



Investigation of the tensile deformation behaviour in Ni-based superalloy inconel alloy 617 using EBSD-based finite element simulations and optical flow method

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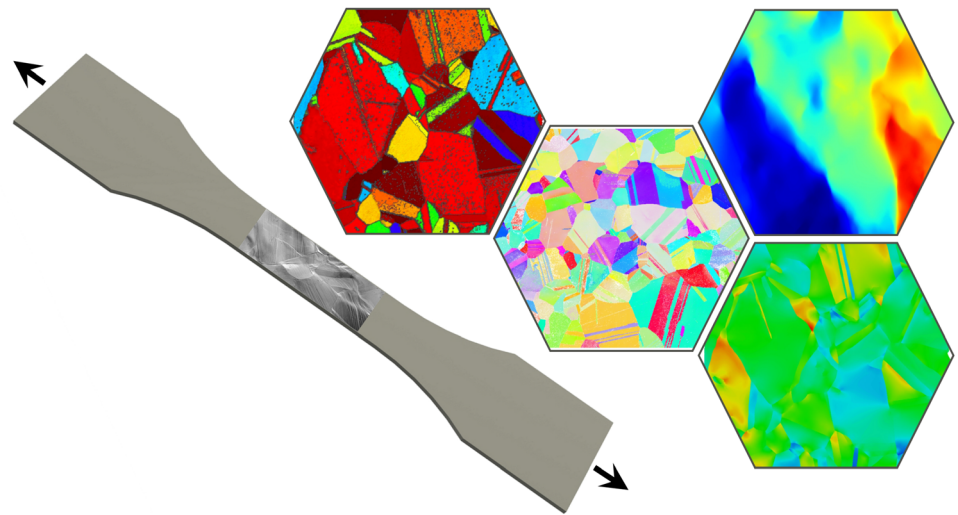
ABSTRACT

To improve component design, the fundamental understanding of the fatigue behaviour of gas turbine materials is essential. Since Ni-alloys exhibit pronounced elastic anisotropy, the local grain orientation strongly affects the stress and strain distribution in the material under mechanical loadings. This work addresses the characterisation of anisotropic elastic–plastic deformation and its consequences for crack initiation of nickel-base superalloy IN617 under tensile loading. Samples were loaded in situ in a scanning electron microscope (SEM) to correlate the deformation behaviour with the grain structure and the grain orientation determined by electron backscatter diffraction (EBSD) measurements. To calculate the resulting stresses and strains, the EBSD data were used to develop a model by finite element method (FEM) considering the grain structure and orientation. The results of the elastic–plastic finite element (FE) simulation were compared with the theories of the E-m model based on the Schmidt factor (m) and anisotropic Young's modulus (E). A mathematical image registration method called “optical flow method” (OFM), which is capable to calculate the transformation of EBSD measuring points during deformation, was applied to the EBSD data. The strains calculated by the optical flow method and by FE simulation were compared for two samples. The findings revealed large strains in the later crack initiation area found in both the OFM and FEM. The developed FEM model was verified by the successful correlation of hypotheses of the E-m model with the simulated mechanical behaviour. Furthermore, the impact of the microstructural neighbourhood on the mechanical behaviour was emphasised.

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GRAPHICAL ABSTRACT



Introduction

High-temperature materials such as nickel-based superalloys provide long-term stability and adequate mechanical properties at temperatures permanently exceeding 500 °C [1, 2]. The applications include aero engine or power plant turbine blades and discs for cast alloys. Sheet material such as the here investigated alloy IN 617 is used for high-temperature ducting, combustion cans or hot gas transition liners [3]. Cast components, e.g. turbine blades, are manufactured by three different routes resulting in single crystals, directionally solidified structures or randomly oriented polycrystals [4]. A subsequent thermal treatment is often used to increase mechanical strength by coherent $\text{Ni}_3(\text{Al,Ti})$ γ' precipitation hardening [5]. Regardless of the individual microstructure or application, all nickel-based superalloys have a crystal anisotropy factor of more than 2.5. Depending on the texture, Young's modulus (E) can vary from 130 to 330 GPa [6], which affects the resulting shear stresses in the slip systems [7], and causes an inhomogeneous stress distribution, local stress peaks and potentially early fatigue failure [8]. The deformation behaviour of nickel-based superalloys has been investigated considering various

parameters and observing phenomena on the macro-, micro- and nanoscale [6, 7, 9–15]. Many investigations show a relation of strain and slip band or twin formation that is always linked to a local plastic deformation in static or cyclic loadings, observed and quantified for example by digital image correlation in combination with electron backscatter diffraction (EBSD) [12, 16, 17]. Simulations of artificially created grain structures subjected to uniaxial loading, considering the local stiffness tensors and Schmid factors (m) were conducted using finite element method (FEM). In previous research, the consideration of local stiffness tensors and local Schmid factors (m) was described as the E-m model [7, 9, 15, 18, 19]. The simulated response of the material to loading was analysed considering the E-m model.

Schmid's law describes the resulting shear stress τ_{res} under a uniaxial tensile load σ considering the slip systems of crystalline material given by the angle of the slip plane normal λ and the angle of the slip direction κ with respect to the tensile axis which are combined to the Schmid factor m [19].

$$\tau_{\text{res}} = \sigma \cdot \cos(\lambda) \cdot \cos(\kappa) = \sigma \cdot m \quad (1)$$

As a background for the subject of this work, the general theories with respect to E and m can be

summarized as follows: grains with low E show elevated elastic deformation after initiation of deformation [20, 21] and large elongation before plasticity [22]. High shear stress [18, 19], plastic deformation and formation of slip bands [7, 15] have been observed for grains with high E - m . Moreover, strongly dissimilar E - m of adjacent grains leads to stress concentrations [8, 18] that are potential fatigue crack initiation sites [7, 9].

In contrast to conservative deterministic methods that imply high safety factors in component design, a probabilistic model for lifetime prediction was developed based on experimental data and finite element (FE) simulations of artificial samples [7, 11, 14]. All these investigations considered local Young's moduli and Schmid factors with respect to the phenomena outlined above.

The study presented in this paper focuses on the deformation behaviour of nickel-based superalloy Inconel 617 under tensile load and experimentally analysed by in situ loadings in the SEM and accompanying EBSD measurements. The results of two samples (sample 1 and sample 2) were discussed and referred to a sample of a previous study [23]. To calculate the local strain, two methods were used and compared. First, the EBSD data were used for an image registration method, that uses the orientation information to identify and to transfer each measuring point from the initial to loaded state, called optical flow method (OFM). Second, the EBSD data were used to develop a virtual model for FE simulations of the same loading scenario. Finally, the results of both methods were compared, verified and correlated with the E - m model described above.

Materials and methods

Inconel 617

The NiCr23Co12Mo alloy IN 617 is widely used in turbine components [3] and was chosen for this investigations due to its grain structure which can be approximated by Voronoi tessellation [24]. This enables a comparatively simple identification of grains and straight-forward interpretation of the results due to sharp and straight grain boundaries. Because the in situ SEM/EBSD measurements were only taken at one side of the sample, and to justify the quasi-two-dimensional approach used in the

