doi:10.1017/S1431927622008777
Microsc. Microanal. 28 (Suppl 1), 2022

Transformation during In Situ Heating of Bismuth Antimony Telluride Nanoplates

Xiner Lu[1] and Kai He[1,2*]

[1.] Department of Materials Science and Engineering, Clemson University, Clemson, South Carolina, United States
[2.] Department of Materials Science and Engineering, University of California, Irvine, California, United States
* Corresponding author: kai.he@uci.edu

Bismuth tellurides (Bi_2Te_3) and their derivatives are known as the most promising thermoelectric materials. These two-dimensional (2D) van der Waals layered metal chalcogenides also hold great promise for a variety of applications, such as topological insulators for electronic and optoelectronic devices, electrocatalysts for energy conversion, and anode materials for rechargeable batteries [1-3]. To design these materials with enhanced properties and performance, it is vital to understand the transformation processes of Bi_2Te_3-based compounds for better controlling the dynamics in real devices. Transmission electron microscopy (TEM) is a powerful tool in characterizing the morphology and structure, while in situ TEM enables the capability of observing their evolutions during dynamic processes [4].

Here, we successfully synthesized $(Bi_{0.5}Sb_{0.5})_2Te_3$ hexagonal nanoplates via the hydrothermal approach and conducted TEM and energy-dispersive X-ray spectroscopy (EDS) characterization to examine the shape, structure and elemental information of the nanoplates. Figure 1A shows the as-synthesized $(Bi_{0.5}Sb_{0.5})_2Te_3$ nanoplates with the uniform hexagonal morphology and the core-shell heterostructure. High-resolution TEM (HRTEM) image shown in Figure 1B demonstrates the good single-crystallinity of as-prepared $(Bi_{0.5}Sb_{0.5})_2Te_3$. We further performed high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) imaging and EDS mapping to examine the elemental distribution of the core-shell structured $(Bi_{0.5}Sb_{0.5})_2Te_3$ nanoplates, which confirms that Bi_2Te_3 is located in the core with Sb_2Te_3 in the shell (Figure 1C). It is suggested that the formation of heterostructure is due to the different activity between Bi and Sb in reaction with Te during the crystal growth.

We performed in situ heating experiments on $(Bi_{0.5}Sb_{0.5})_2Te_3$ nanoplates at elevated temperatures to study their structural evolution in response to thermal input under vacuum conditions. Figure 2 shows a series of time-resolved TEM images of a single nanoplate during in situ heating in the temperature ranging from 30°C to 450°C. It is worth noting that we deliberately deposit Pt nanoparticles on the background as location markers to assist the image alignment and drift correction. Because of the high melting point of Pt nanoparticles, they have no effect on the evolution of nanoplates. Through the cross-correlation alignment, we found that the SiN membrane is subject to the nonrigid thermal expansion, inducing uneven bending at elevated temperatures. We found that the nanoplate did not show any obvious change during thermal annealing below 300°C. Starting at 400°C (even before the melting point), the sublimation of Bi_2Te_3 crystal occurred along the same crystallographic direction. The nanoplate was sublimated rapidly in 120 s, retaining the low-energy edge facets. After heating to 450°C, only three residual parts of the Bi_2Te_3 core remained, but the outer Sb_2Te_3 shell was still maintained. In summary, our in situ TEM observations show the real-time evolution of $(Bi_{0.5}Sb_{0.5})_2Te_3$ hexagonal nanoplates during thermal annealing processes. Further investigation on core-shell heterostructure change and the origin of the early sublimation of Bi_2Te_3 crystal will provide fundamental knowledge for development of thermoelectric applications of Bi_2Te_3-based materials and their heterojunctions [5].

