S. Sadamatsu1, M. Tanaka, K. Higashida, K. Kaneko, M. Mitsuhara, S. Hata, and M. Honda.
Crack tip dislocations observed by combining scanning transmission electron microscopy and computed tomography
Advanced Materials Research Vols. 89-91 (2010) pp 473-478

Abstract.
Crack tip dislocations and dislocations introduced by three point-bending tests at high temperature are observed by combinating scanning transmission electron microscopy and computed tomography (STEM-CT). Commercially available P type (001) single crystal silicon wafers were employed. A series of STEM image was acquired from -60º to +60º in tilt range with 2º in tilt step. The diffraction vector was maintained close to $g(hkl) = 220$ during the acquisition by adjusting the [110] direction of the sample parallel to the tilt axis of the holder. Reconstructed images of dislocations revealed dislocation structures in three-dimension.

高橋　可昌、坂本　惇司、田中　将己、東田　賢二、野口　博司
Fe-3.2wt.％Si合金単結晶の傾斜疲労き裂先端における繰返し変形のTEM観測
日本機械学会論文集（A編）　76巻762号（2010-2）pp.125-127.

Local cyclic slip behavior around an oblique fatigue crack tip in single-crystalline Fe-3.2wt.%Si alloy is precisely observed by using cross-sectional transmission electron microscopy (TEM). The observation successfully reveals the existence of different dislocation structures (vein, ladder, labyrinth, cell) around the crack tip. The results clearly suggest that the crack growth is strongly related to the formation of dislocation cell structures. Additional fractographic observation suggests that the crack growth, the path of which is found to be the boundary between hard cell region and soft matrix, accompanies the formation of striation pattern along boundary.

Y. Takahashi, M. Tanaka, K. Higashida, K. Yamaguchi, H. Noguchi
An intrinsic effect of hydrogen on cyclic slip deformation around a {1 1 0} fatigue crack in Fe–3.2 wt.% Si alloy
Acta Materialia 58 (2010) pp.1972–1981

Abstract
The effect of gaseous hydrogen on cyclic slip behavior around a fatigue crack tip introduced along the {1 1 0} plane in a Fe–3.2 wt.% Si alloy is precisely investigated by cross-sectional transmission electron microscopy and fractography. The results clearly suggest that the fatigue crack growth rate is promoted by hydrogen, whereas the number of dislocations emitted per load cycle is reduced. In addition, dislocation distribution is

localized around the crack, causing quasi-brittle crack morphology. A sustained load test confirms that no subcritical crack growth caused by cleavage or micro-void coalescence exists along the {1 1 0} plane, which indicates that the observed increase in the fatigue crack growth rate is correlated solely to the intrinsic effect of hydrogen on the cyclic slip-off process around the crack tip.

Kenji Higashida, Masaki Tanaka and Sunao Sadamatsu
Characterization of Crack-tip Dislocations and Their Effects on Materials Fracture
Materials Science Forum Vols. 654-656 (2010) pp. 2307-2311

Abstract.
Three-dimensional structure of crack tip dislocations were investigated by combining scanning transmission electron microscopy (STEM) and electron tomography (ET) in silicon single crystals. P-type (001) silicon single crystals were employed. <110> cracks were introduced from an indent on the (001) surface. The specimen was heated at 873K in order to introduce dislocations at the crack tips. The specimen was thinned to include the crack tip in the foil by an iron milling machine. STEM-ET observation revealed the three-dimensional structure of crack tip dislocations. Their Burgers vectors were determined by using an invisibility criterion. The local stress intensity factor was calculated using the dislocation characters obtained in the observation in this study, indicating that the dislocations observed were mode II shielding type dislocations.

Masaki Tanaka, Masaki Honda, Sunao Sadamatsu and Kenji Higashida
3-D structures of crack-tip dislocations and their shielding effect revealed by electron tomography
Journal of Electron Microscopy 59, (2010) S55–S60.

Abstract
Three-dimensional structures of crack-tip dislocations in silicon crystals have been examined by combining scanning transmission electron microscopy and computed tomography. Cracks were introduced by a Vickers hardness tester at room temperature, and the sample was heated at 823 K for 1 h in order to introduce dislocations around the crack tips. Dislocation segments cut out from loops were observed around the crack tip, the three-dimensional structure of which was characterized by using by electron tomography. Their Burgers vectors including the sings were also determined by oscillating contrasts along dislocations. In order to investigate the effect of the dislocations on fracture behaviours, local stress intensity factor due to one dislocation was calculated, which indicates the dislocations observed were shielding type to increase fracture toughness.

