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Scanning and transmission electron microscopy investigations of defect arrangements in a two-phase γ-TiAl alloy

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ABSTRACT

Different methods of scanning and transmission electron microscopy (SEM, TEM) were applied on a γ-TiAl alloy TNB-V5 after a thermo-mechanical fatigue test. Electron channelling contrast imaging (ECCI) and electron backscattered diffraction were carried out on bulk specimen. In addition, ECCI and scanning transmission electron microscopy in the SEM were carried out on a TEM foil in the electron opaque and the electron transparent region, respectively. The investigations were completed by transmission electron microscopy in the form of standard bright field imaging as well as by taking corresponding diffraction patterns. The results demonstrate in an impressive way that the ECCI technique applied in scanning electron microscopy can successfully supplement or in some cases replace imaging of dislocation arrangements in TEM.

1. Introduction

The commonly used technique for the investigation of the deformation microstructure on the nano/microscale is transmission electron microscopy (TEM). It allows the investigation of lattice defects like dislocations, stacking faults and/or microtwins in individual grains. In addition, information on the crystallographic orientation of the investigated grain is available. However, the investigated number of grains is very much restricted due to the limited size of the electron transparent region. In this context, advanced techniques applied in scanning electron microscope help to overcome these restrictions. Complementary to TEM, the combination of the electron channelling contrast imaging (ECCI) technique and electron backscattered diffraction (EBSD) in a high-resolution field emission SEM (HR-FE-SEM) can be applied to bulk specimens.

The ECCI technique can be used for the characterisation of microstructures after plastic deformation, e.g. [1–5], because it allows the visualisation of condensed dislocation arrangements near the surface of bulk specimens, e.g. dislocation walls/cells after fatigue [1,6–9]. Even the imaging of individual dislocations [5,10–12] is possible under defined diffraction conditions. The main advantages of the ECCI technique compared to transmission electron microscopy (TEM) are that (i) images can be taken from bulk specimens making this technique very attractive for the examination of defects during in-situ deformation and (ii) much better statistics on the dislocation arrangements can be obtained. The application of the ECCI technique in combination with EBSD measurements in a SEM gives a great potential for investigating and improving the knowledge on the underlying deformation mechanisms and to clarify the deformation behaviour of metals, e.g. tensile and cyclic deformation of TRIP steels [11,12]. ECCI performed under exact Bragg condition reveals excellent contrast for investigations of dislocations [13]. Defects like individual dislocations lead to a local lattice distortion and as a consequence the Bragg condition is locally not fulfilled. Therefore, individual dislocations will appear as bright lines in a dark matrix. Applying an inverted signal of BSE contrast, inverted ECC images are obtained, where dislocations appear as dark lines in a bright matrix in analogy to TEM bright field micrographs.

Titanium aluminide (γ-TiAl) alloys with their low density (3.7–4.7 g/cm^3), high melting temperature (in the range of 1700 K) and good oxidation resistance are attractive candidates for high temperature applications in the automotive and aerospace industry and have been in focus of scientific interest since many years [14]. Most γ-TiAl alloys are based on a two phases microstructure which contains the tetragonal γ-TiAl phase with a $L1_0$-structure and the hexagonal α_2-Ti_3Al phase with a DO_{19}-structure. Four different types of microstructures formed by these two phases are known [15]: near gamma (NG), duplex (DP), nearly lamellar (NL) and fully lamellar (FL). γ-TiAl alloys of the 3rd generation containing higher concentrations of Nb (5–10%) exhibit a high strength combined with acceptable room temperature ductility and damage tolerance due to their fine-grained microstructure.

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The deformation behaviour of the γ-phase is determined by dislocation glide processes in the $L1_0$-structure [16]. Dislocation glide is activated on {111} γ planes along the close packed directions ⟨110⟩. Both, ordinary dislocations of Burgers vector $b=(1/2)\langle 110]$ as well as super-dislocations with the Burgers vector $b=(1/2)\langle 112]$ or $b=\langle 101]$ can occur. A second possible deformation mechanism is the formation of deformation twins along $(1/6)\langle 11-2]$ on the {111} γ plane. The complex dissociation in partial dislocations forming planar defects like antiphase boundaries or stacking faults was observed for super dislocations of type (1/2)⟨112] and ⟨101] [17]. A dissociation in partial dislocations is not reported for ordinary dislocations of type (1/2) ⟨110]. The deformation behaviour of two phases γ-TiAl alloys up to high temperatures (973 K) is determined by the easy activation of ⟨110] dislocation glide and mechanical twinning [17]. The motion of super-dislocations is suppressed in this temperature regime [18]. Henaff and Gloanec [19] showed that in Ti–48Al–2Nb–2Cr specimens which experienced LCF at temperatures as low as 723 K the dislocation structure consists of ordinary dislocations which showed signs of recovery and climb processes. Corresponding features are also remnant dislocation loops, which were reported by Jouiad et al. [20] in the same alloy for LCF at 1023 K. Also Appel et al. [21] found climbed and cross slipped dislocation segments in a Ti–45Al–8Nb–0.2C alloy after LCF at 823 K. But, besides these features also dense dislocation walls are reported by Appel et al. [22] in the same alloy also at 823 K.

The main objective of the present paper is to demonstrate the application of the ECCI technique to study the dislocation arrangements in comparison with TEM investigations and STEM investigations in a SEM. For these investigations the γ-TiAl alloy (TNB-V5) with a duplex microstructure after a thermo-mechanical fatigue test [23] was chosen.

