Diffraction-Based Multi-Scale Residual Strain Measurements

Namit Pai[1], Sanjay Manda[1], Bhargav Sudhalkar[1], Bethany Syphus[2], David Fullwood[2], René de Kloe[3], Stuart Wright[4], Anirban Patra[1], Indradev Samajdar[1,*]

[1]Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai, 400076, India
[2]Mechanical Engineering, Brigham Young University, Provo, UT 84602, USA
[3]Gatan-Edax, Ringbaan Noord 103, 5046 AA Tilburg, The Netherlands
[4]Gatan-Edax, 5794 W. Las Positas Blvd., Pleasanton, CA 94588, USA

Abstract:

Modern analytical tools, from micro-focus X-ray diffraction (XRD) to electron microscopy based microtexture measurements, offer exciting possibilities of diffraction-based multi-scale residual strain measurements. The different techniques differ in scale and resolution, but may also yield significantly different strain values. This study, for example, clearly established that high resolution electron backscattered diffraction (HR-EBSD) and high resolution transmission Kikuchi diffraction (HR-TKD) (sensitive to changes in interplanar angle $\left(\frac{\Delta\theta}{\theta}\right)$), provides quantitatively higher residual strains than micro-Laue XRD and transmission electron microscope (TEM) based precession electron diffraction (PED) (sensitive to changes in interplanar spacing $\left(\frac{\Delta d}{d}\right)$). Even after correcting key known factors affecting the resolution of HR-EBSD strain measurements, a scaling factor of ~1.57 (between HR-EBSD and micro-Laue) emerged. We have then conducted 'virtual' experiments by systematically deforming an ideal lattice by either changing an interplanar angle (α) or a lattice parameter (a). The patterns were kinematically and dynamically simulated, and corresponding strains were measured by HR-EBSD. These strains showed consistently higher values for lattice(s) distorted by α, than those altered by a. The differences in strain measurements were further emphasized by mapping identical location with HR-TKD and TEM-PED. These measurements exhibited different spatial resolution, but when scaled (with ~1.57) provided similar lattice distortions numerically.

***Keywords*:** Residual Strain, X-ray Diffraction (XRD), High Resolution EBSD (HR-EBSD), Transmission Electron Microscopy, Diffraction Pattern Simulation.

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

1. Introduction:

Residual strains, and corresponding elastic stresses, develop when a specimen is subjected to non-uniform elastic-plastic strain gradients (Verlinden et al., 2007; Lodh et al., 2018; Thool et al., 2020). The latter may be imposed from a variety of processes (Lodh et al., 2018; Thool et al., 2020; Heindlhofer, 1948; Osgood, 1954; Noyan & Cohen, 2013; Cullity, 1956; Almen & Black, 1963; Lodh et al., 2017). In a crystalline material, the residual strains represent lattice distortions or changes in the unit cells (Verlinden et al., 2007). Corresponding non-equilibrium structures are retained by defects, such as dislocations and dislocation boundaries. Based on the scale, the strains and stresses differ (Lodh et al., 2018; Thool et al., 2020; Lodh et al., 2017). They are referred as macro (type I) to meso (type II) and micro (type III) (Verlinden et al., 2007). To a designer, the macroscopic residual stresses are important (Kumar et al., 2016; Wang & Gong, 2002), but micro and mesoscopic stresses and strains are also emerging as an important aspect in any microstructural investigation (Lodh et al., 2019, 2018, 2017; Thool et al., 2020; Revelly et al., 2015). Information on local lattice distortions is a critical but often invisible component of the overall microstructure (Verlinden et al., 2007). This is the subject of multi-scale residual strain measurements, the focus of the present study.

Traditional residual strain measurements include techniques ranging from dimensional changes (mechanical) to property alterations (Verlinden et al., 2007; Heindlhofer, 1948; Osgood, 1954; Withers & Bhadeshia, 2001). However, only diffraction based measurements can bring out the full strain matrix, which may then be converted with appropriate continuum elasticity model(s) to residual stress values (Van Houtte & De Buyser, 1993). Further, modern analytical tools, from micro-focus X-ray diffraction (XRD) to electron microscopy based microtexture determination, hold tremendous potential for multi-scale diffraction based residual strain measurements (Lodh et al., 2019; Prakash et al., 2023; Wilkinson et al., 2006; Ghamarian et al., 2014; Yu et al., 2019). Among these techniques, micro-Laue based single-crystal residual stress measurement (Lodh et al., 2019, 2018, 2017; Thool et al., 2020) is non-trivial to implement in a laboratory XRD setup. However, this can be effectively used to measure grain-by-grain residual strain(s) with progressive plastic deformation or annealing (Lodh et al., 2017; Thool et al., 2020). The limitation of this technique is in the X-ray micro-focus, which is $\sim 50\ \mu m$ based on the spot size achievable in our laboratory setup. The XRD approach estimates relative strain from changes in interplanar spacing $\left(\frac{\Delta d}{d}\right)$, which is also a traditional estimate of lattice distortion (Lodh et al., 2017; He, 2003).

Similar to XRD, neutron diffraction is another $\frac{\Delta d}{d}$ based technique that is routinely employed for measurement of micro/macro residual strains (Krawitz & Holden, 1990), diffraction elastic constants (Baczmanski et al., 1993) and differential scattering (Soper, 2011) in materials. The key advantage lies in the greater penetration depth of neutrons, ranging in the order of millimetres as compared to micron-depths achieved using X-rays. Alternatively, μ-Raman spectroscopy, with a reported strain precision of 10^{-4}, primarily relies on the Raman peak shifts to quantify the local stress variations in microelectromechanical (MEMS) devices and is currently an evolving field of research (Srinivasan et al., 2018; Choi & Griffin, 2016; Wolf, 1999; Senez et al., 2003). However, the present study mainly focuses on the use of laboratory scale micro-Laue single crystal XRD technique for measurement of residual strains in the material.

Other notable techniques include the related methods of high resolution electron backscattered diffraction (HR-EBSD) (Wilkinson et al., 2006; Fullwood et al., 2015) and high resolution transmission Kikuchi diffraction (HR-TKD) (Yu et al., 2019). Both use the same algorithm of the so-called cross-correlation, and are sensitive to changes in interplanar angle $\left(\frac{\Delta\theta}{\theta}\right)$. Their spatial resolution is determined by the electron-atom interaction volume (Goodhew & Humphreys, 2000); and is arguably $\sim 20\ nm$ in EBSD (Zaefferer, 2007; Ruggles et al., 2016; Schwarzer et al., 2009) and even smaller in HR-TKD performed on thin foils (Ghamarian et al., 2014; Humphreys et al., 1999; Trimby, 2012; Sneddon et al., 2016).

The technique of transmission electron microscope (TEM) based precession electron diffraction (PED) (Rauch et al., 2010) has originally been proposed ~2009. This is based on measurements of interplanar spacing, $\left(\frac{\Delta d}{d}\right)$, from the diffraction spots (Ghamarian, 2017; Ghamarian et al., 2014). The method has the potential of providing spatial resolution of below $2\ nm$ but is restricted by the sensitivity of the respective diffraction vectors (Ghamarian et al., 2014). The key advantage of TEM lies is in its adaptability; the same instrument can be utilized to measure strains using various strain mapping techniques. Béché et al., (2013) provides an in-depth comparison of five different TEM based techniques for strain measurements; each of these result in a spatial resolution below $5\ nm$. These include the convergent and nano beam electron diffraction (CBED and NBED), high resolution TEM (HRTEM) and high-resolution scanning TEM (HRSTEM) and the dark field electron holography (DFEH). Each technique provided a different strain precision, ranging from 2×10^{-4} (for CBED) to 10^{-3} (for HRSTEM) (Armigliato et al., 2006; Hüe et al., 2008; Béché et al., 2013) and a different spatial

resolution. Further, their detailed study also involved taking into account the sample specifications (thickness and geometry), data treatment time and the associated computational resources (ranging from 1 day (for CBED) to <1 min (for HRTEM and DFEH)) combined with the selection of reference frame.

In brief, they inferred that the application of TEM based strain mapping techniques ultimately relies on analysing which technique will be the most suited for the problem in-hand (Béché et al., 2013). Recently, the nano-scale strain distributions on strained Ge microdisks have been studied using the PED technique in STEM; the corresponding strain contours have shown a reasonable agreement with three-dimensional finite element models as well (Bashir et al., 2019). Further, utilizing the scanning NBED with direct electron detectors (rather than the slower conventional CCD imaging) in combination with efficient (diffraction) pattern recognition algorithms have enabled mapping of strains with relatively large field of view ($\sim 1\ \mu m$) even in the defected structures (Ozdol et al., 2015).

In summary, a range of micro to nanoscale diffraction techniques presently exist for strain mapping of metallic specimens; with each technique subtending its own merits and demerits, based on the research objectives, length scale and material under consideration. The present study explores a few of them, namely the micro-Laue XRD, HR-EBSD, HR-TKD and TEM-PED. Each of these multi-scale diffraction-based techniques differ in scale and in resolution. More importantly, they are sensitive to two different aspects of lattice distortion, $\left(\frac{\Delta\theta}{\theta}\right)$ (for HR-EBSD and HR-TKD) versus $\left(\frac{\Delta d}{d}\right)$ (for micro-Laue and TEM-PED). Two questions naturally emerge: (i) are these strain measurements numerically similar and (ii) if not, is there a relationship between them. Addressing these and a general comparison of the techniques were the motivations behind this study.

2. Materials and Methods

This study used fully recrystallized interstitial free steel of $\sim 130\ \mu m$ micron average grain size. For details on the prior processing and chemistry, the reader may refer to Manda et al. (Manda et al., 2023). In particular, sub-size micro-tensile specimen(s) (for dimensions refer to supplementary figure S1) were fabricated by electro discharge machining. The gauge regions were prepared by a combination of mechanical polishing, followed by electropolishing. The latter involved an electrolyte of 80:20 methanol and perchloric acid, and 18 volts dc at 253K. As in supplementary figure S1, EBSD scans were performed with interrupted but progressive

tensile deformations. These were conducted with a Deben™ 1 kN stage operated at 10^{-3} s^{-1}, and a maximum strain of 0.15 was imposed. It is to be noted that this stage was compatible with both our XRD and scanning electron microscope (SEM). This enabled us to conduct multi-scale diffraction based residual strain measurements on the same progressively deformed tensile specimens. In particular, we have used XRD based micro-Laue diffraction and SEM based HR-EBSD. Further, we have also performed post-deformation characterizations with HR-TKD in the SEM and TEM based PED.

Micro-Laue diffraction was conducted in a Bruker™ D8 Discover XRD system. The critical components were a X-ray micro-source with Montel™ multi-layer focusing mirrors, laser plus video tracking, and Vantec™ area detector. The reader may refer to Lodh et al. (2022, 2019, 2018, 2017), and section 3.1., for a detailed description of this setup and associated algorithm. A FEI™ Quanta 3D-FEG (Field Emission Gun) SEM equipped with an EDAX™ Velocity-plus EBSD system was used for HR-EBSD (0.5 μm step size) as well as HR-TKD (10 nm step size) measurements. Patterns were acquired at an accelerating voltage of 20 kV, current of 16 nA and a working distance of 12.5 mm, with a sample tilt of 70° (for HR-EBSD). 16-bit Kikuchi patterns with a 2×2 binning size (each image comprising of 230×230 pixels), were acquired and stored for all points within the region of interest for further offline post-processing. All measurements involved identical beam plus detector conditions. Additionally, all experimentally measured (HR-EBSD) residual elastic strains reported in the present work are averaged out over a $70\ \mu m \times 70\ \mu m$ region near the geometric centre of the grains. Such a scheme was followed to avoid erroneous strain measurements, primarily due to large orientation gradients occurring near the grain boundaries (see figure A.1a).