田中　將己，東田　賢二，金子　賢治，光原　昌寿，波多　聰
電子線トモグラフィーによる転位の三次元可視化技術
顕微鏡 Vol. 45, No. 2 (2010) pp.103-108.

要　旨 近年，各種透過電顕法とComputed tomography（CT）とを組み合わせた電子線トモグラフィーが注目を集め結晶性材料の観察へ活発に応用展開されている．その展開の一つとして，回折コントラストを利用した電子線トモグラフィーによる格子欠陥の三次元構造解析が挙げられる．本稿では，回折コントラストを利用した格子欠陥の観察法の中でも特に転位のトモグラフィーに焦点を当て，その手法についての解説を行うと供に，暗視野法，走査透過電子顕微鏡法，超高圧電子顕微鏡法，dual-axis 法を用いた転位のトモグラフィーについても紹介する．

高橋可昌、坂本惇司、田中将己、東田賢ニ、野口博司
Fe-3.2wt. %Si合金単結晶の傾斜疲労き裂先端における繰返し変形におよぼす水素の影響のTEM解析
日本機械学会論文集(A編) 76巻767号(2010-7)pp.1002-1004.

The effect of hydrogen on local cyclic behavior around an oblique fatigue crack tip in single-crystalline Fe-3.2wt3%Si alloy is precisely investigated by using cross-sectional transmission electron microscopy (TEM). The observation successfully reveals that the crack propagation is strongly correlated to the formation of dislocation cell structure in an inert atmosphere, whereas no cell structure is formed around the crack tip in a hydrogen atmosphere.

Sunao Sadamatsu, Masaki Tanaka, Masaki Honda, Kenji Higashida
Crack tip dislocations observed by TEM-tomography in silicon single crystals
Journal of Physics: Conference Series 240 (2010) 012142

Abstract.
3D observations of dislocations at a crack tip were attempted by transmission electron microscopy and computed tomography in order to reveal the 3D structure of dislocations emitted around a crack tip. {011} cracks were introduced into a (001) silicon single crystal wafer by using an indentation method at room temperature. The specimens indented were heated and kept at high temperatures to introduce dislocations from the crack tip. The specimen holder was tilted ±31° by 2° step and dislocation images were taken at every step. The diffraction vector was kept nearly 220 during the tilting operation. The Burgers vectors of the dislocation segments were determined, which included the signs of Burgers vectors. The dislocations observed here were those which accommodate mode II stress intensity around the crack tip. 3D observations using electron tomography reveal these complex crucial processes around the crack tip, which should contribute to understanding the dislocation process improving fracture toughness of crystalline materials.

Yoshimasa Takahashi, Masaki Tanaka, Kenji Higashida, Kazuhiro Yasuda, Syo Matsumura, Hiroshi Noguchi
A combined environmental straining specimen holder for high-voltage electron microscopy
Ultramicroscopy 110(2010) pp.1420-1427.

abstract
A novel specimen holder that enables in situ observation of crack-tip deformation and/or fracture under a controlled environment is developed for a high-voltage electron microscope (HVEM). Awindow-type environmental cell (EC) that incorporates a uniaxial straining apparatus is built into a side-entry-type single-tilt specimen holder. The gas control in EC, straining apparatus design, limited field of view for crack-tip observation, and specimen preparation for the specimen holder are presented in detail. Experimental results successfully demonstrate that the developed specimen holder is quite useful for the dynamic observation of crack-tip deformation and/or fracture subjected to a hostile environment, such as hydrogen gas.

Yoshimasa Takahashi, Junji Sakamoto, Masaki Tanaka, Kenji Higashida and Hiroshi Noguchi.
Characterization of dislocation structures around a mixed-mode fatigue crack tip in a single-crystalline iron–silicon alloy
Scripta Materialia 64(2011)pp.157-160.

Dislocation structures around a mixed-mode fatigue crack tip introduced into a single-crystalline iron–silicon alloy is characterized by cross-sectional electron backscatter diffraction and high-voltage electron microscopy. The results show that the crack growth is preceded by the formation of a cell band and the crack grows along cell boundaries. Crack growth rate and width of the cell band are constant despite the monotonic increase in stress intensity, which is anomalous in terms of the conventional fracture mechanics concept.