

## Development of Dislocation Structures during Cyclic Deformation of Aluminum Single Crystals

Toshiyuki FUJII

Department of Innovative and Engineered Materials, Tokyo Institute of Technology  
4259-J2-45 Nagatsuta, Midori-ku, Yokohama 226-8502, Japan

Aluminum single crystals with a single slip orientation were cyclically deformed under constant plastic-strain amplitudes in the range between  $1 \times 10^{-3}$  and  $5 \times 10^{-2}$  at 77 K. Cyclic hardening to saturation was obtained at all applied strain amplitudes in the cyclic hardening curves. The cyclic stress-strain curve (CSSC) showed a plateau with the stress level of 43 MPa. At strain amplitudes within the plateau region of the CSSC, persistent slip bands (PSBs) were formed and were approximately parallel to the primary slip traces. Transmission electron microscopy observation revealed that the dislocation microstructure developed in the PSBs was the labyrinth-like structure with a periodic wall spacing of about 500 nm. The dislocation walls in the PSBs were almost parallel to the  $\{100\}$  plane. The peculiar features of dislocation microstructure can be explained by considering the ease of secondary slip associated with the high stacking fault energy of aluminum.

**Keywords:** *Cyclic deformation, cyclic stress-strain curve, persistent slip band, dislocation microstructure, labyrinth-like structure.*

### 1. Introduction

There have been many studies on cyclic deformation of single crystals of fcc metals, especially of copper single crystals oriented for single slip [1-12]. The CSSC of copper single crystals deformed at room temperature has been established and exhibits a plateau in the intermediate plastic-strain amplitude between  $1 \times 10^{-4}$  and  $1 \times 10^{-2}$ . In the plateau region, ladder and vein structures are characteristically observed. Contrary to the extensive studies on copper single crystals, only a few studies have been conducted so far on the cyclic deformation of aluminum single crystals [13-18]. Nevertheless, the following specific features have been revealed in the cyclic deformation of aluminum single crystals at room temperature: no stress saturation is observed in cyclic hardening curves and no ladder structure is formed at any plastic-strain amplitude. The significant differences in cyclic deformation behavior at room temperature between copper and aluminum have been explained to be due to a higher homologous temperature,  $T/T_m$  where  $T_m$  is the melting point, and higher stacking fault energy of aluminum. If this is true, the essential effects of the stacking fault energy on the cyclic deformation of aluminum single crystals will be elucidated by conducting fatigue tests at enough low temperature, i.e. 77 K ( $T/T_m = 0.08$ ). Therefore, in this study, plastic-strain-controlled fatigue tests of aluminum single crystals with a single slip orientation are performed at 77 K and the cyclic stress-strain responses and the dislocation microstructures are investigated.

### 2. Experimental Procedure

Pure aluminum (99.8 mass%) single crystal sheets with the  $(21\bar{1})$  surface were grown by the Bridgman method. The single crystals were cut into specimens for fatigue tests with the gauge dimensions of  $4 \times 6 \times 10 \text{ mm}^3$  so that the stress axis becomes parallel to the  $[419]$  direction.

The single crystals were cyclically deformed in symmetric push-pull tests at 77 K under constant plastic-strain amplitudes,  $\gamma_{pl}$ , in the range of  $1 \times 10^{-3}$  and  $5 \times 10^{-2}$  using an electro-hydraulic testing

machine (Shimazu Servo Pulser). The specimens were immersed in liquid nitrogen to maintain a constant temperature during the fatigue tests. Strain was measured with an extensometer and constant strain rate of  $2.34 \times 10^{-3} \text{ s}^{-1}$  was employed using a triangular command signal. The fatigue tests were interrupted at various amounts of cumulative plastic strain defined as  $\gamma_{\text{cum}} = 4N\gamma_{\text{pl}}$  ( $N$ : number of cycles).

The fatigued specimens were sliced into 3 mm discs parallel to the  $(21\bar{1})$  plane and were ground down to 0.2 mm thick. Final thin foils were prepared by a standard twin-jet electropolishing technique using a solution of 10% perchloric acid, 8% 2-butoxyethanol and 82% methanol, and then microstructural observations were carried out on a JEM 2011 TEM at 200 kV.

