2258
doi:10.1017/S1431927622008686

Microsc. Microanal. 28 (Suppl 1), 2022
© Microscopy Society of America 2022

Low-dose HRTEM Study of Stacking Fault Structures in Cubic Ice

Yulin Lin[1,2], Tao Zhou[2], Aiwen Lei[1] and Jianguo Wen[2*]

[1] Institute for Advanced Studies (IAS), College of Chemistry and Molecular Sciences, Wuhan University, Wuhan, Hubei, P.R. China

[2] Center for Nanoscale Materials, Argonne National Laboratory, Lemont, Illinois

[*] Corresponding author: jwen@anl.gov

Cubic ice (ice I_c) as metastable solid phases of hexagonal ice (ice I_h) play a critical role in the nucleation of ice [1]. An ice I_c with a fully cubic crystal structure was obtained from ice XVII by Lorenzo Ulivi in 2020 [2]. Nonetheless, the structure of ice I_c in nature is not pure cubic stacking but a mixture with hexagonal stacking and stacking fault due to the small free-energy difference of ice I_c and ice I_h. Although diffraction methods can efficaciously distinguish the two different phases in disorder ice [3], the stacking sequence can be directly obtained by TEM which is able to provide more detail of the ice I_c stacking fault. With the low-dose high resolution TEM (HRTEM), we observed the stacking fault structure from vaper deposition ice I_c in 100 K. By processing the atomic position information in the HRTEM images, the ice stacking sequence can be analyzed automatically. Under the low temperature condition, the cubic stacking is the dominant stacking model but the stacking direction is frequently interrupted and changed by the stacking fault during the growth of ice.

The ice from vapor deposition at lower temperature has higher cubicity. **Fig 1(a)** is the selected area diffraction image of the low temperature deposition ice film formed on an amorphous carbon TEM grid. The diffraction pattern can be indexed by ice I_c, the distinct streaks indicate the existence of stacking faults in the ice film. From the dark field image **Fig 2(b)**, the diffraction contrast and FFT image show that almost every ice grain contains serried stacking faults. The low-dose HRTEM image **Fig 1(c)** (total dose below 10^4 e^-/nm^2) shows that there are two kinds of stacking faults in the ice stacking structure the growth fault and deformation fault. As shown in the structure schematic **Fig 1(d)**, the growth fault is the stacking sequence of cubic stacking from ABC to CBA. Another is the deformation fault the cubic stacking is interrupted by the introduction of two hexagonal sequences.

Using the Laplacian of Gaussian filter, the location of each spot of 50 water molecules layers is calculated and highlighted by the yellow circle as shown in **Fig 2(a)**. There are 7 short ABC stacking, 8 short CBA stacking, and 9 twin boundaries. The starting and ending positions of each stacking segment are marked by a different colored lines in the right box. We further calculated the 200 layers of ice I_c. As shown in the **Fig 2(b)**, the 3 layers of continuous cubic stacking is the most common. Since the stacking fault in ice Ic can also be seen as introducing hexagonal ice stacking, which reduces the cubicity of low temperature growth ice Ic. The accurate stacking structure of ice Ic improve the understanding of the dynamic process of ice formation at low temperature such as out space and stratosphere [4].

https://doi.org/10.1017/S1431927622008686 Published online by Cambridge University Press

