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Controlled Formation of Honey Carbon Nanotube Thin Films by Tailoring the Ratio of Admixture Concentration and Annealing Time

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Honey is not only used for human nutrition but is also being studied lately as a promising biomaterial with potential for use as a sustainable dielectric film in devices [1-2]. We report here the characterization of films made of honey mixed with carbon nanotubes (CNTs), aimed at tunnelable properties of the films for memristor device applications. Tuning the honey-CNT film thickness and the concentration of the CNTs will play a vital role in the performance of the device. While the structural characterization of biomaterials including the honey-CNT films is challenging, we demonstrate a strategic combination of several characterization techniques to systematically investigate the effects of different CNT concentrations in honey-based films and the annealing durations on the structures of the films.

The samples were prepared as an admixture of honey, water and CNTs at three concentrations: 0.05, 0.1, and 0.2 mg/mL. Films were spin-coated on fused-quartz and annealed at 90°C for 0 hrs, 4 hrs, and 12 hrs, respectively. Optical microscopy, laser scanning confocal microscopy (Keyence VK-X3000), and Raman spectroscopy (Horiba Jobin Yvon HR800) were used for characterization.

The images obtained by laser confocal microscope in Fig. 1 are 3D heightmaps of the honey-CNT film surfaces. In Fig. 1(a), the pre-anneal honey film with CNT clusters embedded in the film is 1~2 μm thick. The top surface is smooth, with few clusters visible. Fig. 1(b) shows an annealed film (4 hrs) with an average total thickness of ~300nm, exposing CNT clusters encased in honey. The small CNT clusters are represented in cyan or green and larger clusters in yellow or red. The sample that was annealed for 12 hrs is shown in Fig. 1(c), with more small CNT clusters visible, although the reduction of the film thickness is almost the same as the sample annealed for 4 hrs. This suggests that after annealing for 4 hrs, reduction of the honey-CNT film thickness is complete. The Raman spectrum in Fig. 1(d) is from the pre-annealed honey film, with characteristic peaks of the honey. These peaks are also present in Fig. 1(b) showing the honey is undamaged by annealing. The peak at ~430 cm^{-1} is from the fused quartz substrate because the annealed film is thinner. In Fig. 1(f) a large cluster shows the characteristic spectrum of the CNTs (black) while a small cluster (purple) shows characteristic peaks of the honey, CNTs and substrate.

Fig. 2 displays the dark field and bright field optical images, where bright field images show large CNT clusters and film topography, and dark field images show small CNT clusters not visible in the bright field image. Fig 2(a) indicates the film at the lowest CNT concentration (0.05mg/mL) with large clusters and a few small ones. By increasing the CNT concentration to 0.1mg/mL in Fig. 2(b) and 0.2 mg/mL in Fig. 2(c), the overall number of clusters increases without a significant increase to the size of individual clusters. The difference of color in Fig. 2(a) compared to Fig. 2(c) is attributed to a thicker film after annealing when a large number of CNT clusters are present. The results also suggest that the honey film areas with a few or no CNT clusters are thinner when annealed.

Annealing the honey-CNT film decreases the thickness from 1~2 micrometers to ~300nm thick. This makes the embedded CNT clusters visible, and Raman confirms that the chemical composition of the honey film is undamaged by annealing at 90° C. The concentration of CNTs in the honey admixture can be varied to increase the number of clusters without significantly changing the CNT cluster size. By tailoring the admixture concentration and annealing time, improved honey-CNT films are expected [3].

