IN-SITU TEM STUDY ON THE COLLAPSE PROCESS OF SFT DURING PLASTIC DEFORMATION—Y. Matsukawa, Yu. N. Osetsky, R. E. Stoller, and S. J. Zinkle (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this research is to obtain the information about the collapse process of stacking fault tetrahedra (SFTs) during plastic deformation by in-situ straining experiments in a transmission electron microscope (TEM).

SUMMARY

Dynamic observation of the collapse process of SFTs during plastic deformation in quenched gold was carried out using a TEM. SFTs having perfect pyramid structure were collapsed by direct interaction with gliding screw dislocations. Although Wirth et al. pointed out that truncation of SFT before interaction with gliding dislocations was an important factor for the SFT collapse [1], the present experimental results clearly showed that truncation of SFT was not a crucial factor for the collapse mechanism. An SFT collapsed in a unique way: only the base portion divided by the gliding plane of dislocation annihilated, while the apex portion remained intact. Judging from the fact that similar collapse process was observed in recent computer simulations using molecular dynamics codes [2], this collapse process is a major one. However, it is still unclear whether this is only one process for SFT collapse, because this collapse process leaves a small SFT as a remnant, whereas SFTs are completely swept out in the dislocation channels actually observed in the deformation microstructure of irradiated fcc metals [3-12]. Further research works will be required for fully understanding of dislocation channeling, which is believed to be a key for ductility reduction of irradiated metals [3].

PROGRESS AND STATUS

Introduction

Recent improvements in resolution of transmission electron microscope (TEM) have established that small defect clusters (≤ 2nm) in the microstructure of neutron irradiated pure fcc metals, so called ‘black dots’, are in many cases stacking fault tetrahedra (SFTs) [13-15]. Thus, there is substantial

interest in the interaction of SFT with gliding dislocations as a key in atomistic scale mechanism for variation of mechanical properties of fcc metals in nuclear reactor environments.
The SFT is a vacancy-type defect cluster having a complex crystallographic geometry: the tetrahedral outer shape consists of intrinsic stacking faults with displacement vectors R=1/12<111> on four crystallographically equivalent {111} planes, and the edges of the tetrahedron are composed of sessile stair-rod dislocations with Burgers vector of b=1/6<110> [16]. The complex structure indicates that SFTs are highly stable under shear stress. However, defect-free regions (dislocation channels) are created locally as deformation progresses in both quenched and irradiated metals [4-12]. This has led to great interest in resolving how SFTs annihilate during plastic deformation and dislocation channels are created.

There are two proposed annihilation mechanisms: (1) annihilation due to large stress fields, associated with localized deformation, and (2) annihilation by direct interaction with individual dislocations, which would operate in a heterogeneous manner and lead to formation of defect-free slip bands. Regarding the former idea, Hiratani et al. investigated the stability of a SFT by numerical analysis based on elasticity theory for the stress field of a glide dislocation [17]. Their calculation showed that the stress field from a single dislocation could never annihilate an SFT. However, in the situation where there is localized deformation, large numbers of dislocations would form a dislocation pile-up, resulting in high stress field at the pile-up front, which could favor destabilization of SFT. Further investigation is necessary to determine the importance of this mechanism. Regarding the second idea, Kimura and Maddin approached this issue forty years ago from the viewpoint of dislocation reactions [18-19]. However, the validity of this model remains unclear because of a lack of experimental evidence.

Recent molecular dynamics computer simulations have investigated the dynamic interaction of a SFT with gliding dislocations [1, 2]. Wirth et al. showed that an SFT remained intact even after multiple (up to 6) interactions with a gliding dislocation [1]. However, an overlapping truncated SFT configuration was annihilated by interactions with a single gliding edge dislocation. These authors concluded that the perfection of the SFT, i.e. perfectly formed or truncated, is an important factor for SFT collapse. However, more recently, Osetsky et al. showed an SFT having perfect pyramid shape collapsed by gliding dislocations [2]. Based on the information obtained by these computer simulations, the crucial factor for SFT collapse has been discussed. Against this background, assessments by experiments using real materials have been expected.

