



Electrodeposited Hierarchical Silver Network Transparent Conducting Electrodes with Excellent Optoelectronic Properties and Mechanical Flexibility

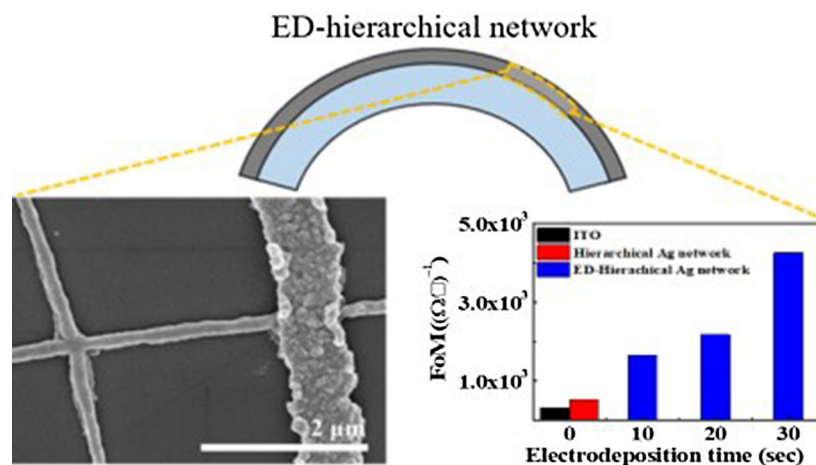
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Received: 10 July 2023 / Accepted: 26 July 2023 / Published online: 24 August 2023
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Abstract

Mechanically flexible transparent conductive electrodes (TCEs) with high optoelectronic performance are essential for flexible or wearable optoelectronic devices, which are currently receiving a considerable amount of attention. In this study, we investigate the structural, electrical, optical and mechanical properties of electrodeposited hierarchical silver network TCEs consisting of two layers of silver nanowires (AgNWs) and a silver micromesh. Hierarchical structures are known to improve the optoelectronic properties of network-type TCEs. To fabricate an electrodeposited hierarchical network, a AgNW solution is first spun onto a substrate to form randomly distributed AgNWs, and a silver micromesh is then formed on the AgNWs. Subsequently, silver is electrodeposited onto the hierarchical network. As a result of the electrodeposition, AgNW-AgNW and AgNW-silver micromesh contacts are effectively welded, and the dimensions of the AgNWs and the silver micromesh are optimized to maximize the figure of merit of the TCE. Furthermore, the electrodeposited hierarchical silver network shows excellent mechanical flexibility and much less degradation of its sheet resistance than that experienced by ITO upon repeated convex and concave bending. Its resulting optoelectronic and mechanically flexible performance is superior to that of commercialized ITO.

Graphical Abstract



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Keywords Transparent conducting electrodes · Hierarchical network · Electrodeposition · Silver micromesh · Silver nanowire

1 Introduction

Transparent conducting electrodes (TCEs) that can be used for flexible or wearable optoelectronic devices must have low sheet resistance and high light transmittance, as well as mechanical flexibility [1–13]. Carbon nanotubes (CNTs), graphene, metal nanowires, and metal meshes are being studied as suitable materials for flexible TCEs [14–19]. The typical optoelectronic properties of mechanically flexible TCE materials, namely, the sheet resistance at 90% optical transmittance, are as follows. CNT: $84 \Omega/\square$ [20], graphene: $30 \Omega/\square$ [21], metal nanowires: $8\text{--}20 \Omega/\square$ [22], metal micromeshes: $5\text{--}10 \Omega/\square$ [23]. Among the abovementioned materials, metal micromeshes offer the best optoelectronic performance [24–26].

When network-type TCEs are employed in optoelectronic devices, charge carriers are present in the empty space of network-type TCEs such as CNTs, nanowires and micromesh [27–29]. Therefore, the relatively large empty space in the metal micromeshes may limit their application in optoelectronic devices. As a potential solution to this problem, nanoscale wires were added to a metal micromesh to form a hierarchical metal network [26]. A multiover network has also been reported to be more suitable than a single-order network in ensuring lateral movement of charge carriers in the empty space of the network TCEs [30–32].

