Experimental validation of microstructure evolution in crystalline materials

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Abstract. The influence of initial grain orientation and geometrical restrictions (grain boundaries, sample geometry) on the microstructural evolution was investigated at ambient temperature. Single- and polycrystalline austenitic steel samples, strained under uniaxial tension, show pronounced orientation changes which are strongly dependent on initial orientation. The numerical simulation results from a crystal plasticity FEM model match the experimental ones.

1. Introduction
Under load, the crystal anisotropy of polycrystalline metals and geometrical restrictions (grain boundaries or forming dies) cause strong inhomogeneities of stress and strain and the crystal orientation changes on the local scale. Especially texture evolution and grain fragmentation during the plastic deformation are of technical interest for the metal forming industry. A recent work [1] supported the plan to examine this structural evolution by identifying the physical processes in coarse grained crystals. With this motivation uniaxial tension tests on single (sc) and polycrystals (pc), actually a bi-crystal, were performed. The analysis was performed on a free sample surface by using EBSD-technique and stereophotogrammetric deformation analysis. Particular attention has been paid to clarify the influence of initial orientation on the structural evolution. For this study a really important advantage of coarse grained samples is the nearly columnar grain structure, which provides a bridge to validate numerical simulation results. For the simulation, a crystal plasticity finite element model was used [2, 3].

2. Sample preparation and experimental setup
The analysed material was a coarse pc austenitic steel, which consists of millimeter-sized grains. The chemical analysis is given in Table 1.

| Table 1. Chemical contents (wt\%) of the analysed austenitic steel Böhler A220. |
|-----------------|---|---|---|---|---|---|---|
| Fe              | Cr | Ni | Mo | Mn | Si | N  | C  |
| 63.2            | 17.5 | 14.5 | 2.7 | 1.7 | 0.3 | 0.07 | 0.03 |

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Tension test samples were machined out of sheets as shown in Figure 1(b). The coarse grained structure makes it possible to cut out samples with a cross section of 1x1mm$^2$ being either sc or pc over the whole inner gauge length of 5mm (Figure 1(a)). Subsequently, these samples were mechanically ground, polished and electrolytically polished (electrolyte A2) to remove the deformation layer from the cutting and mechanical grinding process. The samples were deformed in an open displacement controlled mode to engineering strains of about 30% using a tension test unit (Kammrath&Weiss) as shown in Figure 1(c). This equipment utilized two moveable crossheads, which held the center of the sample at a fixed position during the elongation. The experiments were conducted at a constant crosshead speed of 10µm/s and at ambient temperature.

The analysis procedure consists of a combination of the measurements of the in-plane strains and the local crystal orientation during and after deformation test. Measurements on the crystal orientation were carried out with an EBSD-SEM system (a TSL EBSD system interfaced to a LEO 1525). The initial crystallographic orientations and the grain boundary profile of the sc and the pc sample are plotted as Inverse Pole Figures (IPFs) related to the tension direction in Figure 2. The $\langle 11\overline{2}0 \rangle$ axis of the sc sample was parallel to the loading direction. For the pc sample the two largest grains show a soft $\langle 7\overline{3}17 \rangle$ and a hard $\langle 5\overline{5}7 \rangle$ orientation parallel to the loading direction. In-plane strains were calculated from local displacement fields by means of an in-situ stereophotogrammetric deformation analysis [4, 5]. To support the measurement of the local strain, a randomly distributed pattern was applied to the sample surface by a fine mist of paint. To demonstrate the potential of crystal plasticity FEM simulations, the incremental strain in tension direction is compared to the experimental one.

3. Results and discussion
3.1. Macroscopic stress strain response
In Figure 3 the engineering stress vs. displacement data of the sc and pc samples are plotted. The sc sample was strained without any interruption up to an $\epsilon$ of 34%. In the case of the pc sample, the experiment was conducted incrementally in two steps to correlate the crystal orientation changes with the local strain evolution.
Under normal circumstances a stage I zone is not typical for a pc sample however in our case we are analyzing a bicrystal. For both investigated samples the three hardening stages appear during the deformation. A plausible explanation for this response might be the initial crystallographic orientation and the grain boundary profile. As shown in Figure 2(b), the left coarse grain - having a soft single slip orientation with a Schmid factor of 0.48 - stretches over the whole inner gauge length without any grain boundary in between. In absence of constraining effects it follows that the pc sample deforms like a soft single crystal. The smaller stage I zone, the higher hardening moduli in the stage II and the more pronounced softening at stage III in comparison to the sc sample (in spite of the higher Schmid factor) is due to the constraint from the grain boundary.

**Figure 3.** Engineering stress strain curves for sc and pc sample.

**Figure 4.** Inverse pole figure in tension direction with a point to origin misorientation plot of a 34% strained single crystalline sample.

### 3.2. Local microstructure evolution

**Single crystalline sample**

Figure 4 shows the crystal orientation related to the tension direction and a point to origin misorientation plot of the sc sample after $\epsilon=34\%$. Between the sample flanks the dislocation can move freely to the sample surface and no orientation gradient is found. Conversely, pronounced orientation changes occur at the sample flanks due to geometrical restrictions which cause a rigid body rotation of the inner area.

**Polycrystalline sample**

As already mentioned, the strongest deformation in the pc sample is localized in the soft crystal (Figure 5). From comparison of incremental strain data to crystal orientation data, visualized as path plots in Figure 6, the following results can be presented: The first strain increment of 14% leads to a strong localization and a very heterogenous distribution of strain in the soft grain without any effect on the misorientation gradient. In spite of the small strains, which appear in the hard crystal, the fragmentation process is more developed than in the soft one. With increasing $\epsilon$ the crystal fragmentation occurs in both crystals. This phenomenon motivates to investigate patterns of different slip system activation on the sample surface which are illustrated as close-up scans in Figure 5. A fading procedure is used to combine IPFs and digital images. In case of the soft crystal strain heterogeneities and, for the hard crystal, different activation of slip systems cause the fragmentation process. For the soft crystal there is one major slip system with a maximum Schmid factor and, therefore, it is hard to activate a second slip system. In the hard crystal two slip systems have the same Schmid factor. That means, the resolved shear
stress on each slip system is equal. As a result small orientation fluctuations or geometrical restrictions, leading to a more complex strain tensor, can favour one slip system very easily. For the crystal fragmentation process the initial orientation has a strong influence [6]. As shown for the pc sample in Figure 6 the predicted local strains from the simulation model match the experimental ones.

![Inverse Pole Figures and Incremental In-plane Strain Maps](image)

**Figure 5.** Inverse Pole Figures and incremental in-plane strain maps in tension direction illustrate the local plastic behaviour of the pc sample. The results along the paths indicated by dotted lines are compared in Figure 6.

![Comparison of Experimental and Simulated Strain Paths](image)

**Figure 6.** A comparison of experimentally determined and simulated incremental strain paths (dashed lines are numerical results) with misorientation paths of the pc sample. The position of the paths is plotted in Figure 5.

**Conclusion**

Dislocation slip and shear constraints are the physical mechanisms which control the texture evolution. In double slip orientated grains, the fragmentation is more pronounced as in single slip orientated ones.

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