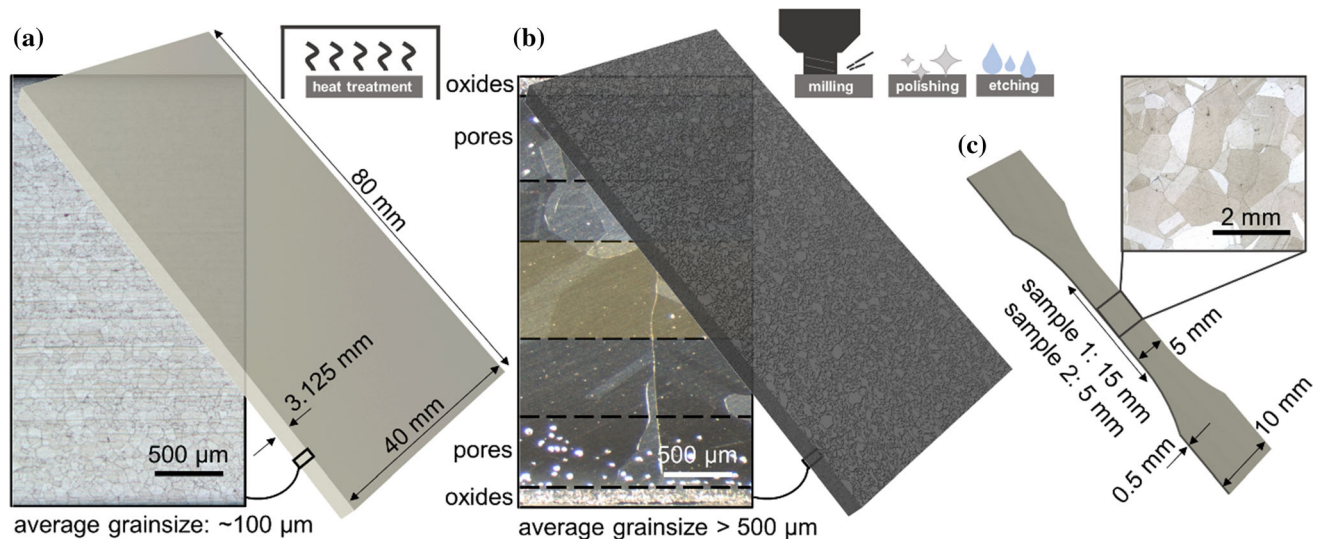
simulations, thin samples with a thickness of 0.5 mm were investigated. The average grain sizes after thermal treatment of 0.585 mm (sample 1) and 0.507 mm (sample 2) were determined by the line intercept method. As these values are larger than the thickness of the samples, it can be inferred that there is largely only one grain in the thickness direction of the samples, legitimating the quasi-two-dimensional approach. Sample 1 was investigated for large elastic and plastic deformation until rupture accompanied by EBSD measurements. Due to the large grain size, the small sample thickness and the highly anisotropic elastic properties, the samples may not necessarily rupture in the gauge, but in the tapered gauge-shaft transition. To increase the probability of failure in the observed area, a comparatively big gauge length of 15 mm was manufactured for sample 1. Sample 2 was loaded until smaller elastic deformation accompanied by EBSD measurements with a high resolution to complement the findings gained from sample 1. Since the EBSD measurements are very time-consuming, the length of the measurement gauge was reduced, so that one measurement is sufficient to cover almost the whole gauge in a reasonable time.

The chemical composition provided by the distributor is listed in Table 1. The low Al and Ti contents compared to cast alloys, e.g. René80 (~ 3 wt% Al and ~ 5 wt% Ti) [25], indicate that $\text{Ni}_3(\text{Al,Ti})$ γ' precipitation hardening is rather limited, which simplifies plastic deformation.

The steps of manufacturing the samples and the respective microstructure of exemplary sections are depicted in Fig. 1. First, an Inconel 617 sheet was divided into smaller pieces by water cutting, showing the initial grain structure with an average size of ~ 100 μm (Fig. 1a). Thermal treatment was performed at 1200 °C for 170 h in air to coarsen the microstructure. Preliminary tests revealed that temperatures equal or higher than 1200 °C are sufficiently high to activate grain growth which, nevertheless, takes considerable time, as shown in the literature, e.g. [27]. However, higher temperatures lead to a more pronounced oxidation and the formation of pores. Considering temperature, time, the resulting grain size as well as oxide and pore formation, a heat treatment at 1200 °C for 170 h showed the most promising results (Fig. 1b). The heating and cooling rates of the thermal treatment are listed in Table 2.

Table 1 Chemical composition of Inconel 617 in wt% [26]

	Ni	Cr	Co	Mo	Fe	Al	Ti	C	Mn	S	Si	Cu
Min	44.50	20.00	10.00	8.00		0.80		0.05				
Max		24.00	15.00	10.00	3.00	1.50	0.60	0.15	1.00	0.015	1.00	0.50

**Figure 1** Sample geometries and the respective microstructure at each manufacturing step for **a** water cut IN617 sheet in initial state and microstructure, in viewing direction to the sheet edge **b** IN617 sheet after thermal treatment, showing an oxide layer and

enhanced pores close to the surface, in viewing direction to the sheet edge **c** finally milled sample geometry with polished and etched surface for the investigations.

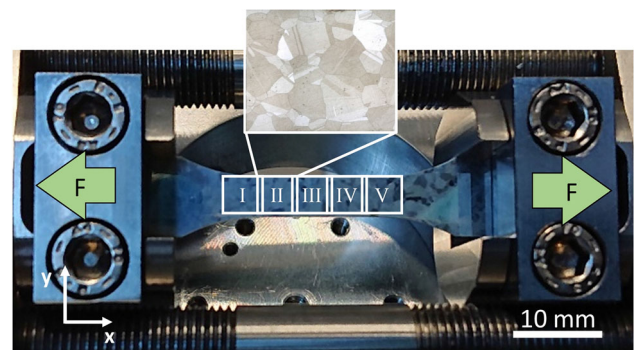
Table 2 Heating and cooling rates of the thermal treatment

RT to 1200 °C	1200–800 °C	800–200 °C	200 °C–RT
5 °C/s	1.5 °C/s	0.25 °C	0.04 °C

An oxide layer of $\sim 100 \mu\text{m}$ and an increased porosity up to of $500 \mu\text{m}$ below the oxide layer are nevertheless apparent after the thermal treatment. Hence, the sheets were milled from both sides until 0.7-mm-thick samples were extracted from the undamaged material volume, as highlighted in Fig. 1b. Finally, the samples were grinded to a thickness of 0.5 mm, polished and etched (Adler-etchant) for investigation.

Tensile loading and EBSD measurements

The tensile load was applied by a Kammrath & Weiss tension–compression-module mounted in the vacuum chamber of an FEI Quanta 600 SEM. The load frame with mounted specimen is shown in Fig. 2. The areas I–V mark the sections that were measured consecutively by EBSD for each applied load level.

**Figure 2** Tensile-compression module for in situ SEM/EBSD measurements in the areas I–V.

The EBSD measurements were taken using a hkl Channel 5 EBSD system with a working distance of 18 mm and a 20-kV beam. The EBSD measurements were accompanied by secondary electron (SE) images for further documentation of the deformation. Two samples were investigated in this work. The parameters of the EBSD measurements are listed in Table 3.

Table 3 Parameters of EBSD measurements

	Resolution: measured points per mm ²	Indexing rate in %	Step size in relation to 100 µm displacement in %
Sample 1	6.66×10^3	75	10
Sample 2	8.24×10^5	94	2
Reference sample [23]	1.75×10^7	91	10

Optical flow method

Considering two images $I_i : \Omega_i \rightarrow \mathbb{R}^2$, $i = 1, 2$, where $\Omega_i \subset \mathbb{R}^2$ is the image domain of image I_i and $M \subset \mathbb{R}^d$ is the range of measured data, say $M = [0, 1]$ for grey-valued images. Then any transformation $\varphi : \Omega_1 \rightarrow \Omega_2$ which maps image I_1 onto image I_2 must satisfy the transport equation

$$I_1(x) = I_2(\varphi(x)), \quad x = (x_1, x_2) \in \Omega_1 \quad (2)$$

The computation of a transformation φ satisfying Eq. 2 is a classical problem in image processing and usually referred to as image registration, see [28, 29] for an overview. Given a transformation φ then the displacement field $u : \Omega_1 \rightarrow \mathbb{R}^2$ and the displacement gradient $\nabla u : \Omega_1 \rightarrow \mathbb{R}^{2 \times 2}$ are defined by Eq. 3.

$$u(x) = \varphi(x) - x, \quad x \in \Omega, \quad \nabla u = \begin{pmatrix} \frac{\partial u_1}{\partial x_1} & \frac{\partial u_1}{\partial x_2} \\ \frac{\partial u_2}{\partial x_1} & \frac{\partial u_2}{\partial x_2} \end{pmatrix} \quad (3)$$