In this work, we improved the optoelectronic properties of a hierarchical silver network TCE by electrodeposition. An electrodeposited (ED) hierarchical silver network

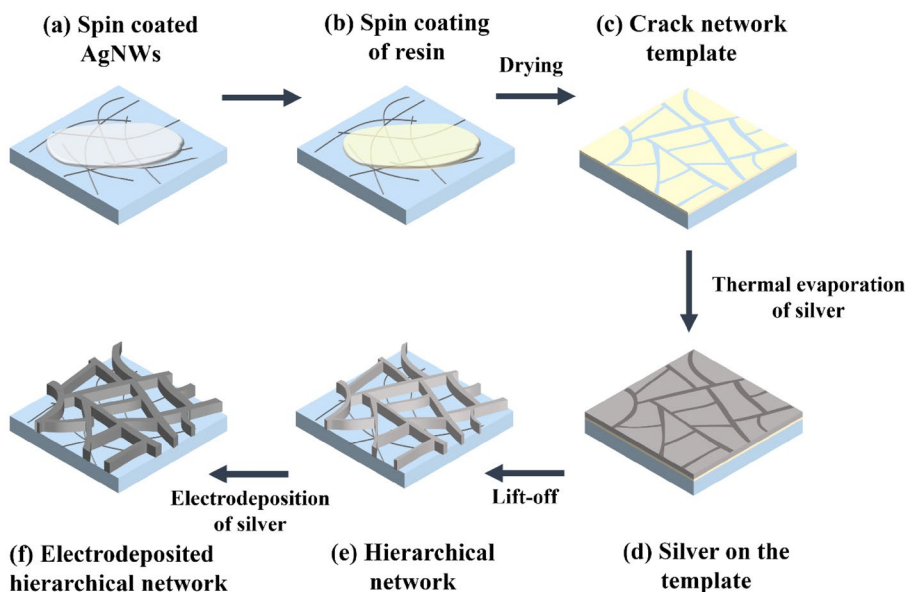
consisting of silver nanowires (AgNWs) and a silver micromesh was prepared. The silver micromesh provides main paths for electrical current flow, and the AgNWs provide local paths within the empty space of the micromesh. The electrodeposition of silver onto a hierarchical silver network enabled proper welding of AgNW-AgNW and AgNW-silver micromesh. The dimension of the silver micromesh was also adjusted to increase the figure of merit (FoM) of the hierarchical silver network TCE by electrodeposition. As a result of electrodeposition, the FoM of a hierarchical network TCE increased to 8.1 times before electrodeposition, which was 13.7 times that of a commercialized ITO thin film. Furthermore, the ED-hierarchical silver network showed much better mechanical flexibility than the ITO thin film upon repeated convex and concave bending. Therefore, the ED-hierarchical silver network is expected to be suitable for various optoelectronic device applications.

2 Experimental Section

2.1 Fabrication

The fabrication of an ED-hierarchical silver network TCE consists of three main steps: the first main step is to form randomly distributed AgNWs, the second main step is to form a silver micromesh on the AgNWs to form a hierarchical silver network, and the final main step is to electrodeposit

Fig. 1 Schematic illustration of the processing step for an electrodeposited hierarchical silver network: **a** spin-coated AgNWs, **b** spin-coating of an acrylic resin layer, **c** a crack network template, **d** thermal evaporation of silver on the crack-network template, **e** deposition of the remaining silver micromesh onto the AgNWs to form a hierarchical silver network and **f** electrodeposition of silver onto the hierarchical silver network



