Characterization of Neutron-Irradiated HT-UPS Steel by High-Energy X-ray Diffraction Microscopy

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Abstract

This paper presents the first measurement of neutron-irradiated microstructure using far-field high-energy X-ray diffraction microscopy (FF-HEDM) in a high-temperature ultrafine-precipitate-strengthened (HT-UPS) austenitic stainless steel. Grain center of mass, grain size distribution, crystallographic orientation (texture), diffraction spot broadening and lattice constant distributions of individual grains were obtained for samples in three different conditions: non-irradiated, neutron-irradiated (3dpa/500°C), and irradiated+annealed (3dpa/500°C +600°C/1h). It was found that irradiation caused significant increase in grain-level diffraction spot broadening, modified the texture, reduced the grain-averaged lattice constant, but had nearly no effect on the average grain size and grain size distribution, as well as the grain size-dependent lattice constant variations. Post-irradiation annealing largely reversed the irradiation effects on texture and average lattice constant, but inadequately restored the microstrain.

Keywords

Austenitic steel; High-energy X-ray diffraction microscopy; Irradiation effect; Meso-scale

Introduction

High-temperature ultrafine-precipitate-strengthened (HT-UPS) austenitic stainless steel is a candidate for structural and cladding applications in the next-generation advanced nuclear reactors because of its improved creep resistance at high temperatures over traditional 316 stainless steels (316 SS) and potentially better radiation resistance [1-3]. By controlled additions of carbide-forming elements such as Ti and Nb, fine MC carbides formed during thermomechanical treatments or plastic deformation can serve as pinning sites for dislocation movement [3-6], which improves the creep resistance by reducing the strain accumulation during primary creep and prolonging the secondary creep regime. Those MC carbides may suppress the defect accumulation under irradiation, leading to enhanced radiation resistance relative to 316 SS. In neutron irradiated 316 SS at temperatures between 300°C to 700°C [7-10], Frank loops, dislocation networks, bubbles/voids, precipitation and solute segregation are common features of microstructural evolution. They can lead to property changes including irradiation hardening, embrittlement, dimensional instability, and decreased creep resistance [9, 11-13]. Presently, there are limited data on neutron-irradiated HT-UPS, and the structure-property relationships have not been extensively investigated, which motivates this study.

Transmission electron microscopy (TEM) of polycrystalline alloys subject to irradiation, either post-mortem or *in-situ*, offer a great deal of detailed knowledge about defect formation and evolution, phase transformation and stability, etc. at atomic and nanometer scales. The microstructural information obtained through TEM is on thin specimens and typically covers a few grains at maximum. On the other hand, X-ray diffraction techniques such as wide-angle scattering and small-angle scattering interrogate bulk-scale sample volumes to obtain statistically significant data [14-16]. While TEM and X-ray techniques are complementary, comparative

information at the grain scale in a polycrystalline sample is typically unobtainable. Several studies have pointed out that information at this intermediate length scale may be important for understanding the structure-property relationship [17-21]. For materials subjected to irradiation, the modifications of grain-level microstructures due to the high-energy particle-atom interactions remain largely unaddressed in current literature, yet could be important in affecting the materials performance.

High energy diffraction microscopy (HEDM), also known as 3D-X-ray Diffraction (3D-XRD), is a non-destructive characterization technique capable of delivering information from thousands of grains, in the bulk, at the individual grain level. In this study we employ HEDM to resolve the effect of neutron irradiation on grains in the HT-UPS steel. While there are several variants of HEDM, we used the far-field HEDM (FF-HEDM) technique to obtain the strains, crystallographic orientations, and center of masses (COMs) of thousands of grains in a HT-UPS alloy before/after neutron irradiation and after post-irradiation annealing, providing rich 3D information well beyond the TEM and traditional diffraction measurements [22-26]. This work presents unique data sets that demonstrate grain-level irradiation effects with good statistics, and opens the opportunity for *in-situ* thermal-mechanical deformation tests for a deeper understanding of the structure-property relationship in nuclear reactor materials.

Materials and Methods

The nominal composition (wt%) of the HT-UPS stainless steel samples examined in this work is shown in Table 1.

The material was solution annealed at 1200°C for 1 h prior to irradiation. Three samples were studied: as-received, neutron irradiated to 3 dpa at 500°C, and same condition irradiated followed

by 600°C annealing for 1 h. Neutron irradiation was conducted at the Advanced Test Reactor at Idaho National Laboratory (INL). Specimens were provided by the Pre-irradiated Sample Library of the Nuclear Science User Facilities (NSUF) at the INL. Detailed information of each sample is listed in Table 2. The two irradiated disk samples had low activity, < 5 mR/h at a distance of 30 cm per sample. They were individually mounted and sealed in double-contained Kapton sample holders at the Irradiated Materials Laboratory at the Argonne National Laboratory (ANL). The as-received coupon sample was mounted on the same type of the specimen holder without Kapton tubing. They were then transferred to the APS 1-ID beamline for FF-HEDM experiments.

