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3D mapping of residual stresses in growing grains of partially recrystallized Gum Metal

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ABSTRACT

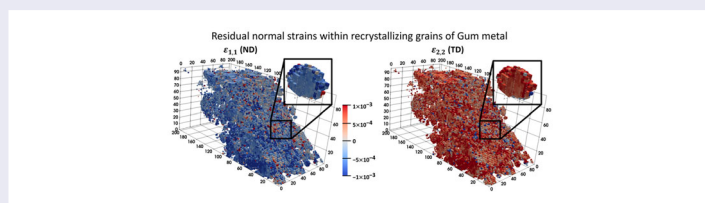
The crystallographic orientations and residual stresses within recrystallizing grains in partially recrystallized β titanium—Gum Metal—were examined non-destructively in 3D using synchrotron Differential Aperture X-ray Microscopy. Contrary to common assumptions, significant local stresses and stress variations are observed within the recrystallizing grains. The results reveal that the development of these local residual stresses depends on the plastic deformation mode and material's elastic constants, rather than grain properties such as size and orientation, or even the material's yield stress. This work provides insights valuable for the design of advanced materials with heterogeneous microstructures.

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Synchrotron radiation;
residual stresses; 3D
reconstruction;
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Recrystallization is an important industrial process widely employed to restore the ductility of deformed metals [1]. Recent studies have demonstrated the possibility of breaking the strength-ductility trade-off by introducing a partially recrystallized heterogeneous microstructure [2–5]. It has generally been assumed that nuclei free of stress and defects develop from the deformed matrix during recrystallization. However, recent findings have revealed the presence of both low angle boundaries and significant residual stresses within individual recrystallizing grains [6–8]. To understand the impacts of these local residual stresses on the enhanced strength-ductility synergy of partially recrystallized materials, a detailed quantification of these stresses is imperative.

With recent advancements in synchrotron techniques, it is now possible to characterize local stresses in bulk samples with the necessary spatial and strain resolution [9–12]. Previous studies on pure iron have revealed significant intragranular stress variations, ranging from 150–200 MPa, within individual recrystallizing grains in the partially recrystallized microstructure [6]. Moreover,

a connection between the average stress and the sample axes suggests an influence of the deformation mode [6]. To understand the formation mechanisms of local residual stresses, it is crucial to conduct such measurements using different materials, and correlate the results with factors such as plastic deformation mode, as well as the material's yield stress, elastic properties and grain size.

The present work examines intragranular deviatoric strains/stresses in partially recrystallized Gum Metal, which is a β Titanium alloy. There are two reasons for studying Gum Metal: (i) Gum Metal has, like pure iron, a body-centered cubic crystal structure, but it is much more compliant than iron [13,14], so the effects from anisotropic thermal expansion coefficients can be excluded and the effects of elastic modulus can be addressed; (ii) Gum Metal has a much higher strength compared to iron [15,16], so the impact of the yield stress on the local residual stresses can be examined. Additionally, this study expands the analysis to full 3D, eliminating any possible surface effects and enabling quantification of effects of grain size and grain boundaries. To ease the

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interpretation of the obtained results, the deformation mode is chosen to be the same as that used in the previous work on pure iron [6], and Differential Aperture X-ray Microscopy (DAXM), the same synchrotron technique as that used in the previous work [6], was chosen to avoid any possible influence from different technique uncertainties. DAXM offers a sub-micrometer spatial resolution. In addition, this non-destructive 3D technique is preferred for the present investigation over the relevant 2D techniques, such as high-resolution electron backscatter diffraction and its variants [17–19], as well as transmission electron microscopy [20,21], because it provides representative bulk stress results without presuming conditions such as zero stress normal to the sample surface.