TEM thin foils, used in HR-TKD as well as PED, were prepared from 3 mm discs using a Struers™ Tenupol-5 twin-jet electropolisher. A sample tilt of $-20°$ was used for the (off-axis) HR-TKD measurements. The TEM-PED residual strain measurements were conducted on a NanoMegas™ system within a Thermofisher™ Themis 300 TEM, at an operating voltage of 300 kV. Further details on these measurement techniques are given in the next section.

3. Diffraction Based Multi-Scale Residual Strain Measurements

The different residual strain measurement techniques are described in subsequent sub-sections: XRD based micro-Laue diffraction (section 3.1), SEM based HR-EBSD and HR-TKD (section 3.2), and TEM based PED (section 3.3). These techniques differed in algorithm, and also in spatial plus angular resolutions. At this stage, it is important to note the rationales

in referring micro-Laue XRD and TEM-PED as techniques sensitive to $\frac{\Delta d}{d}$ and HR-EBSD and HR-TKD as techniques sensitive to $\frac{\Delta \theta}{\theta}$. The strain measurement using TEM-PED technique primarily relies on the shift of the diffraction spots, which alters the resulting distortion matrix $\boldsymbol{D}$. The in-plane residual strain components are then estimated using equation 5. The shift in diffraction spots in TEM-PED, irrespective of (direction of) $\vec{g}$, is an outcome of the changes in the interplanar spacing of the crystal $\left(\frac{\Delta d}{d}\right)$. In a similar manner, any change in $d_{\phi\psi}$, irrespective of the goniometer rotations, is an outcome of the changes in interplanar spacing $\left(\frac{\Delta d}{d}\right)$. However, HR-EBSD as well as HR-TKD techniques rely on the shift in $\vec{q}$ (see equations 2 and 3), which is primarily sensitive to changes in interplanar angles $\left(\frac{\Delta \theta}{\theta}\right)$.

The micro-Laue setup in our study uses an approximate circular X-ray spot of $\sim 50\ \mu m$ with an estimated depth of penetration of less than $5\ \mu m$. The technique has an angular reproducibility of ~0.01º (He, 2003; Krost & Bläsing, 2009; Slowik & Zięba, 2005). HR-EBSD and HR-TKD offer similar angular resolution ($0.006°$) and a higher spatial resolution restricted by electron-atom interaction volume ($\sim 20\ nm$) (Wilkinson et al., 2006; Humphreys, 2004; Yu et al., 2021; Jiang et al., 2013). It is important to note that the spatial resolution in EBSD is non isotropic (Farooq et al., 2008). The tilting of the sample by $70°$ in EBSD results in the intersection shape of the electron beam (at the entry point of the specimen) to take on an elliptical shape, with an aspect ratio of $1:3$ (Schwarzer et al., 2009; Sneddon et al., 2016; Fullwood et al., 2022). The beam subsequently enters the sample to a certain depth, where a 'virtual point source' is generated by inelastic scattering, which then produces the outwards directed elastically scattered electrons, that form the EBSD patterns (Winkelmann, 2010). The non-isotropic nature of the EBSD resolution is hence an outcome of the intersection of this internal cone of electrons with the tilted sample surface. In summary, this leads to the reduction of spatial resolution along the vertical direction by ~3 times (Schwarzer et al., 2009; Sneddon et al., 2016). Further, the interaction volumes and diffraction paths for HR-EBSD on a bulk sample and HR-TKD on thin-foil are quite different. For a bulk sample, the information depth lies in the range $10 - 40\ nm$ at 20 kV. The depth values can decrease further for denser materials (Winkelmann, 2010; Dingley, 2004). In HR-TKD the lateral resolution is defined by the effective beam diameter at the exit point (bottom surface) of the TEM foil. This would typically be of the order of $10\ nm$ and is defined by the actual beam spread in the sample (by the foil thickness, material density, and keV). TEM-PED offers the very best in spatial

resolution (below $\sim 2\ nm$) but less so in angular resolution ($< 0.4°$). This technique is restricted to very thin TEM foils (Ghamarian, 2017; Ghamarian et al., 2014; Viladot et al., 2013).

Figure 1.png

Figure 1: Diffraction based multi-scale residual strain measurements. These include (a) micro-Laue single-crystal X-ray diffraction (XRD), (b) high resolution electron backscattered diffraction (HR-EBSD) in scanning electron microscope (SEM) and (c) precession electron diffraction (PED) in transmission electron microscope (TEM). It is to be noted that (a) and (c) are sensitive, respectively, to changes in interplanar spacing (Δd), and (b) interplanar angle ($\Delta\theta$).

3.1. *XRD based micro-Laue diffraction:*

Unlike the traditional XRD based residual strain measurements (Verlinden et al., 2007; Van Houtte & De Buyser, 1993), which are relatively routine for polycrystalline material, single crystal micro-Laue diffraction remains extremely specialized. Such measurements have been restricted to high energy synchrotron radiation (Margulies et al., 2002). More recently, however, Lodh et al. (2022, 2019, 2018, 2017) have developed a similar, though not identical, approach utilizing laboratory micro-focus XRD. We have adopted the same in this study. As indicated in figure 1a, and further described by Thool et al. (2020), the gauge of the micro-tensile specimen(s) has been subjected to prior EBSD scan(s) at different stages of tensile deformation (see supplementary figure S1). We have tracked the corresponding grains delineated by appropriate fiducial markers. Laser plus video tracking has been used to facilitate the placing of $\sim 50\ \mu m$ X-ray beam in the center of individual grains. Appropriate rotations (θ, ϕ, ψ) in goniometer angles, derived from EBSD-estimated grain orientations, were imposed. As stated earlier, the average orientations from a $70\ \mu m \times 70\ \mu m$ region near the geometric centre of the grains were used to determine the rotations (θ, ϕ, ψ) in goniometer angles. This was done to minimize the errors associated with the determination of goniometer angles due to the spread in EBSD estimated average grain orientations, especially in the deformed specimens. In addition, the laser plus video tracking facility along with a $\sim 0.01°$ of angular reproducibility ensured that no significant errors were introduced in the determination of (θ, ϕ, ψ) angles for micro-Laue residual strain measurements. Further, we have obtained six different 'brightest' Laue spots (Lodh et al., 2017), and the centroids were used to estimate $d_{\phi\psi}$ for each grain. Following this, $\varepsilon_{\phi\psi}$ was calculated as $\varepsilon_{\phi\psi} = \frac{d_{\phi\psi} - d_0}{d_0}$, where d_0 is the

estimated unstrained interplanar spacing obtained from the annealed powder specimen (Noyan & Cohen, 2013; Kumar et al., 2016). In this study, we have obtained and used $d_0^{011} = 2.0266$ Å, $d_0^{200} = 1.4309$ Å and $d_0^{211} = 1.1697$ Å, respectively. The resulting $\varepsilon_{\phi\psi}$ was then transformed to the grain average strain ε_{kl} in the specimen co-ordinate system as given in (Lodh et al., 2022, 2019, 2018, 2017):

$$\varepsilon_{\phi\psi} = \varepsilon_{11}\cos^2\phi\sin^2\psi + \varepsilon_{12}\,sin\,2\phi\sin^2\psi + \varepsilon_{22}\sin^2\psi\sin^2\phi + \varepsilon_{33}\cos^2\psi + \varepsilon_{13}\,cos\,\phi\,sin\,2\psi + \varepsilon_{23}\,sin\,\phi\,sin\,2\psi \quad \ldots(1)$$

The solution of equation 1 gives the residual strain tensor for an individual grain. Since there are six unknown strain components, at least six independent reflections are needed to solve the linear equations. All these make the technique fairly sophisticated. The schematic shown in figure 1a briefly explains the above methodology. We have conducted these measurements, of intergranular residual elastic strains, on 50 different (and randomly selected) grains subjected to progressive tensile deformation.

3.2.*SEM based HR-EBSD and HR-TKD:*

The automated EBSD or TKD measurements involve the Hough transformation to extract the approximate lattice plane traces found in the Kikuchi diffraction pattern (Wright & Adams, 1992; Adams et al., 1993). These measurements are usually based on the angle(s) between the planes or Kikuchi bands, which are then analyzed, using a 'look-up' table, to obtain corresponding crystallographic orientations. The primary difference between the EBSD and TKD lie in the mode of pattern generation. Though both techniques rely on an incoherent scattering event to act as a basis (virtual point source) for further elastic scattering events, the exit beam diameter of the scattered electrons is much lower in TKD, compared to its EBSD counterparts (van Bremen et al., 2016). In addition, the negative tilt angles used in TKD leads to incident electrons being forward scattered, in-contrast to the EBSD technique, where (incident) electrons are backscattered from the sample surface to the detector.

The use of an electron transparent specimen, in conjunction with the negative tilt angle results in the diffraction patterns originating from a small region close to lower surface of the specimen (Trimby, 2012; Sneddon et al., 2016). This leads to a significant improvement in the spatial resolution in comparison to the EBSD technique, which subtends a larger interaction volume (Trimby, 2012). More recently, the use of on-axis TKD technique, which utilizes a zero-tilt condition, was reported to provide significant improvements in the pattern intensities and data acquisition rates (Niessen et al., 2018). Additionally, unlike the conventional TKD

measurements used in the present study, the on-axis TKD technique does not result in a gnomonic distortion of the diffraction patterns (Yuan et al., 2017; Niessen et al., 2018).

Kikuchi patterns from both techniques, EBSD as well as TKD, can be further analyzed by comparing appropriate "regions of interest" (ROI) within a reference pattern. This technique is often referred to as Cross-Correlation or HR-EBSD (Wilkinson et al., 2006; Fullwood et al., 2015; Wright et al., 2011). HR-EBSD, for example, can be used to determine relative orientations of the reference and current sample point to very high angular resolution, and to estimate relatively 'minor' lattice strains (Wilkinson et al., 2006; Yu et al., 2021).