Figure 1 is a schematic of the FF-HEDM experimental set-up at the 1-ID-E beamline, the Advanced Photon Source (APS). The X-ray beam energy was 65 keV, and the beam size was 2 mm (horizontal) $\times$ 0.2 mm (vertical). A single panel GE-RT41 detector was placed approximately 910 mm downstream from the sample, nominally centered with the incident beam. The lab XYZ translations were used to place the ω -rotation axis (rotation about Y_L as illustrated in Figure 1) in the beam. The sample XYZ translations were used to place the volume of interest in the beam and on the rotation axis. The instrument calibration procedure can be found in Ref. [27], which gives a lattice strain resolution of $\sim 5 \times 10^{-5}$. For a particular volume illuminated by the incident X-ray beam, a series of GE detector images were acquired in sweeping mode at 0.5° intervals in ω over a range of -180° to $+180^\circ$. The sample was then translated along Y_L by 0.2 mm to interrogate a different layer of material. For each sample, data for four layers were collected, as illustrated by the four blue boxes in the top right corner of Figure 1.

Data analysis was performed using Microstructural Imaging using Diffraction Analysis Software (MIDAS) [28] developed at the APS. The $\{111\}$ and $\{200\}$ diffraction spots from the austenite phase were analyzed. Twins were treated as independent grains in the analysis. Those spots were first identified using intensity threshold and their positions were corrected for systematic distortions using the instrument parameters obtained from calibration data. The spots are then fitted and indexed [29, 30] to obtain the COMs, crystallographic orientations, and strains of the constituent grains in the illuminated volume.

The spatial resolution of the FF-HEDM technique for this set of experiments was 10 μm in grain radius. Not every identified grain had all the expected diffracting spots matching with observations for the following reasons: (1) certain grains might be highly deformed, and (2) the diffraction spots for some grains might be too weak to be effectively distinguished from the background. The completeness parameter [26] was then used to filter unreliable indexing results. Completeness of an indexed grain is defined as the ratio between the number of matched diffraction spots to the number of overall expected spots (which is $(2^3+2\times 3)\times 2=28$ in the case of using $\{111\}$ and $\{200\}$ spots for full 360° rotation). Only grains that had a completeness of 0.7 or higher were accepted, the same as in a number of studies [31-35]. The COMs of those accepted grains were then further checked and filtered if their COMs lied outside of the illuminated volume, which occurred for about 1% of the accepted grains.

Results

Figure 2 shows the COM of the grains in the three samples: as-received (AR), neutron-irradiated (irr) and irradiated+600°C annealed (irr+ann). In total 943 grains are detected in the AR sample, 843 in the irr sample, and 1457 in the irr+ann sample. The sizes of the dots in Figure 2 are scaled

with the diameters of the grains. For each sample, the volumes of the indexed grains are summed and the sum is compared with the overall illuminated volume. For the AR and irr+ann samples, the detected grains contribute to more than 90% of the total illuminated volume, while for the irr sample the number is 75%. The missing volume could be from the grains that were discarded during completeness and COM screening, or from small grains that gave too weak diffraction intensity to be identified. The colors designate the completeness of the grains where blue is 0.7 completeness and red is 1.0 completeness. In general, grains in the irr sample have lower completeness compared to the AR and the irr+ann samples; the average completeness of the grains in the irr sample is 0.84, while it is 0.90 for the AR sample and 0.89 for the irr+ann sample. The fact that the irr sample has the lowest detected volume fraction and its grains have the lowest averaged completeness imply that irradiation introduces damage to the constituent grains in the polycrystalline aggregate.

The grain size histograms of the three samples are shown in Figure 3 (a), with the inset showing the lognormal fittings of the profiles. The peaks of the fitting profiles are 22 μm for the AR sample, 17 μm for the irr sample and 19 μm for the irr+ann sample. The volume-weighted average of radii is 44 μm for the AR sample, 41 μm for the irr sample and 40 μm for the irr+ann sample. It is seen that irradiation to 3 dpa at 500 C does not have a significant effect on the average grain size and grain size distribution of the HT-UPS steel. Figure 3 (b) shows again the COMs of the grains, but this time the colors designate the grain radii where the blue corresponds to 10 μm and red corresponds to 100 μm . As can be seen, the size distribution is fairly random, i.e. there is no abnormal grain growth. To validate the grain sizes measured by the HEDM technique, we performed metallography of an as-received sample which was also cut from the same plate. A representative optical image is shown in Figure 4 (a). The radii of austenite grains

with the size $>10\text{ }\mu\text{m}$ were measured, and the size distribution was compared with that measured by HEDM, shown in Figure 4 (b). It is observed that: 1) The size ranges are comparable ($10\text{ }\mu\text{m}$ to $100\text{ }\mu\text{m}$); 2) the peak positions are similar (around $25\text{ }\mu\text{m}$). Such a good agreement between the results from the two techniques confirms that the HEDM measurement on grain sizes is valid.

