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Assessing the intergranular crack initiation probability of a grain boundary distribution by an experimental misalignment study of adjacent slip systems

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Abstract

Crack initiation at grain boundaries due to blocked slip transfer of dislocations is a main failure mechanism during the fatigue of metals. A quantification of the resistance effect of a grain boundary is needed to assess a textured or texture-free microstructure for fatigue strength. Geometric approaches based on the misalignment of slip systems in adjacent grains are widely used. Hence, we validated the geometric transmission factor of Shen et al. in coarse-grained high-purity aluminum under the assumption that the combination of a large slip activity and a blocked slip at a grain boundary leads to intergranular crack initiation and revealed that a detailed knowledge of the 3D-orientation of the grain boundary is essential. Thereby we gathered information about the 3D-microstructure using FIB-cross-sectioning. Hence it is possible to evaluate potential crack initiation sites for a specific microstructure or to estimate the fatigue strength of a textured microstructure in terms of a crack initiation probability.

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Keywords: grain boundary resistance; slips system alignment; 3D microstructure; boundary crack initiation; PSBs

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Nomenclature

α	angle between the intersection lines of involved slip planes and grain boundary
β	angle between both involved slip directions
$\mathbf{b}$	normalized Burgers vector
b_R	magnitude of the residual Burgers vector
δ	surface trace angle of the grain boundary
η	depth tilt angle of the grain boundary
i,j	indices
$\boldsymbol{l}$	intersection vector between slip planes and grain boundary plane
$\mathbf{n}_{GB}$	normalized grain boundary normal vector
$\mathbf{n}_i$	normal vector of slip plane of slip system i
SF	Schmid factor
T	transmission factor
ω_{ij}	geometric grain boundary resistance factor between slip systems i and j
Ω	impact parameter

1. Introduction

The fact that at least half of all mechanical failures are due to fatigue shows the importance of this topic for the minimization of risks (Stephens et al. (2002)). Although fortunately, most of these failures happen without personal injury, there are dramatic catastrophes due to the fatigue of metals. The analysis of fatigue crack growth can of course only assess present cracks. However, the stage of crack initiation might encompass a considerable part of the fatigue lifetime (Krupp (2007)), especially in the range of the fatigue limit that is usually used for the dimensioning of essential construction elements.

The microstructure of the material thereby controls the period of the crack initiation phase. Strain localization e.g. at persistent slip bands in f.c.c. materials (PSB) or at grain boundaries are key processes for the initiation of fatigue cracks (Basinski and Basinksi (1992), Vehoff et al. (2004), Zhang and Wang (2008)).

Weidner et al. (2006,2008) showed that PSBs develop after several tens of load cycles in polycrystals during fatigue at plastic strain amplitudes up to the 10^{-3} regime (Mughrabi and Wang (1988), Morrison and Moosbrugger (1997)), even at asymmetrical loading conditions (Holste et al. (1994)). At the same time, the PSB density increases with the strain amplitude (Rasmussen and Pederson (1980)). PSBs develop not only in pure f.c.c. materials but have among others been found in γ'-hardened nickel alloys (Alexandre et al. (2004), Fritzemeier and Tien (1988)). Excepting grains with <001> and <111> orientation (Buque (2001)), all grains develop PSBs on the primary slip system (Blochwitz et al. (1996)), whereby the strain localization due to the PSBs, that consist of a ladder-like dislocation structure or cell structure at higher loads, causes in- and extrusions at the specimen surface (Schwab et al. (1996)) as a precursor for fatigue crack initiation (Sangid (2013)).