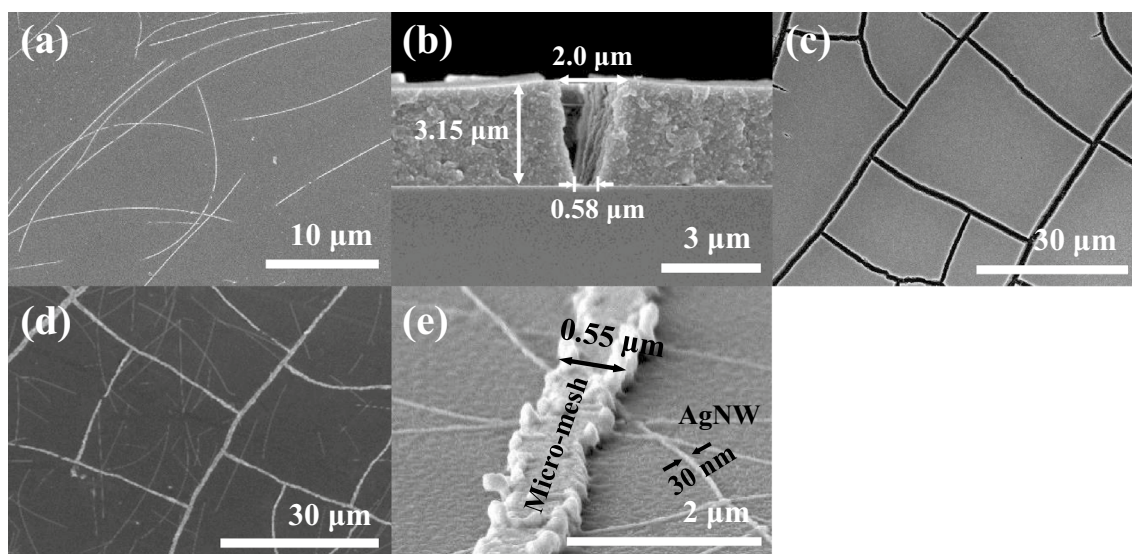


Fig. 2 Microstructure and structural analysis according to hierarchical network formation steps: **a** spin-coated AgNWs, **b** cross-sectional and edge plane views of a crack network template, **d** a plane and **e** a tilted view of a hierarchical silver network

silver onto the hierarchical silver network. Details of the processing steps are shown schematically in Fig. 1.

AgNWs: To form randomly distributed AgNWs, AgNWs dispersed in ethanol (S32E, Nanopyxis) at a concentration of 0.05 wt. % were spun onto a PET substrate with a size of 2.5 cm × 2.5 cm by spin coating at 1000 rpm for 30 s followed by thermal annealing at approximately 120 °C for a few minutes to dry the solvent on a hot plate (Fig. 1a). The length and diameter of the AgNWs were approximately 25 ± 5 μm and 32 ± 5 nm, respectively.

Silver micromesh: To form a silver micromesh on the AgNWs, first, commercially available acrylic-based resin (Lubrizol CarboSet CR735) was spin-coated on the AgNW-coated PET substrate at 3000 rpm for 30 s and dried at 120 °C (Fig. 1b). Volumetric shrinkage of the CR735 thin film during the drying process produced a randomly distributed and well-connected crack network (Fig. 1c). Silver with a thickness of 250 nm was then thermally evaporated onto the crack network (Fig. 1d). Subsequently, the sample was immersed in acetone and sonicated using an ultrasonicator (GT-X1, GT sonic) to remove the sacrificial crack network film. Therefore, the silver thin film deposited onto the crack network film was lifted off, and the silver deposited on the cracked region of the crack network film remained as a silver micromesh on the AgNWs to form a hierarchical silver network (Fig. 1e).

Electrodeposition of silver: Silver was electrodeposited onto a hierarchical silver network, which is a combination of AgNWs and a silver micromesh. In this process, a silver electrodeposition solution was prepared by dissolving 7 g of silver cyanide (Acros Organics, 180,230,500), 1.5 g of potassium carbonate (Alfa-Aesar, A16625), 15 g of potassium cyanide (Sigma-Aldrich, 207,810), and 5 g of sodium

thiosulfate pentahydrate (Alfa-Aesar, A17914) in 85 ml of DI water. A hierarchical silver network as a working electrode and a silver foil as a reference electrode were immersed in the silver electrodeposition solution under a constant current source of −4.5 mA to obtain an ED-hierarchical silver network (Fig. 1f).

2.2 Characterization

The microstructures of the samples, including the AgNWs, a sacrificial crack-network template CR735 film, and hierarchical silver networks before and after electrodeposition of silver, were analyzed using field-emission scanning electron microscopy (FE-SEM, HITACHI SU8230). The optical transmittance of hierarchical network TCEs before and after electrodeposition of silver was analyzed by means of a UV–vis spectrometer (UV-2600, Shimadzu) equipped with an ISR-2600 Plus integrating sphere using an employed substrate as a reference. The sheet resistance of the hierarchical network TCEs before and after the electrodeposition of silver was measured by a 4-point probe (FPP-40 K, Dasol Eng). The mechanical flexibility of an ED-hierarchical silver network TCE was tested by measuring its surface resistance after repeated convex and concave bending with a radius of curvature of 5 mm.

