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Imaging the six-membered ring in Si_3N_4 by a Cs-corrected HRTEM

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Silicon nitride (Si_3N_4) ceramics have been intensively investigated for many years largely because it exhibits unique high temperature mechanics and physical properties. Great efforts were made to understand the local compositions, interface structural as well as local bonding behaviors between dopants, i.e the rare earth, and Si_3N_4 grain using various microscopy techniques. Recent breakthroughs in STEM, for instance, Cs-corrected STEM [1], monochromated STEM [2], and associated chemical analysis permit probing the local atomic structure and bonding characteristics with a resolution close to 1Å, which clearly shown the rare earth distributions along the interface. However, the light element nitrogen in the six-membered ring of Si_3N_4 is still difficult to be resolved.

The tremendous progress in HRTEM has made it possible to achieve sub-Å resolution in a 'mid-voltage' microscope through focus-series reconstructions and hardware Cs – correction to extend resolution defined by Scherzer defocus to an expected information limit less than 1Å [3-4]. By exit – plane wave (EPW) reconstruction methods or a combination of one–Ångstrom microscope (OÅM) and reconstructions, researchers closely examined the crystal structure of silicon nitride, where nitrogen signals in six-memebered ring are hardly discernable [5-6].

For the first time we report here on using an image-side corrected microscope to image β-Si_3N_4 six-membered-ring structure in doped- Si_3N_4 ceramics system. With a small negative spherical aberration and small positive defocus aberrations we succeed in demonstrating structural images of Si_3N_4, i.e. Si-N dumbbell with a spacing of 0.88 Å along the [0001] projection. Single N atom columns between the six-membered rings of SiN_4 tetrahedrons are clearly visible as well, as substantiated by intensive image simulations. Based on the contrast details in the Si_3N_4 structural image obtained, the determination of 6_3 -screw axis directions in the crystal becomes possible.

References

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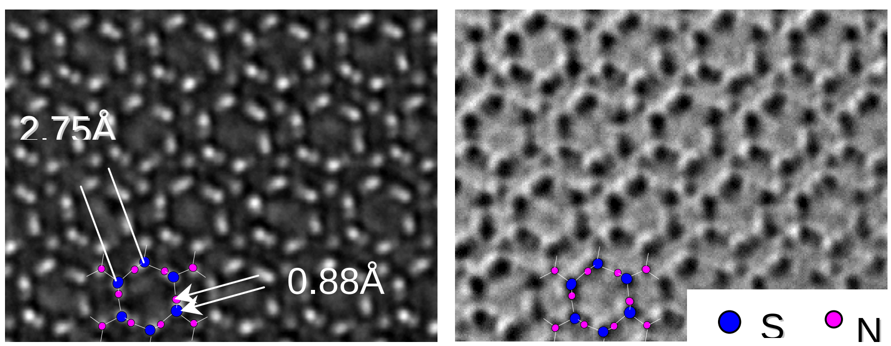

Fig.1 The images were taken at small negative Cs value (-419.1nm) under (a) over focus (+ 3.0 nm), and (b) underfocus (-1.5 nm). Compared with the simulated image, the thickness is about 2.0~ 4.5 nm.