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A synchrotron X-ray and electron backscatter diffraction based investigation on deformation and failure micro-mechanisms of monotonic and cyclic loading in titanium

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ABSTRACT

Synchrotron X-ray diffraction technique has been used to estimate defect structure in terms of dislocation density, crystallite size and micro-strain in commercially pure titanium subjected to tension and cyclic deformation in stress and strain control mode. Statistical analysis of micro-texture data collected from electron backscatter diffraction approximately from the same region as that of synchrotron X-ray has been used to correlate orientation dependent micro-strain and dislocation density with deformation microstructure and micro-texture. Two different orientations, namely, A with prismatic-pyramidal and B with basal orientation along the loading axis has been considered. Weak initial texture yet significant anisotropy in hardening/softening response and failure mode for monotonic tension and cyclic loading paths has been observed. Higher strain hardening response of orientation A during monotonic tensile deformation can be attributed to the evolution of lower micro-strain on basal orientation grains i.e, ⟨0002⟩‖ND along with extensive multi-variant twinning that also restricts crack propagation and delays failure in stress control mode. On the other hand, in strain control mode, orientation B shows higher fatigue life due to the generation of lower micro-strain in the basal orientation grains and single variant twinning that can undergo detwinning easily is responsible for delayed crack nucleation and subsequent failure.

1. Introduction

Macroscopic plastic deformation of materials depends on the mode of loading as well as the strain path followed during deformation. Strain path involving tension-compression, tension-torsion and change in strain rate during quasi-static and dynamic loading shows different deformation behavior in materials. Also, there are different ways in which macroscopic deformation leading to failure in materials occurs for different loading paths [1]. For instance, failure during monotonic tensile loading occurs by necking due to localized stress overload, buckling during compression due to increase in friction force at the contact surface. While failure during torsion test shows no observable phenomenon like necking, rather formation of band of flow localization region leads to failure and hence material shows more ductility before failure during torsion test compared to tensile test. At the sub-micro-structural level, the evolution of dislocation density with change in strain path dictates deformation behavior and contributes to different behavior depending on monotonic, load reversal or cyclic mode of deformation. During monotonic loading, stored dislocation density decreases with increase in strain [2]. While in case of tension-compression cyclic loading, there is multiplication and annihilation of dislocations with overall increase in dislocation density with each cycle and all these affect failure too. The onset of necking in tension corresponds to the saturation dislocation density of statistically stored dislocation that can be stored in a grain and it is rational to assume that the saturation dislocation density can be achieved by large number of cycles leading to failure. However, unlike yielding and necking in tension which can be predicted and are design parameters for any material, failure in tension and more so in cyclic loading is stochastic in nature.

Nevertheless, the role of dislocation interactions is important, and we want to compare dislocation character and fracture features for differently loaded sample. Thus, the present paper is an attempt to bridge the gap in length scale and explain failure as a function of

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crystallographic texture that determines dislocation and twin activity. It requires characterization of the defect structure generated during deformation leading to failure. Synchrotron X-ray source provides statistical information of defects as well as deformation texture information due to its smaller wavelength [3,4]. Even techniques have been developed to carry out in-situ straining of materials accompanied by deformation texture measurement using neutron and synchrotron X-ray source [5].

X-ray line profile analysis (XRLPA) in conjunction with micro-texture analysis is very useful in investigating orientation dependent dislocation activity resulting in anisotropic deformation response [6]. There are different approaches followed for X-ray line profile analysis such as Variance method, Fourier analysis and Convolutional Multiple Whole Profile (CMWP) fitting method, depending on the microstructure feature dimension of concerned material. For instance, the evolution of dislocation during straining of fcc Cu alloy and Al alloy and bcc high entropy alloy under monotonic tensile and compression mode has been studied using variance approach for XRLPA of Synchrotron X-ray and TEM diffraction data [7–9]. On the other hand, XRLPA using CMWP approach for detailed characterization of dislocation of hcp commercially pure titanium and zirconium alloy subjected to various thermo-mechanical processing such as rolling, equal channel angular pressing etc. has been carried out [10–13]. A specific study on the cyclic deformation behavior has been carried out using Electron channeling contrast imaging (ECCI) images in combination with EBSD based strain indicators in Fe-Si steel [14]. Hence, an extensive literature survey reveals that there is no study reporting characterization of orientation dependent ratcheting or cyclic creep behavior using XRLPA.

Cyclic creep behavior is of immense importance for the purpose of improvement of service life of structural component material. In the past, detailed TEM analysis has been carried out on the cyclic creep behavior of polycrystalline copper explaining the phenomenon of acceleration and deceleration of steady state creep rate occurring due to “strain burst” involving disruption and rearrangement of dislocation cell structure [15]. Such organization of dislocation cell structure of saturated dislocation density occurs in the habit plane along the slip direction, which is responsible for strong anisotropy in fatigue life during ratcheting [16].

