In-situ study of microstructural evolution and local strain distribution during tensile loading of near-micrometre grain size aluminium

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Abstract. A combined digital image correlation (DIC) and electron backscatter diffraction (EBSD) study of local strain distribution and microstructural evolution during tensile loading has been carried on samples of near-micrometre grain size Al prepared by spark plasma sintering. To achieve the required spatial resolution and allow combined DIC and EBSD measurements a method of surface patterning using colloidal silica was developed. The DIC results reveal a wide variety in heterogeneity of plastic strain, both in terms of the average strain experienced by each grain and regarding the extent and nature of strain localization within individual grains. No obvious correlation is found between the heterogeneity in plastic strain and either Schmid factor, grain size or the pattern of local grain rotations. The experiments also allow investigation of the pattern of crystal rotation during tension in near-micrometer grain size samples and reveal differences in the mean grain rotation and the in-grain orientation spread.

1. Introduction
In order to understand and control deformation inhomogeneity of materials, it is essential to understand comprehensively the relationship between the deformation inhomogeneity of materials at the grain scale and the link to micro-scale deformation mechanisms. The strain heterogeneity and microstructural inhomogeneity during deformation are interrelated and in single phase materials may depend on the grain orientation, and well as on the size and characteristics (size, orientation, shape) of neighboring grains [1]. The near-micrometer grain size regime is of particular interest with regard to deformation heterogeneity as it has been reported that samples with grain sizes in this regime may exhibit unusual mechanical properties, such as hardening by annealing and softening by deformation, and an obvious yield-drop phenomena [2,3]. These properties are a reflection of a transition in the deformation mechanisms as the average grain size is reduced to a few micrometers. It is of interest therefore to investigate the strain heterogeneity in samples with near-micrometer grain size, as such samples will typically contain a population of grains with sizes that straddle this transition regime. It has been shown that for such experiments samples of Al prepared by spark plasma sintering (SPS) are well suited, as in such samples the grains are in a fully recrystallized condition with an approximately random texture and a low dislocation density [4].

Digital image correlation (DIC) is a powerful technique employed for investigating the surface deformation and provides information regarding full-field displacements and strains [5]. Recent
developments in achieving fine-scale speckle patterns, by remodeling of a deposited layer of gold [6] have shown the potential for this method to achieve sub-micron resolution in deformed metals. Electron backscatter diffraction (EBSD) is now a standard laboratory technique for mapping crystal orientations on a sample surface, and as a scanning electron microscope microscope (SEM) based technique, allows the possibility for in-situ studies of a range of metallurgical phenomena, including phase transformations [7], recrystallization and grain growth [8], and plastic deformation [9,10]. Although the combination of both DIC and EBSD for in-situ studies of plastic deformation and strain heterogeneity is therefore of great interest, a challenge remains in obtaining fine-scale speckle patterns for DIC that do not interfere adversely with the EBSD signal.

2. Material and methods

Samples of Al with an average grain size of 4.4 μm were prepared by using the SPS technique. For the SPS samples, spherical Al spherical powder with an average particle size of 5μm was sintered to disks of diameter 20 mm and height 6 mm following the procedure described in [4]. Dog-bone shape tensile samples were cut from the as-sintered disks by electron-discharge machining. The width, gauge length and thickness of the tensile samples were 1.8, 9 and 0.6 mm, respectively.

Prior to tensile deformation, the specimens were manually ground to 4000-grit silicon carbide paper, and then electrochemically polished in a 10% perchloric acid and 90% alcohol solution to achieve a flat polished surface free of mechanical damage and residual deformation. To achieve a uniform distribution of suitable DIC markers a beaker of colloidal silica solution containing particles with a mean size of 50 nm was first given an ultrasonic treatment for 40 mins, after which a small amount was dropped onto the sample surface. Indents were added to the tensile sample to allow easy identification of the area of interest, and to allow re-alignment of the sample between the alternating EBSD and DIC measurements.

