Available online at www.sciencedirect.com

SCIENCE DIRECT®

Journal of Nuclear Materials 329–333 (2004) 919–923

journal of nuclear materials

www.elsevier.com/locate/jnucmat

Dynamic observation of the collapse process of a stacking fault tetrahedron by moving dislocations

Yoshitaka Matsukawa *, Steven J. Zinkle

Metals and Ceramics Division, Oak Ridge National Laboratory, Bethel Valley Road, P.O. Box 2008, Oak Ridge, TN 37831-6138, USA

Abstract

Dynamic observation of the microstructure of quenched gold during deformation in a transmission electron microscope revealed that stacking fault tetrahedra (SFTs) having perfect pyramid structure were collapsed by direct interaction with moving screw dislocations. Although a recent molecular dynamics computer simulation study found that truncation of SFT before interaction with moving dislocations is a necessary condition for the SFT collapse, the present experimental results clearly show that truncation of SFT is not a crucial factor for the collapse mechanism.

1. Background

Recent improvements in resolution of transmission electron microscope (TEM) have established that small defect clusters ($\leqslant 2$ nm) in the microstructure of neutron irradiated pure fcc metals, so-called 'black dots', are in many cases stacking fault tetrahedra (SFTs) [1–3]. Thus, there is substantial interest in the interaction of SFT with moving 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 $R = 1/12\langle 1\,1\,1\rangle$ on four crystallographically equivalent $\{1\,1\,1\}$ planes, and the edges of the tetrahedron are composed of sessile stair-rod dislocations with Burgers vector of $b = 1/6\langle 1\,1\,0\rangle$ [4]. 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 [5–12]. 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 [13]. 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 40 years ago from the viewpoint of dislocation reactions [14,15]. 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 moving dislocations [16,17]. Wirth et al. showed that an SFT remained intact even after multiple (up to 6) interactions with a moving dislocation [16]. However, an

* Corresponding author. Tel.: +1-865 574 9295; fax: +1-865 574 0641.

E-mail address: ym2@ornl.gov (Y. Matsukawa).

0022-3115/$ - see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2004.04.069

overlapping truncated SFT configuration was annihilated by interactions with a single moving edge dislocation. These authors concluded that the perfection of the SFT, i.e., perfectly formed or truncated, is an important factor for SFT collapse.

The most direct experimental method for investigation of the SFT annihilation involves in situ straining experiments using a transmission electron microscope (TEM). Recently. Robach et al. confirmed the annihilation of SFTs by moving dislocations in copper containing small SFTs produced by ion irradiation [18]. There are several other reports based on in situ straining experiments performed on ion and/or neutron irradiated fcc metals [9–12]. However, the size of SFT introduced by ion/neutron irradiation is small (<2 nm), comparable to the resolution limit of TEM in the diffraction contrast imaging techniques [18]. As a result, few experimental data are available to identify the detailed interaction process with moving 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 100 nm to less than 2 nm [20]. Information obtained by direct observation allows insight of the annihilation process of SFT.

2. Experimental procedure

SFTs were introduced into 99.9975% purity gold specimens, whose thickness was 100 μm, by quenching from 1273 K in an open vertical furnace to 233 K in $CaCl_2$ solution. As addressed elsewhere, a key parameter controlling the size of SFT was annealing time at 1273 K [20]. The specimens in the present experiments were all kept at 1273 K for 1 h. Since vacancies are mobile at room temperature in gold, the thermal history after quenching also affects the formation behavior of SFT [19]. The specimens were kept at 233 K for 1 h, then at 298 K for 2 h and finally at 373 K for 1 h, before electropolishing. Tensile specimens for in situ straining experiments had a rectangular shape (10 mm × 2.5 mm × 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 276 K. In situ straining experiments were carried out at room temperature. The electron microscope accelerating voltage was 200 kV, 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 1 μm/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.

3. Results and discussion

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 [21]: the truncated SFT in their model is missing the top portion

