Materials Science & Engineering A 668 (2016) 166–179

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Low cycle fatigue in aluminum single and bi-crystals: On the influence of crystal orientation

J. Nellessen [a], S. Sandlöbes [a,b,*], D. Raabe [a]

[a] Max-Planck-Institut für Eisenforschung GmbH, Department for Microstructure Physics and Alloy Design, 40237 Düsseldorf, Germany
[b] Institut für Metallkunde und Metallphysik, RWTH Aachen University, 52074 Aachen, Germany

ARTICLE INFO

Article history:
Received 12 April 2016
Received in revised form
12 May 2016
Accepted 14 May 2016
Available online 16 May 2016

Keywords:
Low cycle fatigue (LCF)
Dislocation structure formations
Electron channeling contrast imaging (ECCI)
Aluminum single and bi-crystals

ABSTRACT

Aluminum single crystals with three different double-slip orientations ($[\bar{2}32]$, $[\bar{3}\bar{3}2]$, $[130]$) and two aluminum bi-crystals – one with a high-angle grain boundary and one with a low-angle grain boundary - were cyclically deformed up to 100 cycles under constant displacement control. The distribution of the local strain and the local strain amplitudes was captured by in-situ digital image correlation (DIC). Dislocation structure analysis was performed by electron channeling contrast imaging (ECCI) and the evolution of local misorientations was recorded by high resolution electron backscatter diffraction (EBSD). The DIC results show a homogeneous strain amplitude distribution in the single crystals while the measured strain amplitude in the low-angle grain boundary bi-crystal sample differs significantly. ECCI observations reveal the presence of dislocation cells elongated along the trace of the primary {111} slip plane in all investigated crystals and the formation of deformation bands parallel to the trace of {110} planes. Deformation bands (DB) were observed in all samples but their frequency and misorientation with respect to the matrix was found to sensitively depend on the crystal orientation and the local strain amplitude. Our results on the bi-crystals show that the grain orientation mainly determines the local stresses and therefore also the formation of the associated dislocation structures rather than the grain boundary character.

1. Introduction

Cyclic deformation and fatigue-related damage in metals and alloys is one of the major failure reasons of materials in structural applications [1]. It is estimated that 90% of all mechanical service failures are attributed to progressive structural damage caused by cyclic loading conditions [2]. Compared to numerous investigations performed on fatigued Cu single crystals over the last decades (e.g. [3,4]) the knowledge on the cyclic deformation behavior of aluminum is limited. In contrast to copper single crystals which show a saturation behavior over a wide range of applied strain amplitudes [5], aluminum single crystals exhibit a hardening – softening – secondary hardening sequence without saturation when subjected to cyclic loading [6]. Vorren and Ryum [6] and Videm and Ryum [7] studied the influence of the strain amplitude on the cyclic hardening in $[\bar{1}23]$ oriented single-slip and [001] oriented multiple-slip Al single crystals and observed that the initial hardening rate, the softening behavior and the secondary hardening strongly depend on the applied strain amplitude. They reported [6,7] that the initial hardening rate as well as the maximum stress increases with increasing strain amplitude, an effect which was more distinct for the multiple-slip orientation, whereas higher strain amplitudes decrease the amount of softening. These observations were later confirmed by several other investigations [8–11]. Studies on single-slip, double-slip and multiple-slip oriented single crystals reveal higher stress levels and a more pronounced hardening and softening behavior in double- and multiple-slip oriented crystals [12,13]. Fujii et al. [8] proposed that the amount of softening is directly related to the activity of the cross-slip system: $[\bar{1}12]$ oriented crystals show no softening while $[\bar{1}14]$ and $[\bar{1}120]$ oriented crystals exhibit a pronounced softening stage.

Regarding the microstructure evolution, several studies revealed that dislocation cells with approximate dimensions of 3–5 μm are the dominant type of dislocation structures formed in fatigued Al single and polycrystals, independent of the crystal orientation and over a large range of strain amplitudes [8,9,11–15]. In addition, several authors observed the formation of deformation bands during cyclic deformation of Al [e.g. 16–17], but a thorough understanding of the correlation between the cyclic deformation behavior, dislocation structure and deformation band formation and the crystal orientation in Al has not been investigated in detail yet.

* Corresponding author.
E-mail address: Sandloebes@imm.rwth-aachen.de (S. Sandlöbes).

http://dx.doi.org/10.1016/j.msea.2016.05.054
0921-5093/

In order to investigate these relationships we apply a novel experimental approach developed and established for the analysis of dislocation structures in cyclically deformed polycrystalline stainless steel [18] to analyze low cycle fatigue in Al single crystals. This approach combines digital image correlation (DIC), electron backscatter diffraction (EBSD), slip trace analysis and electron channeling contrast imaging (ECCI). Specifically, the present study aims to further investigate the role of the crystal orientation on the cyclic stress response, the formed dislocation structures, the local misorientations and on the formation of deformation bands in aluminum single crystals during cyclic loading. In addition, aluminum bi-crystals with different types of grain boundary character were subjected to cyclic loading and analyzed by slip trace analysis, dislocation observations and the measurement of local misorientations in order to reveal the influence of grain boundaries. These investigations are aiming towards a more fundamental and quantitative understanding of the underlying mechanisms of local strain accumulation and eventually crack initiation during cyclic deformation relevant not only for low but also for high cycle fatigue. Particularly the simulation and prediction of cyclic deformation, strain localization and failure – both in low and high cycle fatigue – requires more detailed and quantitative understanding of the parameters influencing the formation of dislocation structures in the early stages of fatigue.