From the displacement gradient we can compute the strain tensor $E : \Omega_1 \rightarrow \mathbb{R}^{2 \times 2}$ given by

$$E = \frac{1}{2} (\nabla u + \nabla u^T + \nabla u^T \nabla u) = \frac{1}{2} (\nabla u + \nabla u^T) \quad (4)$$

For grey-valued images digital image correlation methods (DIC) are used in state-of-the-art software packages for strain analysis, e.g. [30–32]. Recently, variational models have been shown more sensitive to abrupt changes by using priors with higher-order derivatives, cf. [33–35]. In what follows we consider EBSD data, i.e. $M = SO(3)/S$, where

$$SO(3) = \{R \in \mathbb{R}^{3 \times 3} : RR^T = I, \det(R) = 1\} \quad (5)$$

is the rotation group, consisting of the three-dimensional rotation matrices, and $S \subset SO(3)$ is the symmetry group of the underlying crystalline structure. Since EBSD data are sensitive to spatial rotations induced by the deformation, the consistency assumption (see Eq. 2) of the measurement values must be adapted. This behaviour is seen by

measuring one specimen using two reference systems which differ by a spatial rotation. More precisely, let φ be a rigid transformation of the form

$$\varphi(x) = \begin{pmatrix} \cos(\alpha) & -\sin(\alpha) \\ \sin(\alpha) & \cos(\alpha) \end{pmatrix} \begin{pmatrix} x_1 \\ x_2 \end{pmatrix} + \begin{pmatrix} v_1 \\ v_2 \end{pmatrix}, \quad x \in \Omega_1 \quad (6)$$

where $\alpha \in [0, 2\pi]$ is a rotation angle and $v \in \mathbb{R}^2$ is a translation vector. If the surface normal of the specimen points into the x_3 -direction, then the orientation measurements of image I_1 must be rotated by

$$R_\varphi = \begin{pmatrix} \cos(\alpha) & -\sin(\alpha) & 0 \\ \sin(\alpha) & \cos(\alpha) & 0 \\ 0 & 0 & 1 \end{pmatrix} \in SO(3) \quad (7)$$

Hence, taking this change of the orientation measurements into account we arrive at the adapted transport equation.

$$I_1(x) = R_\varphi^T I_2(\varphi(x)), \quad x \in \mathbb{R}^2 \quad (8)$$

For that reason, new variation models which are aware of such orientation changes have been proposed in [23], where the numerical results show that these models are able to reconstruct displacement fields with small and large displacements for synthetic and real data.

Transformation of EBSD data to finite element models

To simulate the deformation according to the experimental setup, the EBSD data were transferred to an FE model in several steps using the MATLAB toolbox MTEX 5.8.1, the finite element mesh generator gmsh 3.0.6, the programming language Python 3.6.0 and the software suite Dassault Simulia Abaqus 2021. The flow chart given in Fig. 3 illustrates the procedure.

The measured EBSD data (see Fig. 3a) were first edited in the MATLAB toolbox MTEX following the documentation [36] to index the data according to the measured orientations, recalculate and identify the grain geometries, fill the grains to eliminate non-

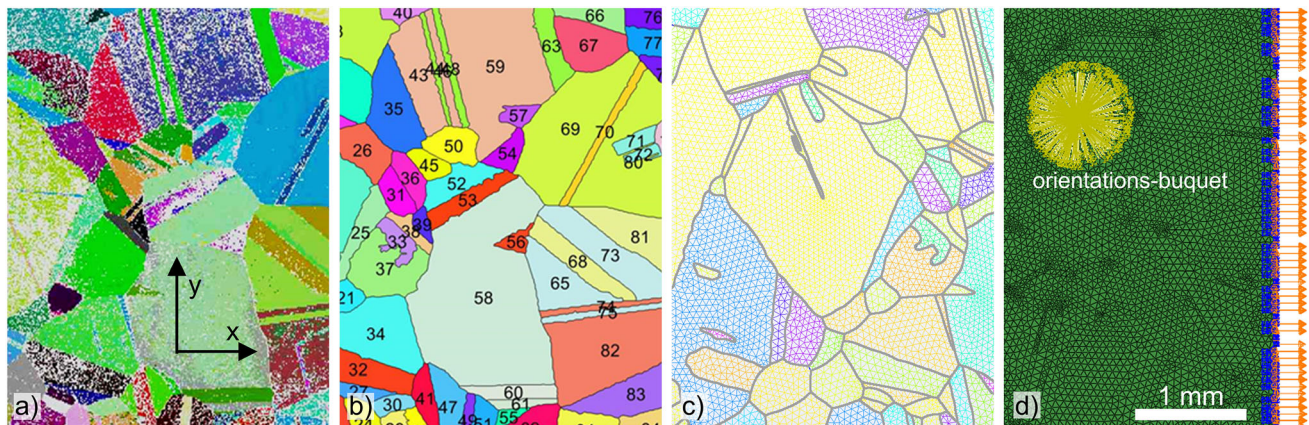


Figure 3 Flow chart of FE model generation with **a** measured EBSD data; **b** recalculated microstructure; **c** meshing; and **d** implementation of texture, material properties and boundary conditions.

Table 4 Material properties used in the FE model

Density [26]	8.5 t/m ³	
Stiffness matrix in GPa [37]	$\begin{bmatrix} 280 & 191 & 191 & & & \\ 191 & 280 & 191 & & & \\ 191 & 191 & 280 & & & \\ & & & 127 & & \\ & & & & 127 & \\ & & & & & 127 \end{bmatrix}$	
Relation between stress and plastic strain	Yield stress in MPa	Plastic strain in %
	200	0
	400	0.53
	470	2.0
	500	3.3

indicated areas and smoothen the grain boundaries to remove sharp edges that disturb the FE simulation (see Fig. 3b). Finally, the orientations were transferred to Euler angles with respect to the grain ID and the recalculated grain geometries were exported to a geometry file (*.geo) to be meshed in the next step using gmsh (see Fig. 3c). The tetrahedral mesh (C3D6) is relatively coarse in the centre of the grains but refined at grain boundaries according to the gmsh algorithm. Gmsh allows to export an input file suitable for Dassault Simulia Abaqus 2021 that is used for the simulations. A Python script was used to assign the measured orientations to the individual grains. In the last step the model is finally prepared for the simulation in Abaqus by implementing directional material properties (see Table 4) and boundary conditions (see model in Fig. 3d). Values for density and stiffness were taken from the literature, whereas the plastic stress–strain relation was taken from the tensile test data of sample 1 (Fig. 4).

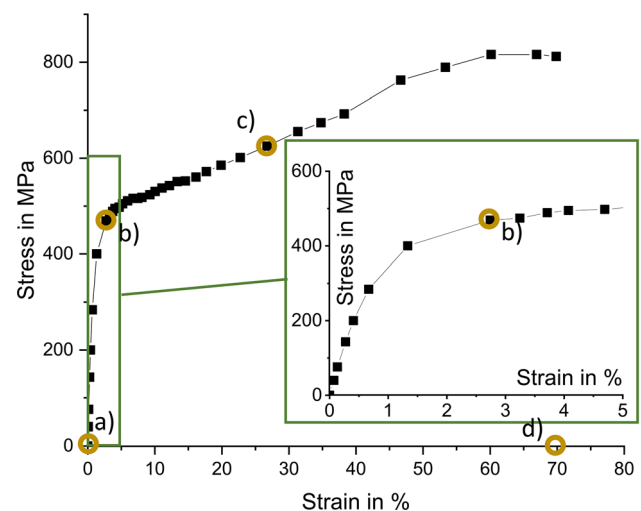


Figure 4 Stress–strain behaviour of sample 1 and points **a–d** referred to Fig. 5.

Results

Sample 1

The first sample was deformed until rupture while measuring the whole gauge length by EBSD (see Fig. 4, all steps between the points a and b) up to a total longitudinal strain of $\varepsilon_{xx} = 2.74\%$. At higher

levels of deformation, EBSD did not provide usable data, but the observation by SE imaging was continued. Different stages during the tensile test of sample 1 until rupture are marked in the stress–strain diagram in Fig. 4.

