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Quantification of grain rotation in bulk nanostructured copper via in situ heating white beam synchrotron X-ray diffraction

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Abstract

Grain rotation during microstructural relaxation under heating has been studied extensively through transmission electron microscopy and simulations. However, there is a shortage in examining grain rotations at larger length and volume scales in bulk materials. It is critical to understand the thermal stability of bulk nanostructured metals, since those enhanced mechanical properties have been well recognized. This study aimed to employ a white beam microdiffraction X-ray technique under in situ heating from 300 K to 1073 K at 12 K/min on a nanostructured copper processed by high-pressure torsion, yielding an initial grain size of ~260 nm before the heating. Evaluation across a wide range of temperatures reveals transition temperatures associated with microstructural relaxation processes. By tracking separate Laue diffraction peaks from individual grains, changes in their orientations can be estimated and quantified. This approach becomes particularly effective when the number of grains within the probed volume is reasonably small. At temperatures above 940 K, about 7.5% of Cu grains are rotating at speeds of $\sim 2 \times 10^{-3}$ °/s. The radial direction of the disk specimen is found to be the preferred direction of grain rotation. Further analysis found no correlation between peak intensity and peak size, related to grain sizes and deviatoric strain, respectively, with rotation conditions or speed. These findings demonstrate that the diffraction technique utilizing white beam X-rays is a complementary and novel characterization tool for understanding microstructural evolution, especially in grain rotations, of bulk crystalline materials.

Keywords: grain orientation; high-pressure torsion; in-situ diffraction; nanostructure; synchrotron X-ray; white beam Laue diffraction.

1. Introduction

Understanding thermal relaxation and microstructural stability in metals is an essential research subject. General relaxation of metals has some well-understood stages, such as recovery, recrystallization, and grain growth. But, particularly, severe strain-induced nanostructure recovers more easily than the coarse-grains with relatively lower strain, and it is often challenging to capture the true nature of the microstructure evolution, which occurs across different length-scales from nano- to micro-meters, without using in situ experimental techniques. Recent studies using in situ heating neutron diffraction experiments successfully captured with time the non-linear transitions of microstructure changes, including the lattice defects in severely nanostructured 316L stainless steel and CoCrFeNi high-entropy alloys [1,2], where the diffraction techniques are beneficial by capturing relatively large, probed volumes of microstructure. General overviews of in situ neutron and synchrotron diffraction, and confocal laser scanning microscopy, especially on nanostructured materials, have been reviewed elsewhere [3,4].

However, grain rotations have only ever been considered in nano-sized grains during heating and deformation, often mainly due to the small, probed regions with volumes limited by the characterization technique. For example, the rotations of nano-scale columnar grains were captured ex-situ by a transmission electron microscope on a gold thin film after different hours of annealing and providing the first modeling of grain rotations through self-diffusion [5]. This model of grain rotation, accommodated by the cross-over from grain boundary to lattice diffusion, was later limited to the special case of a hexagonal grain shape; in other words, the model was generalized to a general grain shape by adopting the diffusion-accommodated grain boundary sliding processes [6]. A modeling study analyzing grain coarsening, grain boundary evolution, and grain rotation using a three-parameter phase field demonstrated that grain boundary migration and grain rotation could occur simultaneously [7]. These research techniques have primarily focused on nanoscale grains, often considering only a single degree of rotational freedom, using thin film experiments or performing computational modeling in two dimensions. Most recently, in situ four-dimensional phase-space scanning transmission electron microscopy (4D-STEM) was used to statistically describe a strong correlation between grain rotations and grain growth of nano-sized grains in

platinum thin films [8]. However, there is still a lack of in situ heating studies of grain rotation in bulk volume, where grains have three-dimensional neighboring grains.

An alternate approach to studying grain rotation requires not only the behavior of nano-grains but also their evolution as they grow thermally, to comprehensively understand the thermal microstructural evolution of nanostructured metals. Besides the above noted in situ heating neutron diffraction experiments, a recent report of in situ heating high-energy synchrotron X-ray diffraction on nanocrystalline Cu provides a quantitative evolution of microstructure, including microstrain, dislocation density, grain size, and lattice constant, leading to thermal expansion coefficient, from nanostructure in time and temperature scales [9].

By contrast, white beam (polychromatic) X-ray diffraction provides a continuous spectrum of X-ray energies, enabling visualization of dynamic crystal structure changes in probed volumes within a bulk material. Considering the complexity of the Laue diffraction patterns, performing quantitative analysis is difficult and has mostly been used on single crystals [10]. However, the characteristics of the white beam offer unique advantages when properly utilized. White beam X-rays provide a spectrum of wavelengths that can maintain diffraction even when the crystal lattice rotates away from a specific diffraction condition. Focused white beam Laue diffraction is routinely used for mapping strain gradients in microstructure [11], but since these methods involve scanning the location, it is inherently slow and thus not suitable for time-resolved studies. An early experiment utilized white-beam X-rays to characterize the grain size effect on grain rotation mechanisms in nickel nanoparticles under high pressures [12]. Furthermore, a previous study conducted in situ heating white-beam Laue diffraction experiments on a fine-grained AZ91 Magnesium alloy analyzes the erratic and spontaneous grain reorientation during abnormal grain growth [13].

X-ray diffraction is capable of probing bulk samples and resolving grain sizes well into the nanometer to micron ranges. When focusing for applying a nanometer beam size, it is suited for studying grain rotations in bulk polycrystalline ultrafine-grained materials. Accordingly, the objective of this study is to quantitatively measure grain rotation in large, relaxed grains of copper from nanostructure during in situ heating experiments and assess these behaviors against existing models to verify the mechanisms associated with larger grains.

2. Experimental Material and Procedures

Commercial purity copper disks were sliced from an extruded copper rod (>99.5% purity) with a diameter of 10.0 mm and thicknesses of ~0.85 mm. A total of two disks were then produced using high-pressure torsion (HPT) processing [14] under a compressive pressure of 6 GPa for 15 turns. One disk was analyzed by transmission electron microscopy (TEM) to measure the initial conditions after HPT, while the other was used for an in situ heating polychromatic X-ray diffraction experiment. The TEM sample was prepared at ~1 mm from the disk edge using a focused ion beam, Helios 650, with initial milling at 3 keV followed by thinning and polishing at 1 keV. A TEM image was taken and collected with a FEI Titan using a tilted incident beam dark-field technique, operating at 200 kV.

In situ heating X-ray diffraction experiments were performed at the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory, utilizing the microdiffraction beamline 12.3.2, under proposal ID ALS-11964. The beamline provides a micron focused beam size to $1 \times 1 \mu\text{m}^2$. The beam shape, however, is a Lorentzian in both directions elongated tails which covers usually more grains than expecting from a $1 \mu\text{m}^2$ spot. The according small footprint then allows to obtain single-grain reflections from a 100 nm scale or even smaller, once grains are recovered. A Pilatus 1M detector was used with a resolution of 981×1043 pixels and a detection area of $169 \times 179 \text{ mm}^2$. Figure 1(a) shows the schematic sample setup for the in situ heating diffraction experiment where the disk sample was placed on the furnace with a 35° counterclockwise (CCW) tilt from the incident beam and a detector placed $\sim 120 \text{ mm}$ away from the sample with a tilt angle of 72° CCW with respect to the incident beam.

