

# Nano- to macroscale *in situ* observation of cracking and deformation in structural materials using X-ray microscopy

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**Introduction.** In this study, X-ray microscopy was utilized to reveal the micromechanical mechanism of structural materials. Multiscale and multimodal measurements have been carried out to show the changes in microstructure and/or heterogeneity.

**CFRP.** Carbon-fiber reinforced plastics (CFRP) composites are widely used as weight-reducing structural materials in aerospace and automobile. Crack formation in CFRP under various stress modes was observed *in situ* at the nanoscale by X-ray microscopy with a special micro-testing stage<sup>(1)</sup>. The initiation of voids and cracks clearly arises not only from local stresses but also from two competing nanoscale mechanisms of fiber/plastic interface debonding and in-resin crack initiation (Fig. 1)<sup>(2)</sup>. The initiation and propagation of cracks at the nanoscale are significantly affected by the geometrical distribution of the local fibers.

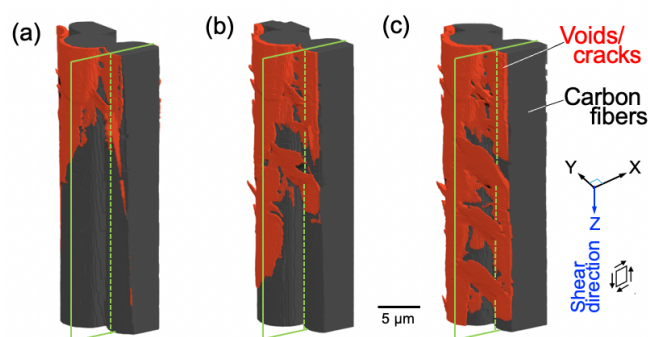


Fig. 1 Segmented image of cracks (red) and carbon fibers (black) in CFRP under mode I+II loading with increasing shear strain from (a) to (c)<sup>(2)</sup>.

**Metal-metal contact surfaces.** The damping of metal contact surfaces considerably influences the dynamic characteristics of machines. The deformation of the metal surface asperity caused by a tangential load was analyzed using the combination of X-ray computed tomography (X-CT) and finite element method (FEM) simulations to understand the damping mechanism. It was suggested that the contribution of the elastic-plastic deformation and slip of asperities is one of the most important key factors<sup>(3)</sup>.

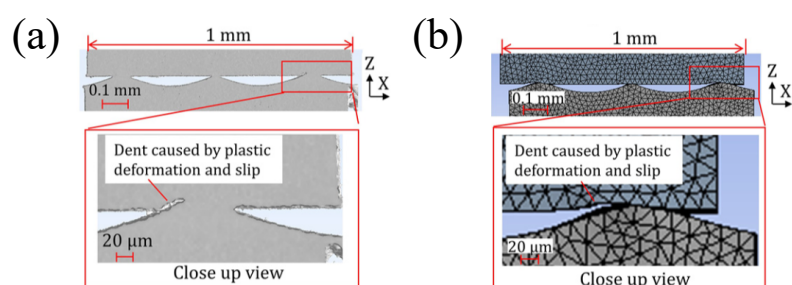


Fig. 2 (left) A cross-sectional image of contact surfaces by X-CT and (right) FEM analysis of the deformation<sup>(3)</sup>.

**Prediction of cracking.** Iron-ore sinter is an essential starting material for iron-making processes. Changes in the iron chemical state in them during reduction reactions were investigated by imaging XAFS (X-ray absorption fine structure) technique<sup>(4)</sup>. It was shown that the reduction progresses heterogeneously due to the complicated microstructure with several phases and the pore network for reductive gas to flow. Cracks were initiated by the volume change caused by reduction. From the image data, crack trigger sites during the reduction were predicted without any knowledge or non-empirically by the combination of applied mathematics (persistent homology) and machine learning<sup>(5,6)</sup>.

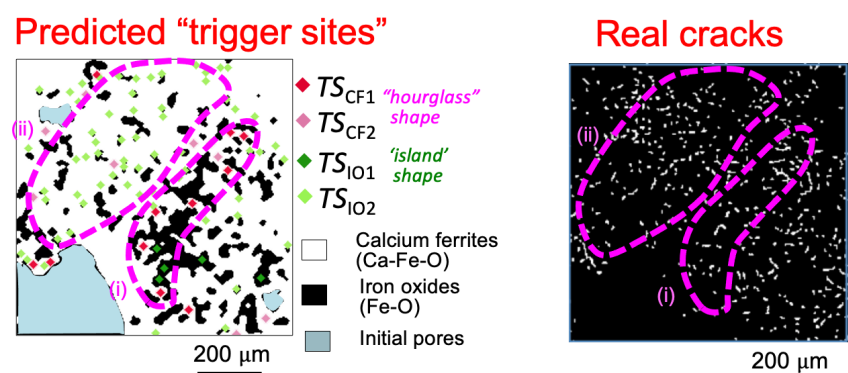


Fig. 3 (left) Predicted locations of trigger sites by the proposed approach and (right) observed cracks' locations.

**Conclusion.** X-ray microscopy can provide essential information for understanding the micromechanical mechanism in various structural materials as far as special experimental techniques are designed and developed accordingly. From a future perspective, we should tackle the development of time resolution of observation and the analysis of big data obtained.

**Acknowledgments.** This work was supported by JST-Mirai JPMJMI20C2 and JPMJMI22C1, and KAKENHI JP19H00834, 20H02028, 20H02046, and 22H05109 by JSPS. Synchrotron radiation experiments were performed with the approval of PF-PAC: 2015S2-002, 2016S2-001, 2019S2-002, and 2022S2-001.

## References.

- (1) Kimura, M. et al., *Sci. Rep.*, **9**, (2019) 19300.
- (2) Kimura, M. et al., *Compos. Sci. Technol.*, **230**, (2022) 109332.
- (3) Kono, D. et al., *CIRP Annals.*, **71** (2022), 485.
- (4) Kimura, M. et al., *Sci. Rep.*, **8**, (2018) 3553.
- (5) Obayashi, I. et al., *J. Appl. Comput. Topol.*, **1**, (2018) 421.
- (6) Obayashi, I. and Kimura, M., *JSIAM Lett.*, **14**, (2022) 151.