The stress–strain diagram shows a linear elastic deformation until $\varepsilon_{xx} = 0.41\%$ at 200 MPa. The tensile tests were performed with a strain rate of $0.4\%/min$

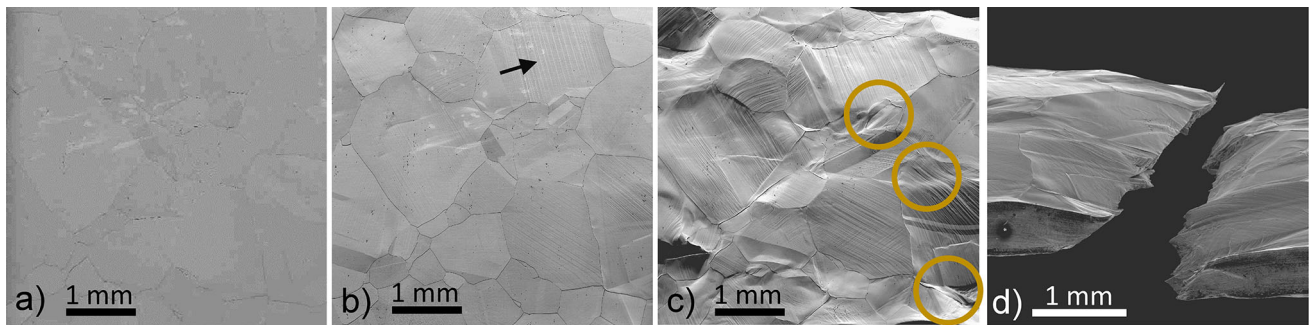


Figure 5 Microstructure of IN617 sample 1 during tensile loading selected total strain levels (ε_{xx}) **a** $\varepsilon_{xx} = 0\%$; **b** $\varepsilon_{xx} = 2.74\%$; **c** $\varepsilon_{xx} = 26.74\%$; and **d** ruptured at $\varepsilon_{xx} = 69.85\%$, see supplementary material S1.

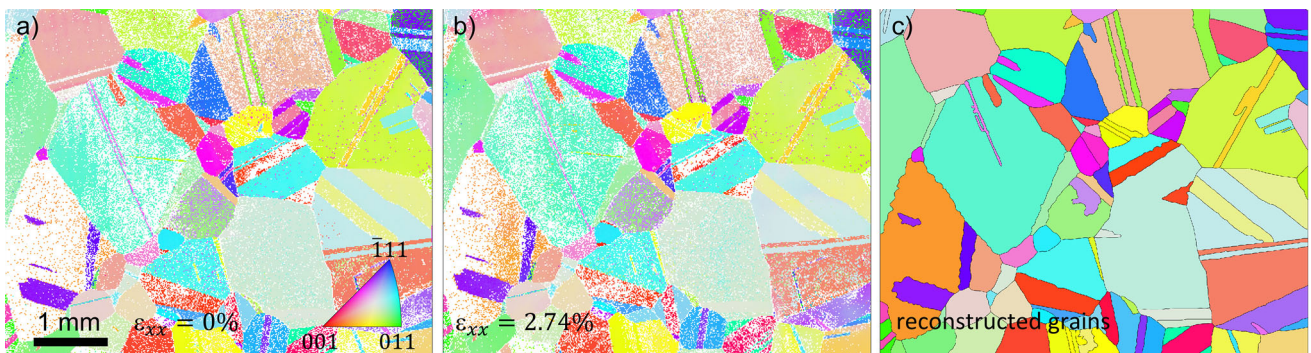


Figure 6 EBSD data at **a** $\varepsilon_{xx} = 0\%$, **b** $\varepsilon_{xx} = 2.75\%$ and **c** reconstructed grain structure of sample 1.

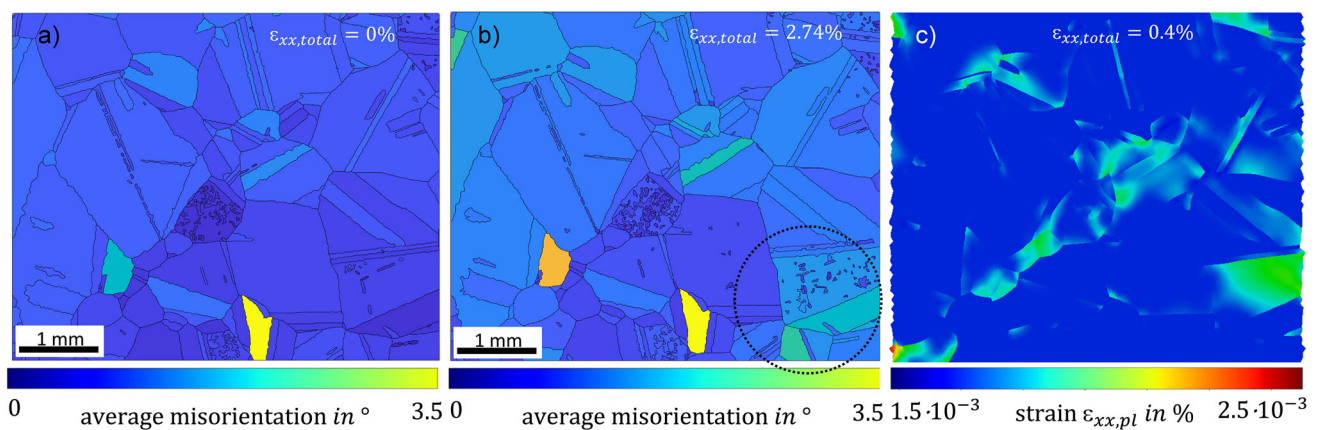
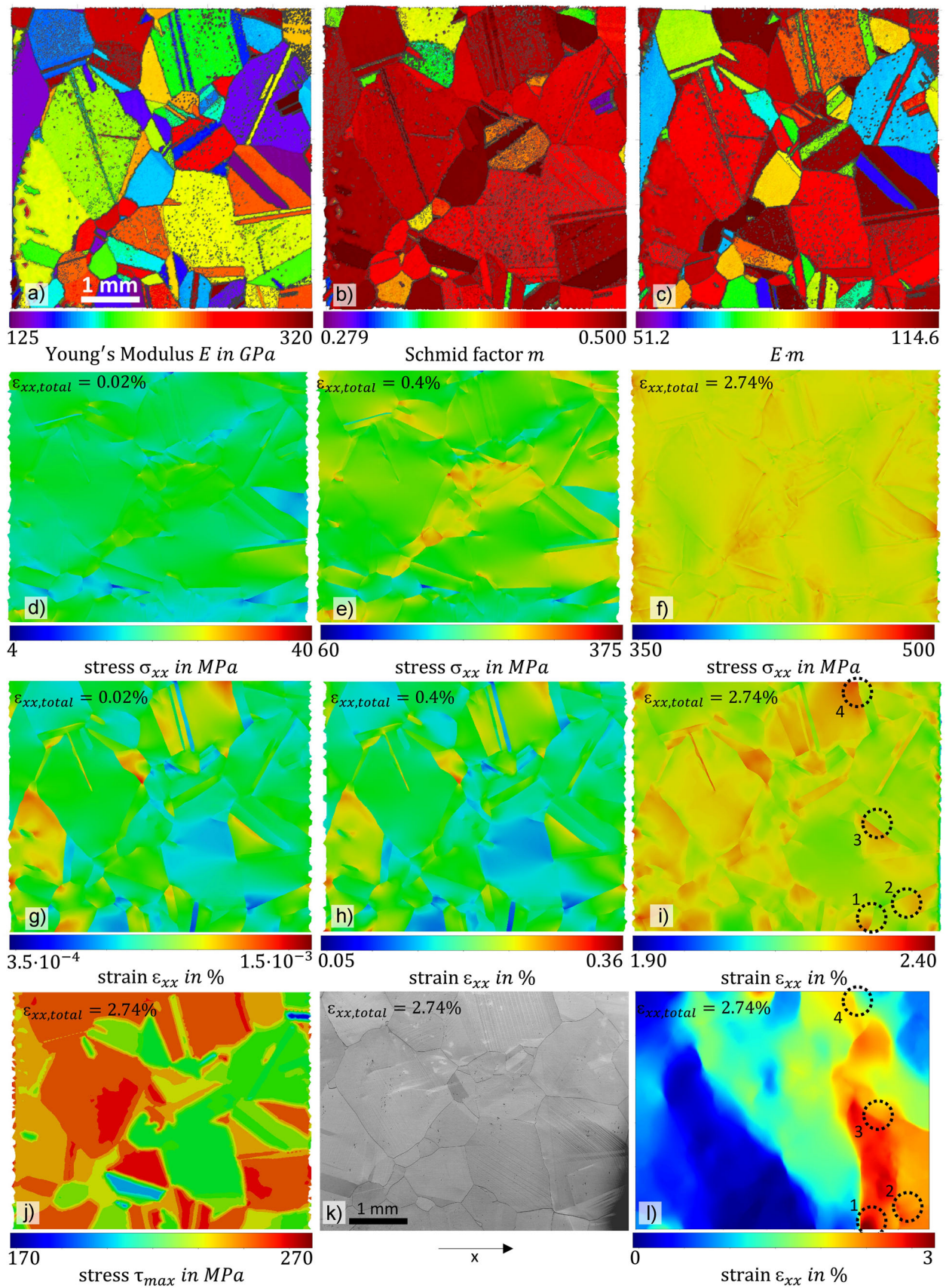