3 Results and Discussion

3.1 Microstructure

Figure 2a shows randomly distributed AgNWs on a PET substrate, corresponding to the schematic shown in

Fig. 1a. The AgNWs do not have to form a network over a long distance because the AgNWs provide local paths within the empty space of a silver micromesh. The top and bottom widths of the U-shaped crack-network template (thickness = 3.15 μm) formed on the preformed AgNWs, corresponding to the schematic shown in Fig. 1c, were approximately 2.0 and 0.58 μm , respectively (Fig. 2b). The plane view of a sacrificial CR735 template formed on the AgNWs shows a well-connected crack network (Fig. 2c). A silver micromesh deposited on the cracked region of the sacrificial template shows a well-connected network formation over a long distance onto the preformed AgNWs (Fig. 2(d)), corresponding to the schematic shown in Fig. 1e. The line width of the silver micromesh was approximately 0.55 μm , which is similar to the bottom width of the sacrificial crack-network template (Fig. 2d). Since the line width of the silver micromesh ($\sim 0.55 \mu\text{m}$) was much larger than the diameter of AgNWs ($\sim 32 \text{ nm}$) (Fig. 2e), the silver micromesh was thought to provide primary paths for electrical current flow, and the AgNWs provide local paths within the empty space of the silver micromesh.

Figure 3 a shows an ED-hierarchical silver network after electrodeposition of silver for 30 s. The ED-hierarchical silver network shows clear AgNW-AgNW welding (Fig. 3b), which can ensure that the AgNWs provide concrete local paths within the empty space of the silver micromesh. The diameter of the AgNWs and the line width of the silver micromesh increased from ~ 32 to $\sim 200 \text{ nm}$ and from 0.55 to 0.72 μm upon electrodeposition for 30 s, respectively (Fig. 3b).

3.2 Optoelectronic Properties

We investigated the optoelectronic properties of the AgNWs alone before discussing the variation in the sheet resistance and the optical transmittance of the hierarchical silver network TCE before and after the electrodeposition of silver. Figure 4a shows the sheet optical transmittance of the AgNWs, corresponding to Fig. 2a. The loss of optical

transmittance of the AgNWs was only $\sim 0.5\%$. The sheet resistance was infinite because the AgNWs did not form a continuous network. These AgNWs did not need to form a continuous network as long as they provided local current paths within the empty space of a silver micromesh in the hierarchical silver networks shown in Figs. 2d and 3b.

An increase in the diameter of the AgNWs and the line width in a silver micromesh upon electrodeposition improved the surface conductance of the hierarchical silver network. However, it also caused a loss of optical transmittance. Figure 4b and c shows the variation in the sheet resistance and the optical transmittance value at 550 nm of the hierarchical silver network TCEs as a function of the silver electrodeposition time. The hierarchical silver network had a sheet resistance of 8.3 Ω/\square and an optical transmittance of 92%. The optical transmittance of the TCEs decreased linearly with the electrodeposition time at a rate as small as $\sim 0.07\%/s$. On the other hand, the sheet resistance significantly decreased upon electrodeposition. Electrodeposition for as little as 10 s dramatically decreased the sheet resistance to 2.3 Ω/\square , with a loss of optical transmittance as low as 1%. After silver electrodeposition for 30 s, the sheet resistance of a hierarchical silver network TCE was only ~ 0.10 times the initial value, with a loss of optical transmittance of only 2.1%. These results demonstrate the effectiveness of the electrodeposition of silver onto a hierarchical network in improving the optoelectronic performance. The optical transmittance of the TCE remained almost constant over the measured wavelength region of the incident light (Fig. 4d) because the light passed through the empty space of the network, and light arriving on the silver surface of the AgNW was mostly reflected.

FoM generally determines the overall optoelectronic performance of a TCE and is expressed as the product of light transmittance and the reciprocal of sheet resistance, as shown below [32].