The investigated Gum Metal sample was originally a wire with a diameter of 1 mm, which was cold rolled to a reduction of 75%. The sample width doubled after rolling. The sample was subsequently heat treated at 800°C for 4 min. This temperature was chosen to avoid the formation of new phases which appear at lower temperatures. Air cooling was used to avoid formation of metastable phases and quenching-induced thermal stresses. A rolling reduction of 75% was chosen as it was low enough to avoid too fast recrystallization at 800°C, and we thus could accurately achieve the desired degree of recrystallization ($\sim 25\%$), but high enough to ensure full recrystallization upon longer annealing time. The nucleation density at this strain level also resulted in an appropriate number of grains per unit volume for the DAXM scan.

The DAXM measurements were performed at 34-ID-E at the Advanced Photon Source, Argonne National Laboratory, USA [9]. A focused polychromatic X-ray beam was used to penetrate into the sample, which had its surface tilted by 45° with respect to the beam. The sample was aligned so that the rolling direction (RD) was normal to the exposed surface. Depth resolution was achieved by moving a thin platinum wire (differential aperture) across the sample surface, selectively obscuring diffracted X-rays from different sample depths. The differential aperture was moved along the transverse direction (TD) for depth resolution along the beam direction. A portion of the diffracted X-rays was collected on a 2D detector positioned such that the detector normal was perpendicular to the beam, and at a 45° angle to the sample surface normal. The sample was scanned around the center of the sample in both the normal direction (ND) and TD to generate a uniform measuring grid. From the patterns recorded on the detector at different sample and aperture positions, a 3D dataset was generated. Reconstruction and indexing of the depth-resolved diffraction patterns were performed using the LaueGo software package [22].

The reconstructed data contains the crystallographic orientation of each measured voxel in the scanned volume with an accuracy of approximately 0.01° [23]. The strain was subsequently fitted using an algorithm to minimize the mismatch between the measured and expected peak positions, which were calculated based on the indexed orientations, yielding a deviatoric strain tensor in each voxel. The normal components of the deviatoric strain tensor can be determined with an accuracy of approximately 10^{-4} for recrystallized materials [11,24], while the shear components can have errors up to 4 times larger [25].

The step size of the scan was chosen to be 1.5 μm in order to obtain a proper compromise between spatial resolution and size of the scanned volume within the available beam time. The examined volume was approximately $100 \times 100 \times 100 \mu\text{m}^3$. The exposure time used was 0.25 s, resulting in a total scan time of approximately 5 days.

The crystallographic orientation of each indexed voxel can be seen in Figure 1(a). Note that the indexing rate decreases with depth (the sample surface is outlined in red in Figure 1(a)) due to X-ray attenuation. The recrystallizing grains are well indexed while the deformed matrix is poorly resolved due to the high defect density. But this is not critical, as only the recrystallizing grains are of interest in the present study. The recrystallizing grains have a drastically lower internal misorientation ($< 0.1^\circ$) than the poorly indexed deformed regions. In order to reduce effects of noise, a minimum size criterion of 10 voxels (approximately equivalent to a 4 μm diameter grain) was applied, and the grain boundaries were defined by a misorientation above 0.1°. The recrystallization volume fraction of the sample was 24.4%. The scanned volume contained 1163 grains with an average size of 6 μm (see the grain size distribution in Fig. S1 in Supplementary Material).

A map showing the Kernel Average Misorientation (KAM) was also generated, see Figure 1(b). Here, in contrast to Figure 1(a), only the recrystallizing grains are shown as they are the focus of this work. The KAM is the average difference in orientation between each voxel and the 6 voxels adjacent to each of its faces. This is calculated with the additional condition that each of the surrounding voxels is part of the same grain according to the prior grain detection. The results show that the local misorientation in the recrystallizing grains is small, but not zero. There are some domains with slightly higher KAMs that are distributed rather evenly throughout the scanned volume. These domains seem to most commonly appear near the boundaries between recrystallizing grains and deformed matrix. It could be speculated if this is an artefact due to a low indexing confidence in these domains.