We have processed high resolution Kikuchi patterns, both EBSD and TKD, using background division and dynamic background subtraction. Kikuchi patterns from the grain center, having maximum image quality (Small et al., 2020), were used as the reference patterns. A comparison between reference and the 'strained' pattern(s), see figure 1b, provided pattern shift ($\vec{q}$) and change(s) in interplanar angles $\left(\frac{\Delta\theta}{\theta}\right)$. As described latter in the results section, this technique needs selection of appropriate ROIs (we have used 48 as well as 64 in our study) and information on accurate pattern center (Fullwood et al., 2015; Basinger et al., 2011). Note that the selection of the reference patterns, ideally from a strain-free crystal, is always relative (Small et al., 2020; Britton & Wilkinson, 2012). The measured $\vec{q}$ vectors are related to both lattice distortion and rotation as (Wilkinson et al., 2006, 2009a; Britton & Wilkinson, 2011),

$$r_2 r_3\left[\frac{\partial u_2}{\partial x_2}-\frac{\partial u_3}{\partial x_3}\right]+r_1 r_3\frac{\partial u_2}{\partial x_1}+r_3^2\frac{\partial u_2}{\partial x_3}-r_1 r_2\frac{\partial u_3}{\partial x_1}-r_2^2\frac{\partial u_3}{\partial x_2}=r_3 q_2-r_2 q_3 \qquad \ldots(2)$$

$$r_1 r_3\left[\frac{\partial u_1}{\partial x_1}-\frac{\partial u_3}{\partial x_3}\right]+r_2 r_3\frac{\partial u_1}{\partial x_2}+r_3^2\frac{\partial u_1}{\partial x_3}-r_1^2\frac{\partial u_3}{\partial x_1}-r_2 r_1\frac{\partial u_3}{\partial x_2}=r_3 q_1-r_1 q_3 \qquad \ldots(3)$$

In brief, when a crystal lattice is subjected to an imposed deformation, the zone axes direction vector $\vec{r}$ shifts by $\vec{q}$ resulting in a lattice distortion of $\frac{\partial u_i}{\partial x_j}$. The elastic strains can then be derived as $\boldsymbol{E^e}=\frac{1}{2}(\boldsymbol{F^{e^T}}\cdot\boldsymbol{F^e}-\boldsymbol{I})$, where $\boldsymbol{I}$ denotes the identity tensor and $\boldsymbol{F^e}$ represents the elastic deformation gradient, given by $\boldsymbol{F^e}=\boldsymbol{I}+\frac{\partial u_i}{\partial x_j}$. Here, i and j denote the basis directions, respectively. Further, to separate normal strains, a boundary condition has to be utilized that forces the stress σ_{33}, normal to the sample surface, to zero. This can be expressed as (Wilkinson et al., 2006, 2009a):

$$\sigma_{33}=0=C_{33}\varepsilon_{33}+C_{32}\varepsilon_{22}+C_{32}\varepsilon_{11} \qquad \ldots(4)$$

Where $C_{33}=C_{11}=231.4\,GPa$ and $C_{32}=C_{12}=134.\,GPa$ are anisotropic elastic constants for the ferrite (Fe) phase (Fullwood, 2020). This defines the so-called traction free boundary

condition (Hardin et al., 2015). An alternative boundary condition, more recently proposed (Ruggles et al., 2020), specifies that the trace of the lattice distortion tensor should be zero. As shown in the appendix figure B.1, both provided similar results. We have hence performed off-line cross-correlations, under traction free boundary condition, to estimate residual elastic strains. These were performed on experimental, HR-EBSD and HR-TKD, as well as simulated (Callahan & De Graef, 2013; Winkelmann et al., 2007; Zaefferer, 2007) Kikuchi patterns. Our cross-correlations used an open source (OpenXY^{TM}) software (Fullwood, 2020).

3.3. TEM based PED:

TEM offers excellent spatial resolution. TEM spot diffraction, however, has relatively poor angular resolution. This is decided by the so-called $\vec{s}$ vector, or deviation from the exact Bragg, and the electron atom-interaction volume (Ghamarian et al., 2014; Ghamarian, 2017). The latter determines the kinematic or dynamical interaction of the transmitted and diffracted beam(s). The recent incorporation of the PED technique (see supplementary figure S2) in thin (below 1/3 of extinction distance) TEM foils can improve the angular resolution of TEM spot diffraction patterns (Vincent & Midgley, 1994). In brief, introduction of an appropriate 'precession' angle provides ability to resolve very small angular deviations ($< 0.4°$) in TEM-PED (Ghamarian, 2017; Ghamarian et al., 2014; Viladot et al., 2013). Naturally, the technique can be used in both orientation and residual strain measurements (Ghamarian et al., 2014; Ghamarian, 2017; Viladot et al., 2013; Rauch & Veron, 2005; Rauch et al., 2010; Portillo et al., 2010). Of course, the latter would still require a reference pattern which is then compared to the 'strained' patterns, see figure 1c.

As shown in figure 1c two non-collinear diffraction spot patterns ($\vec{g}_1$, $\vec{g}_2$ for the reference and the strained crystals, respectively) are acquired. The respective diffraction matrices can be stated as $\boldsymbol{G_0} = [g_{x_1}\ g_{x_2}; g_{y_1}\ g_{y_2}]$ and $\boldsymbol{G} = [g'_{x_1}\ g'_{x_2}; g'_{y_1}\ g'_{y_2}]$ (Zhao et al., 2023). The lattice distortion is then given as the distortion matrix $\boldsymbol{D} = (\boldsymbol{G_0}\ \boldsymbol{G^{-1}})^T$, where T indicates a transpose operation (Zhao et al., 2023). The in-plane residual strain components can be extracted from the distortion matrix using the infinitesimal strain theory, see Thomas et al.(Pekin et al., 2017), as:

$$\begin{bmatrix} \varepsilon_{xx} & \frac{1}{2}(\varepsilon_{xy} - \theta) \\ \frac{1}{2}(\varepsilon_{yx} + \theta) & \varepsilon_{yy} \end{bmatrix} = \boldsymbol{D} - \boldsymbol{I} \qquad \ldots (5)$$

where, ε_{xx}, and ε_{yy} are the strains in X and Y direction, respectively. ε_{xy} represents the in-plane shear component and θ here denotes the lattice rotation. It is important to note that the above expression estimates lattice strain in the reciprocal space, and the signs of ε_{xx}, ε_{yy} and ε_{xy} must be flipped to produce strain measurements in the specimen space. The present study uses the TopSpin™ module of NanoMegas™ PED software for data acquisition and subsequent analysis. Alternative algorithms, based on the polar decomposition of transformation matrix between the strained and unstrained diffraction vectors have also been proposed in literature estimate residual elastic strains with very high precisions ($\pm 0.1\%$) (Ozdol et al., 2015). Further, the reader is referred to Béché et al., (2013) for a detailed analysis on various strain mapping techniques, its associated merits and demerits.

4. Results from Experiments and Pattern Simulations

Figure 2a shows a direct comparison in the development of intergranular von-Mises residual elastic strains, as estimated by the micro-Laue or $\left(\frac{\Delta d}{d}\right)$ and the HR-EBSD or $\left(\frac{\Delta \theta}{\theta}\right)$. This is shown for three randomly selected grains - G1, G2 and G3. It is to be noted that we have taken similar grain surface areas for the two measurements, making the data in figure 2a spatially comparable. Past HR-EBSD studies have also reported somewhat higher residual elastic strain magnitudes, in comparison to the theoretical yield or plasticity limit of 0.2% imposed strain (Small et al., 2020; Britton & Wilkinson, 2011; Zhang et al., 2016, 2014; Zhao & Li, 2021; Mehtani et al., 2020); for example, Small et al. (2020) reported equivalent residual elastic strains of magnitude 0.007 ± 0.008 in additively manufactured Inconel 625 specimen. Residual strains of magnitude 0.007 ± 0.008 indicate an extremely large spread in the reported data. The use of drastic cooling rates in additively manufactured specimens (Inconel 625) often results in large intragranular orientation gradients ($> 10°$), leading to a significant variation in the average residual strain data. Zhang et al. (2014) have noted strains greater than 0.002 (specifically, in grains near non-metallic inclusions) in their nickel-based superalloy subjected to thermal loading, Zhao and Li (2021) reported strain components with peak magnitude of up to 0.01 in stainless steel specimens subjected to three-point bending tests. Similarly, Mehtani et al.(2020) have reported von-Mises residual elastic strains of magnitude $0.01 - 0.03$ in the pseudo-epitaxially grown magnetite (Fe_3O_4) grains, during their studies on oxidation kinetics of interstitial free steel.

Figure 2.png

Figure 2: Comparison of residual elastic strains measured using micro-Laue XRD ($\Delta d/d$) and HR-EBSD ($\Delta\theta/\theta$). These measurements were performed, with progressive tensile deformation, on (a) three identical grains, and (b) 50 random grains. The corresponding best fit slopes are shown in (c). (b,c) show higher residual elastic strains in HR-EBSD and (d) shows the same as statistical distributions.

There have been significant efforts to cross-validate HR-EBSD results with an alternative measurement approach. More specifically, these involve comparing the dislocation density measurements from HR-EBSD with their XRD counterparts (Kalácska et al., 2017; Gallet et al., 2023; Kalácska et al., 2020) and the residual elastic strain component magnitudes between HR-EBSD and synchrotron X-ray diffraction measurements (Deal et al., 2021). Accuracy of HR-EBSD strain measurements have also been evaluated corresponding XRD measurements on a patterned $Si_{1-x}Ge_x$ thin film structure, and a reasonable convergence has been reported (Vaudin et al., 2015; Fullwood et al., 2015). However, such prior studies have often been limited to semi-conductor/thin film structures, which exhibit mechanical properties significantly different from the material used in the present study. Further, in addition to material/mechanical properties, the magnitude of (imposed) deformation in their study is significantly different in comparison to the present work. As seen in figure 2a, b, a similar numerical convergence does hold valid for lower magnitudes of imposed strain in the present study as well. In addition, a methodical grain level comparison, of evolution of HR-EBSD strains with laboratory scale XRD measurements, is lacking in literature. This has been achieved in figure 2a, with direct comparison between residual elastic strain measurements by HR-EBSD and micro-Laue. At large tensile deformations, HR-EBSD clearly showed higher residual elastic strain values. The difference between the strain estimates did depend on the magnitude of the strain as well as the elastic stiffness. The authors would like to state here that the term ‘higher residual elastic strains’, used henceforth, refer to residual elastic strains significantly greater than the theoretical yield limit of 0.2%.

Further, the data in figure 2a is based on only three randomly selected grains. From the large number of grains characterized in this study, 50 were randomly selected for a statistical comparison (see figure 2b,c). The residual elastic strain(s) increased as expected with imposed tensile deformation (Thool et al., 2020). There were, however, significant scatter between different grains (see figure 2b and error bars in figure 2c). Statistically, but definitively, the relative increase in residual elastic strain, with tensile deformation, was more in HR-EBSD

than in the micro-Laue XRD. This last point is further represented in figure 2d as strain distributions at the highest tensile deformation step (0.15 imposed tensile strain). In particular, HR-EBSD 'average' residual elastic strain was ~2.06 times of that of micro-Laue. Further, the HR-EBSD strain estimates had wider distribution or scatter. This last point, to be deliberated latter in the manuscript, actually indicated higher angular resolution for HR-EBSD and/or HR-TKD.

It is important to note here that the micro-Laue residual elastic strain measurements, which are sensitive to $\frac{\Delta d}{d}$, do show large magnitudes for certain grains/orientations. This can be attributed to multiple factors, more specifically, the improper strain free lattice spacing substitution in equation 1, as well as the geometrical errors associated with the determination of the Laue spot centroids (see section 3.1). In addition, it is important to note that minor variation in lattice parameters can result in large difference in the measured strains (see figure 4a). We have ensured that all such possible errors were minimized during the present study. For brevity, this topic has not been deliberated further in the manuscript. The measurement uncertainty for micro-Laue based von-Mises residual strain estimates, calculated by repeating the measurements thrice for five random deformed grains, for the same position and at the same state of imposed strain was observed to be 8.4×10^{-4} (at 0.15 imposed tensile strain). Interestingly, for the HR-EBSD measurements, the measurement uncertainty was nearly negligible.