Fig. 3 a-b Plane views of the hierarchical silver network electrodeposited for 30 s. The diameter of the AgNWs and the line width of the silver micromesh increased from ~ 32 to $\sim 200 \text{ nm}$ and from 0.55 to 0.72 μm after electrodeposition for 30 s, respectively. The ED-hierarchical silver network shows clear AgNW-AgNW welding, as indicated in the circle in Fig. 3b

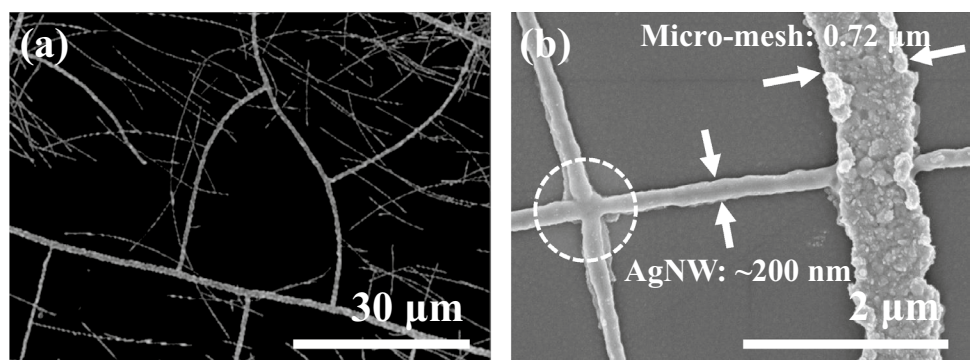


Fig. 4 **a** Optical transmittance of the spin-coated AgNWs, **b** optical transmittance at 550 nm, **c** sheet resistance of the hierarchical silver networks as a function of electrodeposition time, and **d** optical transmittance of the electrodeposited hierarchical silver network with different electrodeposition times

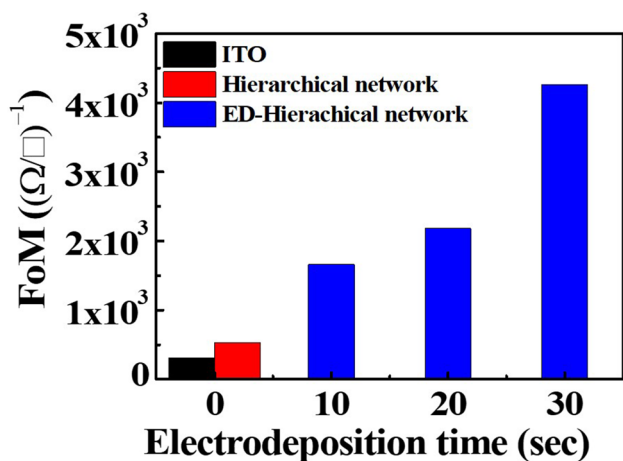
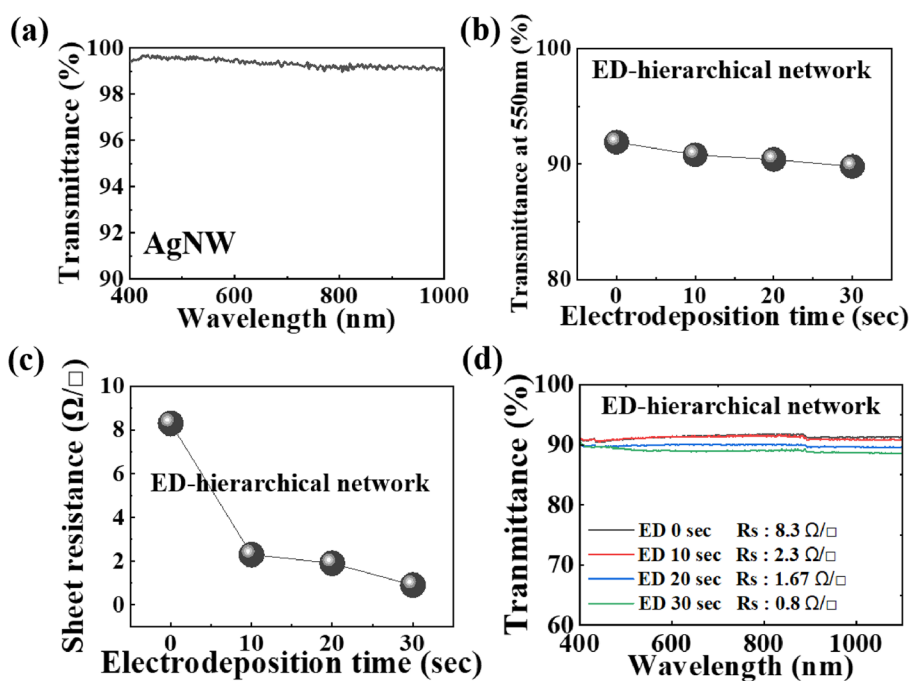


Fig. 5 Figure of merit of ITO and the hierarchical networks as a function of electrodeposition time

$$FoM = \frac{\sigma_{dc}}{\sigma_{opt}} = \frac{188.5}{R_s \times (T_{550}^{-1/2} - 1)} \quad (1)$$

where T_{550} is the optical transmittance at a wavelength of 550 nm and R_s is the sheet resistance.