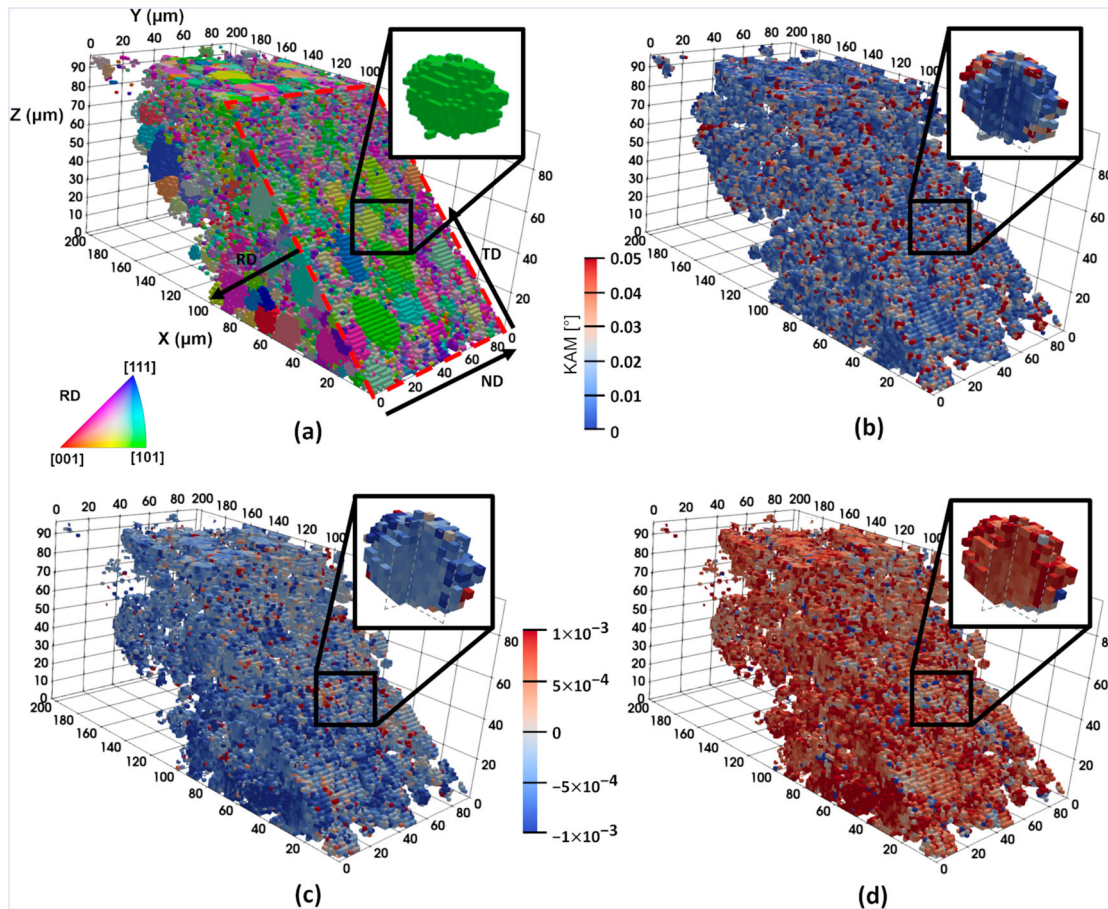


Figure 1. Orientation map of all indexed voxels colored based on the IPF key (a). The KAM of all recrystallizing grains in the examined volume (b). Deviatoric strain components $\varepsilon_{1,1}$ and $\varepsilon_{2,2}$, which represent the normal strains along ND and TD, respectively, of individual voxels within recrystallizing grains are shown in (c) and (d), respectively. For (c) and (d), only voxels that lead to a sufficient number of diffraction peaks to accurately fit the strain are shown, while all voxels that could be indexed within the recrystallizing grains are shown in the KAM map in (b). This causes some differences between (b) and (c)/(d). A specific grain, chosen arbitrarily from the marked region, is magnified with a quarter cut out to reveal the grain interior in (b), (c), and (d). The orientation of this grain is illustrated in the inset in (a), which also documents that only a few voxels of this grain are visible on the sample surface. The units of the axes in (b)–(d) are the same as those in (a).