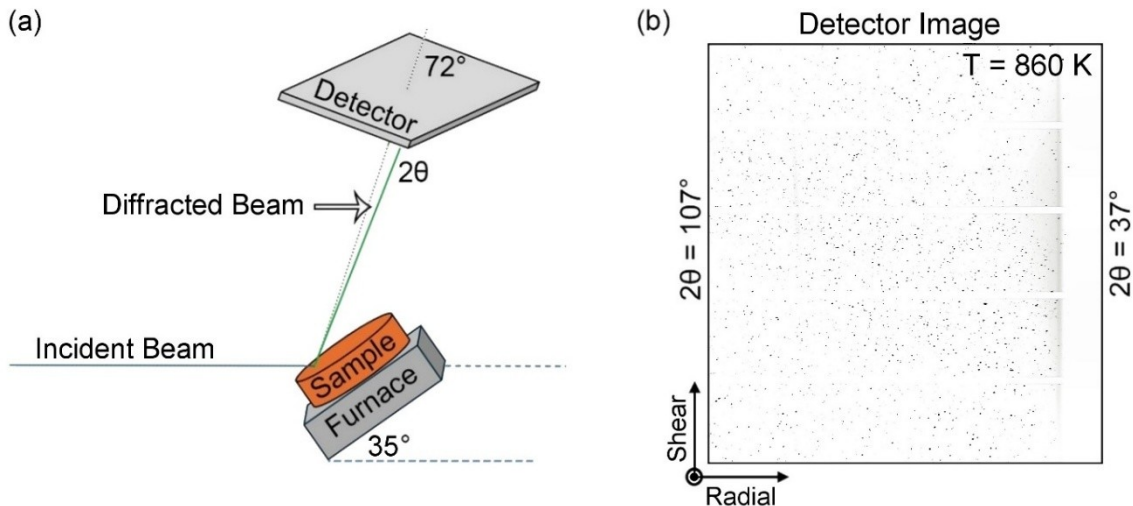


Figure 1 (a) shows a schematic drawing of an in situ heating synchrotron diffraction setup at ALS, and (b) is an example of a white-beam Laue diffraction pattern of a polycrystalline copper at 860 K with a scattering vector about normal to the sample surface.

An Anton Paar DHS900 furnace with a maximum temperature of 1073 K was used to heat the HPT-processed copper sample. The sample was in a nitrogen environment to prevent oxidation during heating. A heating rate of 12 K/min (0.2 K/s) was used from 300 K through 1073 K with a 5 s detector exposure time. The polychromatic beam distribution, as shown in *Supplementary Figure 1*, had an energy distribution between 11 keV and 20 keV. The incident beam was located approximately 1 mm away from the disk edge. The diffraction pattern was then analyzed using ImageJ (Fiji) stack particle and particle tracking macro to count and track the peaks with increasing temperature. Then, custom MATLAB and Python3 scripts were used to perform further analysis. Figure 1(b) shows an example of the white-beam X-ray diffraction pattern taken at an instantaneous temperature of 860 K, where each black dot represents a Laue diffraction peak. The placement of the detector shows diffraction angle (2θ) ranges from 37° to 107° . The horizontal detector direction represents dependence on the radial direction to the HPT disk, and the vertical detector direction aligns with the shear direction.

3. Results

The dark-field TEM micrograph of the HPT-processed Cu sample with the shear direction oriented to the right is shown in Fig. 2. The majority of the grains appear equiaxed, with some elongated grains aligned along the shear direction. The measured average grain size is 256 ± 60 nm, which is consistent with previous studies on HPT-processed pure copper, reporting average grain sizes in the range of 150–400 nm [15-20] dependent on the total applied shear strain, the purity of the copper, and the time elapsed after HPT processing due to the inherent recovery behavior of HPT-processed nanocrystalline Cu [21] containing an excess amount of lattice defects [9]. Various defect structures, such as dislocation structures and low-angle grain boundaries, can be recognized within grains, measuring one to two orders of magnitude less in size, and revealing the highly distorted, nanostructured material.

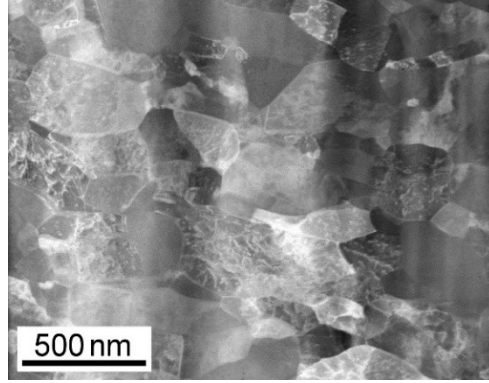


Figure 2 Dark field TEM image of the initial microstructure of HPT copper. Shear direction is left to right.

Figure 3 shows a summary of the quantified Laue diffraction data against temperature, showing the instantaneous total sum of the peak intensity, I_T , of all identified peaks in blue, the instantaneous total area of all peaks, A_T , in red, and the instantaneous total diffraction peak count, N_T , in green. The gray dotted lines indicate the transition temperatures in the trends of the three quantified diffraction characteristics, thus implying that the microstructural evolution can be divided into five distinct temperature regions.

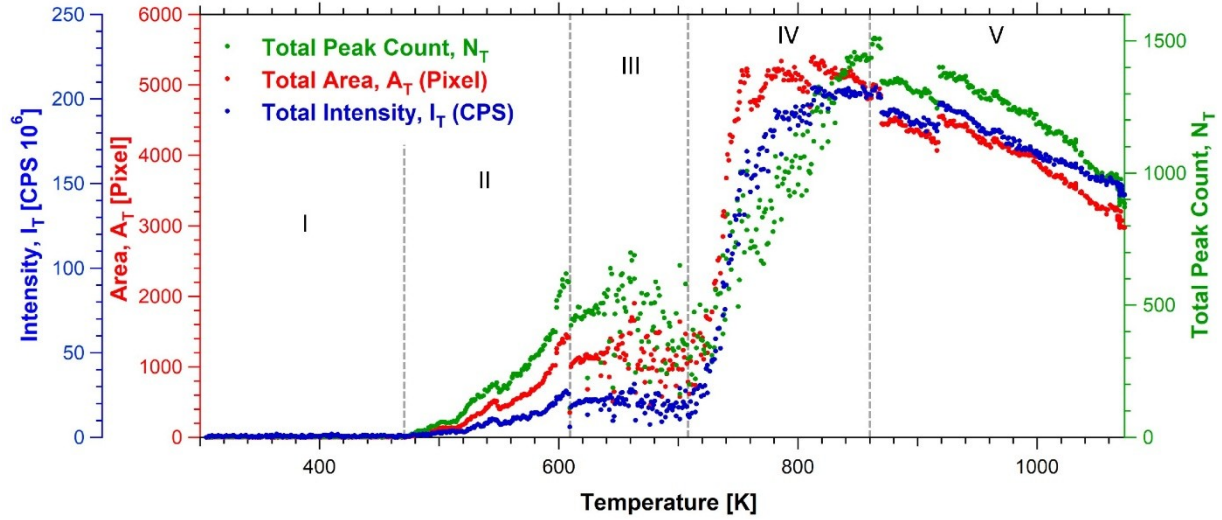


Figure 3 Total peak intensity (I_T), total peak area (A_T), and total peak count (N_T) against temperature plot divided into I-V temperature regions.