The effect of irradiation and post-irradiation annealing on the diffraction spots from single, representative grains are shown in Figure 5 (a), which shows in total 14 spots from $\{111\}$ and $\{200\}$ reflections in the first 180° rotation for one grain in each sample. Those grains are all $\sim 100\text{ }\mu\text{m}$ in radius. Compared with spots from the un-irradiated grain and the post-irradiation annealed grain, those from the irradiated grain show smeared intensities. Statistically, changes in full-width at half maximum of all the $\{111\}$ and $\{200\}$ spots were quantified by fitting a two dimensional peak profile function to each spot. The results are shown in Figure 5 (b) and (c), respectively. The spot width in the radial direction (ρ) (Figure 5 (b)) is sensitive to the grain size and non-uniform strain, while the width in azimuthal direction (η) (Figure 5 (c)) is a measure of crystallographic reorientation of a grain and the formation of sub-grain structures. As seen, peaks from the irr sample are broader in both directions than the other two samples. Post-irradiation annealing partially recovers the modification in the ρ direction as evidenced by the peak of the irr+ann sample lying in between the AR and the irr samples. The recovery was almost complete in the η direction, which is in agreement with the texture result to be shown in Figure 6. The completely recovered peak broadening in the azimuthal direction and partially recovered broadening in the radial direction clearly showed that post-irradiation annealing at 600°C for 1 h can return the sample to its initial texture state, but not the local strain level.

The crystallographic orientations of the indexed grains are used to compute the orientation distribution function (ODF) of the aggregate. For a sample, the set of crystallographic

orientations found from the FF-HEDM analysis are used as seed points in orientation space. Each seed point is associated with a Gaussian function in orientation space with angular width, σ , to generate a smooth ODF. In this paper, σ value of 5° was used. All the calculations were performed using the OdfPf package from Deformation Processes Laboratory, Cornell University [36]. The ODF are plotted over the surface of cubic fundamental region using Rodrigues parameterization. The AR sample shows the typical two-fiber texture induced under plane strain compression [37] from the rolling process. After irradiation, the ODF is sharper. Post-irradiation annealing recovered the fiber texture. Recall that for the irradiated sample, only 75% of the total volume was detected, in contrast to over 90% for the AR and irr+ann sample. It is possible that the missing segments in the fibers in the irr sample are related to the undetected volume.

One great advantage of the FF-HEDM technique is its sensitivity to changes in the lattice constant and its ability to measure the strains in the constituent grains. Figure 7 (a) plots the lattice constant of each grain as a function of grain size for all the grains in the three samples; the color denotes the completeness. The lattice constant is obtained by taking the average of a, b, and c in the distorted lattice as indexed by MIDAS. The volume-weighted average of the lattice constants in each sample is 3.5974 Å for the AR, 3.5970 Å for the irr and 3.5973 Å for the irr+ann. A unique and interesting observation is that for each sample, the lattice constant varies as a function of grain size. The lattice constant shows a broader distribution towards smaller values as the grain size decreases. In addition, grains with smaller lattice constants tend to have lower completeness, and those features remain virtually the same even if with an increased threshold of the completeness (from 0.7 to 0.8), as shown in Figure 7(b). While neutron irradiation has an insignificant effect on the distribution profile, it did reduce the average lattice parameter by 0.0004 Å, which is a measurable change using the instrument [27]. To further back

up the observation, as reported in Ref. [27], another approach, which integrated the diffraction signal from the HEDM measurements to form a powder-like 1D diffraction profile and calculated the lattice parameters, reported a 0.0002 Å reduction after irradiation. Such changes can be attributed to the defects created during irradiation, and/or to chemical changes (e.g. solute segregation). Post-radiation annealing recovers the average lattice parameter by 0.0003 Å, which can be attributed to defect recovery and/or precipitation. Figure 7 (c) shows the distribution of the microstrain of all the grains in each sample, taking the average lattice parameter of each sample as the reference. It is seen that the distributions are essentially the same among these three samples. This implies that the irradiation-induced lattice strain change is uniform across the grains.