Figure 7 Average misorientation in the grains at **a** $\varepsilon_{xx} = 0\%$, **b** $\varepsilon_{xx} = 2.74\%$ and **c** plastic deformation simulated by FEM at $\varepsilon_{xx} = 0.4\%$.



◀ **Figure 8** Calculated E , m and $E\cdot m$ (a–c), the stress, strain and shear stress simulation (d–j) for different total strains, slip bands in the microstructure (k) and the strain calculated by optical flow method (l).

in the elastic range and 2%/min in the plastic range. After the onset of plastic deformation, the sample shows a large elongation until rupture. SEM images of the specimen surface close to the position of rupture (area II in Fig. 2) taken at the strains highlighted in Fig. 4 are shown in Fig. 5.

Figure 5 shows the microstructure in the initial state, at $\epsilon_{xx} = 2.74\%$, $\epsilon_{xx} = 26.74\%$ and after rupture. The increasing plastic strain results in distinct changes of the surface topography (Fig. 5b and c): All grains in Fig. 5b show more or less pronounced slip bands in all grains. The arrow indicates a group of very pronounced slip bands in one grain. In Fig. 5c, all grains clearly show a high density of slip bands and a three-dimensional deformation of the surface due to anisotropic deformation. Moreover, in the encircled areas of Fig. 5c, pronounced local deformation caused by the deformation mismatch at and close to the grain boundaries can be observed, which is finally followed by rupture (see Fig. 5d).

The EBSD data obtained in the deformation states shown in Fig. 5a and b were used for the strain determination by OFM. Furthermore, the determination of E , m and $E\cdot m$ as well as the recalculated grain structure based on EBSD data later used for FE simulations is shown in Fig. 6. For both states ($\epsilon_{xx} = 0\%$ and $\epsilon_{xx} = 2.74\%$) 75% of the measured points were indexed correctly. Despite lower indexing quality in some grains, the grain structure was successfully reconstructed by the MTEX algorithm (Sect. 2.4).

The average misorientation of each grain in the undeformed and elasto-plastically deformed state at $\epsilon_{xx} = 2.74\%$ is shown in Fig. 7a and b and the calculated beginning plastic deformation by FE simulation at $\epsilon_{xx} = 0.4\%$ (Fig. 7c), because the FE model is only valid for onsetting plastic deformation, which is explained later in the text. As expected, the plastic deformation results in a global increase of the average misorientation. The most distinct changes are observed the grains undergoing the highest plastic deformation according to the FE simulation shown in Fig. 7c.

Figure 8 shows the calculated E , m and $E\cdot m$ (Fig. 8a–c), the stress, strain and shear stresses obtained by FE simulation (Fig. 8d–j) for characteristic total strains ϵ_{xx} , slip bands in the microstructure (Fig. 8k) and the strain field calculated by the OFM (Fig. 8l). The tensile test results (Fig. 2) indicate the onset of macroscopic plastic deformation at $\epsilon_{xx} = 0.4\%$. The simulated deformation starts at a total strain of $\epsilon_{xx} = 0.02\%$, i.e. in the fully elastic regime. The OFM was applied for EBSD data in the initial state $\epsilon_{xx} = 0\%$ and at $\epsilon_{xx} = 2.74\%$, i.e. in the fully plastic regime. Figure 8j shows the resulting shear stress in the active slip system calculated by multiplication of the stress tensor and the modified Schmid factor of the most favourable slip system according to previous work [8].

The simulated stresses and strains (Fig. 8d–j) were analysed under consideration of Hooke's law and the $E\cdot m$ model with respect to the tensile load in x direction given in Fig. 8k. The interaction of differently oriented grains with anisotropic elasticity complicates stress state. However, the $E\cdot m$ model is, at least qualitatively, confirmed by the FE simulations: In the purely elastic regime ($\epsilon_{xx} = 0.02\%$), most of the grains with lower Young's moduli show higher local deformation than neighbouring grains with higher Young's moduli (see Fig. 8g). Also, at the onset of plastic deformation ($\epsilon_{xx} = 0.4\%$), those grains tend to higher longitudinal strain (see Fig. 8h). Grains with higher $E\cdot m$ exhibit higher shear stresses with respect to the slip system (see Fig. 8j) and correspond to the areas of beginning plastic deformation (see Fig. 7c). Moreover, high $E\cdot m$ is correlated with high normal stresses (see Fig. 8e). Neighbouring grains with dissimilar $E\cdot m$ led to large local differences and stress peaks that under cyclic loading may cause fatigue failure. The simulated stress and strain behaviour of the specimen corresponds to the theories of the $E\cdot m$ model in the regime of onsetting plastic deformation, which verifies the here developed FE model. Additionally, the actual crack initiation occurs between two grains that differ in $E\cdot m$ (Fig. 8c) and that show an increased local strain (Fig. 8i). The approximately homogeneous stress distribution at $\epsilon_{xx} = 2.74\%$ indicates a full plastification of the sample and reveals the limit of the applied FE model, since the stress is calculated by continuum mechanics only, but crystal plasticity would have to be considered for correct simulation of local stresses in case of high plastic strains (see Fig. 8f).

The second approach of characterization was the determination of local strain by optical flow method. Comparing the simulated strain distribution at a nominal strain $\epsilon_{xx} = 2.74\%$ (Fig. 8I) and the associated strain field determined by the OFM, the differences are obvious. Unlike the locally varying simulated strain that strongly corresponds to the materials grain structure, the resolution of strain, calculated by OFM, is limited to three relatively large zones with similar deformations (blue, green and red areas in Fig. 8I). Nevertheless, the magnitude of the determined strain (0–3%) is reasonable and the area with the highest strain correlates to the area of later crack initiation (Fig. 8i and I, circle 1). Additionally, some other areas match in all cases. These areas (Fig. 8i and I, circles 2–4), include grain boundaries between grains with large differences in E-m. Since the OFM compares information of measurement points in the initial and deformed state, the accuracy is depending on the resolution measurement points per strain (see Table 3). The EBSD measurements according to the results shown for sample 1 were taken with a step size of 10% in relation to 100 μm displacement. Therefore, the states of lower deformation were not suitable for the analysis by optical flow, because the differences were hardly detectable. Moreover, the indexing rate was only 75%, which can be balanced by reconstruction of the grain structure for the simulations. For the OFM this means that 25% of the measurement cannot be considered for the strain calculations, but since the failed indexing occurred mainly in one large grain, the data were still considered as reliable. However, since the principal of the OFM has been proven in previous works [23] it was assumed that the accuracy and the resolution of the EBSD data were insufficient to resolve the local deformations in detail.