Region I, below 470 K, shows no identifiable peaks and therefore no quantified data due to the limitations of the technique. Region II, between 470 K and 610 K, shows a gradual increase in all three diffraction peak characteristics, where small amounts appear at just above 470 K and ultimately increase to the total diffraction peak count of $N_T \approx 500$ that are distinct enough to be identified over 600 K. The total sum of the identified peak intensity I_T and peak area A_T reached around 3×10^7 counts per second (CPS) and 1400 pixels coverage, respectively, toward 610 K. Region III, with a temperature between 610 K and 720 K, shows a sporadic spread between 300 and 700 of total peak counts. This causes both I_T and A_T to vary between 1×10^7 to 3×10^7 CPS and 180 to 1,900 pixels, respectively. At temperatures above 720 K, in region IV, there is a sharp increase in N_T toward 1,500 at 860 K. From 720 K toward slightly lower upper temperature, i.e. 817 K, both I_T and A_T show a sharp increase in value, reaching the highest values of $I_T = 2.1 \times 10^8$ CPS and $A_T = 5,200$ pixels. At around 860 K, the diffraction characteristics enter region V, where they gradually decrease with further temperature increases. It should be noted that a section in region V, where all the peak characteristics provide a sharp drop between 850 K and 900 K, is expected to be the technical experimental condition (i.e., incident beam drop) that is not related to microstructural evolution.

The decrease in all three peak characteristics in region V can be attributed to three possible factors. First, grain sizes are growing to the point where the microdiffraction beam is illuminating a smaller total number of grains than at the start of region V, therefore decreasing the number of Laue diffraction peaks. Second, as grains grow and are simultaneously annealed, thus peak numbers and areas both reduce, extinction of radiation sets in, reducing the integrated reflected intensity, although scattering from the same total illuminated volume [22]. Third, it may be caused by the “moving” of the Laue diffraction peaks. This movement of diffraction peaks appears to be a change in grain orientation with increasing temperature and time. This grain reorientation over time can be further defined as “grain rotation”. Although grains are likely rotating at any temperature in the examined temperature range, due to higher peak intensity and reduced number of total peak counts resulting from the coarsened grains, grain rotations are evaluated at temperatures above 940 K.

In this study, grain rotation was identified from a limited number of grains by their movements of diffraction peak positions among numerous stationary diffraction peaks. The total number of identified moving peaks, N_M , on the detector was tracked and counted in the detector at 5 s time intervals, for which the vertical and horizontal pixel locations of the detector capturing all moving peaks between 940 and 1073 K are plotted in Fig. 4(a). It can be seen better in the magnified section that each spot of line, depicted in arbitrary colors, represents individual peak trajectories with respect to stationary diffraction peaks. Due to a large number of stationary diffraction peaks, only the moving peaks were included in Fig. 4(a). Figure 4(b) shows the displacement vectors of the moving peaks on the detector, illustrating their evolution with increasing temperature. It should be noted that the beginning of peak movements indicates the onset of grain rotation, and the setup is typically unable to track the complete rotation of a grain at any given temperature within the examined range. The detection criteria under the white beam Laue diffraction are limited by the still relatively narrow energy spectrum of the incoming beam to fully track the diffraction peaks to their destination. Nevertheless, most peak displacements resulting in grain rotation were large enough to be represented by displacement vectors. Although not further discussed, it is remarkable that the traces in Fig. 4(a) are not straight lines, indicating competing driving forces from the momentary grain surroundings, leading to zigzag pathways [13]. The quantification of grain rotation direction and rotation speed is possible, allowing further identification of the preferred rotation direction

during recrystallization and coarsening under heating. This will be further analyzed in the discussion.

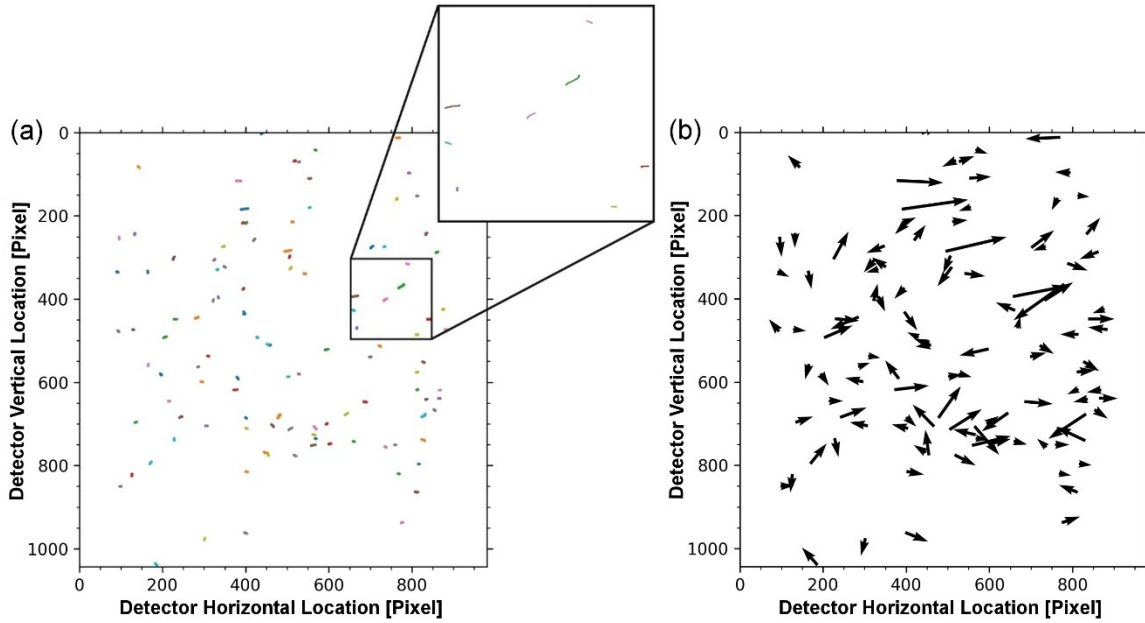


Figure 4 (a) plot of the detector's vertical and horizontal pixels showing the movement of the moving diffraction peaks, and (b) the displacement vector of the diffraction peaks until moving out of the diffraction condition at temperatures between 940-1073 K.

Considering the nature of white-beam X-rays, multiple diffraction peaks can originate from a single grain. Nevertheless, the assumption that the number of grains is proportional to the total diffraction peak counts N_T in Fig. 3 provides a qualitative estimate of the number of illuminated grains. The total number of moving peaks, N_M , can be compared to N_T to provide a ratio at any arbitrarily selected temperature or heating time used in the experiment. Figure 5 displays a plot showing the changes in the ratio of N_M/N_T marked in blue with an increasing temperature above 940 K, together with N_M in red and N_T in green. At ~940 K, the total number of identified peaks is $N_T = 1,328$ with a moving peak count of $N_M = 100$, thus yielding a ratio, N_M/N_T , of ~7.5%, meaning that 7.5% of the grains are changing orientation through rotation, and 92.5% remain stationary at that temperature. As shown in Fig. 3, the total number of instantaneous diffraction peaks, N_T , decreases with increasing temperatures above 940 K, but the ratio of N_M/N_T increases to 8.2% through 985 K due to the increasing number of moving peaks to $N_M > 110$. Thereafter, the ratio of N_M/N_T dropped slowly to 7% until 1020-1030 K and further dropped to 2.5% by 1070 K. Thus, the Cu grains,

initially starting from a nanostructure, have active grain rotation up to 985 K, then show signs of slowing down at higher temperatures. This ratio appears to correlate well with literature that performed a grain rotation experiment by annealing nano-grained thin film platinum with 4D in-situ TEM, where the reported probability of rotation is between 2–20% at 873 K [8].

