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# Quantitative analysis of localized stresses in irradiated stainless steels using high resolution electron backscatter diffraction and molecular dynamics modeling

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ABSTRACT

Quantitative measurements of stress near dislocation channel–grain boundary (DC–GB) interaction sites were made using high resolution electron backscatter diffraction (HREBSD) and have been compared with molecular dynamics (MD) simulations. Tensile stress normal to the grain boundary was significantly elevated at discontinuous DC–GB intersections with peak magnitudes roughly an order of magnitude greater than at sites where slip transfer occurred. These results constitute the first measurement of stress amplification at DC–GB intersections and provide support to the theory that high normal stress at the grain boundary may be a key driver for the initiation of irradiation assisted stress corrosion cracks.



Austenitic stainless steel is a primary structural material and a candidate for generation IV reactors [1]. Understanding degradation and failure mechanisms in this alloy becomes increasingly important as the nuclear industry pushes the lifetime of current reactors and plans for the construction of new plants. A degradation mode of stainless steels that has been a growing problem for several decades is irradiation assisted stress corrosion cracking (IASCC). Due to the complex nature of dynamic processes during irradiation [2–4], isolating the fundamental mechanisms responsible for component failure by IASCC has been exceedingly difficult. However, in recent years localized deformation has emerged as a potential factor in the IASCC process [5]. The introduction of hard barriers such as dislocation loops, precipitates, and stacking fault tetrahedra (SFTs) under irradiation causes a change in deformation mode from homogeneous slip to localized heterogeneous deformation [6,7]. The first mobile dislocations partially annihilate these barriers [8], leaving a relatively soft region of material compared to the hard matrix. Subsequent dislocations move preferentially in these soft regions, confining a majority of the material deformation within narrow channels.

Jiao et al. [9] showed a strong correlation between the average weighted channel height and IASCC susceptibility. McMurtrey et al. [10] identified two different families of dislocation channel–grain boundary (DC–GB) interaction types: discontinuous channels where the dislocation channels were arrested at the grain boundary, and continuous channels where the dislocation channels were transmitted across the grain boundary into the adjacent grain. Cracking propensities for discontinuous DC–GB interaction sites exhibited a greater cracking fraction by a factor of six. The cause of this behavior is believed to be a high local tensile stress due to dislocation pile-up at the head of discontinuous channels. West et al. [11] showed that the inclination angle of cracked boundaries with respect to the loading axis for similar alloys to this study was heavily weighted towards 90 degrees, indicating grain boundary normal stress is an important factor for the crack initiation mechanism. Supporting evidence for this crack initiation mechanism was provided by Fukuya et al. [12] after investigating 316 stainless steel irradiated to 73 dpa in a fast reactor.

One explanation for the difference in cracking behavior is a difference in the grain boundary normal stress between continuous and discontinuous channels. To date, no quantitative measurements of the stress state near the channel–grain boundary interaction site have been made. Quantitative measurements of residual elastic stress can be made with high spatial resolution using a cross correlation technique developed by Wilkinson, Meaden, and Dingley called high resolution electron backscatter diffraction (HREBSD) [13]. Kikuchi patterns generated during traditional EBSD analysis will distort when the material is strained. By measuring changes in the Kikuchi pattern, the magnitude of the residual elastic strain can be determined, which can be used to calculate elastic stress. (For a detailed explanation of the technique see Ref. [14,15].) With HREBSD, quantitative stress measurements near

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discontinuous and continuous channels have been made. The resulting data has been compared and analyzed using molecular dynamics (MD) simulations.

Material used for the study was lab purity stainless steel produced by General Electric Global Research with a nominal composition of 70.36 wt.% Fe, 13.41 wt.% Cr, 15.04 wt.% Ni, 0.016 wt.% C, 1.03 wt.% Mn, and 0.10 wt.% Si that had undergone cold rolling and heat treatment (1200 °C: 2 h) resulting in a final average grain size of 25 μm. Tensile bars were electrical discharge machine (EDM) cut with a 2 mm × 2 mm cross section and a 21 mm gauge length. Samples were mechanically polished in stages from 320 to 1200 grit SiC paper. Each sample was electropolished for 60–90 s using a solution of 10% perchloric acid and 90% methanol at 30 V and −40 °C.

Samples were irradiated using 3.2 MeV protons at 360 °C to a total dose of 5 dpa with a dose rate of $1.0 \times 10^{-5}$ dpa/s at the University of Michigan Ion Beam Laboratory (MIBL). Dose was calculated at a depth of 20 μm using the quick Kinchin–Pease formulation in the SRIM simulation program [16] and a displacement energy of 40 eV. Ion irradiated samples were subjected to constant extension rate tensile (CERT) tests at a strain rate of $3 \times 10^{-7}$ $s^{-1}$ to a total plastic strain of 3% in a high purity argon environment at 288 °C.

An Olympus LEXT confocal laser microscope and an SEM were used to determine the location and type of formed dislocation channels (discontinuous or continuous). 20 nm colloidal silica solution was used to remove approximately 200 nm of material from the sample surface, which is the average height of the dislocation channels. EBSD scans using a Phillips XL30 FEG SEM and TSL OIM 5 software were performed near DC–GB intersections over an ~400 $\mu m^2$ area with a 100 nm step size. Kikuchi patterns collected during scans were analyzed offline with the CrossCourt3 (CC3) software package developed by BLG Vantage. The cross correlation software was run using 50 ROIs distributed uniformly across the reference pattern and single crystal elasticity coefficients for stainless steel [17]. All points with a calculated mean angular error by the CC3 program above $10^{-3}$ rad were removed from this analysis due to the large calculation errors for these points.