Sample 2

The second sample 2 was solely elastically deformed for EBSD measurements and OFM since the FE model does not consider crystal plasticity model (see Fig. 8f) and therefore is only valid until the onset of plastic deformation. However, sample states at larger strains have been observed by SEM for the detection of slip bands. Additionally, a better index rate was achieved in the EBSD and hence, a more accurate determination of strain by the OFM was expected. However, despite a much larger number of measurement points per area, the smaller deformation leads to a lower transformation of single measurement points and the proportion of change between the initial and deformed state is much smaller. The main differences between the EBSD measurements of sample 1 and 2 are listed in Table 3.

The measured EBSD data and the reconstructed grain structure are shown in Fig. 9. Compared with the data of sample 1 in Fig. 6, the higher resolution and the more accurately reconstructed grain structure

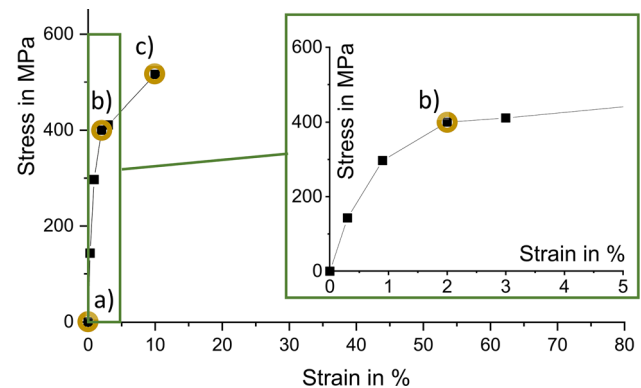


Figure 10 Stress–strain behaviour of sample 2 and points (a–c) referred to * MERGEFORMAT Fig. 11.



Figure 9 EBSD data at a $\epsilon_{xx} = 0\%$, b $\epsilon_{xx} = 0.3\%$ and c reconstructed grain structure of sample 2.

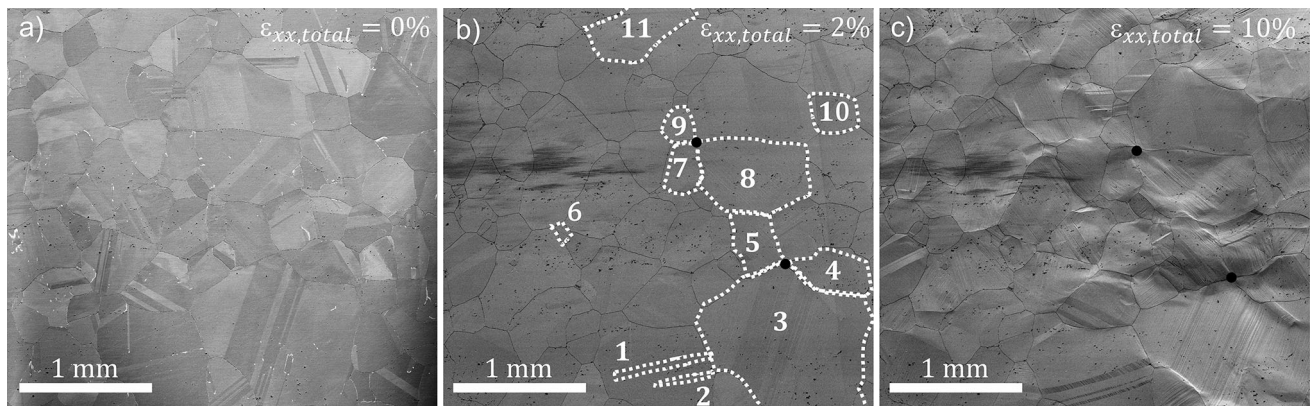


Figure 11 Microstructure of sample 2 under tensile load **a** in initial state, **b** $\epsilon_{xx,total} = 2\%$ and $\epsilon_{xx,total} = 10\%$, see supplementary material S1.

are clearly visible. Additionally, the average grain size is slightly lower (0.507 mm), and the microstructure contains more twins.

Since sample 2 was investigated to complement the findings of sample 1, the following analyses focus on the influence of data resolution on the accuracy of the optical flow method and the correlation between the FE simulations and the E-m model. To detect the initiation of slip band formation, the gauge length was observed, while tensile loads were applied. The load and deformation stages during the tensile test of sample 2 are marked in the stress–strain diagram in Fig. 10. For sample 2, the stress tends to be slightly lower at the same strain levels, but in general the results of both samples correspond to each other.

Figure 11 shows the surface microstructure in the initial state (Fig. 11a), at $\epsilon_x = 2\%$ (Fig. 11b) and $\epsilon_{xx} = 10\%$ (Fig. 11c). Slip bands occurred at $\epsilon_{xx} = 2\%$ in the grains No. 1–11, marked by the dashed lines. In case of grains No. 1 and 2 only the twins showed slip bands and are therefore numbered as separate grains. At a total deformation $\epsilon_{xx} = 10\%$, large elastic and plastic deformation was observed, especially at the triple points between the grains No. 3–4–5 and 7–8–9.

The formation of slip bands indicates the beginning of local plastic deformation that occurs, according to the E-m model, depending on the orientation of each single grain and on the orientation differences between neighbouring grains.

Figure 12 shows the grain specific E, m and E-m (Fig. 12a–c). The simulated stresses and strains are mapped in Fig. 12d–h whereby Fig. 12d shows the maximum shear stress considering the most favourable slip system, analogous to Fig. 8j. The local strain,

determined at $\epsilon_{xx,total} = 0.3\%$ by the optical flow method, is shown in Fig. 12i. The areas with highest strain, determined by the OFM, are highlighted by coarsely dashed lines. Considering the E-m model with respect to slip band formation, almost all affected grains show middle to high E-m, except grain No. 7 which however only partially shows slip bands. Therefore, it is assumed that the slip band formation was induced by the deformation of its neighbouring grain No. 8. Grain No. 11 is almost equally divided into two sections with similar orientation, Young's modulus, Schmid factor and E-m (see Fig. 12a–c). However, an opposing shear strain direction (see Fig. 12g) and remarkably, the majority of slip bands occur in the right section with a slightly higher E-m and shear strain. Grains 3, 4, 5, 8 and 10 also contain twins, and, hence, regions of different crystal orientations, but in these cases slip bands occur all over the grains.

According to the E-m model, the slips systems in grains with high E-m undergo higher resulting shear stress and vice versa, which is confirmed for the grains No. 3, 4, 5, 6, 7, 8, 9 and 10 (see Fig. 12a–d). Grains No. 1, 2 and 11 show high E-m, but low resolved shear stresses in their slip systems and high normal stress (see Fig. 12e). Additionally, high shear stress occurs in many other grains with high E-m, which, however, do not show slip bands, emphasising the importance of the microstructural neighbourhood rather than solely considering E-m for single grains at, and close to, the onset of plastic deformation.

Slip band formation is one effect associated with beginning plastic deformation that is depicted in