Figure 3.png

Figure 3: Minimizing the errors in experimental HR-EBSD (sensitive to $\frac{\Delta\theta}{\theta}$) residual strain measurements and comparing the resultant outcomes with their micro-Laue (von Mises) strains. (a) Frequently reported errors in HR-EBSD and corresponding corrections. (b) These corrections were applied to our statistical data on HR-EBSD versus micro-Laue residual elastic strains. However, even the 'corrected' data did not bring an equivalence between the two approaches. (c) Comparison of the ratios of 'a' type (effect on Δd) and 'α' type (effect on $\Delta\theta$) residual elastic strains with experimental HR-EBSD and micro-Laue (von-Mises) strains. The red dotted best fit line indicates a direct linear relationship between the two entities. (d) Sum of squared error (SSE) distribution, shown as a lognormal fit (red dotted line), for all grains analysed in figure 3c.

There have been attempts to improve HR-EBSD based residual strain measurements (Fullwood et al., 2015; Wright et al., 2011; Basinger et al., 2011; Jiang et al., 2013). Potential errors might arise due to (A) insufficient ROIs, (B) inappropriate selection of the reference pattern and (C) choice of pattern center and (D) remapping, which accounts for orientation gradients in the material (Small et al., 2020; Britton & Wilkinson, 2012; Maurice et al., 2012). In particular, we have considered factors A-D, see figure 3a. Increase in ROIs, from 48 to 64, for example, reduced HR-EBSD estimated single crystal residual elastic strain. However, this drop was marginal. Combining more ROIs with manual selection of reference pattern and correcting for pattern center shift, based on an algorithm proposed by Fullwood et al. (Fullwood et al., 2015; Basinger et al., 2011), reduced the HR-EBSD residual elastic strain more significantly. The remapping technique (Small et al., 2020; Britton & Wilkinson, 2012; Maurice et al., 2012), which uses the finite rotation component of the elastic deformation gradient tensor ($\boldsymbol{F^e}$) to derive the rotated reference pattern, and then estimate the residual shifts ($\vec{q}$) between the rotated reference pattern and Kikuchi pattern from the current pixel, further improves the residual elastic strain estimation. Subsequently, the 'slope' of HR-EBSD versus micro-Laue residual elastic strain dropped from ~2.06 to ~1.57, see figure 3b. However, an absolute numerical, albeit statistical, convergence of the two techniques would require a slope of 1, which appears to be unachievable by simply optimizing HR-EBSD parameters.

It is to be noted that for large grains, pattern center shifts are significant but expected. Such shifts can be compensated by the HR-EBSD code (Britton & Wilkinson, 2012). As shown in supplementary figure S3a, though shifts were real - but even if incorrectly compensated, they affected the HR-EBSD estimated residual elastic strain value(s) only marginally (1% <). Further, the HR-EBSD residual elastic strains emerge from the shift in the $\vec{q}$ vector. As illustrated in the supplementary figure S3b, this is affected by the relative positioning of the zone axes. In brief, zone axes further from the pattern center show numerically higher $\vec{q}$ vector. However, both these factors also do not justify the very large difference, see figure 3b, estimated from our statistical data. Clearly, the HR-EBSD provides larger residual elastic strain estimates than the micro-Laue. To further explore the deviation in numerical convergence between micro-Laue and HR-EBSD techniques, the reader is referred to appendix A. Briefly, figure A.1a shows a grain-level comparison, while figure A.1b depicts a component wise comparison of the residual elastic strains measured using the two techniques.

To further analyse and explore the HR-EBSD residual elastic strain measurements, we estimate the amount of 'a' type (effect on Δd) and 'α' type (effect on $\Delta\theta$) strains from the residual elastic strain tensor $\boldsymbol{E^e}$ as:

$$\varepsilon_a = |E^e_{11}| + |E^e_{22}| + |E^e_{33}| \quad \dots (6)$$

$$\varepsilon_\alpha = |E^e_{12}| + |E^e_{13}| + |E^e_{23}| \quad \dots (7)$$

where, ε_a and ε_α denotes the 'a' and 'α' type residual elastic strains, respectively. Since the elastic strain tensor $\boldsymbol{E^e}$ is calculated in the crystal reference frame, a first order approximation has been utilized, i.e., the absolute values of the diagonal and non-diagonal components of the elastic strain tensor $\boldsymbol{E^e}$ are used, to estimate the 'a' type and 'α' type strains, respectively. Physically, ε_a defines the portion of $\boldsymbol{E^e}$ that would affect the interplanar spacing of a crystal lattice, whereas ε_α is indicative of the shear deformation or the change in interplanar angles in a crystal lattice.

The ratio of these entities, i.e., $\varepsilon_a/\varepsilon_\alpha$, has then been compared with existing experimental datasets (see figure 2a-d). 20 randomly oriented grains were selected from the specimen deformed up to 0.15 (imposed) tensile strain; the corresponding results have been shown in appendix figure 3c. In an ideal scenario, i.e., if numerical convergence were established, these above-mentioned ratios should be identical, i.e., equal to one. However, it can be clearly observed that with increasing magnitude of $\varepsilon_a/\varepsilon_\alpha$, i.e., when the '$\alpha$' type strains are underpredicted, the difference in von-Mises residual strains estimated by the HR-EBSD (sensitive to $\frac{\Delta\theta}{\theta}$) from its counterpart (micro-Laue: sensitive to $\frac{\Delta d}{d}$) increases linearly. It is important to note that the error in estimated von-Mises strains can rise as high $\sim 120\%$ for large variations in $\varepsilon_a/\varepsilon_\alpha$ (see figure 3c). The significant scatter in the data is primarily due to consideration of randomly oriented grains in the specimen (0.15 imposed tensile strain), each of which behaves differently under the imposed deformations.

Table 1: Comparison of residual elastic strain tensors obtained from the micro-Laue (sensitive to $\frac{\Delta d}{d}$) and HR-EBSD (sensitive to $\frac{\Delta\theta}{\theta}$) techniques:

Table 1.docx

We have further validated our observations on the 'a' type and 'α' type strains (see figure 3c) and its effects on the error in strain measurements (between HR-EBSD and micro-Laue) by conducting an identical analysis on the micro-Laue residual strain tensors. This exercise was carried out for three distant scenarios, i.e., for the grains where HR-EBSD residual

strains resulted in a ratio of $\frac{\varepsilon_a}{\varepsilon_\alpha} = 1.1, 2.99$ and 4.43, respectively. The corresponding strain matrices have been shown in Table 1. Clearly, it can be seen that when $\frac{\varepsilon_a}{\varepsilon_\alpha} = 1.1$ (from HR-EBSD), the resulting strain tensors are nearly identical (in magnitude), with the ratio of $\frac{\bar{\varepsilon}_{HR-EBSD}}{\bar{\varepsilon}_{micro-Laue}} = 1.18$. It is important to note that the ratio is not exactly equal to one; the minor difference may be attributed to the different sensitivities of the two measurement techniques to lattice distortion, $\left(\frac{\Delta\theta}{\theta}\right)$ versus $\left(\frac{\Delta d}{d}\right)$. Additionally, the differences in interaction volume for the two techniques, errors in geometric centroid determination and the noise in the HR-EBSD patterns may also contribute to the error.

In contrast, when $\frac{\varepsilon_a}{\varepsilon_\alpha} = 2.99$ and 4.43, the micro-Laue strain is significantly lower than the HR-EBSD strain, with the ratio of $\frac{\bar{\varepsilon}_{HR-EBSD}}{\bar{\varepsilon}_{micro-Laue}}$ being 1.42 and 2.05, respectively (see table 1). A component wise comparison of the residual strain tensors (along with the respective strain contours) shown later in the appendix figure A.1b, also points out towards a similar conclusion. In summary, figure 3c and table 1 conforms to the fact that as the 'a' type (effect on Δd) strain increases relative to the 'α' type (effect on $\Delta\theta$) component, the error in strain measured by HR-EBSD (sensitive to $\frac{\Delta\theta}{\theta}$), relative to the measurements from micro-Laue (sensitive to $\frac{\Delta d}{d}$), increases.

Further, we have also shown the sum of squared error (SSE), a standard output of OpenXY™, for all grains analysed in figure 3c. The SSE values range from 0.08 (minimum) to 0.2 (maximum) with a mean of 0.14. These results indicate that the conclusions derived from figure 3c, i.e., the errors in HR-EBSD strains relative to the micro-Laue strains, are not largely influenced by the noise in the derived HR-EBSD patterns.

The reference patterns for HR-EBSD as well as HR-TKD (explained later in section 5) were selected from a region near the geometric centre of the grain, having the maximum image quality. In all cases, it was assumed that the reference patterns were relatively strain-free. Owing to the large size of grains used in the present study, the geometric centre of the grains remained nearly unaffected with progressive deformation, even at an imposed tensile strain of 0.15 (see GROD maps in figure A.1a). As discussed in section 3.1, the reference d_0 for micro-Laue single crystal XRD, was obtained from annealed powder specimens. The measured residual elastic strains using the $\frac{\Delta\theta}{\theta}$ sensitive and $\frac{\Delta d}{d}$ sensitive methods do not show any notable

errors at lower imposed tensile strains (figure 2 and 3), thus indicating residual elastic strain estimations for all measurement techniques have initiated from an identical baseline.

Figure 4.png

Figure 4: Results from virtual experiments conducted on simulated HR-EBSD patterns. (a) Progressive tensile strains were introduced in an ideal bcc iron (Fe) strain-free lattice ($a = b = c = 2.8667$ Å and $\alpha = \beta = \gamma = 90°$). This was implemented by altering lattice parameter 'a' (while keeping b and c constant) and by changing 'α' (while holding β and γ constant). The corresponding von-Mises strains, as estimated by $\Delta d/d$ and $\Delta\theta/\theta$, ranged from 0.0049 to 0.015. (b) For these strained, and un-strained, lattices HR-EBSD patterns were simulated both kinematically and dynamically. These strains were then estimated using HR-EBSD or cross-correlation algorithm. (c) Scaling factor from patterns simulations (as in figure 4c) from 11 different crystallographic orientations, using traction free boundary conditions. 'Average' scaling factors from pattern simulations (~1.47) and experiments (~1.57, figure 3b) have also been included.