2. Material and deformation tests

Investigations of the microstructure after a thermo-mechanical fatigue test were carried out on the γ-TiAl alloy TNB-V5. The material with the nominal chemical composition Ti–45Al–5Nb–0.2C–0.2B (at%) was produced by Plansee AG (Reutte). The investigated material showed a so-called duplex structure with 40% of globular γ-TiAl grains and 60% of lamellar colonies with γ/α_2-lamellae. The mean grain size of globular γ-TiAl grains is about 2–4 μm, the size of lamellar colonies ranges from 5 to 25 μm and the inter lamellar spacing varies from 50 to 500 nm. Fig. 1 shows an overview of the duplex microstructure (SEM micrograph, backscattered electron contrast).

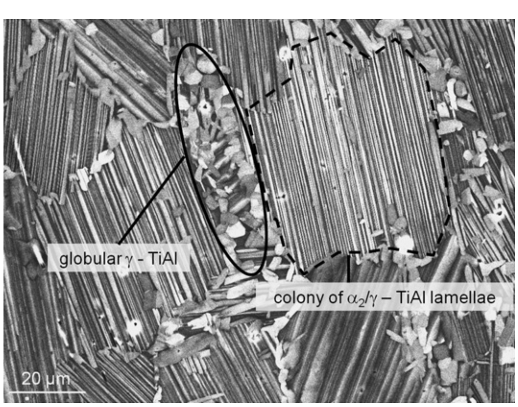

Fig. 1. Duplex microstructure of the investigated γ-TiAl-alloy TNB-V5 consisting of globular γ-TiAl grains and colonies of α_2/γ-TiAl lamellae. SEM micrograph using backscattered electron contrast.

The TMF test was performed on a compact cylindrical specimen with a gauge length of 19 mm and a gauge diameter of 7.0 mm. The specimen surface was electrolytically polished. The TMF test was carried out on a servo hydraulic testing system under total-strain control mode with a mechanical strain amplitude of $\varepsilon_{a,mech}=5.75\times10^{-3}$ and a strain rate of $9.20\times10^{-5}\,s^{-1}$ [23]. The applied temperature interval was $673\,K\leq T\leq 923\,K$. The TMF temperature–strain cycles were applied in-phase (IP) and both, the mechanical strain cycles and the temperature cycles, had a triangular shape. The specimen was heated with a high frequency induction heater at a heating rate of 2 K/s. Axial temperature deviations within the gauge length were below ±5 K [24]. The corresponding cyclic deformation curve and stress–strain hysteresis loops are shown in Fig. 2. Initially, the stress amplitude slightly decreases reaching saturation at about 10 cycles. Furthermore, it becomes evident from the stress–strain hysteresis loops that a compressive mean stress is present from the beginning of deformation. This compressive mean stress is increasing with ongoing deformation as a result of a decreasing upper stress [25].

After failure at $N_f=742$ cycles, the specimen was cut parallel to the loading axis. One half of the specimen was prepared for SEM investigations. From the other part TEM foils were prepared. EBSD and ECCI investigations on the bulk specimens were performed in the plane parallel to the loading axis using field emission scanning electron microscopes (LEO 1530 from ZEISS, MIRA3 XMU from TESCAN). The OIM software from TSL was used for EBSD measurements under a specimen tilt of 70° and an acceleration voltage of 20 kV. The ECCI investigations were performed using a retractable four-quadrant backscattered electron detector allowing working distances of 8 mm. The MIRA 3 XMU works with a

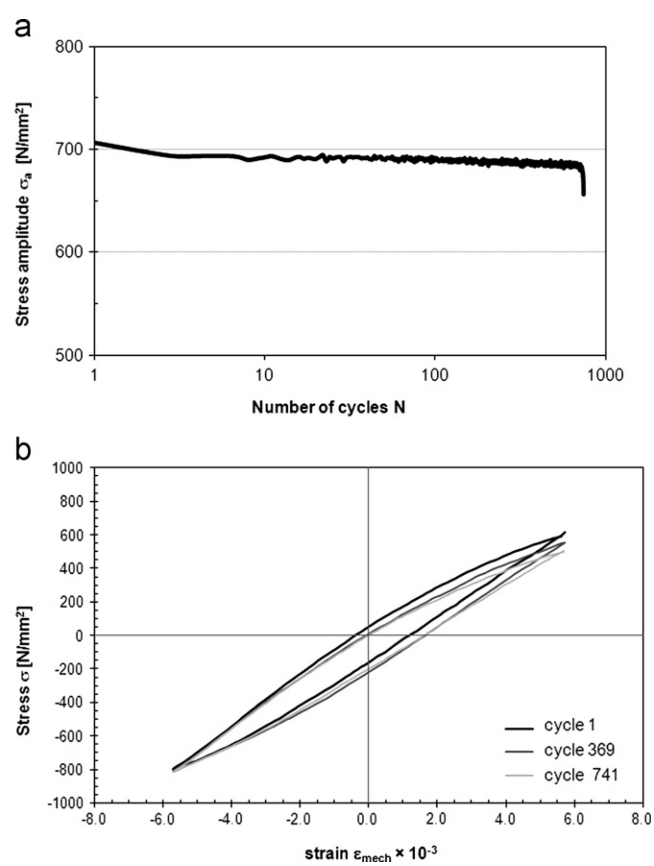

Fig. 2. Cyclic deformation curve (a) and stress–strain hysteresis loops (b) of an in-phase thermo-mechanical fatigue test of a DP-TNB-V5 alloy with $\varepsilon_{a,mech}=5.75\times10^{-3}$ in the temperature interval 673 K ≤ T ≤ 923 K [25].