Tensile deformation was carried out using a home-built push-to-pull stage, equipped with a load cell to record the instantaneous force during tensile loading. The in-situ observations were conducted on a TESCAN MIRA 3 LMH equipped an CMOS-based EBSD detector from Oxford Instruments operating at 20KV. Secondary electron (SE) images were used for the DIC measurements, with the region of interest for DIC covering an area of 69.2 x 69.2μm² (microscope magnification of x3000) at a pixel size 4096 x 4096 pixels. To capture the same area in EBSD maps of size 80 x 80 μm² were collected using a step-size of 0.3μm. Images for DIC analysis were collected before tensile testing (0% strain) and at tensile strains of 0.45%, 1.6%, 3.3%, 5.8% and 8.9%. EBSD data were acquired at strains of 0%, 1.6%, 5.8% and 8.9%. For the DIC calculations the VIC 2D software package was used.

3. Results

A SEM micrograph of the area investigated in study is shown in figure 1(a), with figures 1(b,c) showing higher magnification images of selected areas to illustrate the DIC marker distribution over a range of length scale. A uniform distribution of fine-scale markers is achieved, with the marker diameter ranging from 20 – 200 nm. The coverage of the markers is such that they do not adversely affect EBSD data collected from the same area. Although some aggregation of the larger oxide particles is seen at grain boundaries, this does not adversely influence the EBSD data, as in general boundary regions are associated with lower quality indexing. For the DIC calculations a test-box (subset) size of 91 x 91pixel² (1.5 x 1.5μm²) was used, as illustrated in figure 1(c), using a step-size of 3 pixels, providing sufficient resolution to study strain heterogeneity during tensile deformation on a fine scale within each grain.  

Strain-stress curves obtained by different methods are shown in figure 2. The red markers show values obtained from the load-cell (stress) and movement of hardness indents (strain) during loading, while the blue markers show the tensile strain along the loading direction obtained from the DIC measurements (averaged over the entire mapped area). The black line reproduces the tensile stress-strain curve for a sample prepared with similar Al powder as measured using a standard universal testing frame at an initial strain rate of 1.6 x 10⁻⁵s⁻¹ [4]. A good agreement is seen between all three sets of data.

Maps showing the variation in the strain component along the tensile direction (εxx) at strains of 0.45%, 1.6% and 8.9% are shown in figure 3(a-c). For convenience black lines are overlaid on figures...
3(b,c) to show the positions of the grain boundaries (as determined from the EBSD measurements). Note that in these figures, and in the EBSD maps the tensile direction is along the horizontal direction. Figure 3(d) shows an electron channeling contrast (ECC) image of the result of the tracked area after the final deformation step (8.9%), in which slip bands can be clearly seen in a number of grains.

The DIC data reveal the clear presence of a variety of strain heterogeneity during tensile deformation. Except in a few cases, the highest local strain at each strain step is preferentially concentrated in larger size grains, while the strain associated with small grains is in general low. Not only is the strain uneven among different grains, but also a heterogeneous distribution of strain is seen inside most grains. In some grains significant strain accumulation is found at, or close to, grain boundaries and triple points, while in some areas bands of locally higher strain are observed to pass through grain boundaries and into grain interiors. In other grains, the slip concentration is seen within the grain but without transfer of strain across the grain boundaries. The pattern of strain inhomogeneity in the examined area of the sample becomes increasingly clear with increasing strain. Although at the lowest strain investigated (0.45%) the pattern of strain heterogeneity is rather weak, the areas with higher local higher strain are seen to match those after deformation to larger strains, supporting the validity of the DIC data at the lowest strain. In general the highest local strains at each strain step measured are approximately 2–3 times larger than the average strain.

EBSD maps illustrating the microstructure before and after tensile deformation to a strain of 8.9% are presented in figure 4. The inverse pole figure maps (figures 4a,d) reveal at least qualitatively an increase in orientation spread within each grain. The heterogeneity of grain subdivision, is revealed in more detail in figures 4(b,e), which plots for each grain the misorientation angle between each pixel and the average orientation of the grain. In some grains the tensile deformation results in a significant variation in orientation across each grain, while in other grains, including some of larger size, a much smaller orientation variation is seen. Figures 4(c,e) show the kernel average misorientation (KAM) at each map pixel, calculated using a 3 x 3 kernel with an upper cut-off angle of 4°. This map in effect displays a smoothed version of a boundary misorientation map, and picks up local orientation gradients within the angular resolution of the technique, and as such is commonly used to estimate the geometrically necessary dislocation density at each pixel. The maps reveal the formation inside some grains of low angle boundaries and also the development of orientation gradients close to grain boundaries and in some cases in grain interior regions.
Figure 3. DIC results showing the variation of tensile ($e_{xx}$) strain of after loading to (a) 0.45%, (b) 1.6% and (c) 8.9%. Grain boundary positions (obtained from EBSD mapping) are overlaid in black in (b,c); (d) electron channeling contrast image of the examined area after 8.9% tensile deformation.