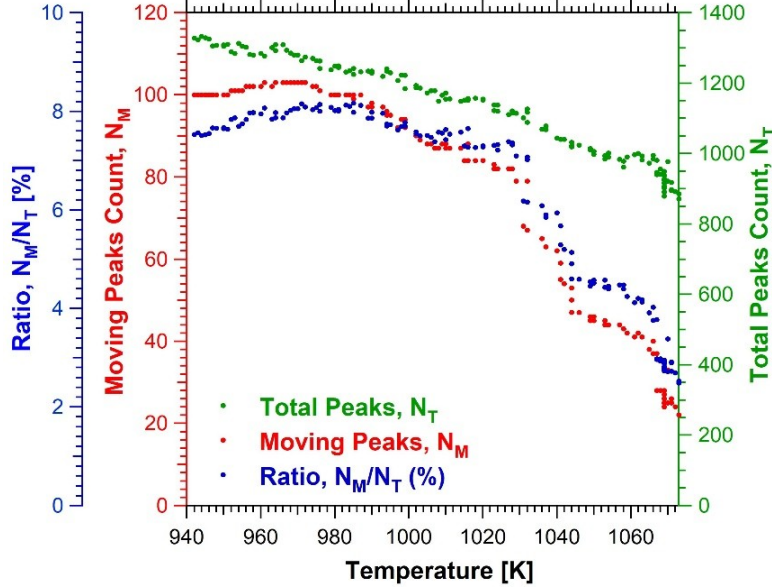


Figure 5 Plot showing trends of the instantaneous total number of grains, N_T , the instantaneous moving grains, N_M , and the ratio of moving grains N_M/N_T at 940 K to 1070 K.

The speed of grain rotation can be estimated from the magnitude of each peak displacement vector shown in Fig. 4(b) against time interval information. Figure 6(a) displays a plot with the estimated average grain rotation speed at temperatures of 940-1070 K. Here, “time” on the upper x-axis denotes $t = 0$ s at 940 K. As shown in Fig. 6(a), the average grain rotation speed varies between 2×10^{-3} °/s to 8×10^{-3} °/s. Specifically, between 940 and 950 K, there is a drastic increase in rotation speed up to 4.5×10^{-3} °/s, which is followed by a significant decrease to 960 K, then an oscillating again and eventually reaching to the highest speed of over 7.6×10^{-3} °/s around 1020 K. A significant decrease in the rotation speed to 2.5×10^{-3} °/s through 1065 K, finishing with an increase in grain rotational speed through 1070 K.

The histograms in Fig. 6(b) show the distributions of grain rotation speed for the moving peaks, N_M , at selected temperatures of 940 K, 985 K, 1020 K, 1055 K, and 1065 K indicated by the red arrows in Fig. 6(a). As seen from the histograms, some slower rotating grains at $\sim 2 \times 10^{-3}$ °/s persist at all five temperatures. As temperature increases, more peaks gradually move to higher

values, thus increasing average rotation speed until 1020 K. Over 1020 K, Fig. 5 showed a significant decrease in the number of rotating grains, as the already highly favorably oriented grains complete their orientation and fall out of the diffraction conditions, leading to a reduction in the average rotation speed. Current observed rotational speeds are comparable to the reported values in the literature. One study reports a speed of 8×10^{-4} °/s on thin-film gold, which is an order of magnitude lower than the current results; however, the annealing temperature is much lower at 523 K [5]. Another study reported a speed of 3×10^{-2} °/s while annealing platinum thin film at 873 K, showing the speed to be one magnitude higher [8]. Although not the same materials, all have an FCC structure, and the reported average rotational speed in this study falls within the range of the two reported values. By contrast, the comparable grain rotation speeds observed in the present HPT-processed copper, even at temperatures above 1000 K, may be influenced by the extremely high density of lattice defects, including grain boundaries, dislocations, and vacancies, produced during HPT. This results in a substantial total stored energy of 1,340 J/kg, which may drive microstructural restoration at such elevated temperatures [9].

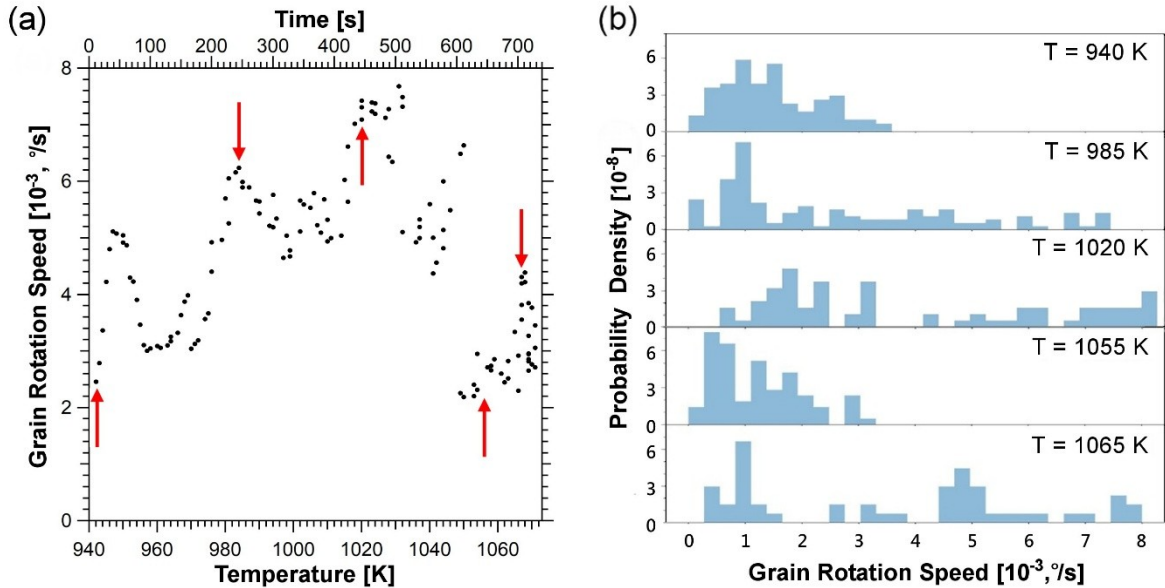


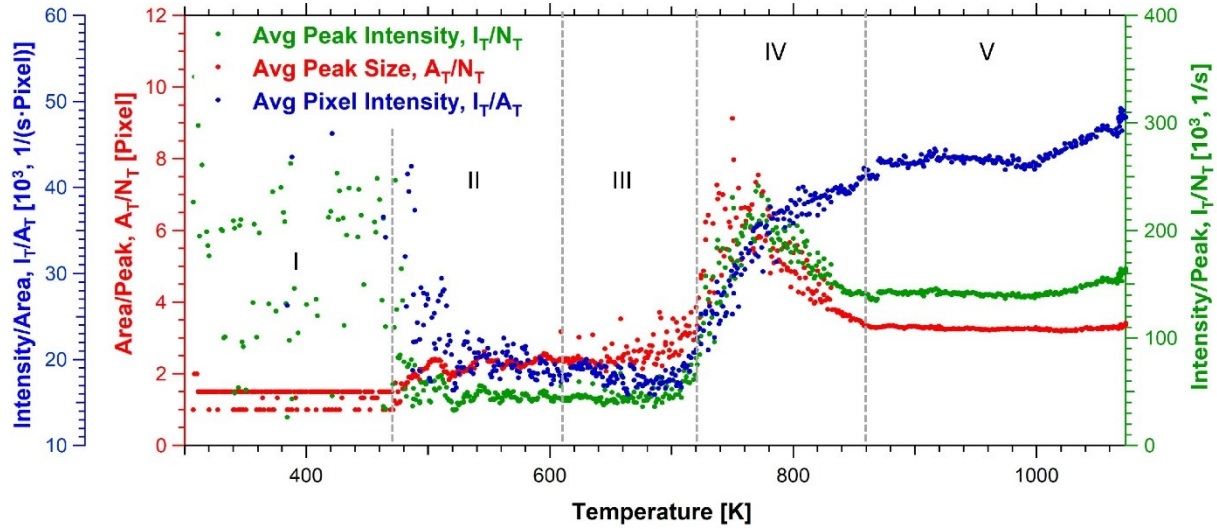
Figure 6 (a) Average grain rotation speed of the moving diffraction peaks against temperature and time count starting from 940 K, and 0 s, respectively, and (b) rotation speed histograms showing distribution of moving peaks at temperatures of 940 K, 985 K, 1020K, 1055, and 1065 K.