One potential cause of this difference is perhaps the different levels of sensitivity, of the respective measurement techniques, to different measures of elastic strain? Afterall, HR-EBSD and micro-Laue XRD are sensitive to measurements of interplanar angle $\left(\frac{\Delta\theta}{\theta}\right)$ and spacing $\left(\frac{\Delta d}{d}\right)$, respectively. To investigate this possibility, 'virtual' experiments were conducted using HR-EBSD pattern simulations (Callahan & De Graef, 2013; Winkelmann et al., 2007; Zaefferer, 2007). As in figure 4a, and expanded in the appendix B, an ideal strain-free bcc iron lattice (Fe) was subjected to progressive strain(s) and corresponding lattice distortions. This was achieved by systematically increasing a 'single' lattice parameter (a) or an interplanar angle (α). The corresponding lattice strains ($\boldsymbol{\varepsilon}$), see figure 4a and appendix B, were then determined from the knowledge of $\left(\frac{\Delta d}{d}\right)$ or $\left(\frac{\Delta\theta}{\theta}\right)$, respectively. The imposed von Mises strain(s) were derived as $\varepsilon_t = \sqrt{\frac{2}{3}\,\boldsymbol{\varepsilon}:\boldsymbol{\varepsilon}}$, and were systematically altered from 0.0049 to 0.015 (see figure 4a). We have used corresponding crystal structures to simulate the HR-EBSD patterns, see figure 4b. The HR-EBSD pattern simulations were conducted with EDAX OIM-Matrix™ software. It is to be noted that both kinematical, where no interaction between beam(s) were considered, and dynamical, with multi-beam interaction, simulations were used.

The latter, as expected (Goodhew & Humphreys, 2000; Callahan & De Graef, 2013; Zaefferer, 2007), appeared more realistic. A description of our pattern simulations is given in the appendix B.

Figure 4b shows pattern simulations, kinematical as well as dynamical, originating from a single representative orientation. It is to be noted that strains were progressively applied on an ideal lattice. From the distorted lattice(s), pattern simulations were then conducted. Finally, from the simulated patterns lattice distortions or strains were estimated by cross-correlation. As discussed earlier, the cross-correlation or HR-EBSD is sensitive to changes in $\left(\frac{\Delta\theta}{\theta}\right)$ only, but were performed on lattices distorted by α or $\Delta\theta$ and a or Δd, respectively. These two estimates were clearly different. As in figure 4b, the strains imposed by distorting the ideal lattice by $\Delta\theta$ showed a higher slope in comparison to their Δd counterparts. More importantly, the strain estimated by HR-EBSD is nearly identical to the imposed strain on ideal lattice (slope~ 1) when the lattices are distorted by α or $\Delta\theta$ as compared to the case when lattice is distorted by by a or Δd. Further, it was checked that the mode of pattern simulation (figure 4b) and traction-free versus trace-free boundary conditions (appendix figure B.1) did not significantly alter this strain estimate. However, crystal orientations or the relative positioning of the zone axes (see supplementary figure S3b) appeared to affect the scaling factor. We used 'fitting' of 11 randomly selected crystallographic orientations (and corresponding ideal lattices), and an average scaling factor of ~1.47 was obtained, see figure 4c. It is interesting to note that the experimental and statistical data produced a scaling factor of ~1.57 (figure 3b), which was similar but not identical to the scaling factor emerging from pattern simulations (figure 4c). It is important to note that the two ratios (scaling factors), the ratio of experimentally measured strains between micro-Laue and HREBSD, and the ratio between the simulated measurements of HR-EBSD cannot be directly correlated. This is because of the fact that the simulated HR-EBSD strains were derived from the cross-correlation algorithm (for lattices distorted by 'α' or '$\Delta\theta$' as well as by 'a' or 'Δd'), which is more sensitive to $\left(\frac{\Delta\theta}{\theta}\right)$. However, the strikingly similar magnitude of scaling factors (see figure 4c) in both cases indicate the importance of a scalar numerical factor when establishing convergence in residual strain estimates using different measures of lattice distortions $\left(\frac{\Delta\theta}{\theta}\right)$ versus $\left(\frac{\Delta d}{d}\right)$.

The scaling factor calculations were primarily based on the effective strains estimated from the HR-EBSD and micro-Laue techniques. Since the use of effective strains is equivalent to a zero-dimensional or a scalar approximation of the residual strain tensors over all grains

present in a polycrystalline ensemble, the same magnitude of average scaling factor may not hold valid directly for the individual grain orientations in a polycrystalline specimen. This can also be concluded from figure 2a, where each grain subtends a different scaling factor, depending on its orientation, elastic modulus and the accommodated strain. Further, the scaling factor is estimated using the ratio of the (best-fit) slopes of HR-EBSD ($\frac{\Delta\theta}{\theta}$ sensitive) and micro-Laue ($\frac{\Delta d}{d}$ sensitive) based von-Mises strains, with imposed tensile deformation. Hence, the numerical scaling factor proposed in the present study does take into account the increasing error between the two modes of strain measurement, with increasing imposed tensile strain.

Micro-Laue and HR-EBSD are significantly different in spatial resolution ($\sim 50\ \mu m$ versus $\sim 20\ nm$). Spatial resolutions of HR-TKD and TEM-PED, on the other hand, are somewhat closer. It was hence decided to explore the differences in strain measurement further with direct observations from HR-TKD ($\left(\frac{\Delta\theta}{\theta}\right)$-sensitive) and PED ($\left(\frac{\Delta d}{d}\right)$-sensitive). It is to be noted that both these measurements were performed on an identical area (see figure 5a) from a 3 mm TEM foil. Considering the foil thickness of a few hundred Å, we have focused on in-plane shear strain (γ_{xy}) for comparison. Reference patterns (HR-TKD and PED) were obtained from the identical 'spot', see figure 5b. Additionally, we have also shown standard SSE output from OpenXY™ in the supplementary figure S4. The low error fraction magnitudes ($\sim 12\%$), especially in a strained specimen, are indicative of the fact that the captured HR-TKD patterns (and the corresponding scan parameters, camera settings) were of sufficient quality..

Figure 5.png

Figure 5: Exploring the numerical convergence in $\left(\frac{\Delta\theta}{\theta}\right)$ sensitive, HR-TKD and $\left(\frac{\Delta d}{d}\right)$ sensitive, TEM-PED based residual elastic strains. (a) Images showing the identical region used for mapping residual elastic strains with HR-TKD and TEM-PED. The red markers highlight the reference topological features used to ascertain identical locations for the respective scans. (b) Residual elastic strains were estimated using high resolution transmission Kikuchi diffraction (HR-TKD) and precession electron diffraction (PED) in the identical regions. The black markers highlight areas with similar strain concentrations in the HR-TKD as well as PED measurements. 'Correction' (using experimental scaling factor of ~1.57) shows similar residual strain maps (in figure 5b, bottom) and (c) plot of absolute magnitudes of HR-TKD versus TEM-PED strains ($|\gamma_{xy}|$).

Several interesting points emerged. Firstly, the orientations (shown as inverse pole figures) were similar. However, the so-called kernel average misorientation or KAM distribution (Thool et al., 2020; Pai et al., 2022) was higher and broader for HR-TKD (see Supplementary figure S5). This is not surprising. HR-TKD (or HR-EBSD) has nearly two-orders of magnitude higher angular resolution than PED. Increasing angular resolution ($\sim 0.006°$) enables HR-TKD to detect minor gradients in orientation effectively. TEM-PED on the other hand, with an angular resolution of $< 0.4°$, would neglect such minor orientation gradients and report them as a single orientation. This results in a wider/broader spread of the misorientation distribution for HR-TKD when compared to TEM-PED measurements. Naturally, the microstructural features appeared different. Though both measurements were taken at a step size of 10 nm, the HR-TKD used a beam current of 16 nA while PED involved a beam current of 0.0017 nA and a precession angle of 10.4 mrad. It is to be noted that the adopted precession angle provided excellent spatial resolution. In brief, the PED map clearly offered higher spatial resolution than the HR-TKD. The respective spatial resolutions were estimated using a MATLAB^{TM} code. This was ~ 1.2 times (in pixels per μm^2) more in PED than in HR-TKD, though same step size was used. In particular, the PED appeared to be more sensitive to the presence of dislocations. Though lateral resolution of TEM-PED is superior in comparison to TKD, higher beam energies and the tendency to probe through the entire sample thickness results in a poor depth resolution (Sneddon et al., 2016).

However, the biggest difference was in the imaging of γ_{xy}, see figure 5b. In HR-TKD, this was ~ 1.73 times of the PED strain values (figure 5b). Imposing the previously estimated scaling factor (between HR-EBSD and micro-Laue – see figure 3b), a slope of ~1.08 was obtained between the HR-TKD and TEM-PED residual strains ($|\gamma_{xy}|$, see figure 5c). This is indeed striking that two different sets of experimental lattice strain estimates (figure 3b versus figure 5c) gave a similar scaling factor. In other words, this study not only established a difference in residual strain estimates using different measures of lattice distortions $\left(\frac{\Delta\theta}{\theta}\right)$ versus $\left(\frac{\Delta d}{d}\right)$, it also brought out a similar scaling adopting two different sets of analytical tools - HR-EBSD versus micro-Laue, and HR-TKD versus TEM-PED.

5. Local Lattice Distortions in Microstructures

The field of microscopy and microstructure, in metallic materials, was started by a Sheffield geologist Henry Sorby (Nuttall, 1981; Higham, 1963). Using thin sections of a

wrought iron, deformed and then partially annealed, Sorby proposed the presence of thermodynamically unstable (deformed) and stable (recrystallized) regions. This was during a time-period, when deformation was often viewed as 'amorphization'. Optical microscopy also described invariant plane strain microstructures, originally by Adolf Marten and then by Bain and Davenport, long before the phenomenological theory of martensite crystallography was formalized (Osmond, 1895). Microscopy naturally evolved further with the arrival of electron columns (Goodhew & Humphreys, 2000) and then the EBSD (Wright & Adams, 1992; Adams et al., 1993). Other than attributes like resolution, the key aspect of any microscopy is in the contrast mechanism. The latter may emerge from the amplitude-phase contrast in classical TEM imaging, to more recent orientation contrast in EBSD and strain contrast in HR-EBSD.

The complete microstructural description consists of grains and phases and also defects (Humphreys & Hatherly, 2012). An important attribute of the latter is in the lattice distortions, the focus of the present study. The lattice distortion translates into residual strains and stresses, even bulk measurements of which may suffer from significant reproducibility issues (Verlinden et al., 2007). The local residual strain can be measured from high resolution TEM imaging and the so-called geometrical phase analysis (Revelly et al., 2015; Ghamarian et al., 2014; Ghamarian, 2017). However, this restricts any such measurement to aberration corrected microscopes and very 'limited' area. Alternatives exist in the form of micro-Laue XRD (Lodh et al., 2022, 2019, 2018, 2017) and SEM (Wilkinson et al., 2006; Fullwood et al., 2015; Wright et al., 2011) plus TEM (Ghamarian et al., 2014; Ghamarian, 2017) based microtexture measurements. It is perhaps redundant to 'preach' on the potentials for such multi-scale diffraction-based measurements, as any metallurgist or materials scientist would readily appreciate this point. Such techniques hold possibilities of revealing 'uncharted' aspects of microstructure.