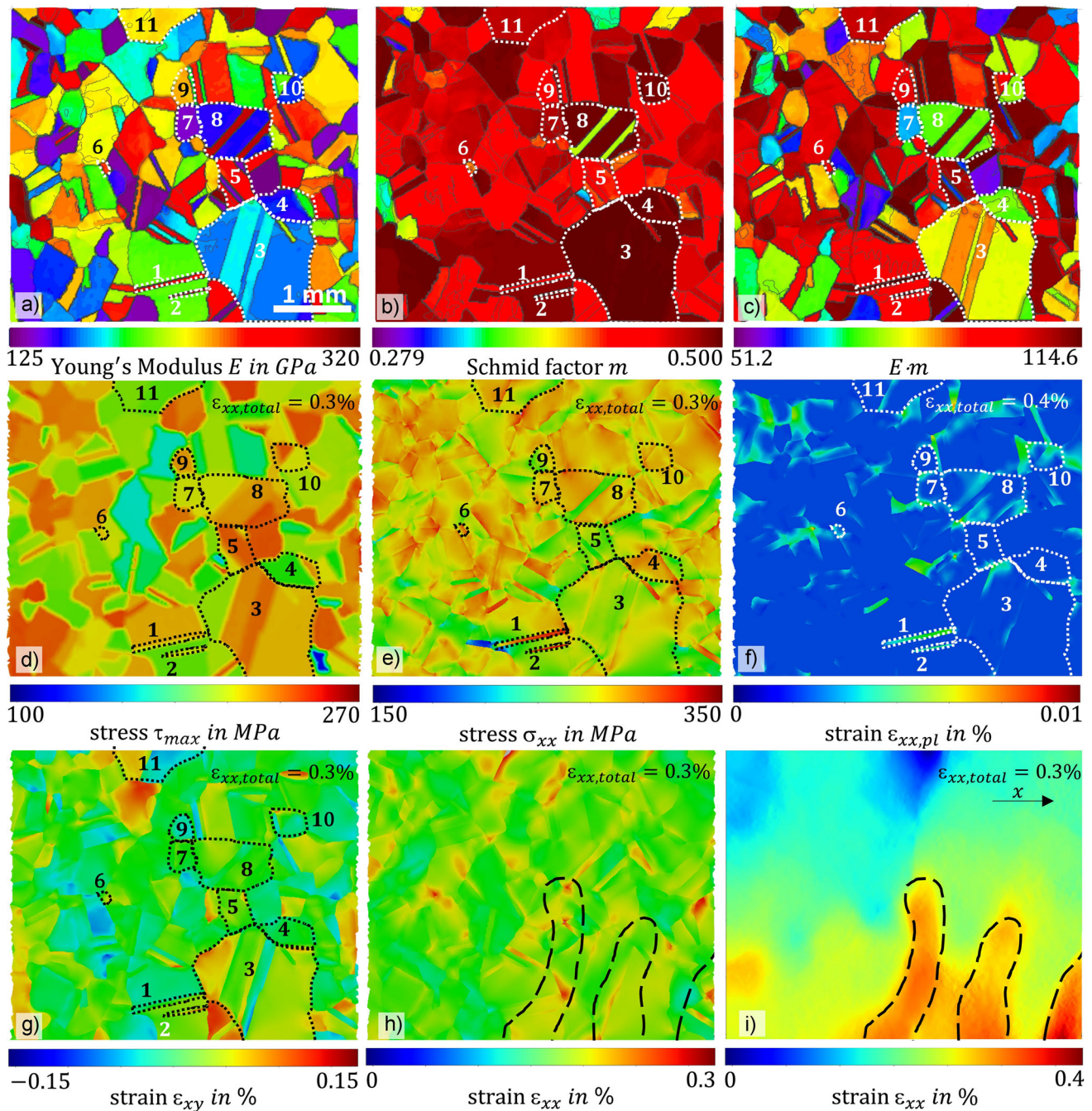


Figure 12 Calculated E , m and $E \cdot m$ (a–c) and materials reaction under tensile load in x-direction: shear stress (d), normal stress (e) and plastic strain (f), shear strain (g), elastic strain (h) and the strain calculated by optical flow method (i).

Fig. 12f at a simulated total strain of $\epsilon_{xx,total} = 0.4\%$. Plastic deformation occurred in or directly near to all grains (No. 1–11) that contained slip bands at $\epsilon_{xx,total} = 2\%$ (see Fig. 11b). Since slip bands were visually detected by SEM observation of the surface, there is a possible delay between the initiation of plastic deformation and visibility of resulting slip

bands. However, all grains that include slip bands show plastic stain in the simulation (see Fig. 12f). Hence, the other few regions that show plastic deformation in the simulation may have been also plastically deformed in the experiment, but without visible slip bands.

The strain determined by OFM based on EBSD data between the initial state and at $\varepsilon_{xx, \text{total}} = 0.3\%$ is shown in Fig. 12i. Three characteristic areas with the highest values around 0.4% are clearly visible. In contrast, Fig. 12h shows that the simulated strain is more equally distributed in the whole area and reaches the maximum of 0.3% at several places. Nevertheless, the area of maximum strain determined by OFM (Fig. 12i) coincides with zones of large simulated strains (Fig. 12h) and contains some areas that reached the maximum deformation determined by FEM. However, the accordance between strain determined by OFM and by finite element simulation is even less than for sample 1 despite the higher resolution and indexing rate, but the lower transformation of measurement points in relation to the investigated area size and the fact that less measurement points were considered in relation to strain or larger step size per displacement, respectively (see Table 3), obviously limited the quality of the OFM analyses.

Summary and conclusions

The mechanical behaviour of quasi two-dimensional samples of the nickel-based superalloy IN 617 under tensile loading has been investigated using SEM/EBSD data. The behaviour was analysed using an image registration method—the optical flow method—that has been presented in a previous work [23] and by a specially developed FE model. The results of two samples were presented.

Sample 1 was deformed far into the plastic regime up to 8% total strain accompanied by EBSD measurements and then loaded until rupture. The large deformation caused a large translation of the individual measurement points, which facilitated the strain determination by OFM. In contrast, sample 2 was only elastically deformed. Therefore, the displacement of a single measurement point relative to the size of the investigated area was much smaller, making an accurate strain determination by OFM more difficult.

The resulting simulated stresses and strains were considered in relation to the established E-m model and Hooke's law. The results confirmed the validity of the E-m model and of the FE model up to the onset of plastic deformation. The FE simulations included continuum mechanics-based plastic deformation, but

no explicit crystal plasticity model. Therefore, the results are only valid up to the onset of plastic deformation (see Fig. 12f).

The strain fields determined by OFM showed only limited correlation with the FE simulations for both samples. Nevertheless, for sample 1, the region of later crack initiation and some effects of the microstructure on the local deformation were detected in agreement with the FE simulations. Sample 2 showed a rather poor overall correlation between the strains determined by OFM and FEM. However, the method was confirmed by a reference sample in previous work. Therefore, it is assumed that the experimental parameters of both samples were insufficient for the OFM.

The major findings of this work are:

- A continuum mechanics-based, elasto-plastic FE model has been successfully developed for IN617, considering the grain structure and elastic anisotropy. The model was successfully validated up to the onset of plastic deformation.
- The E-m model was confirmed by the FE simulations and experimental observations regarding resulting average stresses and strains in the individual grains as well as the formation of slip bands.
- However, the E-m model is not capable to consider the complex interaction of adjacent grains in elastically anisotropic polycrystalline materials.
- The strain determined by OFM correctly indicated area of maximum strain in the FE simulation where also crack initiation took place at the end of the tensile test performed at sample 1. However, the general correlation between the results of OFM and FEM was limited.
- The successful application of the OFM for the determination of strain fields requires both a sufficiently large shift, i.e. a large number of measurement points in relation to the deformation and a very high indexing rate.

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Author contributions

ML, TB, MG, GS helped in conception and planning of the work that led to the manuscript or acquisition; ML executed the experiments; ML, PL developed the finite element models; MG, GS developed the optical flow method; ML, PL, TB, MG, GS analysed and interpreted the data and approved the final submitted version of the manuscript; ML, MG drafted the manuscript; PL, TB, GS critically revised the manuscript.

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Data availability

The datasets used and analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests The authors declare no competing financial interests.