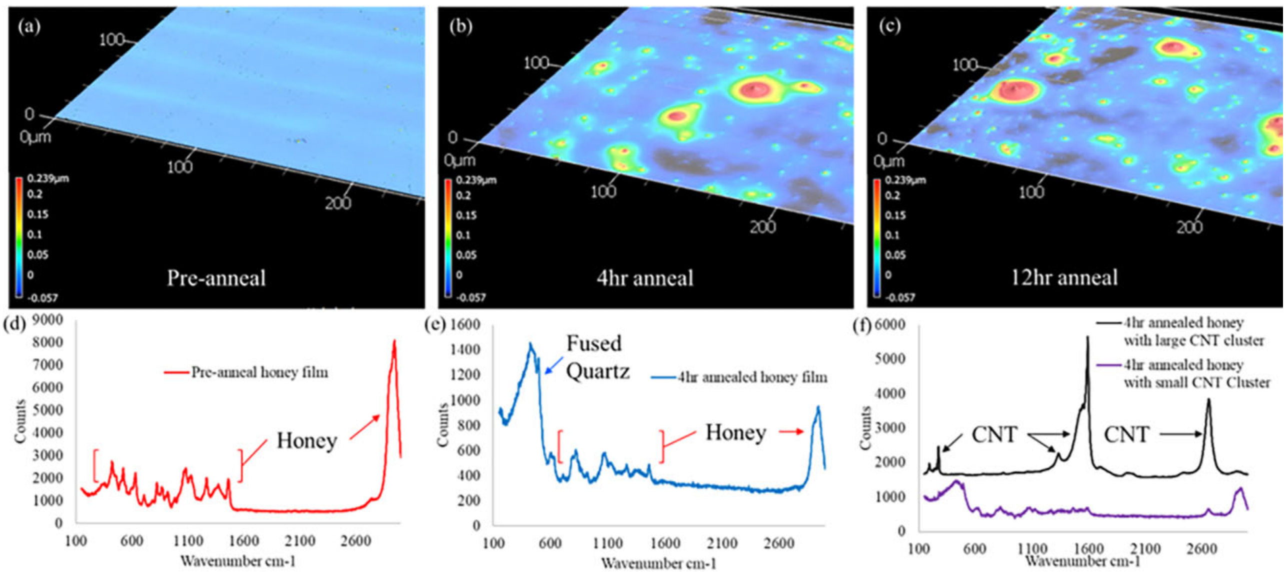

Fig. 1. The annealing effect on the honey-based film. (a) 3D heightmap of top surface of a honey-CNT film without annealing. (b) The top surface after 4 hours of annealing with CNT clusters visible. (c) The same, after 12hrs of annealing. (d) Raman spectrum for a pre-annealed honey film with characteristic honey peaks only. (e) The honey-film Raman spectrum after annealing, where the thinner film results in a peak from the substrate. (f) The spectrum from a large CNT cluster in the annealed film, and the spectrum from a small cluster which has peaks from the substrate, honey, and CNTs.

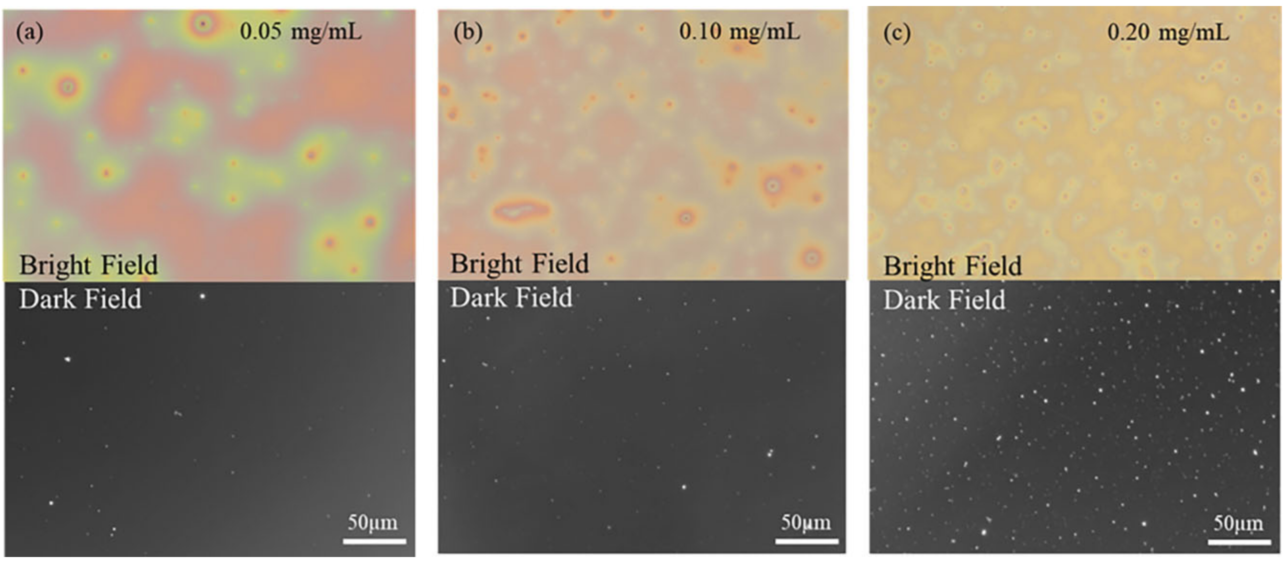

Fig. 2. Optical bright field and dark field images of CNT clusters in the honey-CNT film (12 hr anneal) at different admixture concentrations. (a) 0.05mg/mL, (b) 0.1 mg/mL, and (c) 0.2 mg/mL. As concentration increases, the number of clusters increases but there is no further increase in the cluster size.

References

1. B. Sueoka *et al.*, *Materials Letters* **308** (2022), p. 131169.
2. AA Sivkov *et al.*, *Materials Letters* **271** (2020), p. 127796.
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