Discussion

We have shown that the far-field high-energy X-ray diffraction microscopy (FF-HEDM) is a valuable non-destructive technique to obtain comprehensive crystallographic information of individual grains over a statistically significant ensemble. In this study, the effects of neutron irradiation as well as post-irradiation annealing in a HT-UPS steel were examined at the grain scale rather than conventional grain-averaged behavior. The fact that the average completeness and the fraction of recovered volume are the lowest in the neutron-irradiated sample implies that the quality of diffraction signals from the irradiated sample is lower due to irradiation-induced defects. Neutron irradiation to 3 dpa at 500°C has no significant effect on the spatial and size distribution of grains in the steel alloy, as shown in Figure 3. While the grain-averaged lattice constant is reduced after irradiation, the grain-level lattice strain distribution shows no change after irradiation, as shown in Figure 7. Note that no new diffraction peaks were detected in the irradiated and post-irradiation-annealed specimens, and therefore no measurable second-phase

precipitation occurred during irradiation or post-irradiation annealing. The reduction of lattice constants in the irradiated specimens may be attributed to the presence of residual stress, the formation of radiation defects, or structural relaxation [38-40]. The lattice constant almost increases back to the as-received value following post-irradiation annealing with a slightly sharper distribution profile, indicating a recovery of crystallinity and possibly chemical changes (e.g. precipitation). Effects from irradiation and post-irradiation annealing can be further quantified using broadening of $\{111\}$ and $\{200\}$ diffraction spots from individual grains, as shown in Figures 5 (b) and (c). In contrast to the lattice strain distribution, the peak broadening profiles are significantly modified by irradiation and post-irradiation annealing. Preliminary TEM characterization of the irradiated HT-UPS specimen (to be published) indicates that Frank loops are the primary defects formed under irradiation. The results in Figure 5 and Figure 7 imply that the peak broadening is more sensitive to defect population induced by irradiation rather than the peak shift. Figure 7 also implies that the irradiation-induced elastic strain and/or chemical effect is uniform among grains and irradiation has a minimal effect on the intergranular strain distribution. Further investigations of irradiated microstructure, including TEM and Atom Probe Tomography, are underway.

The grain size dependence of lattice parameters shown in Figure 7 (a) is an interesting finding. For all samples, grains with smaller sizes give broader variations and smaller averaged values in lattice parameter. The lattice strain distribution (Fig. 7(b)) spans from -0.12% to 0.07%, implying significant grain-dependent intergranular strains. Neutron irradiation and post-irradiation annealing show minimal effect on grain size-dependent lattice parameter variations though they do modify the grain-averaged lattice constant. Earlier studies on nanocrystalline materials have reported non-monotonic lattice parameter variations with average crystallite size [41-43]. These

observations were explained as the result of two competing mechanisms: the interfacial stress (surface curvature) induced contraction and the grain boundary free volume induced expansion. However, those studies were focused on crystallites that were below 100 nm where grain boundaries taken up a significant amount of volume. The extrapolation of their results to μm -sized grains yields a lattice strain that is orders of magnitude smaller than those observed here. Therefore, such a grain boundary effect cannot explain the lattice constant variation observed in the HT-UPS specimen. Further investigations are needed to clarify the mechanisms as well as to study the effect from this phenomenon on material's properties.

It is worth mentioning that the information of 3D grain size distribution provided by HEDM may help to answer some fundamental questions in materials strengthening. It has been well recognized that, apart from the Hall-Petch relation [44, 45] which claims that the tensile yield stress of a polycrystalline alloy scales with the inverse square root of its mean grain size, the actual size dispersion also has a significant impact on the overall yield stress, especially for fine-grained materials [46-55]. In current practice, statistically significant grain size distributions are produced by characterizing 2D images of polished surfaces and then applying statistical and geometrical methods to extrapolate the data to 3D[56]; for example, a well-received method is the Schwartz-Saltykov diameter method [57]. These methods are limited by the assumptions and simplifications they adopted. Serial sectioning can be further applied to overcome many of these limitations to create 3D maps [58, 59], which although provides high-quality microstructural images, is time consuming and destructive. In contrast, the information from FF-HEDM method is non-destructive and can be obtained from thousands of grains simultaneously, opening up the possibility for in-situ mechanical test studies. Since the diffraction data is crystallography-

sensitive, this method can even be applied to multi-phase materials to individually characterize each phase in a non-destructive manner.