The most direct experimental method for investigation of the SFT annihilation involves in-situ straining experiments using a transmission electron microscope (TEM). There are several reports based on in-situ straining experiments performed on ion and/or neutron irradiated fcc metals [8-12]. However, the size of SFT introduced by ion/neutron irradiation is small (< 2nm), comparable to the resolution limit of TEM in the diffraction contrast imaging techniques [20]. As a result, few experimental data are available to identify the detailed interaction process with gliding dislocations leading to collapse of SFTs.

In the present study, an in-situ straining experiment has been carried out on SFTs introduced into gold by quenching. SFT size can be controlled relatively easily in quenching: from more than 100nm to less than 2nm [21]. Information obtained by direct observation allows insight of the annihilation process of SFT.

Experimental Procedure

SFTs were introduced into 99.9975% purity gold specimens, whose thickness was 100 μm, by quenching from 1273K in an open vertical furnace to 233K in $CaCl_2$ solution. As addressed elsewhere, a key parameter controlling the size of SFT was annealing time at 1273K. The specimens in the present experiments were all kept at 1273K for 1hr. Since vacancies are mobile at room temperature in gold, the thermal history after quenching also affects the formation behavior of SFT [21]. The specimens were kept at 233K for 1hr, then at 298K for 2hr, and finally at 373K for 1hr, before electro-polishing. Tensile specimens for in-situ straining experiments had a rectangular shape (10 mm x 2.5 mm x 100 μm), and the central section was electro-polished by the twin jet method. Electrolytic solution for the polishing was KCN 67 g/l water solution, and the polishing temperature was 276K. In-situ straining experiments were carried out at room temperature. The electron microscope accelerating voltage was 200kV, which introduces negligible irradiation damage in gold. An FEI Tecnai20 TEM with Twin pole piece was used for the in-situ straining tests, with a GATAN Model 671 single tilt cooling straining stage. The crosshead speed of the straining holder is variable ranging from 0.01 to 1μm/s. Motion pictures of the in-situ straining were captured with a GATAN Model 622 camera, at a frame rate of 30 frames/s, recorded on DV tapes, and then computer processed.

Results

The SFTs introduced by quenching are essentially perfect pyramid shape without any truncation. Although some truncated SFTs are also present in Fig. 1(A2, A3, B2), their truncation style is entirely different from the style predicted by Silcox and Hirsch [22]: the truncated SFT in their model is missing the top portion of the tetrahedron, while the truncated SFTs in Fig. 1 are all missing portions of their edge region. The truncation of the SFTs in Fig. 1 is simply caused by intersection of the specimen surface. We have not observed any cases of truncated SFTs except those created by intersection with the foil surface during specimen preparation by electro-polishing. SFTs grow by absorbing vacancies and forming a so-called V-ledge on the stacking fault planes [23-24]. Thus, nearly all SFTs should inevitably have ledges of one atomic layer thickness on their stacking fault planes. This statement is supported by high-resolution images shown by Ajita et al [16]. However, possession of atomic-layer ledges is an entirely different issue from the truncation where the top