### 3. Results and Discussion

#### 3.1 Cyclic stress-strain response

Cyclic hardening curves of all specimens deformed at 77 K showed cyclic hardening to saturation. This behavior is quite different from what is observed at room temperature ( $T/T_m = 0.31$ ), where the cyclic hardening curves exhibit cyclic softening at intermediate stages of fatigue [13, 15, 17]. Therefore, the testing temperature is indeed an important factor to control the stress-strain response in cyclic deformation. By plotting the saturation stresses at different plastic strain amplitudes, as shown in Fig. 1, the CSSC was drawn. It is clearly found that the CSSC shows a plateau with the stress of about 43 MPa over a range of plastic-strain amplitude from  $2 \times 10^{-3}$  to  $2 \times 10^{-2}$ . The plateau stress is almost the same as that obtained by Vorren and Ryum [13]. If we refer to the results of copper single crystals fatigued at room temperature, it is highly expected that PSBs and a characteristic dislocation structure should be developed during the plateau region.

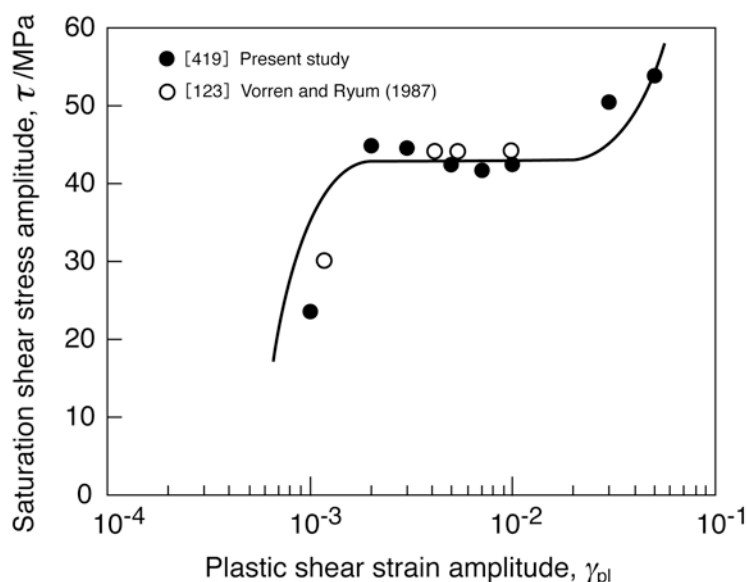


Fig. 1 Cyclic stress-strain curve of aluminum single crystals fatigued at 77K. The results obtained by Vorren and Ryum [13] are also included.

#### 3.2 Microstructural development

The fatigue tests were interrupted when the stress amplitudes clearly saturated at given plastic-strain amplitudes, and then dislocation microstructures developed in the specimens were observed on

TEM. Fig. 2 shows a dislocation structure formed in a specimen cycled to saturation at  $\gamma_{pl} = 3 \times 10^{-3}$ . As can be seen, it is obvious that the PSB parallel to the  $(1\bar{1}1)$  primary slip plane was formed during cyclic deformation at a constant strain amplitude within the plateau region in Fig. 1. The wall structure, consisting of periodically arranged dislocations with an average separation of about 500 nm, was developed in the PSB. From the trace analysis of the dislocation walls, it was found that the wall plane was approximately parallel to the  $(001)$  plane. This fact implies that the observed dislocation structure cannot be characterized by the well-known ladder structure but by an extended wall or a “labyrinth-like” structure, since the wall plane of the ladder structure would become parallel to the  $(011)$  plane, i.e. perpendicular to the primary slip direction. Considering the past observations [15, 18], the so-called labyrinth structure, consisting of two mutually perpendicular  $\{100\}$  dislocation walls, is frequently formed by operation of multiple slip systems in aluminum single crystals fatigued at room temperature. In this study, therefore, we conclude that the labyrinth-like structure was developed by simultaneous activation of the secondary slip system even at 77 K, while the PSBs were also observed.



Fig. 2 Dislocation microstructure developed in a PSB of a specimen of aluminum single crystal cyclically deformed at  $\gamma_{pl} = 3 \times 10^{-3}$  until  $\gamma_{cum} = 96$  at 77 K. The foil normal is close to  $[21\bar{1}]$ .