Conclusion

In this study we investigated the effect of neutron irradiation and post-irradiation annealing on grain size, texture, grain-level lattice constant and broadening of diffraction spots in a HT-UPS steel, using the 3D high-energy X-ray diffraction microscopy (HEDM) technique. It was shown that neutron irradiation to 3 dpa at 500°C introduced damage to the crystallinity of the grains and modified the texture but had nearly no effect on average grain size and size distribution. Broadening of diffraction spots from individual grains was largely modified by irradiation-induced defects. Post-irradiation annealing at 600°C for 1 h reversed the irradiation effects on texture, but inadequately restored the strain state. It was also shown that there was an apparent correlation between the lattice constant of an individual grain and the grain size, with a smaller averaged value and a broader variation in lattice constant in smaller grains. Neutron irradiation reduced the grain-averaged lattice constant but did not affect the lattice constant–grain size correlation. Post-irradiation annealing largely restored the average lattice constant. The rich information of grain-level microstructures obtained using this 3D-XRD technique offers a new opportunity for a better understanding of the structure-property relationship in nuclear materials at the meso-scale.

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Reference

- [1] P.J. Maziasz. DEVELOPING AN AUSTENITIC STAINLESS-STEEL FOR IMPROVED PERFORMANCE IN ADVANCED FOSSIL POWER FACILITIES, *Jom-Journal of the Minerals Metals & Materials Society* 41 (1989) 14-20.
- [2] J.T. Busby. Advanced materials for nuclear reactor systems: Alloys by design to overcome past limitations. International conference on fast reactors and related fuel cycles (FR09): Challenges and opportunities. Kyoto, Japan: International Atomic Energy Agency, 2009. p.147-148.
- [3] R.W. Swindeman, P.J. Maziasz. The mechanical and microstructural stability of austenitic stainless steels strengthened by MC-forming elements, 1992.
- [4] J.P. Shingledecker, P.J. Maziasz, N.D. Evans, M.J. Pollard. Creep behavior of a new cast austenitic alloy, *International Journal of Pressure Vessels and Piping* 84 (2007) 21-28.
- [5] K.H. Lo, C.H. Shek, J.K.L. Lai. Recent developments in stainless steels, *Materials Science and Engineering: R: Reports* 65 (2009) 39-104.
- [6] T. Sourmail. Precipitation in creep resistant austenitic stainless steels, *Materials Science and Technology* 17 (2001) 1-14.
- [7] P.J. Maziasz. OVERVIEW OF MICROSTRUCTURAL EVOLUTION IN NEUTRON-IRRADIATED AUSTENITIC STAINLESS-STEELS, *Journal of Nuclear Materials* 205 (1993) 118-145.
- [8] S.J. Zinkle, P.J. Maziasz, R.E. Stoller. DOSE DEPENDENCE OF THE MICROSTRUCTURAL EVOLUTION IN NEUTRON-IRRADIATED AUSTENITIC STAINLESS-STEEL, *Journal of Nuclear Materials* 206 (1993) 266-286.
- [9] T.R. Allen, J.I. Cole, C.L. Trybus, D.L. Porter, H. Tsai, F. Garner, E.A. Kenik, T. Yoshitake, J. Ohta. The effect of dose rate on the response of austenitic stainless steels to neutron radiation, *Journal of Nuclear Materials* 348 (2006) 148-164.
- [10] A. Etienne, B. Radiguet, P. Pareige, J.P. Massoud, C. Pokor. Tomographic atom probe characterization of the microstructure of a cold worked 316 austenitic stainless steel after neutron irradiation, *Journal of Nuclear Materials* 382 (2008) 64-69.