4. Discussion

4.1 Recovery, Recrystallization and Grain Growth

Figure 3 displays three quantified white-beam X-ray characteristics, where the peak intensity can be used to inform grain size, with the assumption that the grain sizes are below 10 μm to minimize the primary extinction effect [22,23]. The diffraction area, counted as the total number of pixels, can be related to peak broadening, thus the relative deviatoric strains of each grain [11], and the peak count can be proportional to the number of grains being illuminated.

These peak characteristics are further analyzed, and a plot in Fig. 7 presents the computed ratio and averages of them from the values shown in Fig. 3, where the average peak intensity per area I_T/A_T are shown in blue, the average peak size A_T/N_T indicated in red, and the average peak intensity I_T/N_T described in green. The five separate temperature regions I-V identified in Fig. 3 are added to the plot. Although limited useful information is provided from the average value of each diffraction peak at arbitrary temperatures, evaluation of the averages can reveal qualitative changes between the three attributes. As shown in Fig. 3, region I, below 470 K, has almost no identifiable diffraction peaks because the grain sizes are in the nanoscale range, which was confirmed by a



different study using high-energy synchrotron X-rays on the consistent HPT-processed Cu [9]. Thus, the diffraction peak intensities are overlapping into a sustained halo. One can see the white-beam Laue experiment as a continuous concentric overlap of diffraction rings for each offered wavelength. As long as monochromatic diffraction rings are smooth, there is no distinction in the white-beam case either. Eventually, some individual grains may grow and show diffraction spots on top of the halo. In this region, the plot in Fig. 7 shows that the few identifiable peaks can cause wild variations in the average intensity, but the detector lacks the resolution to gain any useful information within this region.

Figure 7 The average peak characteristics of average intensity per area I_T/A_T in blue, average peak size A_T/N_T in red, and average peak intensity I_T/N_T in green, calculated using the data from Fig. 3.

Over 470 K, region II shows stable values in all ratios of I_T/A_T , I_T/N_T and A_T/N_T , except the early region II showing high peak intensities due to small grain sizes influences, under increasing N_T , A_T and I_T with increasing temperature as shown in Fig. 3. Literature attributes this region to both the recovery and recrystallization stages of microstructural evolution reported in cold-rolled copper [24] and continuously rolled copper [25]. Although grains may not grow during recovery, the grain's mosaic spread sharpens [26], which makes up distinguishable peaks, particularly from the small, illuminated volume. This trend continues in Region III over 610 K through 710 K, with sporadic variations in the data. This temperature range is defined as “exaggerated recrystallization” where the grain sizes are constantly varying in size [15], but can also be caused by physical sample rotation or displacement during the experiment. In Fig. 3, there are consistent increasing trends of I_T , A_T and N_T in regions II and a sustained trend in region III, conversely Fig. 7 shows the average peak intensity and area remain constant with increasing temperature in the stages. Specifically, the average peak intensity that is proportional to average grain size is constant in Fig. 7, while the number of identified peaks N_T grows at a steady rate in Fig. 3, suggesting reasonably constant average grain sizes even with the introduction of newly recrystallized grains in regions II and III. A recent study on the HPT-processed copper estimated a gradual increase in average grain size at 470-710 K, using the method of monochromatic high-energy micro-beam X-ray diffraction [9], maybe capturing such an increase in the sizes of small, recrystallized grains.

Over 710 K to ~770 K in region IV, a sharp increase in both average intensity and peak count marks the grain growth stage, where the most significant changes in grain size occur. A heating experiment using differential scanning calorimetry on continuously rolled copper confirms the occurrence of grain growth in the temperature range [25]. Over this temperature range towards 860 K, the significance of grain growth tends to disappear in terms of the number of grains, thus average peak intensity and area decrease with increasing temperature. When a noticeable decrease in N_T occurs over 860 K in region V, there is reasonably constant average peak intensity and area, while I_T/A_T increases until 1073 K. Thus, it described the primary microstructural features in this range of grain growth given by high enough I_T and reduced N_T of total number count with relaxed average lattice strain state described by the decreasing A_T . The continuous decrease in N_T is primarily attributed to grain growth, as statistically, grain rotations do not significantly reduce intensity because some peaks move out of the field of view while others simultaneously move in.

As noted in the results section, grain size increased large enough to decrease the number of illuminated grains in Region V, where approximately 8% of the diffraction peaks are moving away from the diffraction conditions. There is still a noticeable gradual increase in average peak intensity in I_T/A_T and I_T/N_T after 1000 K, which is evident for further grain growth with minimal lattice strain, thus increasing the peak intensity by more scattering volume of the grain, and low mosaic spread, keeping the peak shape narrow [26].

4.2 Influential Factors of Grain Rotation

The results in Fig. 5 will be further analyzed to evaluate the influential factors of grain rotation in the HPT-processed Cu above 940 K in the present white-beam Laue X-ray diffraction experiment. Fig. 8 shows two normalized histograms of probability density of a specific interval intensity against (a) peak intensity as count per second, and (b) diffraction peak size in pixels for the total peak population N_T marked in green and all the moving peak population N_M marked in red within the temperature range of 940 K - 1073 K. While not a direct comparison, the peak intensity can provide a qualitative contrast for the effect of grain size on grain rotation, along with peak size as an indirect measure of deviatoric strain effect on grain rotations. As presented in Fig. 8(a), the distributions between the total peak population and moving peak population are quite similar, with the exception that moving peak histogram appears more jagged compared to the total peak population due to sparser data, especially at higher intensity of $>2 \times 10^5 \text{ s}^{-1}$, count per second with even more irregular pattern. As for comparing peak size, Fig. 8(b) also displays a similar feature from the sparse moving peak population compared to the total population size. Nonetheless, the two histograms show similar probability distributions, but with some outliers for moving peak populations at peak sizes, i.e., 10 pixels showing little to no probability, and 24 pixels with higher probability density than the total populations. It is worth noting that these outliers are isolated counts and therefore not representative. Nevertheless, they appear only at the moving peaks, indicating that the peaks are indeed Laue reflections and not artifacts of the detector. Overall, the histograms shown in Fig. 8(a) and (b) imply very similar probability densities of N_T and N_M regarding the diffraction peak intensity and size at the examined temperature range.