However, even when only the voxels with high indexing confidence (8 or more diffraction peaks per indexed orientation) are included, fewer of these domains are found. A lower number of diffraction spots will reduce the confidence of the indexed orientations and strain fitting, causing a higher KAM and potentially higher strain values to be calculated. However, an alternative reason could be that these high defect domains are the result of the growth of the recrystallizing grain into the deformed matrix. The fact that these domains become fewer when the required number of spots is increased can then be explained by the fact that they are close to the edges of grains and thus generate weaker spots as the grain volume interacting with the microbeam is smaller than it is in the center of the recrystallizing grains. This latter explanation is the more likely one since a significant number of these regions remain when only very high confidence voxels are included.

3D maps of two example deviatoric elastic strain components for all recrystallizing grains are presented in Figure 1(c,d). From these figures, it can be observed that there is a strong macroscopic pattern in the strain, overlaid with some local variations. For each deviatoric elastic strain component, histograms (Figure 2) are used to show the levels in each voxel within the recrystallizing grains. Large variations are seen both within individual grains and across different grains. For example, the standard deviation of the grain average normal strain along TD, $\varepsilon_{2,2}$, is 1.5×10^{-3} and the average intragranular standard deviation of $\varepsilon_{2,2}$ is 1.3×10^{-3} . Besides the strain variations *within* recrystallizing grains, there is also a clearly noticeable pattern across the whole examined volume. Specifically, the average normal strain is tensile along TD and compressive along RD and ND. The average normal strains along ND ($\varepsilon_{1,1}$) and TD ($\varepsilon_{2,2}$) are -4×10^{-4} and 7×10^{-4} , respectively (see Figure 2). The few voxels