- [11] G.R. Odette, D. Frey. Development of mechanical property correlation methodology for fusion environments, *Journal of Nuclear Materials* 85–86, Part 2 (1979) 817-822.
- [12] J.P. Foster, T.M. Karlsen. IRRADIATION CREEP AND IRRADIATION STRESS RELAXATION OF 316 AND 304L STAINLESS STEELS IN THERMAL AND FAST NEUTRON SPECTRUM REACTORS, 15th International Conference on Environmental Degradation of Materials in Nuclear Power Systems-Water Reactors (2011) 1339-1350.
- [13] M.B. Toloczko, F.A. Garner. Stress and temperature dependence of irradiation creep of selected FCC and BCC steels at low swelling. in: Grossbeck ML, Allen TR, Lott RG, Kumar AS, (Eds.). *Effects of Radiation on Materials: 21st International Symposium*, vol. 1447. 2004. pp. 454-467.
- [14] M.M. Li, L.Y. Wang, J.D. Almer. Dislocation evolution during tensile deformation in ferritic-martensitic steels revealed by high-energy X-rays, *Acta Materialia* 76 (2014) 381-393.
- [15] L.Y. Wang, M.M. Li, J. Almer. In situ characterization of Grade 92 steel during tensile deformation using concurrent high energy X-ray diffraction and small angle X-ray scattering, *Journal of Nuclear Materials* 440 (2013) 81-90.
- [16] L.Y. Wang, M.M. Li, J. Almer, T. Bieler, R. Barabash. Microstructural characterization of polycrystalline materials by synchrotron X-rays, *Frontiers of Materials Science* 7 (2013) 156-169.
- [17] S.L. Wong, M. Obstalecki, M.P. Miller, P.R. Dawson. Stress and deformation heterogeneity in individual grains within polycrystals subjected to fully reversed cyclic loading, *Journal of the Mechanics and Physics of Solids* 79 (2015) 157-185.
- [18] H. Lim, J.D. Carroll, C.C. Battaile, T.E. Buchheit, B.L. Boyce, C.R. Weinberger. Grain-scale experimental validation of crystal plasticity finite element simulations of tantalum oligocrystals, *International Journal of Plasticity* 60 (2014) 1-18.
- [19] T.J. Turner, P.A. Shade, J.C. Schuren, M.A. Groeber. The influence of microstructure on surface strain distributions in a nickel micro-tension specimen, *Modelling and Simulation in Materials Science and Engineering* 21 (2013) 015002.
- [20] R. Pokharel, J. Lind, A.K. Kanjarla, R.A. Lebensohn, S.F. Li, P. Kenesei, R.M. Suter, A.D. Rollett. Polycrystal Plasticity: Comparison Between Grain-Scale Observations of Deformation and Simulations, *Annual Review of Condensed Matter Physics*, Vol 5 5 (2014) 317-346.
- [21] P. Dawson, J. Gerken, T. Marin. *Modeling Heterogeneous Intragrain Deformations Using Finite Element Formulations*, 2011.
- [22] U. Lienert, S.F. Li, C.M. Hefferan, J. Lind, R.M. Suter, J.V. Bernier, N.R. Barton, M.C. Brandes, M.J. Mills, M.P. Miller, B. Jakobsen, W. Pantleon. High-energy diffraction microscopy at the advanced photon source, *Jom* 63 (2011) 70-77.
- [23] W. Ludwig, A. King, P. Reischig, M. Herbig, E.M. Lauridsen, S. Schmidt, H. Proudhon, S. Forest, P. Cloetens, S.R. du Roscoat, J.Y. Buffiere, T.J. Marrow, H.F. Poulsen. New opportunities for 3D materials science of polycrystalline materials at the micrometre lengthscale by combined use of X-ray diffraction and X-ray imaging, *Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing* 524 (2009) 69-76.
- [24] J. Oddershede, S. Schmidt, H.F. Poulsen, L. Margulies, J. Wright, M. Moscicki, W. Reimers, G. Winther. Grain-resolved elastic strains in deformed copper measured by three-dimensional X-ray diffraction, *Materials Characterization* 62 (2011) 651-660.
- [25] J. Oddershede, S. Schmidt, H.F. Poulsen, H.O. Sorensen, J. Wright, W. Reimers. Determining grain resolved stresses in polycrystalline materials using three-dimensional X-ray diffraction, *Journal of Applied Crystallography* 43 (2010) 539-549.
- [26] H.F. Poulsen. An introduction to three-dimensional X-ray diffraction microscopy, *Journal of Applied Crystallography* 45 (2012) 1084-1097.
- [27] J.-S. Park, X. Zhang, H. Sharma, P. Kenesei, D. Hoelzer, M. Li, J. Almer. High-energy synchrotron x-ray techniques for studying irradiated materials, *Journal of Materials Research* 30 (2015) 1380-1391.