A significant hurdle to any such effort, of strain-based microstructural representation, is in quantifying the differences in estimated strain magnitudes by different analytical tools. We have established that strain estimates from $\left(\frac{\Delta\theta}{\theta}\right)$ are higher than those from $\left(\frac{\Delta d}{d}\right)$. This was shown with both statistical experimental data (figure 3b) and also with numerical simulations (figures 4a-c). Even using the same HR-EBSD algorithm, the lattices distorted by $\left(\frac{\Delta\theta}{\theta}\right)$ exhibited higher strain values (see figure 4b,c) than those altered by $\left(\frac{\Delta d}{d}\right)$. The numerical differences cannot be fixed using simple measurement algorithm adjustments (figure 3) or protocol (supplementary figure S3). The difference originated from the template of imposed

lattice distortion. The lattices distorted by changing $\Delta\theta$ appeared more strained than those altered by Δd, see figure 4b. It is interesting that experiments and pattern simulation both brought out these differences, and a somewhat similar scaling factor (figures 3b and 4c) emerged. The clear linear relationship, with strain level, is striking, and it enabled us to obtain a convergence in strain magnitudes (see figure 4c and 5c).

Overall, our observations in figure 3 can be justified as follows: phenomenologically, for the same state of deformation in a grain, the strain measured by HR-EBSD and micro-Laue single crystal residual elastic strain measurements should be identical. One potential cause of this difference is perhaps the different levels of sensitivity of the respective measurement techniques to different measures of elastic strain. The Kikuchi patterns are considered to be a gnomonic projection of a crystal lattice on the phosphor screen. The angles between the two bands corresponds to the interplanar angles in a crystal lattice, whereas, the width of Kikuchi band corresponds to the interplanar spacing d_{hkl} (Schwarzer et al., 2009). The cross-correlation based residual strain calculations rely on the shift in zone axes' ($\vec{q}$) on the phosphor screen, to estimate the lattice distortion gradient tensor at a given pixel (Wilkinson et al., 2006, 2009b). As seen in figure 1b, such a technique is primarily sensitive to $\frac{\Delta\theta}{\theta}$ (change in interplanar angles), in contrast to the micro-Laue technique, which is primarily sensitive to $\frac{\Delta d}{d}$ (change in interplanar spacing). The varying interpretation of the same imposed state of deformation by the two techniques can lead to the errors/discrepancies between micro-Laue and HR-EBSD/TEM-PED and HR-TKD residual strains.

In addition, a larger 'a' type strain, which mainly results in an uniform change in the interplanar spacing (d_{hkl}) would not accurately reflect on the zone axes' shifts; thus, resulting in a large deviation in the HR-EBSD/HR-TKD ($\frac{\Delta\theta}{\theta}$ sensitive) estimated strains as compared to the $\frac{\Delta d}{d}$ based techniques (see figure 3c). It is important to note that the present study does not comment on the reliability of either of the two diffraction-based residual strain measurement techniques, $\frac{\Delta\theta}{\theta}$ sensitive and $\frac{\Delta d}{d}$ sensitive; we only present a comparison, followed by a possible justification of the errors/discrepancies and calculation of a scaling factor to establish numerical convergence between these multi-scale diffraction-based strain measurement approaches. Based on the statistical dataset as well as the reasoning provided in the present study, the authors believe that a similar (qualitative), but not identical (quantitative), numerical scaling factor should emerge for all cubic materials deformed under tensile loading conditions. The variations in absolute magnitudes however, may arise as a result of the difference in the

elastic stiffness, microstructure, and the measurement parameters associated with the technique.

Towards a broader perspective, it is hoped that the present study will help researchers to exploit the full potentials of multi-scale diffraction-based lattice strain measurements as a microscopic technique and contrast mechanism.

6. Conclusions

- We have used different multi-scale diffraction-based residual strain measurements. These were sensitive to changes in interplanar angle ($\left(\frac{\Delta\theta}{\theta}\right)$ – HR-EBSD and HR-TKD) or interplanar spacing ($\left(\frac{\Delta d}{d}\right)$ – micro-Laue XRD and TEM-PED).
- The measurements differed in scale and resolution, but more importantly they were numerically different. For example, both HR-EBSD and micro-Laue XRD showed an increase in residual strain values with imposed tensile deformation. However, even after optimizing the HR-EBSD parameters - the increase in strains were ~1.57 times higher in HR-EBSD.
- We have then distorted an ideal bcc lattice, virtually, by progressively altering its interplanar angle (α) and lattice parameter (a), respectively. These effectively changed $\Delta\theta$ and Δd. From kinematically and dynamically simulated patterns, the corresponding strains were calculated by HR-EBSD. The strain estimates were consistently higher for lattices distorted by $\Delta\theta$ compared to lattices distorted by Δd. The scaling factor (~1.47) was somewhat similar to the experimentally observed ratio of ~1.57.
- HR-TKD and TEM-PED measurements, conducted on the identical location, showed higher strains for HR-TKD. However, similar magnitudes of strain distributions were obtained when scaled with the earlier (HR-EBSD versus micro-Laue) factor of ~1.57.

7. Acknowledgements

The authors gratefully acknowledge funding received from the Department of Science and Technology (DST), India - Science and Engineering Research Board (SERB), India for this research under grant number: CRG/2020/000593. Support from the National Facility of Texture and OIM and Themis-300 TEM laboratories are also acknowledged. Namit Pai is also grateful for funding received from the Prime Minister's Research Fellowship to support his

doctoral studies. David Fullwood and Bethany Syphus were funded by National Science Foundation (NSF) grant CMMI 2147126.

8. Appendix A

Figure A.1a shows the evolution of the grain reference orientation deviation (GROD) and von-Mises residual elastic strains with increasing tensile deformation, measured using the HR-EBSD technique. The corresponding average values from its $\frac{\Delta d}{d}$ counterpart (micro-Laue) have also been provided. It is to be noted that the HR-EBSD strain measurements have been shown after accounting for the corrections, i.e., (A) insufficient ROI, (B) inappropriate selection of the reference pattern, (C) choice of pattern centre and (D) Remapping, discussed previously in section 4. It can be seen that HR-EBSD provides significantly larger residual strains in comparison to the micro-Laue; the differences get amplified with increasing deformation. This observation can also be noted from the component level comparison shown in figure A.1b, albeit the scaling factor differs significantly for each individual strain component.

An interesting observation from figure A.1a is that the elastically harder grains tend to develop higher magnitude of elastic strains. In contrast, the softer grains deform plastically, thus developing significant intragranular orientation gradients within them (see black markers in figure A.1a). Such a behaviour has been captured by the micro-Laue (sensitive to $\frac{\Delta d}{d}$) and HR-EBSD (sensitive to $\frac{\Delta \theta}{\theta}$) strain measurements. Despite the similar trends, the differing magnitudes highlight the need of a scaling factor between the two techniques.

Figure A.1.png

Figure A.1: Comparison of residual elastic strains measured using the micro-Laue (sensitive to $\frac{\Delta d}{d}$) and HR-EBSD (sensitive to $\frac{\Delta \theta}{\theta}$) technique in terms of (a) von-Mises strain and (b) component-level comparison. The markers refer to regions depicting large orientation gradients and hence, the strain concentrations.

9. Appendix B

To investigate the difference between the measurement of residual strains based on $\left(\frac{\Delta\theta}{\theta}\right)$ and $\left(\frac{\Delta d}{d}\right)$, we have conducted simulations of HR-EBSD patterns. An ideal strain free (BCC) lattice, see figure 4a, was subjected to progressive strains of two types. The corresponding deformation gradients were estimated as:

1. Idealized expansions were applied along the 'a' direction with no Poisson contraction in the perpendicular directions, see figure 4a. The corresponding deformation gradient:

$$\boldsymbol{F} = \begin{bmatrix} \lambda & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad \ldots (\mathrm{B}.1)$$

   where, $\lambda = d/d_0$. d and d_0 represent the strained and unstrained lattice spacings, respectively. The residual strains (see figure 4a) were represented as type $\left(\frac{\Delta d}{d}\right)$.

2. Idealized shear strains were applied at 45° to the 'a' direction, see figure 4a. The corresponding deformation gradient:

$$\boldsymbol{F} = \begin{bmatrix} 1 & \tan(\theta) & 0 \\ \tan(\theta) & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad \ldots (B.2)$$

   where, θ denotes the change in interplanar angle. This approach represented residual strain as type $\left(\frac{\Delta\theta}{\theta}\right)$.

We have used this approach for 11 crystallographic orientations, see figure 4c. Out of these, figure 4b shows results from 'orientation 1' with both kinematical and dynamical simulations. The corresponding patterns were input to OpenXY™ (Fullwood, 2020), and estimates of residual strains were obtained by an off-line cross-correlation of simulated Kikuchi patterns. As the results for kinematical and dynamical pattern simulations were similar (figure 4b), we have only used the computationally inexpensive kinematical simulations for the remaining calculations. It is to be noted that HR-EBSD estimates adopting traction free (figure 4b) or trace free (appendix figure B.1) boundary conditions were nearly identical. We have then determined an average scaling factor between $\left(\frac{\Delta\theta}{\theta}\right)$ type strain measurements and $\left(\frac{\Delta d}{d}\right)$ type measurements for a given effective strain level, for all 11 orientations – see figure 4c. An average scaling factor of ~1.47 was thus obtained. It is to be noted that the estimated scaling factors were different for orientations 5 and 11. These were orientations where zone axes were at the edge of HR-EBSD Bragg spread, potentially introducing higher $\vec{q}$ vector (see Supplementary figure S3b).

Figure B.1.png

Figure B.1: Kinematical pattern simulations, similar to figure 4b, but conducted using trace free boundary conditions ($\sigma_{11} + \sigma_{22} + \sigma_{33} = 0$). The slopes were similar to those obtained by adopting traction free boundary conditions (figure 4b).

To further verify the simulated residual elastic strain measurements, we have estimated the change in interplanar spacing ($\frac{d-d_0}{d_0}$ for lattices distorted by a or Δd) and the change in interplanar angle 'α' (for lattices distorted by α or $\Delta\theta$) from the corresponding strain tensors, derived at various deformation levels (see figure 4a). The resultant magnitudes have then been compared with their imposed counterparts. A reasonable convergence has been achieved by our HR-EBSD pattern simulations, especially in the predictions of the 'α' angle. For brevity, the results have been only been presented for the orientation 1 (see figure 4c and appendix figure B.1) in Table B.1 and Table B.2. The imposed change in Table B.1 and B.2 refer to the use of deformation gradients described in equations B.1 and B.2 to distort the ideal crystal lattice, whereas the estimated changes refer to the back-calculations carried out from the OpenXY^{TM} estimated deformation gradients.

Table B.1: Comparison of the imposed versus estimated change in interplanar angles (for lattices distorted by α or $\Delta\theta$)

Table B.1.docx

Table B.2: Comparison of the imposed versus estimated change in $\frac{d-d_0}{d_0}$ (for lattices distorted by a or Δd)

Table B.2.docx

10. Data Availability

All data necessary to evaluate the claims made in this article are summarized in the published figures. Data in expanded form are available from the corresponding author upon reasonable request.

11. References

ADAMS, B. L., WRIGHT, S. I. & KUNZE, K. (1993). Orientation imaging: the emergence of a new microscopy. *Metallurgical Transactions A* **24**, 819–831.

ALMEN, J. O. & BLACK, P. H. (1963). *Residual stresses and fatigue in metals*. McGraw-Hill.