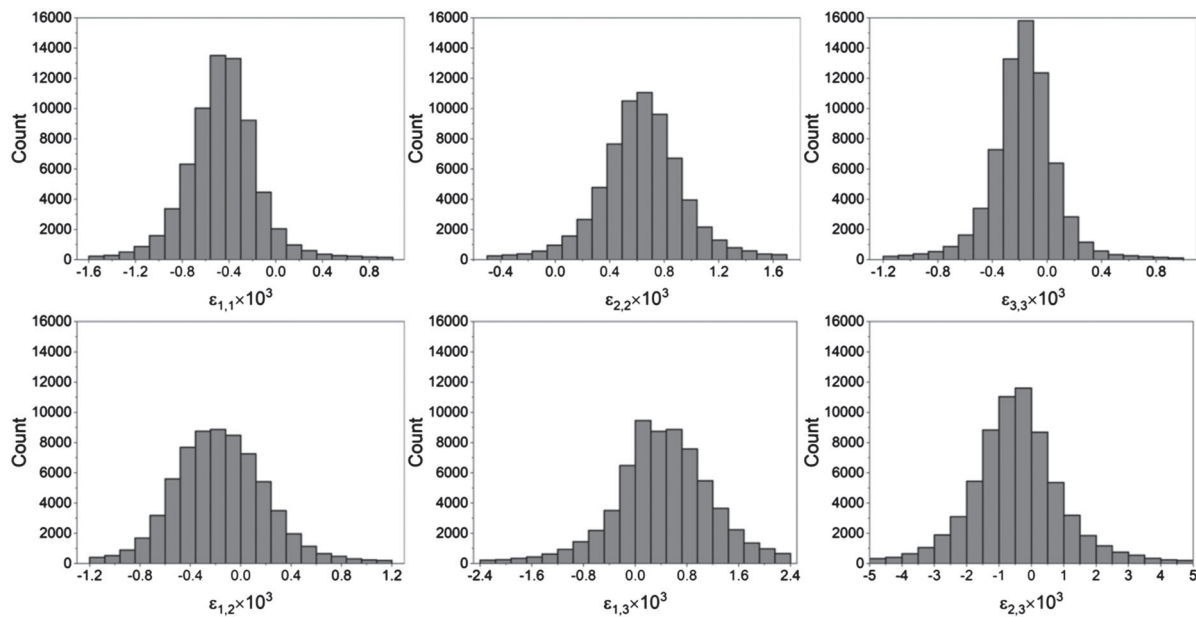


Figure 2. Histograms of each strain component of each voxel within the recrystallizing grains. The 'count' is the number of voxels in each bin. The bin size is scaled with the overall scatter range. 1, 2, and 3 represent the sample coordinate system of ND, TD, and RD, respectively.

different from this general pattern are distributed rather evenly throughout the volume and are seemingly not concentrated in specific grains.

By examining the details of the magnified grain in Figure 1(b–d), voxels displaying high levels of strain may appear to coincide with those having the highest KAM levels. However, there is no strong correlation between KAM and strain values when all voxels are considered (see Fig. S2 in the supplementary material).

It is also worth noting that the normal components of the strain tensor have significantly narrower distributions than the shear components, with $\varepsilon_{2,3}$ having a particularly wide distribution. This is mainly because the shear strain components obtained from micro-diffraction data are noisier, which is related to the geometry of the experimental setup [25]. For this reason, the following analysis will be focused on the normal components.

In order to investigate the causes leading to the variations of the strain and the KAM, possible effects of grain size and crystallographic orientation were analyzed. For this analysis, only the fully internal grains, i.e. grains not touching the sample surface or the surfaces of the gauge volume, were included, so the true 3D grain size can be determined precisely. There were in total 876 such grains. The analysis showed no strong correlation between grain size and KAM/strain, nor between grain orientation and strain (see Figure 3). This suggests that the variations in strain and KAM are not directly related to the characteristics of the individual recrystallizing grains.

The dislocation density within the recrystallizing grains is low and will not lead to significant intragranular residual stresses on their own [26]. The local residual stresses are therefore more likely caused by dislocation cell walls in the immediately adjacent deformed matrix. These dislocation cells develop during cold rolling and remain after annealing. The local residual stress state within individual deformed grains typically varies, depending on the orientation of the grains [27,28]. Since the recrystallizing grains are abutting several different deformed grains, the intragranular stress variations within individual recrystallizing grains may be quite different, even if they have similar crystallographic orientations. This explains why no correlation between elastic strain and crystallographic orientation is observed for the present sample (see Figure 3(c,d)).

It is also very interesting to note that the elastic strains seem much higher in Gum Metal than in pure iron [7]. To examine this difference further, the deviatoric stress tensors were calculated using the general Hooke's law and a stiffness matrix with $C_{11} = 125$ GPa, $C_{12} = 90$ GPa, and $C_{44} = 31$ GPa as measured for recrystallized samples by [29] (see Figure 4(a–c)). Interestingly, the average stress levels are comparable to those found in iron, despite the vastly higher yield stress of Gum Metal (~ 400 – 500 MPa in cold rolled iron vs. ~ 1 GPa in cold rolled Gum Metal). The lower ratio of residual stress to yield stress in Gum Metal may be related to a different recovery behavior of the remaining deformed matrix or to the difference in elastic moduli. Considering the high alloying content of

