

Title	その場TEM観察による2硫化モリブデン膜の歪に依存した物性に関する研究
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Abstract

Study of strain dependence of physical properties in MoS₂ layers

by in situ transmission electron microscopy observation

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Owing to the ultrathin crystal structure, two-dimensional (2D) materials, such as molybdenum disulfide (MoS₂), exhibit unique mechanical and electronic properties that make them promising for the application in electronic devices. Applying strain is an effective method to modify its properties since strain can modulate the crystal structure and electronic structure. However, experimentally investigation on atomic-scale during straining has been rarely reported. Therefore, in this thesis, we developed an in situ stretching holder for the transmission electron microscopy (TEM) observation, and investigated the strain dependence of the physical properties of MoS₂ nanosheets.

We developed an in situ stretching TEM holder, which was designed for the purpose of investigating the strain dependence of 2D materials. The stretching function is performed with the piezo actuator, which generates a displacement by applying the bias voltage and drives the deformation of the titanium plate for stretching the trench in the Si chip. Our in-situ stretching TEM holder enabled us to obtain an atomic TEM image and a linear relationship between the applied bias voltage and gap distance, indicating it is stable and effective for the TEM observation. The displacement step of the holder was measured to be 0.06 nm/mV that is applicable for stretching on atomic scale.

We performed in situ stretching TEM observations of the MoS₂ nanosheet by our stretching TEM holder, for investigating the mechanical response of MoS₂ nanosheet under stretching. The fracture process of a multilayer MoS₂ nanosheet was studied, in which the crack of the sample was propagated along with the zigzag edge layer by layer. Our results suggested that the fracture of MoS₂ nanosheet was an interlayer fracture with a zigzag propagated orientation. Additionally, we observed the rippled structure in the MoS₂ nanosheet, and investigated the structural modulation during stretching. The periodically atomic-contrast change during stretching with a 0.7% tensile strain was revealed, it was attributed to the out-of-plane displacement of the sample during stretching.

We proposed a method for the quantitatively identifying ripple structure of MoS₂ nanosheets on atomic scale. The periodical ripple structure, which formed along the armchair configuration, were observed in the MoS₂ nanosheet. According on the geometry of the rippled structure and the projective feature of TEM, we investigated the relationship between the geometric structure of the ripple and corresponding apparent strain, that the rippled can be identified on atom-scaled with high-resolution TEM (HR-TEM) images. By applying the geometric phase analysis method to the HR-TEM images of ripple structures, we obtained the corresponding apparent strain distribution and proposed that the ripple structure of the MoS₂ layers could be quantitatively estimated on the sub-nanometer scale. We experimentally observed the ripple structure of the MoS₂ layers, and analyzed the corresponding apparent strain. It was found that it was an inclined sinusoidal pattern, which is inclined approximately 7.1° from the plane perpendicular to the incident electron beam, and its period and amplitude were estimated to be 5.5 and 0.3 nm, respectively. Furthermore, the bending model of MoS₂ nanosheet was suggested to be the layer model with no-in-plane distortion, due to the van der Waals interaction between layers of MoS₂ was very weak.

We experimentally investigated the mechanical response of MoS₂ nanosheets under applied strain conditions. The geometric evolution with increasing tensile strain was revealed by using our strain-based analysis on HR-TEM images. It was found that the relationship between the tensile strain and the amplitude/period was not followed the 1/4 power scale law, indicating the continuum mechanics is failed in explaining the bending of 2D materials at the atomic scale. This failure was attributed to the incapable plate model for the bending of the 2D materials. By analyzing the strain dependence of the geometric structure of the ripples, we estimated the Poisson's ratio of MoS₂ nanosheet from our

experimental results during stretching, which showed that the Poisson's ratio increased to 0.57 when the longitudinally tensile strain increased to $\sim 1.6\%$. We proposed an analytical model, which revealed that the nonlinear response of Poisson's ratio with increasing strain was originated from the changing of bond length was not proportional to strain.

In conclusion, the physical properties of atomic-scale MoS₂ layers have been investigated by our homemade TEM holder. Our result revealed the interlayer fracture mechanism, proposed a strain-analysis based method to estimating rippled structure at the subnanometer scale, and investigated the strain dependence of the structure and Poisson's ratio of MoS₂ layers. Our results provide an experimental method for investigating 2D materials under strain conditions, which can contribute to a better understanding of their mechanical properties at the atomic scale and the development of their future applications.

Keywords: in situ TEM, MoS₂, stretching, rippled structure, strain dependence.