Figure 4. EBSD data for the examined area before deformation (a-c) and after deformation to 8.9% strain (d-f); inverse pole figure coloring showing tensile axis direction; (b,e) deviation angle to grain average orientation (c,f) 3 x 3 kernel average misorientation angle maps.
4. Discussion

An advantage of the present experiment, where colloidal silica markers are used for DIC, is that information both on the local deformation and the microstructural evolution and crystal rotations can be obtained on from the same region on a sample surface, providing a rich data set for analysis of deformation heterogeneity. Here we focus on two aspects, namely the Schmid factor and size of each grain. The Schmid factor provides a measure of the ease of primary slip system activation, and it has been reported that grains with high Schmid factor (SF) in FCC materials with conventional grain sizes are prone to slip and strain localization [11].

Some example data is shown in figure 5. Grains 96, 52, and 67 have a similar Schmidt factor (SF = 0.41 – 0.42), but clearly the very small grain (96) sustains less local strain than the two larger grains. Grains 27, 28, 29 have a similar SF (0.45), with significantly less plastic strain taken by grain 28. This grain has a smaller size than the other two grain, but the strain pattern may also be affected by the cluster of smaller un-numbered grains lying between grains 27 and 28. Schmid factor and grain size do not appear to be the only determinant of local strain heterogeneity: grain 50 has a higher SF (0.47) than grains 27-29, but the average local strain for this grain is relatively low and uniform. Further analysis of the data, including both grain orientation, size and neighbors is underway, noting that in some cases transmission of localized strain between grains may also play role. Although not examined in detail in this work, it is worth highlighting that a comparison of the KAM map in figure 4f and the local strain map at the same strain (figure 3d) shows very few similarities.

![Figure 5](attachment:image.png)

**Figure 5.** Comparison of local tensile strain (from DIC results) with Schmid factor (SF) for some selected grains in a region near the center of the investigated area.

The in-situ nature of the experiment also allows the crystal rotation of each grain to be determined. An example of the data available is shown in figure 6, which plots both the crystal direction parallel to the tensile axis for the average orientation of grain 41 (marked in figure 5) after each deformation step, and also shows evolution of the spread of crystal directions parallel to the tensile axis within the grain. For this grain an overall crystal rotation towards the <100> corner of the unit triangle is observed,
consistent with X-ray synchrotron observations of grain rotation in the bulk of larger size grains with this initial orientation [12]. The results here also show that the orientation spread within for grain develops in a different manner, with the spread approximately perpendicular to the overall lattice rotation.

![Figure 6. Inverse pole figure showing crystal rotation during tensile deformation for grain 41 (see figure 5). Red, blue, black and green points correspond to the grain average orientation after 0%, 1.1%, 5.5% and 8.9% tensile strain, respectively; smaller points in yellow show the evolution of the spread in tensile axis within this grain at each strain level.](image)

5. Summary
By careful application colloidal silica (SiO$_2$) markers allow combined high resolution DIC and EBSD investigation, where the spatial resolution is sufficient to follow in-situ tensile deformation of samples with near-micrometer grain size. In the present study, where Al prepared by SPS has been investigated, no significant degradation is seen after tensile straining to 8.9% in either the surface markers or in the EBSD data, where the same area was mapped at tensile strains of 0%, 1.6%, 5.8% and 8.9%.

The DIC results show wide variety in heterogeneity of plastic strain, both in terms of the average strain experienced by each grain and regarding the extent and nature of strain localization within individual grains. No obvious correlation between the heterogeneity in plastic strain and either Schmid factor or grain size is found. Similarly, the observed pattern of local strain heterogeneity differs from the variation in local misorientation (as viewed in a KAM map), highlighting the fact that such maps are not in general a proxy for plastic strain. The present 2D results are limited in that the stress state differs to that in the bulk, and the true grain size is unknown. A parallel X-ray synchrotron investigation is underway where lattice rotations in bulk grains can be obtained, albeit at a lower spatial resolution and without accompanying DIC data for plastic strain heterogeneity.

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