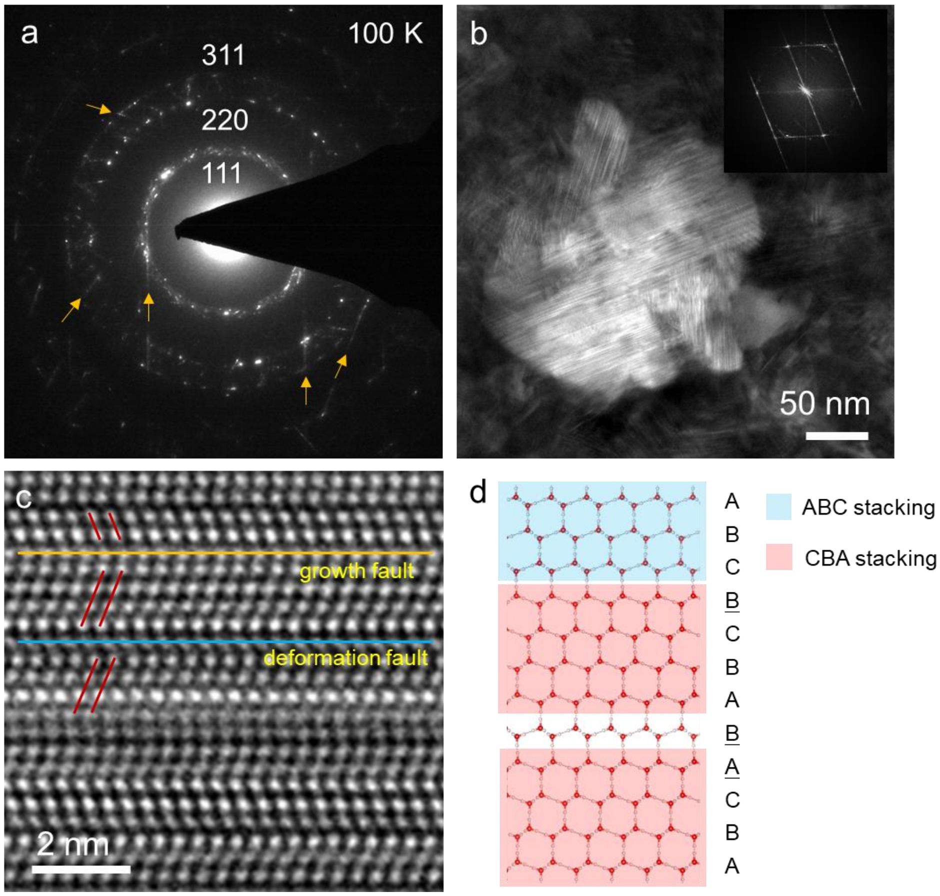

Figure 1. (a) Typical SEAD pattern of low temperature vapor deposition ice. (b) Dark filed image of ice with stacking faults. (c) HRTEM of two kinds of stacking faults. (d) structure schematic

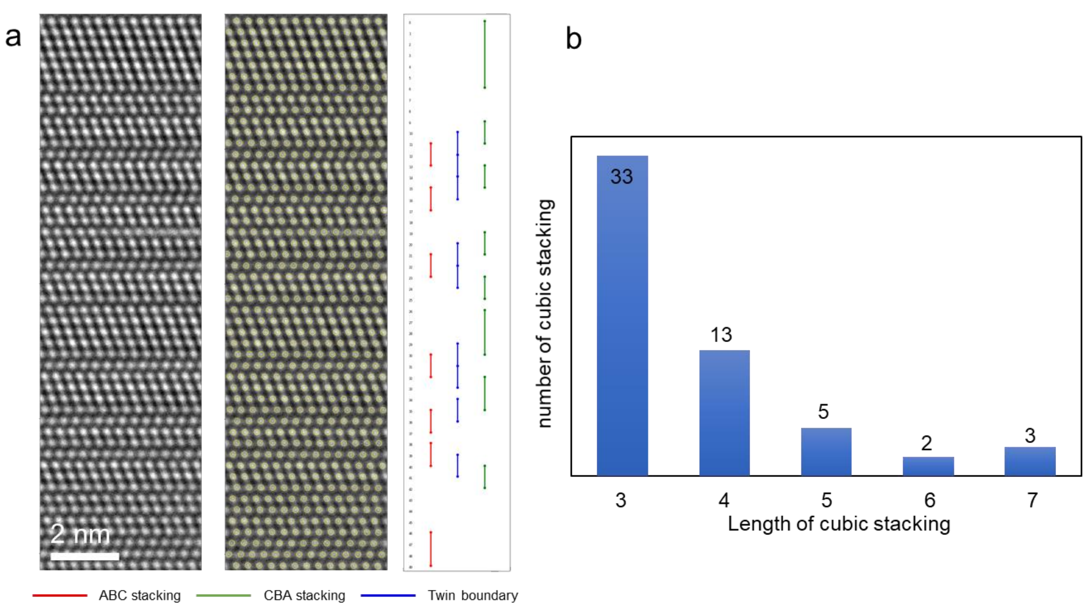

Figure 2. (a) HRTEM of 50 layers ice I_c and the stacking information. (b) Distribution of the cubic stacking length in 200 layers ice I_c.

https://doi.org/10.1017/S1431927622008686 Published online by Cambridge University Press

Reference:

[1] Lupi, Laura, et al. "Role of stacking disorder in ice nucleation." Nature 551.7679 (2017): 218-222.
[2] Del Rosso, Leonardo, et al. "Cubic ice Ic without stacking defects obtained from ice XVII." Nature Materials 19.6 (2020): 663-668.
[3] Kuhs, Werner F., et al. "Extent and relevance of stacking disorder in “ice Ic”." Proceedings of the National Academy of Sciences 109.52 (2012): 21259-21264.
[4] This work was performed at the Center for Nanoscale Materials, a U.S. Department of Energy Office of Science User Facility, and supported by the U.S. Department of Energy, Office of Science, under Contract No. DE-AC02-06CH11357. Yulin Lin is grateful for the Fundamental Research Funds for the Central Universities, China Scholarship Council (No20170627041)

https://doi.org/10.1017/S1431927622008686 Published online by Cambridge University Press