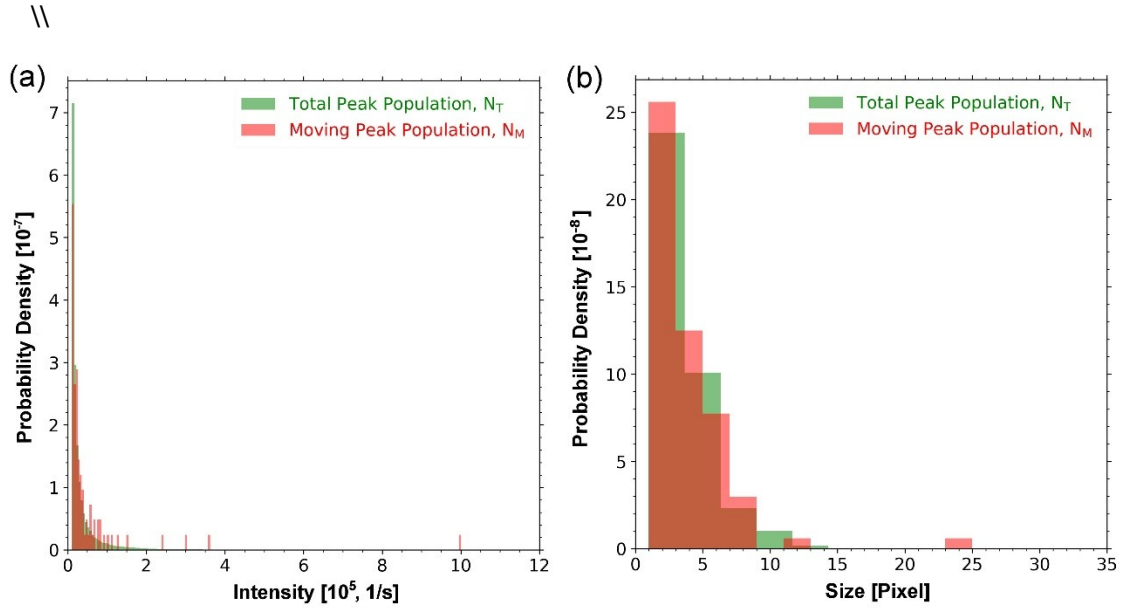


Figure 8 Normalized histograms of probability density of a specific interval intensity against (a) peak intensity as count per second, and (b) diffraction peak size in pixels for the total peak population N_T marked in green and all the moving peak population N_M marked in red at 940 K - 1073 K.

Since determining the visual differences in the histograms in Fig. 8(a) and (b) can be subjective, Kolmogorov-Smirnov (K-S) two-sample statistical tests [27] are employed to objectively determine if the two probability distributions are from the same population of diffraction peaks. The statistical test compares the cumulative distribution functions (CDF) and computes the largest difference between the two to examine a null hypothesis: “two distributions are from the same peak distribution”. Figure 9(a) and (b) show the CDF of both intensity and peak size, respectively, for the total peak population N_T marked in green and all the moving peak population N_M marked in red at

940 K - 1073 K. The K-S statistical tests will compute the largest difference between the curves for N_T and N_M and compare them to the K-S critical value [28] to determine if there is enough evidence that the probability distributions are different. In both peak intensity and size, the K-S tests failed to reject the null hypothesis, concluding no significant evidence for the difference between the two distributions. Unfortunately, the evaluation showed no statistical evidence to prove they are different, while this does not mean that the distributions are the same.

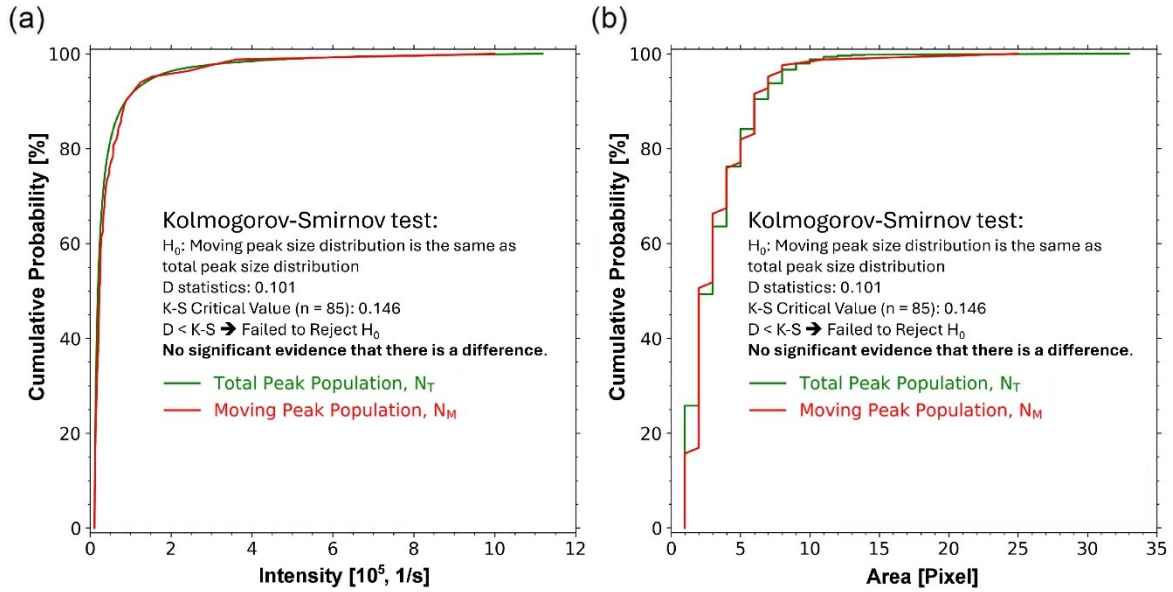


Figure 9 (a) Cumulative distribution function of peak intensity values of moving peak in red and total peak represented in green, and (b) peak size values of moving peak in red and total peak in green.

Nevertheless, these findings align with the first model reported for grain rotations formulated by Harris et alii [5], which is a rotational glide of a grain within the matrix, thus based on Raj & Ashby [29] corrugated grain boundary sliding. Subsequently, Moldovan et alii [30] have generalized the model to arbitrary grain shapes using continuum theories and diffusive flow equations, and introduced the crossover between grain boundary diffusion, dominant at lower temperatures, and lattice diffusion at higher temperatures. In Harris [3], the rate of grain rotation is given in the following equation [5],

$$\frac{d\Theta}{dt} = M \sum_i A_i \frac{d\gamma_i}{d\Theta} \quad (1)$$

where Θ is the instantaneous orientation angle of a grain, t is time, A_i is the area associated with the i_{th} grain boundary of the grain, γ_i is the specific energy referring to the grain boundary energy per unit area, and M is related to the kinetics, denoted as the mobility term that will be explored further in section 4.3. The equation implies that grain rotation may slow down and ultimately cease as grain boundary energy per unit orientation angle decreases. This suggests that rotation may fully accommodate the energy reduction and lead to stable grain orientation. Moreover, Harris [5] as well as Moldovan [6,30] and their collaborators state a strong grain size dependence of L^{-5} to L^{-4} , depending on grain boundary or lattice diffusion, respectively, and where L is the side length of a grain boundary, i.e. proportional to the grain size. Revisiting the results from the statistical evaluation in Fig. 8, the lack of certainty from the K-S tests shown in Fig. 9 supports the grain rotation model, indicating that grain size and deviatoric strain are not proven as the primary factors controlling grain rotation in the evaluation.

