Title	透過型電子顕微鏡による多層MoS膜のその場破壊過程観察
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Citation	
Issue Date	2025-09
Type	Thesis or Dissertation
Text version	ETD
URL	http://hdl.handle.net/10119/20097
Rights	
Description	Supervisor: 大島 義文, 先端科学技術研究科, 博士

Abstract

In-situ transmission electron microscopy observation of fracture process in multilayer MoS_2 films

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s2120421

Mechanical strain plays a pivotal role in modulating both the structural and electronic properties of two-dimensional (2D) materials. While small, controlled strains are widely utilized to tune functionalities such as bandgap and carrier mobility, such minor deformations are insufficient for probing the intrinsic mechanical response and failure mechanisms of atomically thin materials. In particular, the understanding of fracture behavior under uniaxial tensile stress at the atomic scale remains incomplete, largely due to limitations in experimental techniques that can simultaneously provide mechanical control and atomic-resolution imaging. In this study, we address these challenges by developing an integrated in situ transmission electron microscopy (TEM) tensile testing platform and applying it to the fracture investigation of few-layer molybdenum disulfide (MoS_2) nanosheets.

Our work begins with a comprehensive assessment of existing experimental approaches and the identification of key limitations in current tensile testing methods for 2D materials. To enable high-stability mechanical testing under TEM, we first constructed a dedicated *in situ* experimental system comprising a JEOL 2100Plus TEM equipped with a custom-built tensile holder. This holder features a piezoelectric actuator and a titanium support plate to enable voltage-controlled tensile displacement with sub-nanometer accuracy.

Recognizing the instability of conventional tensile chips, such as those relying on independent silicon fragments, we designed a new tensile chip architecture based on a monolithic silicon frame integrated with a SiN_x observation window. A key feature of our design is the introduction of long, narrow slits on the SiN_x membrane, which localize strain and improve stress uniformity within the imaging region. Finite element simulations and in situ performance validation confirmed the linear displacement behavior and mechanical robustness of the platform. Additional improvements, such as the implementation of T-shaped termini and

the minimization of out-of-plane movement, further enhanced the reliability of atomic-resolution deformation tracking.

High-quality few-layer MoS_2 samples were prepared via modified mechanical exfoliation using PDMS, followed by a dry-transfer process facilitated by a PPC/PDMS stamp. We adopt contrast-thickness calibration protocol using optical microscopy (OM), atomic force microscopy (AFM), and Raman spectroscopy, allowing for real-time layer number identification during sample manipulation. Moreover, we introduced a practical method for determining the crystallographic zigzag (ZZ) edge orientation of MoS_2 flakes by correlating natural edge morphology with optical and TEM observations. This orientation calibration was critical for exploring the anisotropic fracture behavior of MoS_2 under uniaxial strain.

Using the custom tensile platform, we performed a series of in situ stretching experiments on few-layer MoS_2 nanosheets with controlled orientations and thickness. Our observations revealed a striking deviation from the brittle fracture behavior typically associated with 2D materials. Specifically, samples stretched along directions near the armchair (AC) axis exhibited clear signatures of ductile fracture, including stepped edge morphologies, irregular crack fronts, and sustained compressive strain near crack tips. These features were observed consistently across multiple samples and were supported by GPA-based strain mapping, which revealed localized strain bands and interlayer sliding regions indicative of energy dissipation during deformation.

One of the most remarkable findings of this study is the consistent formation of ~10 nm-wide stepped fracture zones, which emerged during the near AC direction uniaxial tensile failure of few-layer MoS_2. These characteristic fracture regions suggest that crack propagation occurs along distinct paths. The fracture morphology further supports a scenario where cracks propagate preferentially along the ZZ direction, consistent with theoretical predictions of lower fracture energy along this direction. Similar ductile features were also observed in MoS_2 samples stretched along the ZZ orientation at low strain rates, reinforcing the generality of the observed behavior.

In addition to crack initiation and propagation, we identified the role of pre-existing structural features, such as holes and pre-cracks generated during the pre-straining process, as fracture precursors. These defects influenced the stress field and promoted crack nucleation, especially in regions characterized by ripple-like stress distributions formed propagated along two AC directions away from tensile axis. These intersecting ripples exhibit a periodicity of approximately 10 nm, which accounts for the formation of alternating stripes with ~10 nm spacing and varying contrast observed after the pre-strain treatment.

In summary, this study presents a comprehensive framework for investigating fracture mechanics in 2D materials at the atomic scale. By developing a stable, precise, and orientation-calibrated *in situ* TEM tensile testing platform, we successfully visualized the dynamic evolution of fracture processes in few-layer MoS_2. Our findings challenge the conventional view of 2D materials as inherently brittle and instead reveal a ductile failure mechanism characterized by stepwise fracture zones, interlayer sliding, and stress field redistribution. These insights not only deepen our understanding of mechanical failure in 2D systems but also provide practical guidance for designing mechanically robust nanoelectronic and optoelectronic devices based on van der Waals materials.

KEYWORDS: few layers 2H-MoS_2 nanosheet, tensile loading, ductile fracture, real-time *in situ* TEM, atomic resolution

List of publication(s):

1. Xiong, Wei, Xie, Lilin, and Oshima, Yoshifumi. Development of an in-situ TEM method for elucidating tensile fracture processes of 2D materials at atomic scale. ***Jpn. J. Appl. Phys.*** **(2025)** doi:10.35848/1347-4065/ade945.

Presentations:

- International Conference

1. Wei Xiong, Limi Chen, Lilin Xie, Kohei Aso and Yoshifumi Oshima, "Precise measurement of ripple structure of MoS_2 nanoribbon when stretching", 20th International Microscopy Congress (IMC20), 10-15 September 2023, Korea. (Poster)

- Domestic Conference

1. Wei Xiong, Lilin Xie and Yoshifumi Oshima, "Ripple structure of MoS_2 nanosheets evaluated by in situ stretching transmission electron microscopy", 9th International Symposium on Organic and Inorganic Electronic Materials and Related Nanotechnologies, 5-8 June 2023, Ishikawa. (Oral)
2. Wei Xiong, Limi Chen, Lilin Xie, Kohei Aso and Yoshifumi Oshima, "Precise measurement of ripple structure of MoS_2 nanoribbon when stretching", JAIST International symposium of Nano-Materials for Novel Devices, 11-12 January 2024, Ishikawa. (Poster)
3. Wei Xiong, Lilin Xie and Yoshifumi Oshima, "Interlayer fracture of multilayer MoS_2 evaluated by in situ transmission electron microscopy", 10th International Symposium on Organic and Inorganic Electronic Materials and Related Nanotechnologies, 11-14 June 2025, Fukui. (Poster)