2. Experimental procedure

Aluminum single crystals of 99.999% pure Al were grown by the Bridgman method. Aluminum bi-crystals were provided by the Institut für Metallkunde und Metallphysik (IMM) at RTWH Aachen University in the form of small plates with approximate dimensions of 20×6 mm ($L \times W$) and a thickness of 2 mm. Flat bone-shaped single crystal samples with $[\bar{2}32]$ // LD, $[\bar{3}\bar{3}2]$ // LD and $[130]$ // LD orientations (LD: loading direction), respectively, were cut from the aluminum single crystals using electric discharge machining (EDM). The three single crystals were all in double-slip orientation, each placed on a different symmetry border of the standard triangle. The sample geometry throughout this study was the same as described in [18] with slight variations in the gauge length. The dimensions of the gauge length of the single crystalline samples were 4 mm in length, 2 mm in width and 2 mm in thickness. The gauge length of the bi-crystalline samples was reduced to 2 mm due to limited sample dimensions. Two bi-crystals, one with a low-angle grain boundary (LAGB) and one with a high-angle grain boundary (HAGB), were investigated. All samples were metallographically prepared by grinding using 400, 600, 1000, 2500 and 4000 SiC grinding paper and mechanically polishing using 3 μm diamond suspension prior to deformation. The final polishing step was performed with a 0.05 μm silica (SiO_2) suspension. Both, aluminum single- and bi-crystals were crystallographically characterized by EBSD to ensure that each sample had the desired crystallographic orientation with respect to the loading direction (Al single crystals) and to characterize the position and the character of the grain boundaries (Al bi-crystals). These EBSD measurements were carried out using a CamScan 4 tungsten filament scanning electron microscope (SEM) with an accelerating voltage of 20 kV. The crystallographic characterization of the undeformed aluminum single- and bi-crystals by EBSD measurements is summarized in Table 1.

Cyclic deformation experiments were carried out on a high-precision screw-driven deformation instrument manufactured by Kammrath & Weiss equipped with a 5 kN load cell and an in-situ DIC setup provided by GOM (Gesellschaft für optische Messtechnik) in displacement control [19,20]. The accuracy of the screw driven device is in the range of ± 0.1 μm. Individual DIC images were taken at each 1/4 cycle, i.e. at maximum displacement, at zero displacement, at minimum displacement and at the next zero displacement. Aluminum single crystals were cyclically deformed for 50 and 100 cycles, respectively, with a displacement of ± 20 μm and a deformation speed of 2 μm/s (corresponding to a strain rate of $5 \times 10^{-4}\ s^{-1}$). Sets of four samples for each single crystal orientation were tested in order to ensure statistical significance. The shown cyclic stress response curves are representative for each orientation. The two bi-crystalline samples were subjected to cyclic loading for 50 cycles using a displacement of ± 10 μm (owing to the smaller gauge length compared to the single crystals) and a deformation speed of 1 μm/s (corresponding to a strain rate of $5 \times 10^{-4}\ s^{-1}$). The limited dimensions of well-defined bi-crystals unfortunately disabled the production of more than one sample per bi-crystal.

Table 1.
Miller indices of the three investigated aluminum single crystals with respect to the loading direction (LD) and Miller indices and misorientation between grain 1 (G1) and grain 2 (G2) of the low-angle grain boundary (LAGB) and high-angle grain boundary (HAGB) aluminum bi-crystal.

Single crystals	LAGB bi-crystal	HAGB bi-crystal
Single crystal 1	Grain 1 (G1)	Grain 1 (G1)
$[\bar{2}32]$ // LD	$[56\bar{1}]$ // LD	$[10235]$ // LD
Single crystal 2	Grain 2 (G2)	Grain 2 (G2)
$[\bar{3}\bar{3}2]$ // LD	$[161\bar{7}\bar{2}]$ // LD	$[182\bar{3}6]$ // LD
Single crystal 3	Misorientation between G1 & G2:	Misorientation between G1 & G2:
$[130]$ // LD	12.2°@ $[142\bar{2}1\bar{3}]$ // LD	36.1°@ $[141\bar{3}\bar{2}3]$ // LD

The surface slip traces formed during deformation were recorded after deformation and a detailed slip trace analysis was performed for each sample by correlating the crystallographic information obtained by the EBSD measurements with the observed slip traces; allowing the identification of the respective active slip systems. All Al samples were re-polished after deformation and surface trace analysis prior to dislocation analysis using the electrolyte A2 by Struers (30 V, 30 s). ECCI dislocation observations were performed using a Zeiss FIB XB1540 SEM and a Zeiss Merlin SEM both equipped with a field emission gun. A working distance of approximately 6 mm and an accelerating voltage of 30 kV were used for optimized ECCI contrast [21–27]. However, due to the low Z-number of Al, the obtainable ECCI contrast is weak when compared to materials with a higher Z-number, e.g. steels. Low-resolution EBSD measurements were performed after deformation using the same set-up as for the initial characterization of the samples in order to visualize the misorientations. In addition, high resolution ESBD measurements were carried out on the deformed and re-polished samples to investigate the local misorientations in defined regions. These measurements were performed using a high resolution field emission gun scanning microscope (JEOL 6500 F) with an accelerating voltage of 15 kV and a step size of 0.15 μm for single crystal samples and 0.3 μm for bi-crystals, respectively.

3. Experimental results

3.1. Mechanical behavior

3.1.1. Aluminum single crystals

The global stress response in terms of the stress amplitude of the $[\bar{2}32]$ // LD, $[\bar{3}\bar{3}2]$ // LD and $[130]$ // LD oriented Al