However, the materials considered so far have fcc crystal structure and slip is the dominant mode of deformation. Very few studies have been carried out on materials with hcp crystal structure which has also twinning as dominant mode of deformation. Hot rolled magnesium alloy showed in-plane anisotropy in ratcheting response [17]. However, reason behind the anisotropy in mechanical property of hcp system is not well explained in literature. This may be due to twinning phenomenon which occupies almost 50% of parent grain and induces change in orientation of twinned region too. Also, the difficulty in quantifying its effect arises from the numerous types of interactions, including primary twin-matrix interaction, primary twin-secondary twin interaction and most importantly dislocation interactions with all of these types of obstacles. Hence, the role of twinning and its effect on material behavior in polycrystalline titanium is very important which involves more complex interaction compared to magnesium and it has been the subject of most recent researches however, is still not fully understood.

Therefore, in the present paper correlation of defect structure with orientation dependent slip/twin activity during monotonic tension and cyclic loading in commercially pure titanium has been carried out. The statistical information of defect structure obtained from synchrotron X-ray diffraction in transmission mode has been combined with deformation texture information of from electron backscatter diffraction to correlate the observed anisotropy in tensile and cyclic deformation and failure in commercially pure titanium.

Table 1
Sample designation of tensile, stress control and strain control samples for A and B orientation.

Orientation	Tensile	Stress control	Strain control
A	AT	AR	AC
B	BT	BR	BC

2. Experimental

Cold rolled and annealed plate of grade 2 commercially pure titanium was taken for the present investigation. Flat tensile and fatigue test specimen of ASTM standard E 606 were machined with loading axis parallel to rolling direction (RD) and transverse direction (TD) from normal direction (ND) and rolling direction RD plane, respectively. Fatigue tests were carried out in both stress control and strain control mode. In stress control mode, asymmetric stress cycle was applied which is also known as ratcheting test. The specimen dimension and specimen orientation are reported in [18]. Samples from normal plane and rolling plane were referred as A and B orientation respectively and the corresponding sample designation for tensile (T), stress control (*R*) and strain control (C) were listed in Table 1. Stress amplitude ($\sigma_a = 0.8\sigma_y$) and mean stress ($\sigma_m = 0.4\sigma_y$) was used for stress control mode and for strain control cyclic loading, strain amplitude of 0.83% was used. Deformation texture was measured in PETRA III@DESY/Hamburg using synchrotron X-ray radiation in transmission geometry at approximately 1 mm away from fracture tip. The high energy beamline HEMS P07B with energy of 87 keV corresponding to a wavelength of 0.143155 Å was used. In order to obtain sufficient local resolution required for deformation texture measurement $0.3 \times 0.3\,mm^2$ slit was used. The experimental settings for this instrument required that the samples were cut into 15 mm long and 1.5 mm × 1.5 mm (thickness x width) rods. A rotation stage was used to obtain 37 images with PE XRD 1621 type solid state area detector every 5° (+90° to −90° span around vertical axes), generating a set of complete Debye-Scherrer rings for each image and allowing to gather information on all orientation planes in the same measurement. Exposure time for a single shot scan (only one image) was 3 s per image by adding 5 frames and for texture measurement (37 images) fast scans with exposure time of 1 s per image were carried out in 10 frames. Detector calibration was performed using Al_20_3 powder NIST standard. Data evaluation includes corrections of primary intensity, absorption and exposed volume followed by pole figure data transfer in correct format using Fit 2D software. The data obtained from the pole figures were used to calculate the complete orientation distribution function (ODF) using Resmat software [19] and complete pole figures was recalculated. Also, the x-ray diffraction data were used for line profile analysis using variance method [20]. At about 1 mm away from the fracture tip the specimen surface was electropolished using A3 electrolyte and subjected to micro-texture investigation using a field-emission gun scanning electron microscope (JSM-7100F FE-SEM) fitted with Oxford Channel 5 electron back scatter diffraction (EBSD) attachment. An area of 200 × 200 μm^2 was scanned with a step size of 0.5 μm.

3. Results

3.1. Initial microstructure and texture

Fig. 1(a) represents the initial microstructure of cold rolled and annealed plate of commercially pure titanium showing equiaxed alpha grains. The misorientation angle distribution shown in Fig. 1(b) shows two peaks at about 30° and 75° corresponding to grains tilted away from ND towards TD in same and opposite direction, respectively. Fig. 1(c) shows inverse pole figures (IPF) along loading direction for the