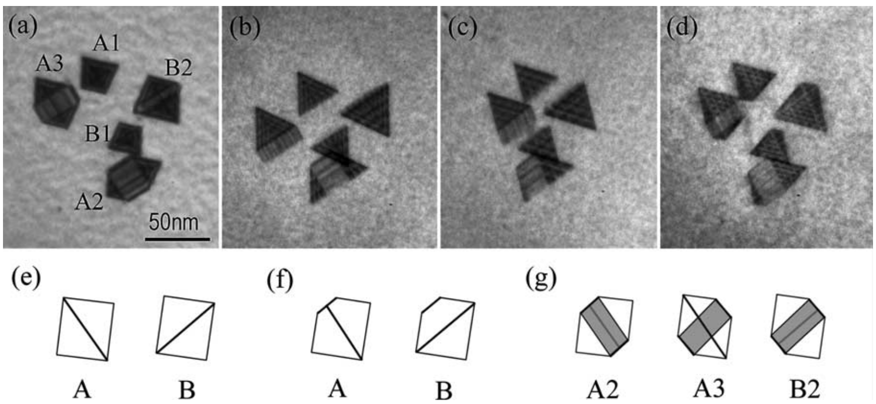

Fig. 1. Typical example of SFTs in quenched gold. Two types of crystallographically equivalent SFTs are indexed as A and B in these figures. A1 and B1 have perfect pyramid shape; however, the others are truncated. The truncation is simply due to intersection by foil surface. A2 and B2 are intersected 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 [0 0 1] zone axis. (b–d) Observed from directions tilted from (a). (e–g) Schematic diagrams of SFTs in various conditions observed from [0 0 1] 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.

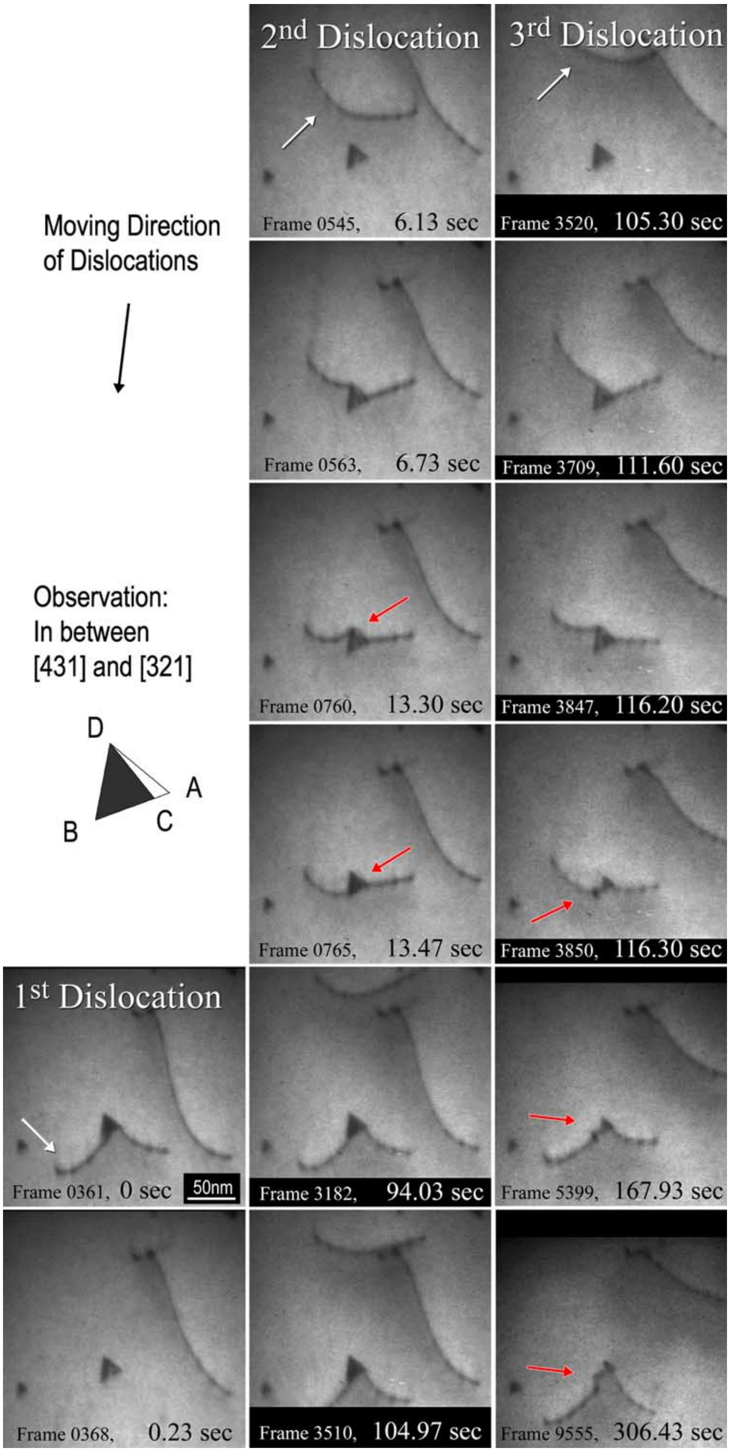

Fig. 2. Typical example of the collapse process of SFT by moving dislocations. Observation was made in an orientation exactly the middle in between [4 3 1] and [3 2 1]. Stacking fault contrast changes when a moving dislocation passes through an SFT, indicating that the moving dislocation pass through without leaving Orowan loop behind. The SFT collapses into a smaller SFT, and only the base portion is annihilated, which corresponds to the two pieces separated by cutting with a moving dislocation. Judging from the interaction behavior with the SFT, dislocations are gliding on $(\bar{1}\,\bar{1}\,1)$, i.e., parallel to ABD. They are mixed dislocations, essentially having screw character (see text). The SFT edge length is 27 nm, dislocation length is 121 nm and specimen foil thickness is 99 nm.

