Development of double tilt TEM holder and observation of nanoindentation of thin-filmed silicon

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Silicon is used in a wide range of industries as an important semiconductor material. With the development of nanomaterials, more sophisticated devices are required to be fabricated, so it has become important to understand the properties of silicon at the nanoscale. Since there are various phase transitions in silicon at high temperature or high pressure, and various forces are applied during device processing or use, understanding the structural property transformation of silicon materials at the nanoscale after being subjected to forces becomes a great topic. Nanoindentation experiments are widely used as a material characterization technique to characterize the structural phase transition of silicon. Many indentation experiments on bulk silicon have been reported, but not many nanoindentation experiments have been performed to observe silicon in situ due to the high sample requirements, so more experimental support is still needed to fully understand the changes of nanoscale silicon materials after stress. Therefore, in this thesis, we developed an in situ holder for transmission electron microscopy (TEM) observation and investigated the structural transformation of silicon nanosheets under indentation experiments.

We developed an in situ TEM holder designed to study the structural transition of silicon nanosheets under indentation experiments. First, in order to be able to observe the atomic structure, the sample holder needs to be developed with a double-tilt function. The double-tilt function is provided by a piezoelectric actuator that provides the initial displacement, which is generated by applying a bias voltage and driving a lever structure used to amplify the displacement, which is connected to the sample stage and the amplified displacement drives the stage to complete the rotation. Our in-situ double-tilt TEM holder allows us to obtain atomic TEM images and the applied bias voltage versus the tilt angle of the sample stage, demonstrating that it is stable and effective for TEM observations. An ultrasonic motor was then used to provide displacement in connection with the loading table on which the indenter was mounted, bringing the indenter close to the sample and providing the displacement required for indentation experiments by connecting an additional tubular piezoelectric actuator to the indenter. A linear relationship between the bias voltage applied to the tubular piezoelectric actuator and the indenter displacement can be obtained from the obtained TEM images. It shows that a stable indentation experimental operation can be performed.

We fabricated thin-film silicon samples with a thickness of 20 nm using FIB equipment, and fabricated indenters of different sizes to investigate the effect of indenter size on the indentation results. In situ nanoindentation experiments were performed on the thin-film silicon samples using a TEM mount to investigate the response of the thin-film silicon samples under pressure. Large indenter (R=120 nm) and small indenter (R=15 nm) were used, respectively.

Our results show that during the indentation experiments on the thin-film silicon material using the large indenter, a fan-shaped region of poor crystallinity was created near the residual indentation region, which we believe underwent plastic deformation. We believe that this area has been plastically deformed, and that a surface defect belonging to the group of crystalline planes (113) has occurred. This defect is often reported in electron irradiation experiments on silicon. During indentation experiments on thin-film silicon materials using small indenters, partial rotations of silicon crystallites were produced in the residual indentation region and were confirmed by geometric phase analysis (GPA). At the same time, a region with the presence of Moire pattern was produced near the residual indentation region, which was surrounded by amorphous silicon. Amorphous silicon is believed to be generated during the indentation experimental loading process of β-Sn structured silicon during the rapid unloading process. And the results of the analysis of the Moire pattern region indicate the presence of two high-pressure phases, Si-V, and Si-VI, in this region.

Keywords: in situ TEM, Si- thin film, Nanoindentation, high-pressure phases, defects