- [28] <https://www1.aps.anl.gov/Science/Scientific-Software/MIDAS>.
- [29] H. Sharma, R.M. Huizenga, S.E. Offerman. A fast methodology to determine the characteristics of thousands of grains using three-dimensional X-ray diffraction. II. Volume, centre-of-mass position, crystallographic orientation and strain state of grains, *Journal of Applied Crystallography* 45 (2012) 705-718.
- [30] H. Sharma, R.M. Huizenga, S.E. Offerman. A fast methodology to determine the characteristics of thousands of grains using three-dimensional X-ray diffraction. I. Overlapping diffraction peaks and parameters of the experimental setup, *Journal of Applied Crystallography* 45 (2012) 693-704.
- [31] L. Wang, J. Lind, H. Phukan, P. Kenesei, J.S. Park, R.M. Suter, A.J. Beaudoin, T.R. Bider. Mechanical twinning and detwinning in pure Ti during loading and unloading - An in situ high-energy X-ray diffraction microscopy study, *Scripta Materialia* 92 (2014) 35-38.
- [32] S. Schmidt, H.F. Poulsen, G.B.M. Vaughan. Structural refinements of the individual grains within polycrystals and powders, *Journal of Applied Crystallography* 36 (2003) 326-332.
- [33] H.F. Poulsen, S. Schmidt, D.J. Jensen, H.O. Sørensen, E.M. Lauridsen, U.L. Olsen, W. Ludwig, A. King, J.P. Wright, G.B.M. Vaughan. 3D X-RAY DIFFRACTION MICROSCOPY. Strain and Dislocation Gradients from Diffraction. IMPERIAL COLLEGE PRESS, 2014. pp. 205-253.
- [34] J. Oddershede, M. Majkut, Q. Cao, S. Schmidt, J.P. Wright, P. Kenesei, J.E. Daniels. Quantitative grain-scale ferroic domain volume fractions and domain switching strains from three-dimensional X-ray diffraction data, *Journal of Applied Crystallography* 48 (2015) 882-889.
- [35] L. Nervo, A. King, J.P. Wright, W. Ludwig, P. Reischig, J. Quinta da Fonseca, M. Preuss. Comparison between a near-field and a far-field indexing approach for characterization of a polycrystalline sample volume containing more than 1500 grains, *Journal of Applied Crystallography* 47 (2014) 1402-1416.
- [36] Cornell University Deformation Processes Laboratory.
<http://anisotropy.mae.cornell.edu/onr/Matlab/matlab.html>.
- [37] A. Kumar, P.R. Dawson. Computational modeling of FCC deformation textures over Rodrigues' space, *Acta Materialia* 48 (2000) 2719-2736.
- [38] P. Ehrhart. INVESTIGATION OF RADIATION-DAMAGE BY X-RAY-DIFFRACTION, *Journal of Nuclear Materials* 216 (1994) 170-198.
- [39] W. Maysenholder. LOWEST-ORDER APPROXIMATIONS TO RELAXATION VOLUMES OF MONOVACANCIES IN CUBIC METALS FROM PAIR POTENTIALS AND FINNIS-SINCLAIR POTENTIALS, *Philosophical Magazine a-Physics of Condensed Matter Structure Defects and Mechanical Properties* 53 (1986) 783-791.
- [40] R.S. Averback, P. Bellon. Fundamental Concepts of Ion-Beam Processing. in: Bernas H, (Ed.). *Materials Science with Ion Beams*, vol. 116. 2010. pp. 1-28.
- [41] G.K. Rane, U. Welzel, S.R. Meka, E.J. Mittemeijer. Non-monotonic lattice parameter variation with crystallite size in nanocrystalline solids, *Acta Materialia* 61 (2013) 4524-4533.
- [42] M.B. Ponnuchamy, A.S. Gandhi. Lattice expansion and contraction in nanocrystalline yttria-stabilized zirconia powders, *Scripta Materialia* 83 (2014) 21-24.
- [43] A.K. Srivastav, N. Chawake, B.S. Murty. Grain-size-dependent non-monotonic lattice parameter variation in nanocrystalline W: The role of non-equilibrium grain boundary structure, *Scripta Materialia* 98 (2015) 20-23.
- [44] E.O. Hall. The deformation and ageing of mild steel III. Discussion of results, *Proceedings of the Physical Society. Section B* 64 (1951) 747-753.
- [45] N.J. Petch. THE CLEAVAGE STRENGTH OF POLYCRYSTALS, *Journal of the Iron and Steel Institute* 174 (1953) 25-28.
- [46] S. Berbenni, V. Favier, M. Berveiller. Impact of the grain size distribution on the yield stress of heterogeneous materials, *International Journal of Plasticity* 23 (2007) 114-142.