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 [22,23]. 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. [4]. However, possession of atomic-layer ledges is an entirely different issue from the truncation where the top 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.

Fig. 2 shows the typical collapse process of an SFT interacting with moving dislocations. Judging from the interaction behavior with the moving 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. The SFT collapses after interaction with three successive moving dislocations. After the SFT collapse, a smaller SFT is left behind, and finally a super jog is formed on the moving dislocation. As shown in Fig. 3, the position of the smaller SFT corresponds to the top part of the two pieces cut by the first dislocation interaction with the original SFT. This indicates that only the base portion annihilates through the collapse process while the top portion survives.

Fig. 2 shows clearly that an SFT collapses not when the moving dislocations are approaching the SFT, but when a moving dislocation is in physical contact with the SFT. This indicates that the cause of the collapse is not the stress field of approaching dislocations, but the direct interactions with the dislocation core. These experimental results do not necessarily deny the possibility that SFTs could be collapsed by a large stress field, which could arise under highly localized stress conditions; however, they show clearly that direct interaction with the dislocation core can cause collapse of a SFT.

When the second moving dislocation passes through the SFT, stacking fault contrast inside the SFT clearly changes. This indicates that the moving dislocation does not pass through without cutting the SFT, i.e., leaving an Orowan loop behind, but instead has cut through the inside of the SFT, or traced the SFT surface by multiple cross-slip as proposed by Kimura and Maddin [14]. Diffraction contrast imaging of the SFT following the passage of one or more dislocations did not reveal any evidence for an Orowan loop surrounding the SFT.

Several variations of the collapse process were observed in addition to the process described above. For example, although in Fig. 2 an SFT collapsed after multiple (3) interactions with moving dislocations, it is unclear whether the number of interactions is an essential parameter for the collapse of SFT. In fact, it was often observed in our studies that SFT collapsed by a single interaction with a moving dislocation. Further discussion on this issue will be presented elsewhere [20].

Wirth et al. have found in MD computer simulation performed at high strain rates at low temperature that an SFT remains intact even after cutting by a moving edge dislocation several (up to 6) times [16]. Only incompletely formed (overlapping truncated) SFT could be collapsed by a moving dislocation in their simulations. In the collapse process, the truncated SFT was absorbed in the moving dislocation, which developed a super jog. The first statement is not consistent with our experimental results, which clearly show that a single moving dislocation can collapse a perfect SFT. This discrepancy might be associated with the difference in strain rate and temperature between their simulations and the present experimental study. However, we can see a similarity in the collapse process of the truncated SFT in their simulation and the collapse process of the per-

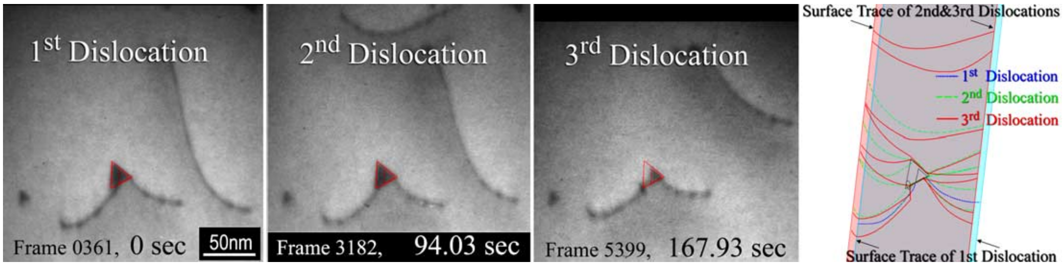

Fig. 3. Analysis of the position of the ends of dislocation line intersected by foil surface and the position of SFT interacted with the dislocations provide the following information about geometry of the interaction process shown in Fig. 2: (1) The first moving dislocation interacts with the SFT relatively close to the top, compared with the second and the third ones. (2) The second and the third dislocations glide on almost the same $(\bar{1}\,\bar{1}\,1)$ plane.

fect SFT observed in the present study. The similarity in the collapse processes indicates that the collapse mechanism of the truncated SFT in their simulation may provide valuable information for understanding the collapse process of the perfect SFT.