ARMIGLIATO, A., SPESSOT, A., BALBONI, R., BENEDETTI, A., CARNEVALE, G., FRABBONI, S., MASTRACCHIO, G. & PAVIA, G. (2006). Convergent beam electron diffraction investigation of strain induced by Ti self-aligned silicides in shallow trench Si isolation structures. *Journal of applied physics* **99**. https://pubs.aip.org/aip/jap/article/99/6/064504/293132 (Accessed December 4, 2023).

BACZMANSKI, A., WIERZBANOWSKI, K., HAIJE, W. G., HELMHOLDT, R. B., EKAMBARANATHAN, G. & PATHIRAJ, B. (1993). Diffraction Elastic Constants for Textured Materials-Different Methods of Calculation. *Crystal Research and Technology* **28**, 229–243.

BASHIR, A., MILLAR, R. W., GALLACHER, K., PAUL, D. J., DARBAL, A. D., STROUD, R., BALLABIO, A., FRIGERIO, J., ISELLA, G. & MACLAREN, I. (2019). Strain analysis of a Ge micro disk using precession electron diffraction. *Journal of Applied Physics* **126**. https://pubs.aip.org/aip/jap/article/126/23/235701/156467 (Accessed December 4, 2023).

BASINGER, J., FULLWOOD, D., KACHER, J. & ADAMS, B. (2011). Pattern center determination in electron backscatter diffraction microscopy. *Microscopy and Microanalysis* **17**, 330–340.

BÉCHÉ, A., ROUVIÈRE, J. L., BARNES, J. P. & COOPER, D. (2013). Strain measurement at the nanoscale: Comparison between convergent beam electron diffraction, nano-beam electron diffraction, high resolution imaging and dark field electron holography. *Ultramicroscopy* **131**, 10–23.

VAN BREMEN, R., GOMES, D. R., DE JEER, L. T., OCELÍK, V. & DE HOSSON, J. T. M. (2016). On the optimum resolution of transmission-electron backscattered diffraction (t-EBSD). *Ultramicroscopy* **160**, 256–264.

BRITTON, T. B. & WILKINSON, A. J. (2011). Measurement of residual elastic strain and lattice rotations with high resolution electron backscatter diffraction. *Ultramicroscopy* **111**, 1395–1404.

BRITTON, T. B. & WILKINSON, A. J. (2012). High resolution electron backscatter diffraction measurements of elastic strain variations in the presence of larger lattice rotations. *Ultramicroscopy* **114**, 82–95.

CALLAHAN, P. G. & DE GRAEF, M. (2013). Dynamical electron backscatter diffraction patterns. Part I: Pattern simulations. *Microscopy and Microanalysis* **19**, 1255–1265.

CHOI, S. & GRIFFIN, B. A. (2016). Local residual stress monitoring of aluminum nitride MEMS using UV micro-Raman spectroscopy. *Journal of Micromechanics and Microengineering* **26**, 025009.

CULLITY, B. D. (1956). *Elements of X-ray Diffraction*. Addison-Wesley Publishing.

DEAL, A., SPINELLI, I., CHUANG, A., GAO, Y. & BRODERICK, T. (2021). Measuring residual stress in Ti-6Al-4V with HR-EBSD, using reference patterns from annealed material. *Materials Characterization* **175**, 111027.

DINGLEY, D. (2004). Progressive steps in the development of electron backscatter diffraction and orientation imaging microscopy. *Journal of Microscopy* **213**, 214–224.

FAROOQ, M. U., VILLAURRUTIA, R., MACLAREN, I., BURNETT, T. L., COMYN, T. P., BELL, A. J., KUNGL, H. & HOFFMANN, M. J. (2008). Electron backscatter diffraction mapping of herringbone domain structures in tetragonal piezoelectrics. *Journal of Applied Physics* **104**. https://pubs.aip.org/aip/jap/article/104/2/024111/934362 (Accessed December 4, 2023).

FULLWOOD, D. (2020). Openxy. https://github.com/BYU-MicrostructureOfMaterials/OpenXY (Accessed March 6, 2022).

FULLWOOD, D. T., SANDERSON, S., BAIRD, S., CHRISTENSEN, J., HOMER, E. R. & JOHNSON, O. K. (2022). Determining grain boundary position and geometry from EBSD data: Limits of accuracy. *Microscopy and Microanalysis* **28**, 96–108.

FULLWOOD, D., VAUDIN, M., DANIELS, C., RUGGLES, T. & WRIGHT, S. I. (2015). Validation of kinematically simulated pattern HR-EBSD for measuring absolute strains and lattice tetragonality. *Materials Characterization* **107**, 270–277.

GALLET, J., PEREZ, M., GUILLOU, R., ERNOULD, C., LE BOURLOT, C., LANGLOIS, C., BEAUSIR, B., BOUZY, E., CHAISE, T. & CAZOTTES, S. (2023). Experimental measurement of dislocation density in metallic materials: a quantitative comparison between measurements techniques (XRD, R-ECCI, HR-EBSD, TEM). *Materials Characterization* **199**, 112842.

GHAMARIAN, I. (2017). 'Application of ASTAR^{TM}/Precession Electron Diffraction Technique to Quantitatively Study Defects in Nanocrystalline Metallic Materials'. PhD Thesis, Iowa State University.

GHAMARIAN, I., LIU, Y., SAMIMI, P. & COLLINS, P. C. (2014). Development and application of a novel precession electron diffraction technique to quantify and map deformation structures in highly deformed materials—as applied to ultrafine-grained titanium. *Acta Materialia* **79**, 203–215.

GOODHEW, P. J. & HUMPHREYS, J. (2000). *Electron microscopy and analysis*. CRC press.

HARDIN, T. J., RUGGLES, T. J., KOCH, D. P., NIEZGODA, S. R., FULLWOOD, D. T. & HOMER, E. R. (2015). Analysis of traction-free assumption in high-resolution EBSD measurements. *Journal of microscopy* **260**, 73–85.

HE, B. B. (2003). Introduction to two-dimensional X-ray diffraction. *Powder diffraction* **18**, 71–85.

HEINDLHOFER, K. (1948). *Evaluation of residual stress*. McGraw-Hill Book Company.

HIGHAM, N. (1963). *A very scientific gentleman: the major achievements of Henry Clifton Sorby*. Oxford: Pergamon Press; New York: Macmillan.

HÜE, F., HÝTCH, M., BENDER, H., HOUDELLIER, F. & CLAVERIE, A. (2008). Direct Mapping of Strain in a Strained Silicon Transistor by High-Resolution Electron Microscopy. *Physical Review Letters* **100**, 156602.

HUMPHREYS, F. J. (2004). Characterisation of fine-scale microstructures by electron backscatter diffraction (EBSD). *Scripta materialia* **51**, 771–776.

HUMPHREYS, F. J. & HATHERLY, M. (2012). *Recrystallization and related annealing phenomena*. Elsevier.

HUMPHREYS, F. J., HUANG, Y., BROUGH, I. & HARRIS, C. (1999). Electron backscatter diffraction of grain and subgrain structures—resolution considerations. *Journal of Microscopy* **195**, 212–216.

JIANG, J., BRITTON, T. B. & WILKINSON, A. J. (2013). Measurement of geometrically necessary dislocation density with high resolution electron backscatter diffraction: Effects of detector binning and step size. *Ultramicroscopy* **125**, 1–9.

KALÁCSKA, S., DANKHÁZI, Z., ZILAHI, G., MAEDER, X., MICHLER, J., ISPÁNOVITY, P. D. & GROMA, I. (2020). Investigation of geometrically necessary dislocation structures in compressed Cu micropillars by 3-dimensional HR-EBSD. *Materials Science and Engineering: A* **770**, 138499.

KALÁCSKA, S., GROMA, I., BORBÉLY, A. & ISPÁNOVITY, P. D. (2017). Comparison of the dislocation density obtained by HR-EBSD and X-ray profile analysis. *Applied Physics Letters* **110**. https://pubs.aip.org/aip/apl/article/110/9/091912/34380 (Accessed October 16, 2023).

KRAWITZ, A. D. & HOLDEN, T. M. (1990). The measurement of residual stresses using neutron diffraction. *MRS Bulletin* **15**, 57–64.

KROST, A. & BLÄSING, J. (2009). Fast, micrometer scale characterization of group-III nitrides with laboratory X-ray diffraction. *Materials Science and Engineering: A* **524**, 82–88.

KUMAR, G., KANJARLA, A. K., LODH, A., SINGH, J., SINGH, R., SRIVASTAVA, D., DEY, G. K., SAIBABA, N., DOHERTY, R. D. & SAMAJDAR, I. (2016). Burst ductility of zirconium clads: The defining role of residual stress. *Metallurgical and Materials Transactions A* **47**, 3882–3896.

LODH, A., TAK, T. N., PRAKASH, A., GURUPRASAD, P. J., HUTCHINSON, C. & SAMAJDAR, I. (2017). Relating residual stress and substructural evolution during tensile deformation of an aluminum-manganese alloy. *Metallurgical and Materials Transactions A* **48**, 5317–5331.

LODH, A., TAK, T. N., PRAKASH, A., GURUPRASAD, P. J., KERALAVARMA, S. M., BENZERGA, A., HUTCHINSON, C. & SAMAJDAR, I. (2019). Microstructural origin of residual stress relief in aluminum. *Metallurgical and Materials Transactions A* **50**, 5038–5055.

LODH, A., TEWARY, U., SINGH, R. P., TAK, T. N., PRAKASH, A., ALANKAR, A., GURUPRASAD, P. J. & SAMAJDAR, I. (2018). Orientation-dependent developments in misorientation and residual stress in rolled aluminum: the defining role of dislocation interactions. *Metallurgical and Materials Transactions A* **49**, 5946–5952.

LODH, A., THOOL, K. & SAMAJDAR, I. (2022). X-ray diffraction for the determination of residual stress of crystalline material: An overview. *Transactions of the Indian Institute of Metals* **75**, 983–995.

MANDA, S., KUMAR, S., PAL, K., BHATTACHARYYA, A. R., PANWAR, A. S. & SAMAJDAR, I. (2023). Snoek-Dominated Internal Friction Response in bcc Steel: Relating Experiments With a Multi-scale Atomistic Computational Framework. *Metallurgical and Materials Transactions A* **54**, 562–576.

MARGULIES, L., LORENTZEN, T., POULSEN, H. F. & LEFFERS, T. (2002). Strain tensor development in a single grain in the bulk of a polycrystal under loading. *Acta Materialia* **50**, 1771–1779.

MAURICE, C., DRIVER, J. H. & FORTUNIER, R. (2012). On solving the orientation gradient dependency of high angular resolution EBSD. *Ultramicroscopy* **113**, 171–181.

MEHTANI, H. K., KHAN, M. I., DURGAPRASAD, A., DEB, S. K., PARIDA, S., PRASAD, M., FULLWOOD, D., DOHERTY, R. D. & SAMAJDAR, I. (2020). Oxidation behavior of interstitial free steel: The defining role of substrate crystallographic texture. *Acta Materialia* **190**, 43–57.

NIESSEN, F., BURROWS, A. & DA SILVA FANTA, A. B. (2018). A systematic comparison of on-axis and off-axis transmission Kikuchi diffraction. *Ultramicroscopy* **186**, 158–170.

NOYAN, I. C. & COHEN, J. B. (2013). *Residual stress: measurement by diffraction and interpretation*. Springer.