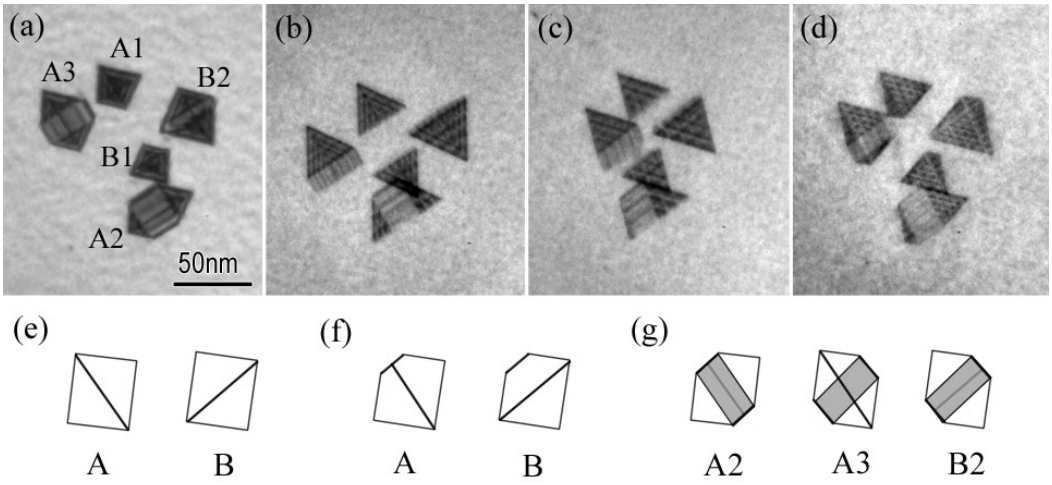

Fig. 1. Typical example of SFTs in quenched gold. Two types of crystallographically equivalent SFTs are indexed as A and B in these figures. A1 and B1 have perfect pyramid shape; however, the others are truncated. The truncation is simply due to intersection by foil surface. A2 and B2 are intersected by a surface, and A3 by another surface. All of the SFTs in quenched gold have essentially perfect pyramid structure. (a) SFTs observed from near [001] zone axis. (b)(c)(d) Observed from directions tilted from (a). (e)(f)(g) Schematic diagrams of SFTs in various conditions observed from [001] zone axis. (e) Image of SFTs without any truncation. (f) Image of SFTs missing a top portion. (g) Image of SFTs missing an edge portion.

portion of the SFT is missing. It is unclear whether an SFT having ledges can be called ‘perfect’ in the strict sense; however, in what follows these SFTs are called ‘perfect’ in order to distinguish them from truncated SFTs.

Figure 2 shows the typical collapse process of an SFT by direct interaction with gliding dislocations. Judging from the interaction behavior with the gliding dislocation, both ends of which intersect the foil surfaces, it is clear that the SFT is located at the center of the foil thickness direction. Therefore, this is a perfect SFT without being truncated by foil surface. SFTs collapsed into a smaller SFT. This collapse process is different from any models previously proposed by Kimura, because in those models such small SFT cannot be the remnant of collapse [18-19]. The gliding dislocation intersected the SFT does not have any remarkable jog at the moment of SFT collapse, indicating that the dislocation did not cross-slip on the original SFT as assumed in Kimura’s model for screw dislocation, but simply intersected the SFT. The geometry analysis revealed that the smaller SFT corresponds to an apex portion of the original SFT: in other words, only the base portion divided by intersection with the dislocation annihilated, while the top portion intact [25-26]. After the SFT collapse, a vacancy cluster having ambiguous shape was created on the intersected dislocation, then the vacancy cluster developed into a super jog. Judging from that dislocation cross-slip is not involved in the collapse process, this type of SFT collapse may be induced irrespective of dislocation-type: not only by screw but also by edge [26]. Although the SFT was collapsed at the intersection with third dislocation in Fig. 2, the number of intersection would not be the primary factor dominating the collapse process: actually, it was often observed that an SFT was collapsed at the intersection with first dislocation [3, 26].

When an SFT is intersected by a dislocation gliding on the (111), steps are created on the two of three stacking fault planes, which were actually intersected, depending on the Burgers vector of the intersecting dislocation, as schematically shown in Fig. 3. The steps on stacking faults are traditionally called V-ledge and I-ledge [27]: the Burgers vector of the dislocation determines which type of ledge is created on which stacking faults. Fig. 4 is the schematic diagram showing variation of atom configuration for V-ledge and I-ledge. At V-ledge, exactly the same stacking sequence of (111) planes is created as the SFT bottom. However, at I-ledge, a quite unusual atom configuration appears: the atoms on adjacent (111) planes are overlapping each other.