- [47] S. Berbenni, F. Wagner, N. Allain-Bonasso. On the Roles of Grain Size Dispersion and Microscale Hall-Petch Relation on the Plastic Behavior of Polycrystalline Metals, *Materials Transactions* 55 (2014) 39-43.
- [48] H.Y. Chao, Y. Yang, X. Wang, E.D. Wang. Effect of grain size distribution and texture on the cold extrusion behavior and mechanical properties of AZ31 Mg alloy, *Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing* 528 (2011) 3428-3434.
- [49] C.F.O. Dahlberg, J. Faleskog. Strain gradient plasticity analysis of the influence of grain size and distribution on the yield strength in polycrystals, *European Journal of Mechanics a-Solids* 44 (2014) 1-16.
- [50] F. Lavergne, R. Brenner, K. Sab. Effects of grain size distribution and stress heterogeneity on yield stress of polycrystals: A numerical approach, *Computational Materials Science* 77 (2013) 387-398.
- [51] N. Nicaise, S. Berbenni, F. Wagner, M. Berveiller, X. Lemoine. Coupled effects of grain size distributions and crystallographic textures on the plastic behaviour of IF steels, *International Journal of Plasticity* 27 (2011) 232-249.
- [52] L. Zhu, S. Shi, K. Lu, J. Lu. A statistical model for predicting the mechanical properties of nanostructured metals with bimodal grain size distribution, *Acta Materialia* 60 (2012) 5762-5772.
- [53] Y. Liu, J. Zhou, X. Ling. Impact of grain size distribution on the multiscale mechanical behavior of nanocrystalline materials, *Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing* 527 (2010) 1719-1729.
- [54] B. Raeisinia, C.W. Sinclair, W.J. Poole, C.N. Tome. On the impact of grain size distribution on the plastic behaviour of polycrystalline metals, *Modelling and Simulation in Materials Science and Engineering* 16 (2008).
- [55] S. Patra, S.M. Hasan, N. Narasaiah, D. Chakrabarti. Effect of bimodal distribution in ferrite grain sizes on the tensile properties of low-carbon steels, *Materials Science and Engineering: A* 538 (2012) 145-155.
- [56] A. Berger, M. Herwegh, J.-O. Schwarz, B. Putlitz. Quantitative analysis of crystal/grain sizes and their distributions in 2D and 3D, *Journal of Structural Geology* 33 (2011) 1751-1763.
- [57] R.T. DeHoff, F.N. Rhines. *Quantitative Microscopy*, McGraw-Hill, New York, 1968.
- [58] A. Zankel, J. Wagner, P. Poelt. Serial sectioning methods for 3D investigations in materials science, *Micron* 62 (2014) 66-78.
- [59] A.D. Rollett, S.B. Lee, R. Campman, G.S. Rohrer. Three-dimensional characterization of microstructure by electron back-scatter diffraction. *Annual Review of Materials Research*, vol. 37. 2007. pp. 627-658.

Tables

Table 1. The chemical compositions (wt%) of the HT-UPS stainless steel examined in this work.

element	Fe	Cr	Ni	Mo	Mn	Si	P	Ti	Nb	V	C	N
wt. %	bal	13.91	16.00	2.59	2.00	0.11	0.04	0.28	0.11	0.53	0.06	0.02

Table 2. Specimen condition and geometry and dimensions.

Sample ID	Condition	Dimensions & Geometry
AR	As-received.	1.7 mm×2 mm×0.15 mm (coupon)
irr	Neutron irradiated to 3 dpa at 500°C	3 mm diameter × 0.16 mm thickness (disk)
irr+ann	Neutron irradiated to 3 dpa at 500°C followed by annealing at 600°C for 1 h	3 mm diameter × 0.2 mm thickness (disk)

Figures

Figure 1. Schematic showing the FF-HEDM setup for radioactive materials, with actual diffraction spots shown. Top right is a closer view of the sample holder, showing the double-layered Kapton encapsulation, the 3mm-diameter disk shaped sample (half buried in holder), and the four consecutive layers probed by the X-ray beam (in blue boxes).

Figure 2. Center-of-Mass (COM) maps for (a) the as-received (AR) sample, (b) the neutron-irradiated (irr) sample and (c) the irradiated and then annealed (irr+ann) sample. The size of each spot reflects the size of the grain. The color denotes the completeness, from 0.7 to 1.

Figure 3. (a) Grain size distribution for the three samples. The inset shows the log-normal fittings of the profiles. (b) COM reconstructions with the color denoting the grain radius, from 10 μm to 100 μm .

Figure 4. (a) Optical micrograph showing the 2D grain structure of the AR sample. (b) Comparison of the grain size distribution measured by optical microscopy and FF-HEDM, showing a good agreement.

(a)

Figure 5. (a) Diffraction spots from $\{111\}$ and $\{200\}$ reflections of a single grain in each sample. (b) The FWHM of the diffraction spots from Fe $\{111\}$ and $\{200\}$, along radial direction (ρ). (c) The FWHM of the diffraction spots from Fe $\{111\}$ and $\{200\}$, along azimuthal direction (η).

Figure 6. Orientation distribution function computed from the crystallographic orientations of the grains for the three samples. Color denotes multiples of uniform distribution.

Figure 7. (a) Lattice constant vs. grain radius for the three samples. The color denotes the completeness, from 0.7 to 1. (b) Lattice constant vs. grain radius for the three samples. The color denotes the completeness, from 0.8 to 1. (c) Lattice strain distribution of all the grains in each sample, taking the average lattice constant, denoted in each subfigure in (a), as the reference lattice constant.