NUTTALL, R. H. (1981). The First Microscope of Henry Clifton Sorby. *Technology and culture* **22**, 275–280.

OSGOOD, W. R. (1954). *Residual stresses in metals and metal construction*. Reinhold Publishing Corporation.

OSMOND (1895). General method for the microscopical analysis of carbon steels. *Bulletin de la Société d'Encouragement pour l'Industrie Nationale* **10**, 476–518.

OZDOL, V. B., GAMMER, C., JIN, X. G., ERCIUS, P., OPHUS, C., CISTON, J. & MINOR, A. M. (2015). Strain mapping at nanometer resolution using advanced nano-beam electron diffraction. *Applied Physics Letters* **106**. https://pubs.aip.org/aip/apl/article/106/25/253107/27065 (Accessed December 4, 2023).

PAI, N., PRAKASH, A., SAMAJDAR, I. & PATRA, A. (2022). Study of grain boundary orientation gradients through combined experiments and strain gradient crystal plasticity modeling. *International Journal of Plasticity* **156**, 103360.

PEKIN, T. C., GAMMER, C., CISTON, J., MINOR, A. M. & OPHUS, C. (2017). Optimizing disk registration algorithms for nanobeam electron diffraction strain mapping. *Ultramicroscopy* **176**, 170–176.

PORTILLO, J., RAUCH, E. F., NICOLOPOULOS, S., GEMMI, M. & BULTREYS, D. (2010). Precession electron diffraction assisted orientation mapping in the transmission electron microscope. In *Materials science forum*vol. 644, pp. 1–7. Trans Tech Publ.

PRAKASH, A., TAK, T. N., PAI, N. N., SEEKALA, H., MURTY, S. N., PHANI, P. S., MAHESH, S., GURUPRASAD, P. J. & SAMAJDAR, I. (2023). Inception of Macroscopic Shear Bands During Hot Working of Aluminum Alloys. *International Journal of Plasticity* 103632.

RAUCH, E. F., PORTILLO, J., NICOLOPOULOS, S., BULTREYS, D., ROUVIMOV, S. & MOECK, P. (2010). Automated nanocrystal orientation and phase mapping in the transmission electron microscope on the basis of precession electron diffraction. *Zeitschrift für Kristallographie* **225**, 103–109.

RAUCH, E. F. & VERON, M. (2005). Coupled microstructural observations and local texture measurements with an automated crystallographic orientation mapping tool attached to a tem. *Materialwissenschaft und Werkstofftechnik: Entwicklung, Fertigung, Prüfung, Eigenschaften und Anwendungen technischer Werkstoffe* **36**, 552–556.

REVELLY, A. K., BECKER, H. W., VISHWANADH, B., MANI KRISHNA, K. V., TEWARI, R., SRIVASTAVA, D., DEY, G. K., SAMAJDAR, I. & PANWAR, A. S. (2015). High-purity Zirconium under Niobium ion implantation: possibility of a dynamic precipitation? *Philosophical Magazine* **95**, 3727–3744.

RUGGLES, T. J., RAMPTON, T. M., KHOSRAVANI, A. & FULLWOOD, D. T. (2016). The effect of length scale on the determination of geometrically necessary dislocations via EBSD continuum dislocation microscopy. *Ultramicroscopy* **164**, 1–10.

RUGGLES, T. J., YOO, Y. S. J., DUNLAP, B. E., CRIMP, M. A. & KACHER, J. (2020). Correlating results from high resolution EBSD with TEM-and ECCI-based dislocation microscopy: Approaching single dislocation sensitivity via noise reduction. *Ultramicroscopy* **210**, 112927.

SCHWARZER, R. A., FIELD, D. P., ADAMS, B. L., KUMAR, M. & SCHWARTZ, A. J. (2009). Present state of electron backscatter diffraction and prospective developments. *Electron backscatter diffraction in materials science* 1–20.

SENEZ, V., ARMIGLIATO, A., DE WOLF, I., CARNEVALE, G., BALBONI, R., FRABBONI, S. & BENEDETTI, A. (2003). Strain determination in silicon microstructures by combined convergent beam electron diffraction, process simulation, and micro-Raman spectroscopy. *Journal of Applied Physics* **94**, 5574–5583.

S\LOWIK, J. & ZIĘBA, A. (2005). Silicon strip detector applied to X-ray diffractometer: Angular resolution and counting rate. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment* **551**, 73–77.

SMALL, K. A., CLAYBURN, Z., DEMOTT, R., PRIMIG, S., FULLWOOD, D. & TAHERI, M. L. (2020). Interplay of dislocation substructure and elastic strain evolution in additively manufactured Inconel 625. *Materials Science and Engineering: A* **785**, 139380.

SNEDDON, G. C., TRIMBY, P. W. & CAIRNEY, J. M. (2016). Transmission Kikuchi diffraction in a scanning electron microscope: A review. *Materials Science and Engineering: R: Reports* **110**, 1–12.

SOPER, A. K. (2011). *GudrunN and GudrunX: programs for correcting raw neutron and X-ray diffraction data to differential scattering cross section*. Science & Technology Facilities Council Swindon, UK https://epubs.stfc.ac.uk/manifestation/6710/RAL-TR-2011-013.pdf (Accessed December 4, 2023).

SRINIVASAN, N., BHASKAR, L. K., KUMAR, R. & BARAGETTI, S. (2018). Residual stress gradient and relaxation upon fatigue deformation of diamond-like carbon coated aluminum alloy in air and methanol environments. *Materials & Design* **160**, 303–312.

THOOL, K., PATRA, A., FULLWOOD, D., KRISHNA, K. M., SRIVASTAVA, D. & SAMAJDAR, I. (2020). The role of crystallographic orientations on heterogeneous deformation in a zirconium alloy: a combined experimental and modeling study. *International Journal of Plasticity* **133**, 102785.

TRIMBY, P. W. (2012). Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope. *Ultramicroscopy* **120**, 16–24.

VAN HOUTTE, P. & DE BUYSER, L. (1993). The influence of crystallographic texture on diffraction measurements of residual stress. *Acta metallurgica et materialia* **41**, 323–336.

VAUDIN, M. D., OSBORN, W. A., FRIEDMAN, L. H., GORHAM, J. M., VARTANIAN, V. & COOK, R. F. (2015). Designing a standard for strain mapping: HR-EBSD analysis of SiGe thin film structures on Si. *Ultramicroscopy* **148**, 94–104.

VERLINDEN, B., DRIVER, J., SAMAJDAR, I. & DOHERTY, R. D. (2007). *Thermo-mechanical processing of metallic materials*. Elsevier.

VILADOT, D., VÉRON, M., GEMMI, M., PEIRÓ, F., PORTILLO, J., ESTRADÉ, S., MENDOZA, J., LLORCA-ISERN, N. & NICOLOPOULOS, S. (2013). Orientation and phase mapping in the transmission electron microscope using precession-assisted diffraction spot recognition: state-of-the-art results. *Journal of microscopy* **252**, 23–34.

VINCENT, R. & MIDGLEY, P. A. (1994). Double conical beam-rocking system for measurement of integrated electron diffraction intensities. *Ultramicroscopy* **53**, 271–282.

WANG, Z. & GONG, B. (2002). Residual stress in the forming of materials. *Handbook of residual stress and deformation of steel* 141–49.

WILKINSON, A. J., DINGLEY, D. J. & MEADEN, G. (2009a). Strain mapping using electron backscatter diffraction. In *Electron Backscatter Diffraction in Materials Science*, pp. 231–249. Springer.

———— (2009b). Strain mapping using electron backscatter diffraction. *Electron Backscatter Diffraction in Materials Science* 231–249.

WILKINSON, A. J., MEADEN, G. & DINGLEY, D. J. (2006). High-resolution elastic strain measurement from electron backscatter diffraction patterns: New levels of sensitivity. *Ultramicroscopy* **106**, 307–313.

WINKELMANN, A. (2010). Principles of depth-resolved Kikuchi pattern simulation for electron backscatter diffraction. *Journal of Microscopy* **239**, 32–45.

WINKELMANN, A., TRAGER-COWAN, C., SWEENEY, F., DAY, A. P. & PARBROOK, P. (2007). Many-beam dynamical simulation of electron backscatter diffraction patterns. *Ultramicroscopy* **107**, 414–421.

WITHERS, P. J. & BHADESHIA, H. (2001). Residual stress. Part 1–measurement techniques. *Materials science and Technology* **17**, 355–365.

WOLF, I. D. (1999). Stress measurements in Si microelectronics devices using Raman spectroscopy. *Journal of Raman Spectroscopy* **30**, 877–883.

WRIGHT, S. I. & ADAMS, B. L. (1992). Automatic analysis of electron backscatter diffraction patterns. *Metallurgical Transactions A* **23**, 759–767.

WRIGHT, S. I., NOWELL, M. M. & FIELD, D. P. (2011). A review of strain analysis using electron backscatter diffraction. *Microscopy and microanalysis* **17**, 316–329.

YU, H., KARAMCHED, P., DAS, S., LIU, J., MIZOHATA, K. & HOFMANN, F. (2021). New perspectives on collision cascade damage in self-ion irradiated tungsten from HR-EBSD and ECCI. *Journal of Nuclear Materials* **554**, 153074.

YU, H., LIU, J., KARAMCHED, P., WILKINSON, A. J. & HOFMANN, F. (2019). Mapping the full lattice strain tensor of a single dislocation by high angular resolution transmission Kikuchi diffraction (HR-TKD). *Scripta Materialia* **164**, 36–41.

YUAN, H., BRODU, E., CHEN, C., BOUZY, E., FUNDENBERGER, J. & TOTH, L. S. (2017). On-axis versus off-axis Transmission Kikuchi Diffraction technique: application to the characterisation of severe plastic deformation-induced ultrafine-grained microstructures. *Journal of Microscopy* **267**, 70–80.

ZAEFFERER, S. (2007). On the formation mechanisms, spatial resolution and intensity of backscatter Kikuchi patterns. *Ultramicroscopy* **107**, 254–266.

ZHANG, T., COLLINS, D. M., DUNNE, F. P. & SHOLLOCK, B. A. (2014). Crystal plasticity and high-resolution electron backscatter diffraction analysis of full-field polycrystal Ni superalloy strains and rotations under thermal loading. *Acta materialia* **80**, 25–38.

ZHANG, T., JIANG, J., BRITTON, B., SHOLLOCK, B. & DUNNE, F. (2016). Crack nucleation using combined crystal plasticity modelling, high-resolution digital image correlation and high-resolution electron backscatter diffraction in a superalloy containing non-metallic inclusions under fatigue. *Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences* **472**, 20150792.

ZHAO, C. & LI, X. (2021). Quantitative Study of Residual Strain and Geometrically Necessary Dislocation Density Using HR-EBSD Method. *Experimental Mechanics* **61**, 1281–1290.

ZHAO, D., PATEL, A., BARBOSA, A., HANSEN, M. H., WANG, A. L., DONG, J., ZHANG, Y., UMALE, T., KARAMAN, I. & SHAMBERGER, P. (2023). A reference-area-free strain mapping method using precession electron diffraction data. *Ultramicroscopy* **247**, 113700.