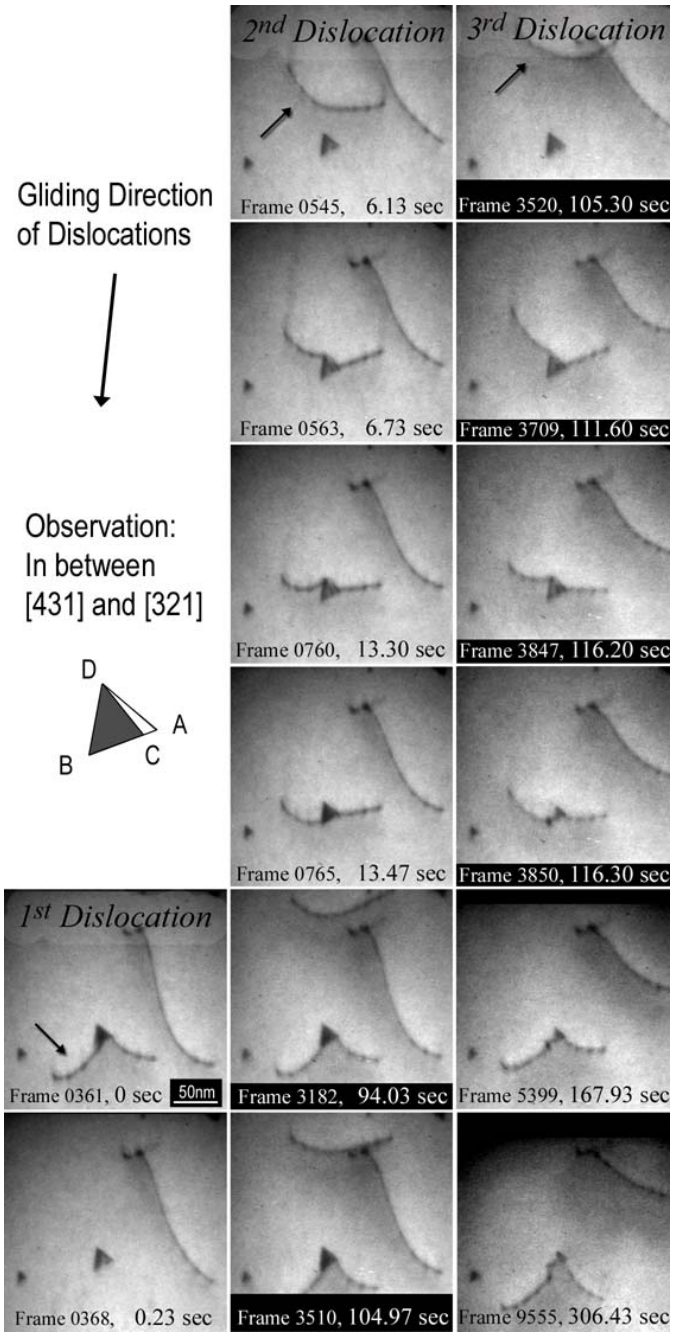

Fig. 2. An example of SFT collapse by gliding dislocations. The SFT collapsed on interaction with the third dislocation. A small SFT and a vacancy cluster were produced by the SFT collapse. The vacancy cluster gradually developed into a super jog on the dislocation collapsed the original SFT. Judging from the interaction behavior with the SFT, dislocations were gliding on $(\bar{1}\,\bar{1}\,1)$, i.e. parallel to ABD. The SFT edge length was 27nm, dislocation length was 121nm, and specimen foil thickness was 99nm.

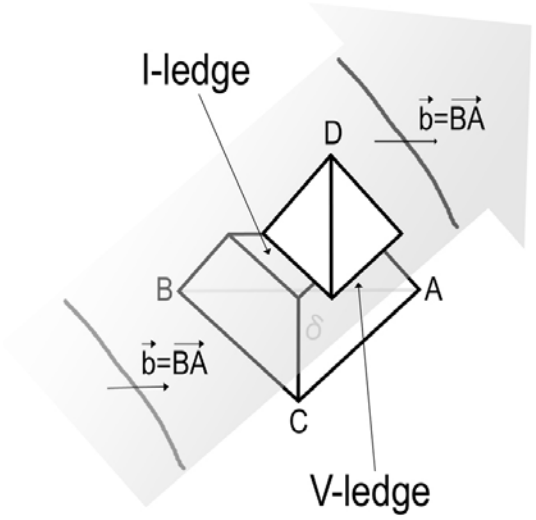

Fig. 3. Intersection by a dislocation produces ledges on two of three stacking fault planes intersected by the dislocation.

This simple assessment of atom configuration in consideration of dislocation's Burgers vector provides a key to understand the SFT collapse process observed in experiments: those overlapping atoms have strong repulsive force that may be the driving force for the SFT collapse. Those overlapping atoms will displace to somewhere to relax the dense configuration. However, at the place where the atom displaced, another dense configuration is created by this displacement. This chain reaction of displacements continues until the displacement finally creates energetically favorable configuration such as Frank loop. An important point in this chain reaction is, judging from the geometry of an I-ledge, that the chain reaction inevitably collapses only the base portion of the SFT. The detail mechanism will be discussed elsewhere [28].