PSBs are sources for fatigue cracks (Polak et al. (2005), et al. Petrenec (2007)) although the role of PSBs in fatigue crack initiation is competitive. At low strain amplitudes, strain localization in PSBs dominates the crack initiation process, whereas at higher strain amplitudes crack initiation occurs where PSBs impinge on a grain boundary (Morrison and Moosbrugger (1997)). The PSBs form dislocation pile-ups and exert an extra-stress at the grain boundary (Mughrabi (1983), Mughrabi et al. (1983)). Zhang et al. (Zhang and Wang (2000,2003,2008), Zhang et al. (2003)) have shown that PSBs interacting with low-angle grain boundaries (LAGB) can cause transgranular PSB-cracks and that high-angle grain boundaries (HAGB) are affected by boundary crack initiation. They subscribed this to a blocked slip due to a geometrical mismatch of the active slip systems in both adjacent grains. Depending on the capability of a grain boundary to transfer slip activity from one grain to another, a grain boundary is more or less susceptible for fatigue crack initiation. Zhang et al. proposed the following three cases:

- Complete slip blockade followed by a grain boundary crack
- A partial pass-through that can also cause a grain boundary crack because the grain boundary must integrate a residual Burgers vector locally that causes an additional stress concentration

- Finally, a pass-through of slip that can cause PSB-cracking because the effective grain size is increased.

Davidson et al. (2007) found that super-grains or super-grain clusters develop in the case of a free pass-through of slip by consecutive LAGBs and that strain accommodates mainly in these de facto larger grains compared to the real grain size (Keller et al. (1989)). Lukas and Kunz (1987) studied the influence of the grain size on the fatigue limit and found that the plastic strain fatigue limit is affected by the grain size, whereas their and our experiments have shown that the total stress or the total strain controlled one almost not.

The aim of this work is to check geometric approaches by a fatigue crack initiation study in near-isotropic coarse-grained aluminum. After a short introduction to common geometric approaches to slip transfer resistance is introduced in chapter 2. Our experiments are explained in chapter 3. Chapter 4 is dedicated to the experimental results for the geometric concept.

2. Geometric concept for the grain boundary resistance against slip transfer

Many models for the geometric resistance effect of a grain boundary against slip transfer have already been proposed in literature. A classical classification to LAGB und HAGB, although often used to explain the interaction between PSBs and grain boundaries, is not sufficient to assess the slip system misalignment, especially not in the case of HAGBs. Hence, the misorientation angle between both adjacent grains is not a valid resistance parameter (Knorr et al. (2015), Schaefer et al. (2016)).

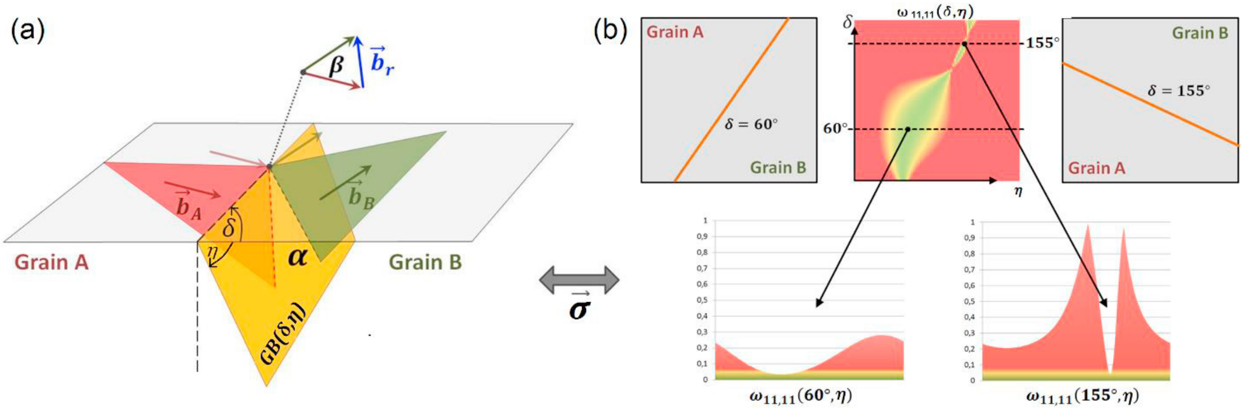


Fig. 1. (a) slip system coupling at grain boundary with surface trace angle δ and depth tilt angle η, α denotes the angle between the slip planes on the grain boundary plane and β denotes the angle between the slip directions, $\boldsymbol{\sigma}$ the direction of the tensile load; schematic of Burgers vectors $\boldsymbol{b}$ of both slip systems and the residual Burgers vector $\boldsymbol{b}_R$; (b) exemplary dependence of the geometric resistance parameter ω from the grain boundary tilt angle η for two selected surface trace angles δ and the slip system coupling i=j=11.