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References

- [1] Heat-resistant materials (1997), 1st edn. ASM specialty handbook. ASM Internat, Materials Park, Ohio
- [2] Reed RC (2006) The superalloys: fundamentals and applications, 1st edn. Cambridge University Press, Cambridge
- [3] Smith GD, Yates DH (1991) In: Volume 5: Manufacturing Materials and Metallurgy; Ceramics; Structures and Dynamics; Controls, Diagnostics and Instrumentation; Education; IGTI Scholar Award; General. American Society of Mechanical Engineers. <https://doi.org/10.1115/91-GT-161>
- [4] Ma D (2018) Novel casting processes for single-crystal turbine blades of superalloys. *Front Mech Eng* 13(1):3–16. <https://doi.org/10.1007/s11465-018-0475-0>
- [5] Perrut M, Caron P, Thomas M, Couret A (2018) High temperature materials for aerospace applications: Ni-based superalloys and γ -TiAl alloys. *C R Phys* 26:657–671. <https://doi.org/10.1016/j.crhy.2018.10.002>
- [6] Zhang X, Oskay C (2016) Polycrystal plasticity modeling of nickel-based superalloy IN 617 subjected to cyclic loading at high temperature. *Modelling Simul Mater Sci Eng* 24:0550099. <https://doi.org/10.1088/0965-0393/24/5/055009>
- [7] Engel B, Beck T, Moch N, Gottschalk H, Schmitz S (2018) Effect of local anisotropy on fatigue crack initiation in a coarse grained nickel-base superalloy. *MATEC Web Conf.* <https://doi.org/10.1051/mateconf/201816504004>
- [8] Engel B, Mäde L, Lion P, Moch N, Gottschalk H, Beck T (2019) Probabilistic modeling of slip system-based shear stresses and fatigue behavior of coarse-grained Ni-base superalloy considering local grain anisotropy and grain orientation. *Metals* 9:813. <https://doi.org/10.3390/met9080813>
- [9] Gottschalk H, Schmitz S, Seibel T, Rollmann G, Krause R, Beck T (2015) Probabilistic schmid factors and scatter of low cycle fatigue (LCF) life. *Mat-wiss u Werkstofftech* 46:156–164. <https://doi.org/10.1002/mawe.201400371>

- [10] Gottschalk H, Moch N, Schmitz S, Beck T Von Mikroskopischen Modellen der Schadensakkumulation zur Ausfallwahrscheinlichkeit von Gasturbinen - Schlussbericht Laufzeit: 01.12.2014–30.11.2017. Von Mikroskopischen Modellen der Schadensakkumulation zur Ausfallwahrscheinlichkeit von Gasturbinen - Schlussbericht. <https://doi.org/10.2314/GBV:1027945082>
- [11] Mäde L, Schmitz S, Gottschalk H, Beck T (2018) Combined notch and size effect modeling in a local probabilistic approach for LCF. *Comput Mater Sci* 142:377–388. <https://doi.org/10.1016/j.commatsci.2017.10.022>
- [12] Stinville JC, Echlin MP, Callahan PG, Miller VM, Texier D, Bridier F, Bocher P, Pollock TM (2017) Measurement of strain localization resulting from monotonic and cyclic loading at 650 °C in nickel base superalloys. *Exp Mech* 57:1289–1309. <https://doi.org/10.1007/s11340-017-0286-y>
- [13] Stinville JC, Lenthe WC, Echlin MP, Callahan PG, Texier D, Pollock TM (2017) Microstructural statistics for fatigue crack initiation in polycrystalline nickel-base superalloys. *Int J Fract* 208:221–240. <https://doi.org/10.1007/s10704-017-0241-z>
- [14] Engel B, Huth M, Hyde C (2022) Numerical investigation into the influence of grain orientation distribution on the local and global elastic-plastic behaviour of polycrystalline nickel-based superalloy INC-738 LC. *Crystals* 12:100. <https://doi.org/10.3390/cryst12010100>
- [15] Bettge D, Österle W (1999) “Cube slip” in near-[111] oriented specimens of a single-crystal nickel-base superalloy. *Scripta Mater* 40:389–395. [https://doi.org/10.1016/S1359-6462\(98\)00446-1](https://doi.org/10.1016/S1359-6462(98)00446-1)
- [16] Stinville JC, Echlin MP, Texier D, Bridier F, Bocher P, Pollock TM (2016) Sub-grain scale digital image correlation by electron microscopy for polycrystalline materials during elastic and plastic deformation. *Exp Mech* 56:197–216. <https://doi.org/10.1007/s11340-015-0083-4>
- [17] Charpagne MA, Hestroffer JM, Polonsky AT, Echlin MP, Texier D, Valle V, Beyerlein IJ, Pollock TM, Stinville JC (2021) Slip localization in Inconel 718: a three-dimensional and statistical perspective. *Acta Mater* 215:117037. <https://doi.org/10.1016/j.actamat.2021.117037>
- [18] Engel B, Ohneseit S, Mäde L, Beck T (2021) Influence of grain orientation distribution on the high temperature fatigue behaviour of notched specimen made of polycrystalline nickel-base superalloy. *Metals* 11:731. <https://doi.org/10.3390/met11050731>
- [19] Schmidt E, Boas W, Hughes FA (1950) Plasticity of crystals. *J R Aeronaut Soc.* 54:718–719. <https://doi.org/10.1017/S0368393100116219>
- [20] Ahluwalia R, Lookman T, Saxena A (2003) Elastic deformation of polycrystals. *Phys Rev Lett* 91:055501. <https://doi.org/10.1103/PhysRevLett.91.055501>
- [21] Chou PC, Pagano NJ (1992) Elasticity: Tensor, dyadic and engineering approaches. Dover Publications Inc, New York
- [22] Gonzalez M, Axisa F, Bulcke MV, Brosteaux D, Vandevelde B, Vanfleteren J (2008) Design of metal interconnects for stretchable electronic circuits. *Microelectron Reliab* 48:825–832. <https://doi.org/10.1016/j.microrel.2008.03.025>
- [23] Gräf M, Neumayer S, Hielscher R, Steidl G, Liesegang M, Beck T (2022) An image registration model in electron backscatter diffraction. *SIAM J. Imaging Sci.* 15(1):228–260. <https://doi.org/10.1137/21M1426353>
- [24] Aurenhammer F (1991) Voronoi diagrams—a survey of a fundamental geometric data structure. *ACM Comput Surv* 23:345–405. <https://doi.org/10.1145/116873.116880>
- [25] Rahmani K, Nategh S (2008) Influence of aluminide diffusion coating on the tensile properties of the Ni-base superalloy René 80. *Surf Coat Technol* 202:1385–1391. <https://doi.org/10.1016/j.surfcoat.2007.06.041>
- [26] Special Metals (2023) INCONEL® Alloy 617 data sheet
- [27] Liu Y-X, Ke Z-J, Li R-H, Song J-Q, Ruan J-J (2021) Study of grain growth in a Ni-based superalloy by experiments and cellular automaton model. *Materials* 14(22):69122. <https://doi.org/10.3390/ma14226922>
- [28] Becker F, Petra S, Schnörr C (2015) In: *Handbook of Mathematical Methods in Imaging*. Springer, New York, pp 1945–2004
- [29] Weickert J, Bruhn A, Brox T, Papenberg N (2006) In: *Mathematical Models for Registration and Applications to Medical Imaging*. Springer, New York, pp 103–136
- [30] Blaber J, Adair B, Antoniou A (2015) Ncorr: open-source 2D Digital image correlation matlab software. *Exp Mech.* <https://doi.org/10.1007/s11340-015-0009-1>
- [31] Chemnitzer Werkstoffmechanik GmbH (2017) VEDDAC—Digital Image Correlation Software
- [32] Correlated Solutions Inc. (2017) VIC 2D—Digital Image Correlation Software
- [33] Balle F, Beck T, Eifler D, Fitschen JH, Schuff S, Steidl G (2019) Strain analysis by a total generalized variation regularized optical flow model. *Inverse Probl Sci Eng* 27:540–564. <https://doi.org/10.1080/17415977.2018.1475479>
- [34] Hartman C, Weiss HA, Lechner P, Volk W, Neumayer S, Fitschen JH, Steidl G (2021) Measurement of strain, strain rate and crack evolution in shear cutting. *J Mater Process Technol* 288:116872. <https://doi.org/10.1016/j.jmatprotec.2020.116872>
- [35] Hewer A, Weickert J, Seibert H, Scheffer T, Diebels S (2013) In: Burghardt T, Damen D, Mayol-Cuevas W, Mirmehdi M

- (eds) Proceedings of the British Machine Vision Conference 2013. British Machine Vision Association, 129.1–129.11. <https://doi.org/10.5244/C.27.129>
- [36] Bachmann F, Hielscher R, Schaeben H (2011) Grain detection from 2d and 3d EBSD data—specification of the MTEX algorithm. *Ultramicroscopy* 111:1720–1733. <https://doi.org/10.1016/j.ultramic.2011.08.002>
- [37] Bayerlein U, Sockel HG (1992) Determination of single crystal elastic constants from de- and dr-Ni-based superalloys by a new regression method between 20°C and 1200°C. *Superalloys*:695–704
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