesh films showed excellent heating performances, which reached maximum steady-state temperature of 225 °C in 60 s, with respect to applied low-voltage (9V), and better thermal stability than that of the pure Cu mesh film.