The most established approach is the concept of Clark et al. (1992), based on the transmission electron microscopy observations of Lee et al. (1990) and Shen et al. (1988). Their transmission factor T combines the alignment of the active slip directions i in both adjacent grains represented by the angle β and the alignment of the slip planes j represented by the angle α that depends on the grain boundary orientation angles (surface trace δ, depth tilt η, both from 0° to 180°), see Fig. 1a.

$$T_{ij} = cos(\alpha_{ij})cos(\beta_{ij}) \tag{1}$$

The intersection vectors of the grain boundary plane with the slip planes and the Burgers vectors of the slip directions involved can also express the transmission factor. We reinterpret the transmission factor as a geometric resistance factor ω_{ij} as shown in Knorr et al. (2015).

$$\omega_{ij} = 1 - T_{ij} = 1 - (\vec{l}_i \cdot \vec{l}_j)(\vec{b}_i \cdot \vec{b}_j) \tag{2}$$

Thereby the normalized intersection vector l_i is given by:

$$\vec{l}_i = \frac{\vec{n}_{GB} \times \vec{n}_i}{|\vec{n}_{GB} \times \vec{n}_i|} \; with \; \vec{n}_{GB} = \begin{pmatrix} sin(\eta)sin(\delta) \\ -sin(\eta)cos(\delta) \\ cos(\eta) \end{pmatrix} \tag{3}$$

where $\boldsymbol{n}_{GB}$ is the normal unit vector of the grain boundary depending on δ and η. This parameter is difficult to apply because the 3D orientation of the grain boundary, especially the important depth angle η, is unknown in standard experiments. Depending on the tilt angle η, the resistance parameter ω can vary over its complete range from 0 to 1 (see Fig. 1b). Werner and Prantl (1990) suggested an alternative usage of the slip plane normal vectors $\boldsymbol{n}_i$ instead of the hard-to-reach intersection vectors $\boldsymbol{l}_i$ as a worst case estimation for α because the angle between the slip planes cannot exceed the angle between their intersection lines with the grain boundary α.

$$\omega_{ij}^{WP} = 1 - (\vec{n}_i \cdot \vec{n}_j)(\vec{b}_i \cdot \vec{b}_j) \tag{4}$$

A more detailed description can be found at Schaefer et al. (2016). In accordance with the findings of Zhang et al. (2013) and Abuzaid et al. (2012), we suppose a third factor with a stronger influence of residual Burgers vector to integrate at the boundary to be checked with our experimental results (chapter 4):

$$\omega_{ij}^{\beta} = \frac{\beta}{90°}(1 - cos(\alpha)) \tag{5}$$

The geometric resistance factor ω is weighted by the Schmid factors SF_i of the slip systems. PSBs that embrace the complete grain and do not only exist very locally nearby a grain boundary due to incompatibility appear only on the primary slip system as shown by Blochwitz et al. (1996). Hence, only the slip system coupling with the highest Schmid factor of each grain is considered because we assume that the condition for fatigue crack initiation due to slip blockade is as proposed in literature: High slip activity meets high slip blockade. If the slip plane containing the primary slip system is the grain boundary in the special case of a coherent twin boundary, the secondary slip system is used because the geometric incompatibility vanishes if the grain boundary plane contains the primary slip system as proposed by Knorr et al. (2015).

So, the grain boundary impact factor Ω is supposed as the geometric impact factor ω weighted by the Schmid factors of the slip systems involved and is in the range of 0 to 1:

$$\Omega = 4 \cdot SF^A \cdot SF^B \cdot \omega^i \tag{6}$$

3. Experimental details

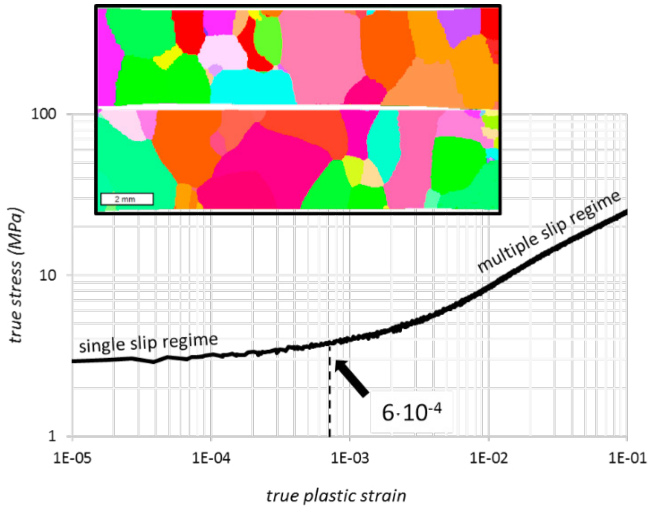
3.1. Specimen preparation

High-purity 4 mm aluminum foils were cut by EDM to a flat tensile specimen with a total length of 50 mm and a width of 4 mm. The gauge section was 10 mm. In order to avoid crack initiation apart from the gauge section very smooth radii were cut. The specimen was heat-treated to get a coarse-grained microstructure as shown in Fig. 2 (15 min at 600 °C, vacuum with a heating and cooling rate of about 600 °C/hour). In order to get a flat and notch-free specimen surface and to be able to get orientation data from EBSD, the samples were mechanically grinded and polished with 6 and 3 µm diamond suspension even on the side surfaces and finally vibratory polished with 50 nm alumina suspension. To obtain good EBSD patterns, the specimen was Ar-ion polished at an inclination angle of 5° with 4 kV acceleration voltage for 2 hours. The final surface roughness of the aluminum sample was below 5 nm, measured by atomic force microscopy.

EBSD orientation maps were collected by an Oxford NanoAnalysis EBSD-system with a Zeiss Sigma VP SEM. Special attention was paid to the orientation of the specimen and the lab coordinate system. The surface trace angle δ was measured from the orientation maps (see Fig. 2).

3.2. Fatigue test and analysis

Tensile pretests were performed on a comparison specimen to find the total strain level for the fatigue test (see Fig. 2). All fatigue tests were performed at room temperature at a total strain amplitude of $6 \cdot 10^{-4}$ at the transition from a single slip conditioned by the coarse-grained microstructure to a multiple slip behavior (Fig. 2). The initial plastic strain amplitude was nearly the same because the elastic strain is minimal at the beginning of the test (yield stress of 3.5 MPa at total strain of $5 \cdot 10^{-5}$).



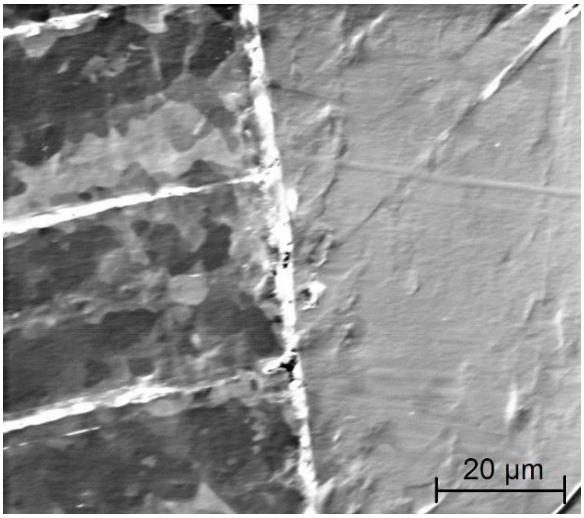


Fig. 2. Left hand side: true plastic strain vs. true stress curve for aluminum specimen; the initial plastic strain amplitude was marked as well as the single and multiple slip regime; the inset shows the orientation map of the front and back surface of the gauge section of the sample; right hand side: PSB (in-/extrusions) impinge on the grain boundary, dislocation cell structure visible in the left grain by ion induced secondary electrons.

The stress amplitude was observed during the test. While dislocation multiplication during plastic strain results in a strain hardening process, the formation of PSBs and crack initiation are characterized by a strain or in the latter case a specimen softening (Mughrabi and Wang (1988)). This softening after an initial hardening was used to find the moment to abort the fatigue test.