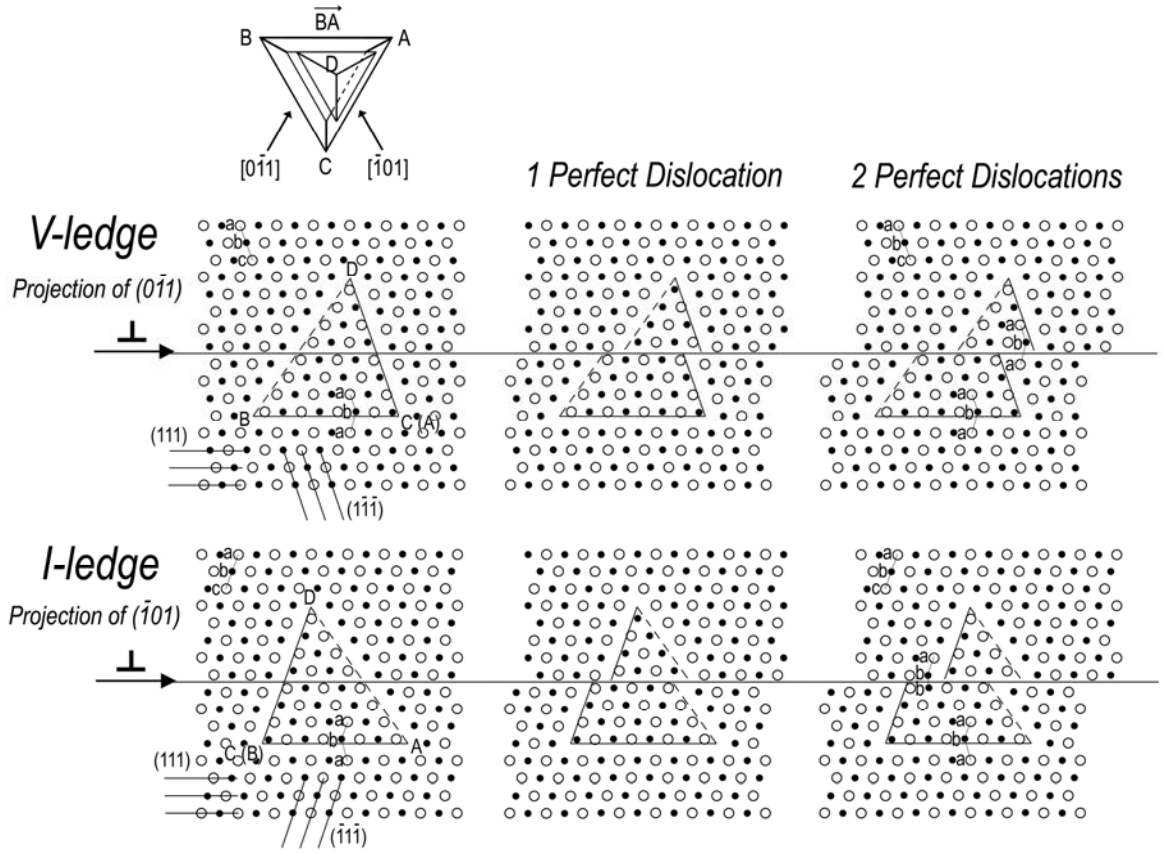

Fig. 4. Variation of atom configuration by intersection with perfect dislocations gliding on a (111) plane, projected on $(0\bar{1}1)$ for V-ledge, and on $(\bar{1}01)$ for I-ledge, respectively. Burgers vector of the intersecting perfect dislocations is 1/2 $\left[1\bar{1}0\right]$. Filled circles represent atoms on a plane 1/4 $\left[\bar{1}01\right]$ below the open circles.

Acknowledgments

This research was sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-ACO5-00OR22725, with UT-Battelle, LLC. We are grateful to Kathy Yarborough for specimen preparation, Cecil Carmichael for furnace operation, and Dr. Neal Evans for maintenance of TEM. Also, we thank Drs. Stas Golubov, Thak Sang Byun, Nao Hashimoto, Joe Horton, and Jim Bentley at ORNL, and Dr. Kazuto Arakawa at Shimane University for valuable comments.