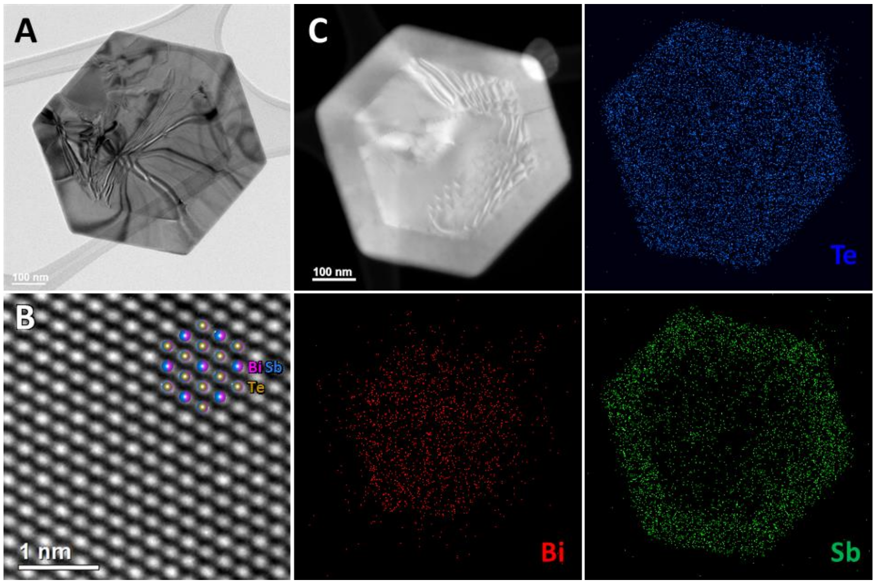

Figure 1. (A) TEM and (B) HRTEM images of as-prepared $(Bi_{0.5}Sb_{0.5})_2Te_3$ nanoplate. (C) HAADF-STEM image and corresponding EDS maps of $(Bi_{0.5}Sb_{0.5})_2Te_3$ heterostructure.

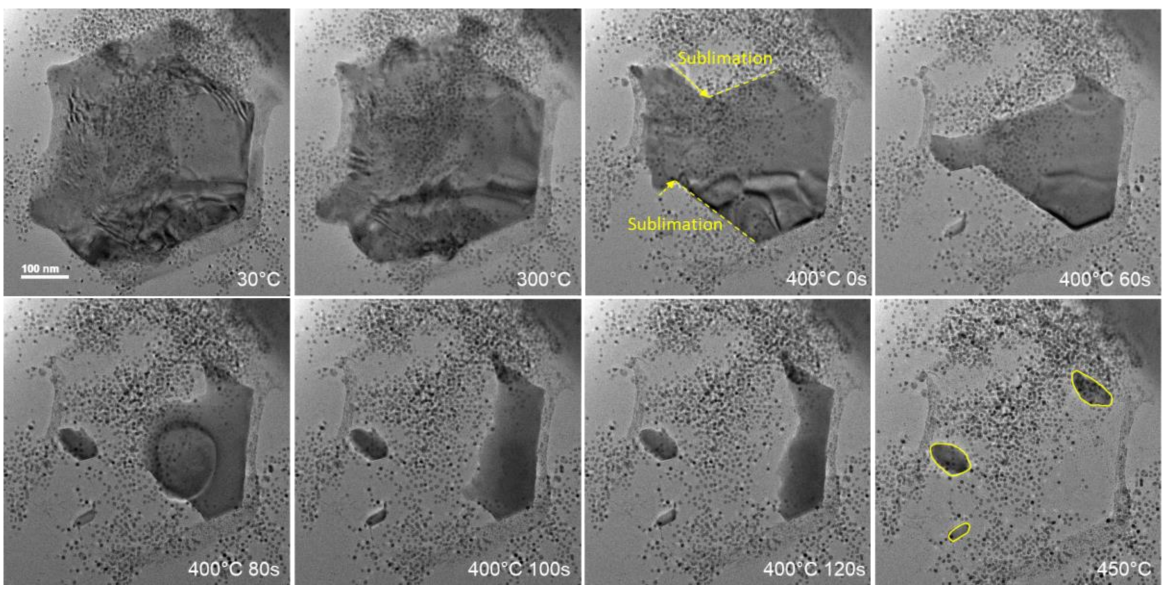

Figure 2. In situ TEM images of a $(Bi_{0.5}Sb_{0.5})_2Te_3$ nanoplate during heating from 30°C to 450°C. The small particles deposit on the background as alignment markers have no thermal impact upon heating.

References:

[1] H. Zhang et al., Nature Physics **5** (2009), 438-442.
[2] D. Kong et al., Energy Environ. Sci **6** (2013), 3553-3558.
[3] J. Cui et al., Matter **4** (2021), 1335-1351.

[4] H. Zheng, X. Lu, K. He, J. Energy Chemistry **68** (2022), 454-493.
[5] This work was supported in part by American Chemical Society PRF Grant No. 62493-DNI10. The authors acknowledge the use of Electron Microscopy Facility at Clemson University.

https://doi.org/10.1017/S1431927622008777 Published online by Cambridge University Press