It is noteworthy to compare the dislocation structure observed in the present study with that found in copper single crystals cyclically deformed at 77 K ( $T/T_m = 0.06$  for copper) [6, 9-11]. If only the testing temperature is a dominant factor to determine the dislocation structure developed during cyclic deformation, the differences in the structures between copper and aluminum would become reduced at 77 K. Although an irregular wall structure has been found in copper single crystals fatigued at 77 K [6, 9-11], no labyrinth structure has been reported to be formed. This means that there are significant differences in the dislocation structures at 77 K between copper and aluminum. It can be understood that the higher stacking fault energy of aluminum makes an activation of secondary slip easier, and then the labyrinth-like structure is developed even at 77 K. Therefore, we conclude that the stacking fault energy is still an important factor to affect the cyclic deformation behavior of aluminum at such low homologous temperature as 0.08.

#### 4. Summary

Plastic-strain-controlled fatigue tests were carried out on aluminum single crystals with a single slip orientation at 77 K. The results and conclusions are summarized as follows.

- (1) The CSSC shows a plateau with the shear stress amplitude of about 43 MPa.
- (2) The PSBs and the labyrinth-like structure are developed during the plateau region.
- (3) Even at low homologous temperature of 0.08, there remain the effects of high stacking fault energy on cyclic deformation behavior of aluminum.

#### Acknowledgement

This research was partially supported by a Grant-in-Aid for Scientific Research (C) (22560690) by the Japan Society for the Promotion of Science.

#### References

- [1] P. Lukáš, M. Klesnil and J. Krejčí: *Phys. Stat. Sol.* 27 (1968) 545-558.
- [2] S. J. Basinski, Z. S. Basinski and A. Howie: *Phil. Mag.* 19 (1969) 899-924.
- [3] P. J. Woods: *Phil. Mag.* 28 (1973) 155-191.
- [4] H. Mughrabi: *Mater. Sci. Eng.* 33 (1978) 207-223.
- [5] H. Mughrabi, F. Ackermann and K. Herz: *Proceedings of the Symposium on Fatigue Mechanisms*, Ed. by J. T. Fong, (ASTM STP675 1979) pp. 69-105.
- [6] Z. S. Basinski, A. S. Korbel and S. J. Basinski: *Acta Metall.* 28 (1980) 191-207.
- [7] F. Ackermann, L. P. Kubin, J. Lepinoux and H. Mughrabi: *Acta Metall.* 32 (1984) 715-725.
- [8] L. Buchinger, S. Stanzl and C. Laird: *Phil. Mag. A*, 50 (1984) 275-298.
- [9] L. L. Lisiecki and O. B. Pedersen: *Acta Metall. Mater.* 39 (1991) 1449-1456.
- [10] U. Holzwarth and U. Essmann: *Appl. Phys. A* 58 (1994) 197-210.
- [11] J. Bretschneider, C. Holste and W. Kleinert: *Mater. Sci. Eng. A* 191 (1995) 61-72.
- [12] C. Watanabe, K. Kanmuri, M. Kato, S. Onaka and T. Fujii: *Phil. Mag. A*, 82 (2002) 1317-1330.
- [13] O. Vorren and N. Ryum: *Acta Metall.* 35 (1987) 855-866.
- [14] A. Giese and Y. Estrin: *Scr. Metall. Mater.* 28 (1993) 803-807.
- [15] M. Videm and N. Ryum: *Mater. Sci. Eng. A* 219 (1996) 1-10.
- [16] M. E. Kassner, M. A. Wall and M. A. Delos-Reyes: *Metall. Mater. Trans. A* 28A (1997) 595-609.
- [17] T. Fujii, N. Sawatari, S. Onaka and M. Kato: *Mater. Sci. Eng.* 387-389C (2004) 486-490.
- [18] T. Fujii, S. Uju, H. Tanaka, T. Murayama, C. Watanabe, S. Onaka and M. Kato: *Proceedings of the Hael Mughrabi Honorary Symposium*, Ed. by K. J. Hsia, M. Göken, T. Pollock, P. D. Portella and N. R. Moody, (TMS, Warrendale, 2008) pp.123-127.