References

[1] B. D. Wirth, V. V. Bulatov, and T. Diaz de la Rubia, J. Eng. Mat. Tech. 124 (2002) 329-334.

[2] Yu. N. Osetsky, personal communication.

[3] S. J. Zinkle, N. Hashimoto, Y. Matsukawa, R. E. Stoller, and Yu. N. Osetsky, MRS Symp. Proc. 792, Eds., L. M. Wang et al. (Materials Research Society, Warrendale Pa., 2004), R1.1.1-1.1.10.

[4] M. Meshii and J. W. Kauffman, Acta Met. 7 (1959) 180-186.

[5] M. S. Bapna, T. Mori, and M. Meshii, Phil. Mag. 17 (1968) 177-184.

[6] A. Okada, K. Kanao, T. Yoshiie, and S. Kojima, Trans. JIM 30, No. 4 (1989) 265-272.

[7] S. Yoshida, M. Kiritani, Y. Deguchi, and N. Kamigaki, Trans. JIM 9 (supplement) (1968) 83-89.

[8] J. V. Sharp, Phil. Mag. 16 (1967) 77-96.

[9] H. Saka, K. Noda, K. Matsumoto, and T. Imura, Proceedings of 4th International Conference on High Voltage Electron Microscopy, Toulouse (1975) 325-327.

[10] K. Noda, H. Saka, K. Shiraishi, H. Yoshida, and T. Imura, Proceedings of 5th International Conference on High Voltage Electron Microscopy, Kyoto (1977) 403-406.

[11] E. Johnson and P. B. Hirsch, Phil. Mag. A 43, No. 1 (1981) 157-170.

[12] J. S. Robach, I. M. Robertson, B. D. Wirth, and A. Arsenlis, Phil. Mag. 83, No. 8 (2003) 955-967.

[13] M. Kiritani, Mat. Chem. Phys. 50 (1997) 133-138.

[14] S. J. Zinkle and L. L. Snead, J. Nucl. Mater. 225 (1995) 123-131.

[15] M. Victoria, N. Baluc, C. Bailat, Y. Dai, M. I. Luppo, R. Schaublin, and B. N. Singh, J. Nucl. Mater. 276 (2000) 114-122.

[16] N. Ajita, H. Hashimoto, and Y. Takaki, Phys. Stat. Sol. (a) 87 (1985) 235-252.

[17] M. Hiratani, H. M. Zbib, and B. D. Wirth, Phil. Mag. A 82, No. 14 (2002) 2709-2735.

[18] H. Kimura and R. Maddin, *Lattice Defects in Quenched Metals*, Proceedings of an International Conference, Argonne National Laboratory, June 15-17, 1964, Ed., R. Cotterill (New York: Academic Press, 1965) 319-386.

[19] P. B. Hirsch, *Vacancies '76*, Proceedings of a Conference on Point Defect Behaviour and Diffusional Process, September 13-16, 1976, Ed., R. E. Smallman and J. E. Harris (London: The Metals Society, 1977) 95-107.

[20] Y. Satoh, H. Taoka, S. Kojima, T. Yoshiie, and M. Kiritani, Phil. Mag. A 70, No. 5 (1994) 869-891.

[21] A. Yoshinaka, Y. Shimomura, M. Kiritani, and S. Yoshida, Japanese J. Appl. Phys. 7, No. 7, (1968) 709-714.

[22] J. Silcox and P. B. Hirsch, Phil. Mag. 4 (1959) 72-89.

[23] M. De Jong and J. S. Koehler, Phys. Rev. 129 (1963) 49-61.

[24] D. Kuhlmann-Wilsdorf, Acta Met. 13 (1965) 257-270.

[25] Y. Matsukawa and S. J. Zinkle, J. Nucl. Mater. (2004) in press.

[26] Y. Matsukawa and S. J. Zinkle, Dynamic Observation of the Collapse of a Stacking Fault Tetrahedron by Gliding Dislocations., Phil. Mag., to be submitted.

[27] D. Kuhlmann-Wilsdorf, Acta Met. 13 (1965) 257-269.

[28] Y. Matsukawa and S. J. Zinkle, The Driving Force for the Collapse of a Stacking Fault Tetrahedron due to Intersection by Gliding Dislocations., Phil. Mag., to be submitted.