Confocal microscopy shows dark contrast at the location of dislocation channels (Fig. 1a). The location of this same region within the SEM is confirmed by overlaying a traditional EBSD orientation map on top of the SEM image, Fig. 1b. Surface steps have been removed entirely near DC–GB sites, allowing for high quality EBSD patterns to be collected near the location of dislocation channels. The removal of these surface steps is critical to EBSD pattern generation since their height (up to 600 nm) causes shadowing of the detector, preventing the acquisition of critical information. The green cross in Fig. 1b denotes the location where the EBSD pattern (Fig. 1c) was collected. A stress field, made by resolving the principle components of the CC3 generated stress tensor normal to the specific grain boundary plane where discontinuous channels intersect, is presented in Fig. 1d. The phenomenon of high tensile stress near an intersection site is visible for the three discontinuous channels in Fig. 1d (indicated with red arrows). Elevated stress values are localized to the point of DC–GB interaction and then dissipate rapidly over the first few microns into the adjacent grain. Shear stresses were also observable, but at values only ~30% of the tensile stress. Fig. 1e–h shows the same information for two continuous dislocation channels intersecting a grain boundary. Such a high stress is not observed at the continuous DC–GB intersection.

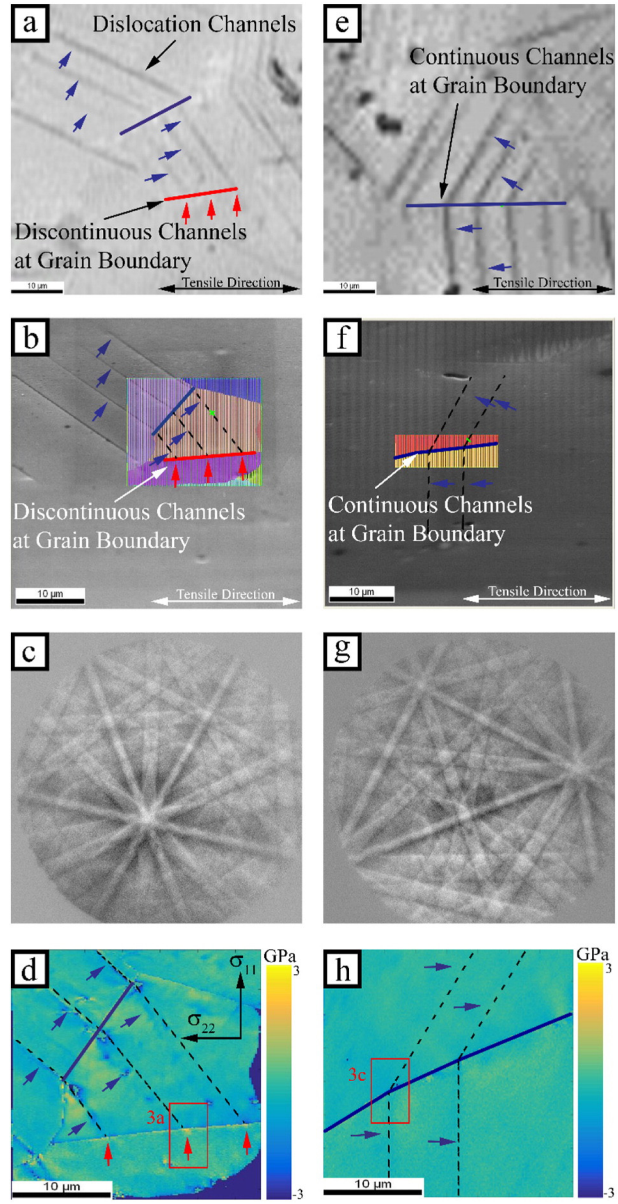


**Fig. 1.** Process for generating stress distributions using CC3 for a discontinuous DC–GB intersection. Confocal height profile (a), SEM with EBSD overlay (b), captured EBSD pattern near a channel with the green cross in 1b showing pattern location (c), and the CC3 calculated GB normal stress distribution for a discontinuous DC–GB intersection (d). Figure e–h shows the same progression for a continuous DC–GB intersection. Black dashed lines denote dislocation channel location, blue arrows show dislocation channels which are continuous at the grain boundary, and red arrows show dislocation channels which are discontinuous at the grain boundary intersection site. Red boxes show areas which are expanded for visualization in Fig. 3. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Digital samples were created to match the ones investigated experimentally. The deformation response was modeled using molecular dynamics (MD) and an embedded atom method (EAM) interatomic potential [10]. Five grains surrounding a specific DC–GB intersection with an average size of 120 nm and the same orientations as in the experimental sample were generated using the Voronoi construction method [18] to form a nano-structured thin film with a thickness of 9 nm. Relaxation, deformation, and quenching were simulated using MD with the LAMMPS [19] implementation and the EAM potential for Ni developed by Voter et al. [20]. All samples were relaxed for 150 ps to obtain equilibrium grain structure, then strained in tension with a strain rate of $3 \times 10^{8}$ $s^{-1}$ and a temperature of 300 K using a Nosè Hoover Barostat [21]. All MD was run with periodicity in the x and y directions with free surfaces perpendicular to the z direction. Following increments of 0.5% strain, samples were quenched from 300 K to 1 K in 50 ps to eliminate remnants of thermal stress. All stress measurements of digital samples reflect post-quench values. The 6-component stress tensor was estimated in LAMMPS as a sum of pairwise forces on each atom and the average volume occupied by a Ni atom in a FCC lattice. Dislocation slip lines were visualized by mapping the volumetric strain of each atom relative to the original, undeformed configuration. This value increases as neighboring atoms move away from one another due to slip, but are unaffected by