The dislocations analyzed in the present study were all identified as screw-type. Since the SFT collapse mechanism proposed by Kimura and Maddin was the one dedicated for screw dislocations, the type of dislocation has traditionally been believed as an important factor to collapse SFTs. Further discussion will be presented elsewhere regarding the collapse mechanism of SFT, including the discussion on the effect of dislocation type (edge vs. screw) [20].

4. Conclusions

An SFT can be collapsed by direct interaction with moving dislocations. Initial truncation of the SFT before the interaction with moving dislocations is not a crucial factor for the SFT collapse. Stacking fault contrast in the SFT changes when a moving dislocation passes through, indicating that the moving dislocation passes through without leaving behind an Orowan loop. The SFT collapses into a smaller SFT, and only the base portion is annihilated, which corresponds to the two pieces separated by cutting with a moving dislocation.

Acknowledgements

This research was sponsored by office of Fusion Energy Sciences, US 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, Yuri Osetsky, Roger Stoller, Thak Sang Byun, Nao Hashimoto, Joe Horton, and Jim Bentley in ORNL, and Dr Kazuto Arakawa in Shimane University for valuable comments.

References

[1] M. Kiritani, Mater. Chem. Phys. 50 (1997) 133.
[2] S.J. Zinkle, L.L. Snead, J. Nucl. Mater. 225 (1995) 123.
[3] M. Victoria, N. Baluc, C. Bailat, Y. Dai, M.I. Luppo, R. Schaublin, B.N. Singh, J. Nucl. Mater. 276 (2000) 114.
[4] N. Ajita, H. Hashimoto, Y. Takaki, Phys. Stat. Sol. (a) 87 (1985) 235.
[5] M. Meshii, J.W. Kauffman, Acta Metall. 7 (1959) 180.
[6] M.S. Bapna, T. Mori, M. Meshii, Philos. Mag. 17 (1968) 177.
[7] A. Okada, K. Kanao, T. Yoshiie, S. Kojima, Trans. JIM 30 (4) (1989) 265.
[8] S. Yoshida, M. Kiritani, Y. Deguchi, N. Kamigaki, Trans. JIM 9 (Suppl.) (1968) 83.
[9] J.V. Sharp, Philos. Mag. 16 (1967) 77.
[10] H. Saka, K. Noda, K. Matsumoto, T. Imura, in: Proceedings of 4th International Conference on High Voltage Electron Microscopy, Toulouse, 1975, p. 325.
[11] K. Noda, H. Saka, K. Shiraishi, H. Yoshida, T. Imura, in: Proceedings of 5th International Conference on High Voltage Electron Microscopy, Kyoto, 1977, p. 403.
[12] E. Johnson, P.B. Hirsch, Philos. Mag. A 43 (1) (1981) 157.
[13] M. Hiratani, H.M. Zbib, B.D. Wirth, Philos. Mag. A 82 (14) (2002) 2709.
[14] H. Kimura, R. Maddin, Lattice defects in quenched metals, in: R. Cotterill (Ed.), Proceedings of an International Conference, Argonne National Laboratory, 15–17 June 1964, Academic Press, New York, 1965, p. 319.
[15] P.B. Hirsch, Vacancies'76, in: R.E. Smallman, J.E. Harris (Eds.), Proceedings of a Conference on Point Defect Behaviour and Diffusional Process, 13–16 September 1976, The Metals Society, London, 1977, p. 95.
[16] B.D. Wirth, V.V. Bulatov, T. Diaz de la Rubia, J. Eng. Mater. Tech. 124 (2002) 329.
[17] Y.N. Osetsky, Y. Matsukawa, R.E. Stoller, J. Nucl. Mater., these Proceedings.
[18] J.S. Robach, I.M. Robertson, B.D. Wirth, A. Arsenlis, Philos. Mag. 83 (8) (2003) 955.
[19] A. Yoshinaka, Y. Shimomura, M. Kiritani, S. Yoshida, Jpn. J. Appl. Phys. 7 (7) (1968) 709.
[20] Y. Matsukawa, S.J. Zinkle, Philos. Mag., in preparation.
[21] J. Silcox, P.B. Hirsch, Philos. Mag. 4 (1959) 72.
[22] M. De Jong, J.S. Koehler, Phys. Rev. 129 (1963) 49.
[23] D. Kuhlmann-Wilsdorf, Acta Metall. 13 (1965) 257.