The specimen surfaces were analyzed by scanning electron microscopy, especially by electron channeling contrast imaging (ECCI), and cracked grain boundaries were listed. For the further analysis, only those cracks were considered that did not originate from the sample side surfaces.

Although there are many advanced techniques to detect a strain localization, as for example Digital Image Correlation (Abuzaid et al. (2012) or HR-EBSD (Guo et al. (2014)), PSBs are easily detectable by their in- and extrusions (Buque et al. (2001)) as shown in figure 3. This is of advantage especially for aluminum samples because the ECCI contrast is too low in the SEM to detect dislocation structures.

After the classification of the grain boundaries into cracked and uncracked ones, the grain boundary tilt angles η were measured using cross sectioning in the focused ion beam (FIB, FEI Helios). In order to get the necessary mechanical stability for the sample preparation and the fatigue testing, the sample was too thick compared to the grain size to fulfill the assumption of straight and vertical grain boundaries. Ion induced secondary electrons provided the necessary contrast to get the information about the grain boundary orientation from the cross section surfaces (Fig.2 and 3).

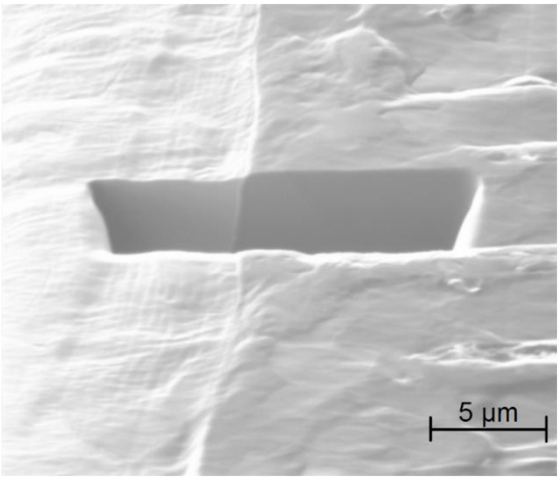


Fig. 3. FIB cross section, ion induced secondary electrons; the depths angle of the grain boundary can be measured after adapting a tilt correction to the images.

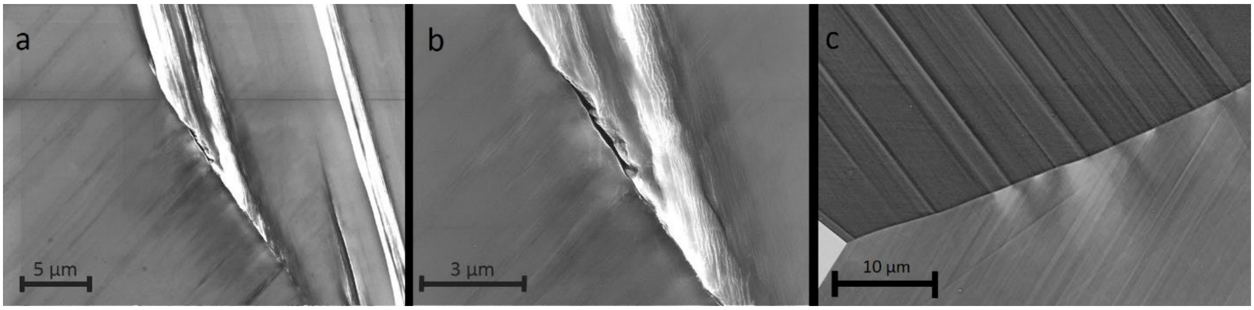


Fig. 4. (a) in- and extrusions impinge on a grain boundary; (b) a crack initiates at the grain boundary; (c) PSBs hitting a grain boundary; the local extra-stress at the grain boundary is visible by a channeling contrast in the adjacent grain due to a local crystal lattice tilt.

4. Analysis using the geometric concepts

All 55 grain boundaries from the near-isotropic aluminum specimen were analyzed for the significance of the 3 geometry parameters proposed in chapter 2. With 1.22, the elastic anisotropy factor of aluminum has the lowest elastic anisotropy among f.c.c. metals. Therefore, we assume that the results are not affected by incompatibility stresses. The results are displayed in Fig. 5.