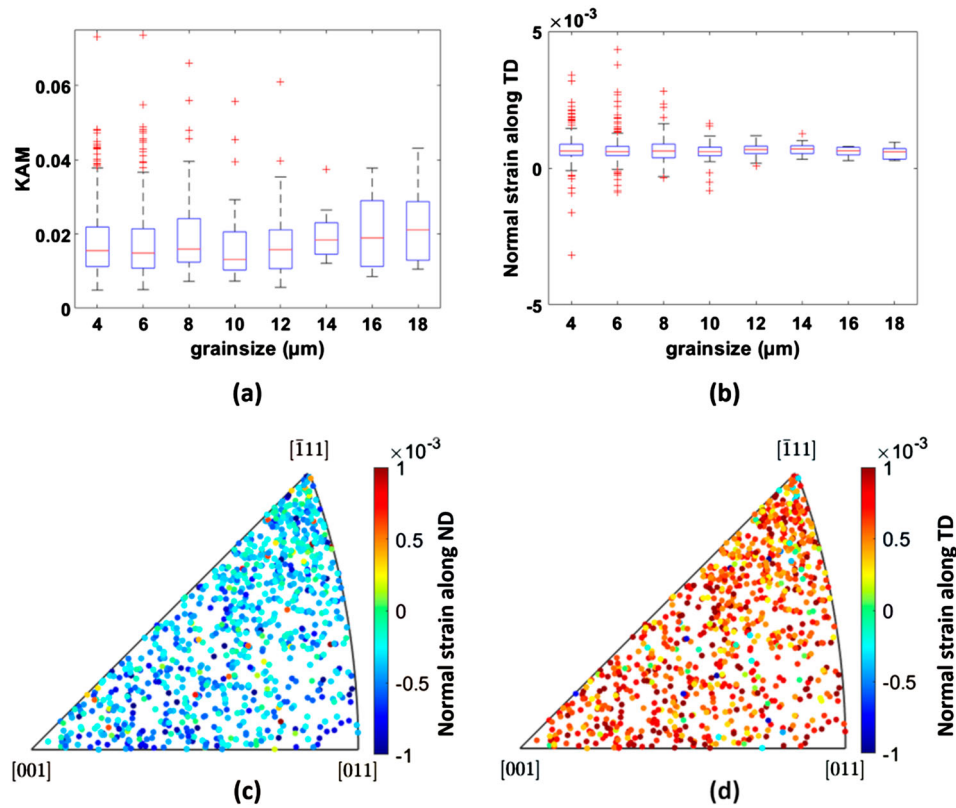


Figure 3. Grain-averaged KAM (a) and normal strain along TD (b) plotted with respect to grain size of the interior recrystallizing grains presented by box plots. The red horizontal lines represent the median values, the blue boxes the two middle quartiles and the whiskers are bounded by 1.5 times the interquartile range (corresponding to 99.3% of data points assuming a normal distribution). The red crosses represent outliers. The grain averaged normal strain along ND (c) and TD (d) of all internal recrystallizing grains are plotted in an inverse pole figure corresponding to the grain averaged crystallographic orientation with respect to ND.