Directions of grain rotation can be another indicator for understanding the grain rotation mechanisms. The displacement vectors in Fig. 4(b) are used to quantify the grain rotation directions. Each displacement vector of the moving diffraction peak is extracted, counted and represented in a directional histogram as shown in Fig. 10, where the horizontal direction indicates the radial direction towards the HPT disk center, and the opposite direction is towards the disk edge and will be indicated by 180° . The shear directions are perpendicular to the radial direction. Furthermore, the color of the bars in the histogram transitions with increasing count.

As observed in Fig. 10, the greatest number of grains tend to rotate toward the disk center, followed by grain rotation opposite the radial direction toward the disk edge. By contrast, a relatively low number of grains rotate in the shear direction, especially between 90° to 112° . The component controlling the grain orientation rate in eq. (1), $\Sigma A_i(dy/d\theta)$, implies the grain rotation is heavily influenced by grain morphology and structure. The Cu sample tested in the present study is produced using HPT processing, which introduces a severely deformed nanostructure in Cu by introducing an excess number of lattice defects [31-33]. A simple shear created by HPT processing can transform an equiaxed grain shape into an ellipsoid form with the long axis along the shear direction [34,35] as seen in Fig. 1. The elongated grains aligned along the shear direction induced by HPT may undergo slight coarsening during subsequent heating. Nevertheless, the ellipsoid grains may rotate preferentially vertically to the longer axis of the grain, thus along the radial

direction. As the Harris-Moldovan model [5,6] on mobility depends sensitively on grain size in each dimension, though rotating in the shear direction is smaller, since elongated, than in the radial direction, where the grain dimension is reduced. Moreover, the nature of HPT shear strain introduces the gradient microstructure in grain sizes as well as grain boundary misorientation and defect structures along the radial direction in the HPT-processed metals with different crystal structures [36]. With simple shear under the compression, a turbulent shear flow occurs during HPT, making it even more complex across the disk thickness, observed in an HPT-processed Al-Mg alloy [37] and by modeling [38,39]. After HPT, complex shear patterns remain in both radial and compression directions over the disk vertical cross-section even after heating, as observed in an HPT-processed CoCrFeNiMn alloy through heating to 923 K [3]. These may support the idea that the shear texture and microstructure of the HPT-processed copper remained during heating, contributing to the grain orientation as suggested in eq. (1), leading to preferential grain rotation along the radial directions both towards and away from the disk center. The asymmetry between the two, at 0° and 180° , may reflect the residual stress gradient from the surface to the interior of the HPT disk, and the fact that diffractograms are taken in reflection, rather than transmission geometry, so that deeper disk layers are less probed due to absorption, resulting in a preferred sense of rotation.

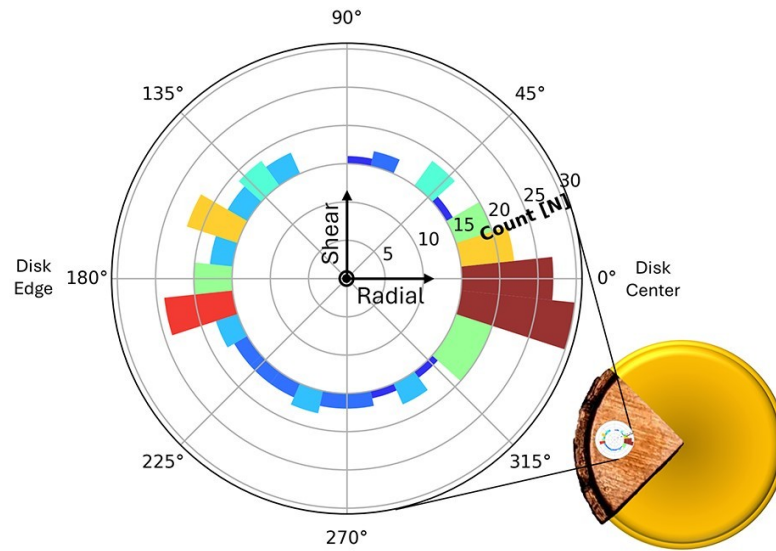
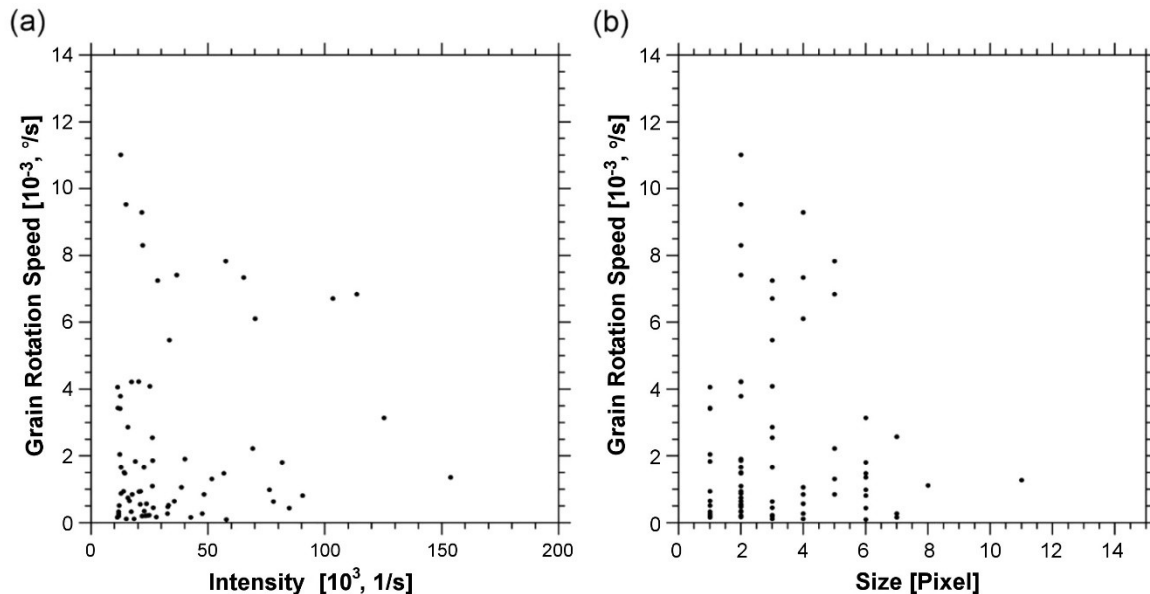


Figure 10 Directional histogram of preferred grain rotation showing horizontal axis being sample radial direction and vertical axis being sample shear direction.

4.3 Kinetics of Grain Rotations

Revisiting the kinetics of thermally activated grain rotations from eq. 1, the term of grain rotation mobility, M , was modified by considering the functions of grain size R and temperature dominating either self-lattice diffusion or grain-boundary diffusion [6], utilizing a diffusion-accommodated grain boundary sliding model [29]. Thus, the rotation mobility of grains M is expressed as $M(R) = C/R^p$ [30], where p is calculated by the grain boundary energy and the mean curvature of grain boundary segment, giving $p = 5$ for grain boundary diffusion and $p = 4$ for self-diffusion, and C is a dimensionless constant dependent on material properties, temperature, grain morphology and the ratio of the diffusion-accommodation mechanisms transiting at a crossover grain size of $R_c = 1.8 \mu\text{m}$ leading to the rate of grain orientation of $d\theta/dt = 0.42 \text{ }^\circ/\text{s}$ [6]. Thus, the study predicted that large-sized grains rotation will be accommodated by the lattice diffusion and have slower mobility, and it is difficult to measure grain orientations experimentally. Nevertheless, during a recent in situ heating high-energy diffraction experiment on the HPT-processed Cu, the grain sizes at 940 K were estimated to be greater than $4.5 \mu\text{m}$ [9]. With an earlier report on thermal grain rotation of a magnesium alloy [13], an in situ heating white-beam diffraction experiment enables the capture of grain rotations quantitatively and qualitatively, even when the grain sizes are much larger than a few microns.