Figure 5 shows the FoM values of ITO and the hierarchical network as a function of electrodeposition time. Even the nonelectrodeposited hierarchical network ($FoM = 526 (\Omega/\square)^{-1}$) showed a much higher FoM value than ITO ($FoM = 311 (\Omega/\square)^{-1}$). As the electrodeposition time increased from 10, 20 and 30 s, the FoM increased to 1657, 2180, and 4263 $(\Omega/\square)^{-1}$, respectively. That is,

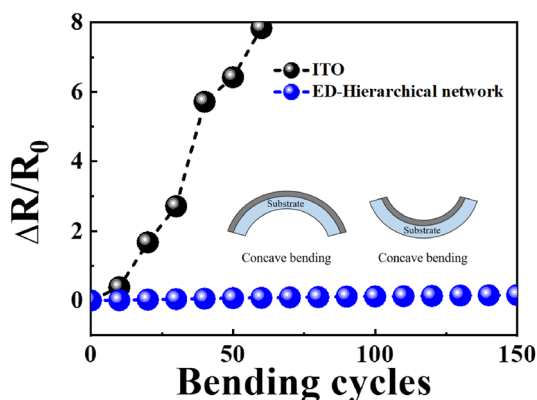


Fig. 6 Comparison of the mechanical flexibility between the ED-hierarchical network and ITO by measuring the resistance change upon repeated convex and concave bending cycles with a radius of curvature of 5 mm

we were able to adjust the optoelectronic performance of a hierarchical silver network by adjusting the electrodeposition time.

3.3 Mechanical Flexibility

One of the most important characteristics to be evaluated for application in flexible or wearable devices is mechanical flexibility. To evaluate the flexibility, a bending test

was performed on ED-hierarchical silver networks formed on a PET substrate. Repetitive convex and concave bending with a radius of curvature of 5 mm was applied, and then the sheet resistance was measured. The sheet resistance of the commercialized ITO film increased to more than 2.5 times the initial value after 30 bending cycles. The ED-hierarchical silver network TCE with the best FoM of $4263(\Omega/\square)^{-1}$ showed resistance changes of 7, 12, and 16% upon 50, 100, and 150 bending cycles, demonstrating that it had much better mechanical flexibility than ITO (Fig. 6).

4 Conclusion

We have demonstrated ED-hierarchical silver network TCEs consisting of AgNWs and a silver micromesh. We were able to significantly improve the optoelectronic performance of a hierarchical network TCE by the electrodeposition of silver. Electrodeposition for as short as 30 s dramatically decreased the sheet resistance of a TCE from 8.3 to 0.8 Ω/\square , with a relatively small loss in the optical transmittance as low as 2.1%. As a result, the FoM of the TCE increased to 8.10 times the initial value and was 13.7 times that of ITO. This significant improvement was achieved by adjusting the dimensions of each AgNW and the silver micromesh by electrodeposition. Furthermore, our ED-hierarchical network TCE showed much better mechanical flexibility than ITO. That is, the TCE showed a much smaller increase in the sheet resistance upon repeated convex and concave bending cycles than ITO. With these advantages, the ED-hierarchical silver network may be one of the best choices as a TCE for flexible or wearable optoelectronic devices.

Acknowledgements This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) that is funded by the Ministry of Science and ICT (Grant No. NRF-RS-2023-00239207, and NRF-2021R1A2C1005815)

Declarations

Conflict of interest We declare that this manuscript is original, has not been published before and is not currently being considered for publication elsewhere. The authors declare that they have no conflicts of interest to disclose that are relevant to the content of this article.

Ethical approval All authors participated in (a) conception and design or analysis and interpretation of the data; (b) drafting the article or revising it critically for important intellectual content; and (c) approval of the final version.

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