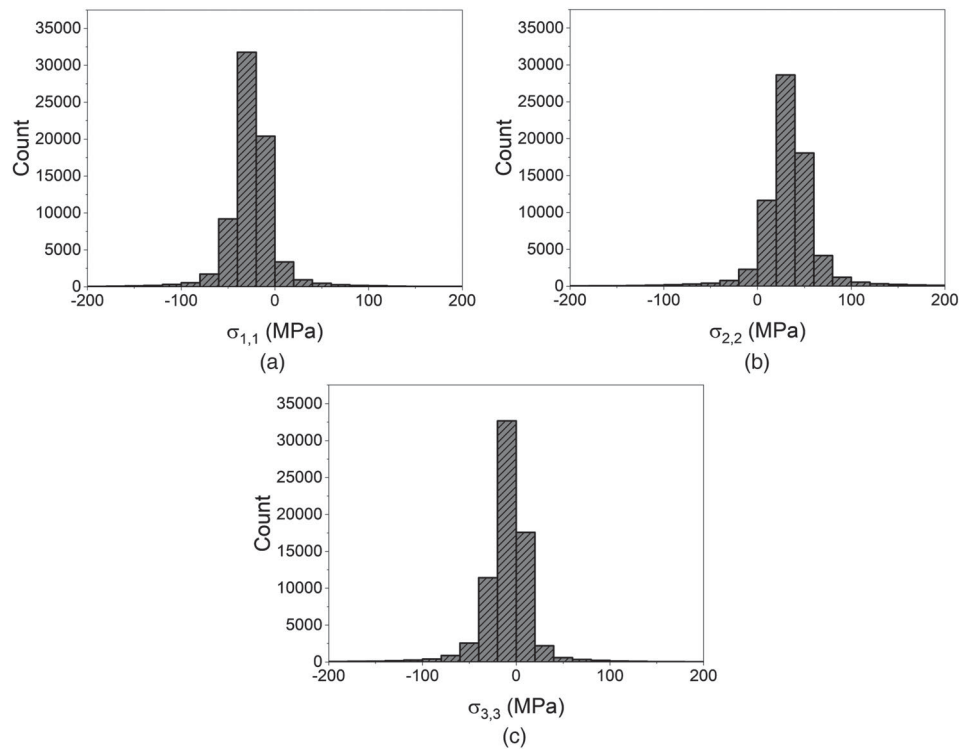


Figure 4. Residual normal stress levels in the recrystallizing grains of Gum Metal are shown in (a-c).

Gum Metal, a comparatively lower recovery rate than that in pure iron would be expected.

On average, the residual stresses are tensile along TD, compressive along ND, and slightly compressive along RD, which is similar to the pattern observed in partially recrystallized cold rolled iron [7]. This suggests that the plastic deformation mode is important for the development of the observed strain/stress tensors. To further understand the connection between the deformation mode and the stress/strain of the recrystallizing grains, the macroscopic strains present in the deformed matrix also need to be considered. It is very likely that this strain/stress state is directly inherited from the as-rolled condition: The sample was much too narrow for the rolling operation to be considered as being a plane strain case. Instead, it is nearer to asymmetric compression. This would be congruent with previous work, where the macroscopic residual stresses were measured in a drawn sample to be positive along the drawing direction and negative perpendicular to the drawing axis [30]. Reversing this to a compression would mean that the residual stress in the deformed matrix is compressive along the compressed direction (ND) and tensile in the directions perpendicular to the compressed direction (RD and TD). The asymmetry would then have to account for the compressive stress along RD, which is relatively small in magnitude when compared to the one seen along ND. At the microscopic level, residual stresses in the deformed matrix are related directly to how the dislocations are stored within dislocation boundaries, which leads to the development of medium- to long-range residual stresses associated with the dislocation cell and cell wall structure [30,31]. BCC metals develop the same type of deformation texture upon medium-to-high strain rolling [2], which may explain the observed similarities in macroscopic stress pattern for the two samples. Characterization of the residual stresses in the deformed state is required to further clarify the formation mechanisms. Nevertheless, a slight difference between the relatively small stress component along RD in Gum Metal and pure iron suggests that the initial sample dimension (diameter of 1 mm in Gum Metal vs. 25 mm in pure iron) may also be an important factor to be evaluated in the future.

In summary, the local stress patterns in $\sim 25\%$ partially recrystallized Gum Metal have been quantified using synchrotron X-ray micro-diffraction. The results show significant local residual strain variations within and between individual recrystallizing grains for all strain components. The average normal strains along ND, TD and RD are $-4 \pm 6.5 \times 10^{-4}$, $7 \pm 9.9 \times 10^{-4}$, and $-2 \pm 8.9 \times 10^{-4}$, respectively. The strain magnitudes exhibit limited reliance on the grain size and

orientation, but stronger correlation with the surrounding deformed matrix. The corresponding local residual stresses are much lower than the macroscopic yield stress of both fully recrystallized and deformed Gum metal. A comparison with the results found in pure iron suggests that the local residual stresses are unlikely to be determined by the material yield strength. This work underlines the importance of including local residual stresses within recrystallizing grains in the understanding of recrystallization and these unexpected residual stresses are very likely to also affect the mechanical properties.

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Impact statement

Contrary to common knowledge, significant local residual stresses exist within recrystallizing grains of gum metal. Such stresses should be considered in design of advanced materials with heterogeneous microstructures.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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