Figure 11 shows the grain rotation speed $d\theta/dt$ against (a) diffraction peak intensity and (b) peak size, respectively. As the rotation mobility $M(R)$ predicts, the rate of grain rotation decreases with increasing grain size, assumed to be related to the intensity of the diffraction peaks as seen in Fig. 11(a). Despite some scatter in the data, high grain rotation speeds tend to occur at relatively low peak intensities among all identified moving peaks, suggesting that smaller grains rotate faster than



the larger counterparts. This observation agrees with the expected behavior described by the grain rotation mobility $M(R)$ and eq. (1). The sporadic spread of data in Fig. 11(b) makes it difficult to visually identify the effect of deviatoric strain, as inferred from peak size, on grain rotation speed. Nevertheless, peak size exhibits a similar trend with the highest speeds appearing at relatively small peak sizes, implying that deviatoric strain may influence grain rotation speed.

Figure 11 (a) Plot of rotation speed with peak intensity (b) and rotation speed with peak size.

Similar to the analysis in Section 4.2, subjective observation of trends does not provide enough justification for definitive conclusions. A Spearman's Rank Correlation [40] is applied to assess the presence of a monotonic trend between rotation speed and diffraction peak attributes. The correlation value is represented as ρ , where $\rho = 0$ implies no correlation, $\rho = -1$ indicates a strong negative correlation, and $\rho = 1$ means a positive correlation. Using the intensity data from Fig. 11(a), the calculated correlation value is $\rho = -2.27 \times 10^{-3}$ with a p-value of 0.98, thereby describing a negligible trend in the negative direction. However, since the probability of quantifying the strength of evidence against a null hypothesis, p -value, is greater than 0.05, the test shows a lack of evidence for a trend between diffraction peak intensity and grain rotational speed. Likewise, the peak size data from Fig. 11(b) also shows a negative correlation value of $\rho = -5.48 \times 10^{-2}$ with a p-value of 0.62. Therefore, there is no significant correlation between peak size and rotation speed as well.

Concluding the present evaluations, there is no dominant dependence of grain rotation speed on either grain size or deviatoric strain. However, the rotation mobility term $M(R)$, and consequently the rate of grain rotation $d\theta/dt$, strongly depend on grain size, morphology and thermal diffusion. Additional microstructural factors, such as densities of lattice defects, including dislocations and vacancies, should be considered. A recent report has explored possible local grain boundary melting to facilitate further diffusion in magnesium alloy at the eutectic temperature [41]. Although grain boundary melting does not play in the present work on copper, it does provide an example of additional mechanisms for thermal grain rotations. Moreover, magnesium shows abnormal grain growth, for which a spontaneous grain boundary activation model has been elaborated [13], opening long-living diffusion channels, and thus enhancing grain rotation of activated grains, even when they become large.

5. Summary and Conclusion

- (1) This study demonstrates an in situ heating white beam Laue X-ray diffraction experiment from 300 K to 1,073 K on a bulk nanostructured Cu processed by high-pressure torsion (HPT), having a grain size of 256 ± 60 nm to evaluate its microstructural evolution under heating.
- (2) The diffraction peak characteristics of intensity, peak area, and total numbers of peaks describing the microstructural evolution under heating can be divided into five distinct temperature ranges labeled between regions I-V. In the high-temperature region (V), over 860K, the number of diffraction peaks decreases with increasing temperature, allowing for tracking individual peak movement. Diffraction peaks in region V are further examined to investigate grain rotation during coarsening.
- (3) Grain reorientations identified by the individual moving diffraction peaks are observed over 940 K (region V) with 7.5% of the total counted diffraction peaks as moving peaks, indicating rotating grains. The results are consistent with the driving force component of the model from eq. 1; however, the mobility component of the model holds primarily for nano-sized grains.
- (4) There is an anisotropy in grain rotation between the radial and shear direction, attributed to elongated grains in the shear direction. Because the rotation speed slows down dramatically with grain size, that size anisotropy reflects in rotation anisotropy.
- (5) The computed average grain rotation speed remains measurable at $\sim 4.1 \times 10^{-3}$ °/s, where the maximum speed of $> 7.8 \times 10^{-3}$ °/s was observed around 1,020 K. Further research and data collection can help develop the model and take into consideration high concentrations of defects produced from nanostructure HPT samples during heating.
- (6) The in-situ heating white-beam Laue method enables the quantification of the rotation of coarsening grains while interrogating large numbers of grains. This unconventional approach is especially effective and novel for quantifying small-length scale features within a bulk volume, which is often unachievable with other techniques, including microscopy techniques.

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CRedit author statement

Isshu Lee: Investigation. Data curation, Writing- Original draft preparation; **Laxman Bhatta:** Investigation; **Jae-Kyung Han:** Investigation; **Nobumichi Tamura:** Methodology; **Klaus-Dieter Liss:** Conceptualization, Writing- Reviewing and Editing; **Megumi Kawasaki:** Supervision, Writing- Reviewing and Editing

Data Availability Statement

The data that support the findings of this study are available from the corresponding author on reasonable request.

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Figure captions

- Fig. 1** (a) shows a schematic drawing of an in situ heating synchrotron diffraction setup at ALS, and (b) is an example of a white-beam Laue diffraction pattern of a polycrystalline copper at 860 K with a scattering vector about normal to the sample surface.
- Fig. 2** Dark field TEM image of the initial microstructure of HPT copper. Shear direction is left to right.
- Fig. 3** Total peak intensity (I_T), total peak area (A_T), and total peak count (N_T) against temperature plot divided into I-V temperature regions.
- Fig. 4** (a) plot of the detector's vertical and horizontal pixels showing the movement of the moving diffraction peaks, and (b) the displacement vector of the diffraction peaks until moving out of the diffraction condition at temperatures between 940-1073 K.
- Fig. 5** Plot showing trends of the instantaneous total number of grains, N_T , the instantaneous moving grains, N_M , and the ratio of moving grains N_M/N_T at 940 K to 1070 K.
- Fig. 6** (a) Average grain rotation speed of the moving diffraction peaks against temperature and time count starting from 940 K, and 0 s, respectively, and (b) rotation speed histograms showing distribution of moving peaks at temperatures of 940 K, 985 K, 1020K, 1055, and 1065 K.
- Fig. 7** The average peak characteristics of average intensity per area I_T/A_T in blue, average peak size A_T/N_T in red, and average peak intensity I_T/N_T in green, calculated using the data from Fig. 3.
- Fig. 8** Normalized histograms of probability density of a specific interval intensity against (a) peak intensity as count per second, and (b) diffraction peak size in pixels for the total peak population N_T marked in green and all the moving peak population N_M marked in red at 940 K - 1073 K.
- Fig. 9** (a) Cumulative distribution function of peak intensity values of moving peak in red and total peak represented in green, and (b) peak size values of moving peak in red and total peak in green.
- Fig. 10** Directional histogram of preferred grain rotation showing horizontal axis being sample radial direction and vertical axis being sample shear direction.
- Fig. 11** (a) Plot of rotation speed with peak intensity (b) and rotation speed with peak size.