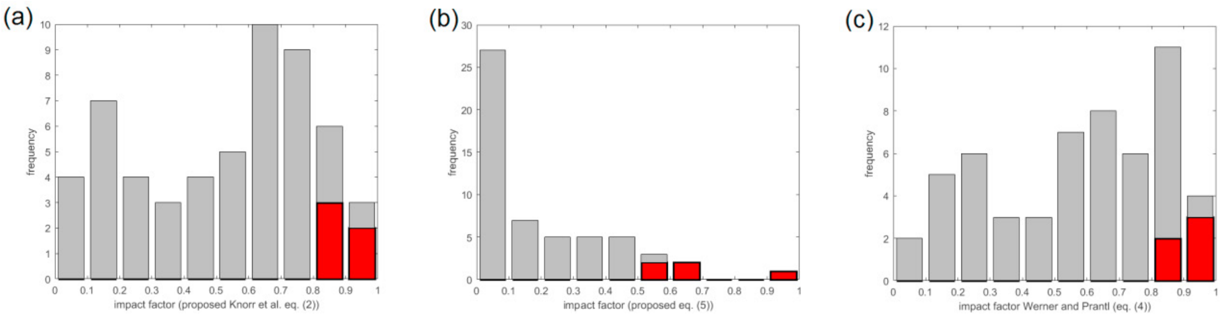


Fig. 5. Histograms of the geometrical grain boundary impact factors Ω: (a) the impact factor derived from Clark et al. (1992) according to Knorr et al. (2015); (b) the modified impact factor with a higher impact of the misalignment of the slip directions β; (c) impact factor derived from Werner and Prantl (1990); the cracked grain boundaries are highlighted.

For all three parameters, the cracked grain boundaries are among those with the highest impact parameter values. Nevertheless, the parameter with an increased amount of the angle β between the slip direction and therefore the residual Burgers vector (Fig. 6b) shows a better significance because all 5 cracked boundaries coincide with the boundaries with the highest impact factors. Therefore, we recommend, that in accordance with literature the common parameter of Clark et al. (1992) (Fig. 6a) should be reweighted to a higher impact of the alignment of the slip directions.

The assumption of Werner and Prantl (1990) might have an enormous effect on the geometric impact factor because a very high slip transfer resistance effect might be assigned to grain boundaries with a lower one due to the worst-case estimation for the misalignment of the slip planes at the grain boundary α. This lowers the significance level as displayed at Fig. 5c.

In contrast to earlier results from the literature, we could not confirm that the misorientation angle is a useful parameter. Although for small misorientations, the slip system misorientation is similar to the misorientation angle of the crystal lattices, this does not apply to higher misorientation angles. The cracked grain boundaries all had misorientation angles between 35° and 55°. This is not significant because for f.c.c. materials, most grain boundaries have misorientation angles in this range (Mackenzie (1964)).

5. Conclusion and Outlook

In isotropic f.c.c. materials, it is essential to know the orientation of the grain boundary planes in order to assess the ability of slip transfer. A worst-case estimation for the misalignment of the slip planes at the grain boundary by the misalignment of slip planes themselves is not expedient. A worst-case estimation for the misalignment tends to overestimate slip transfer resistance of the grain boundary and therefore the crack initiation probability. In good agreement with works from other groups, we have successfully implemented a higher weighting of the misalignment of slip directions or rather the residual Burgers vector causing a local translation at the grain boundary due to slip transfer.

The local incompatibility stresses at the grain boundaries due to elastic anisotropy will be included to the model using nickel specimens in studies in progress. The analytical solution for the incompatibility stress state at a bicrystals boundary suggested by Tiba et al. (2015) or from FEM simulations as performed by Klusemann et al. (2013) can provide information about the local stress state near a grain boundary.

Even the grain size influences the probability of transgranular PSB cracks due to the high strain localization at the PSB themselves in larger grains or grain clusters (additional effect of the grain neighborhood). Mughrabi (1983) suggested a $1/D^3$ dependency, but Tanaka and Mura (1981) predicted a $1/D$ dependency for constant shear stress amplitudes. The grain size effect remains an open question but it might well be included in our parameter in future studies.

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