DEFORMATION LOCALIZATION AND DISLOCATION CHANNEL DYNAMICS IN NEUTRON-IRRADIATED AUSTENITIC STAINLESS STEELS*

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ABSTRACT

The dynamics of deformation localization and dislocation channel formation were investigated in situ in a neutron-irradiated AISI 304 austenitic stainless steel and a model 304-based austenitic alloy by combining several analytical techniques including optic microscopy and laser confocal microscopy, scanning electron microscopy, electron backscatter diffraction, and transmission electron microscopy (TEM). Channel formation was observed at ~70% of the polycrystalline yield stress of the irradiated materials ($\sigma_{0.2}$). It was shown that triple junction points do not always serve as a source of dislocation channels; at stress levels below the $\sigma_{0.2}$, channels often formed near the middle of the grain boundary. For a single grain, the role of elastic stiffness value (Young's modulus) in channel formation was analyzed; it was shown that in the irradiated 304 steels the initial channels appeared in "soft" grains with a high Schmid factor located near "stiff" grains with high elastic stiffness. The spatial organization of channels in a single grain was analyzed; it was shown that secondary channels operating in the same slip plane as primary channels often appeared at the middle or at one-third of the way between primary channels. The twinning nature of dislocation channels was analyzed for grains of different orientation using TEM. In the AISI 304 steel, channels in grains oriented close to $\langle 001 \rangle \parallel \text{TA}$ (tensile axis) and $\langle 101 \rangle \parallel \text{TA}$ were twin free and grain with $\langle 111 \rangle \parallel \text{TA}$ and grains oriented close to a Schmid factor maximum contained deformation twins.

Keywords: deformation localization, dislocation channeling, neutron irradiation, deformation twinning, spatial organization of channels

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1. Introduction

High-dose neutron irradiation and post-irradiation deformation of metallic polycrystals lead to deformation localization and the formation of dislocation channels [1]. It is generally accepted that the majority of plastic deformation occurs in dislocation channels and the characteristics of these channels depend on the type of material, grain orientation and grain size, and stress and strain levels [1, 2, 3]. Most often, channels are free or exhibit a reduced density of radiation- and deformation-induced defects; however, in some cases they contain dislocation debris at channel intersections [4] and dislocation pile-ups near grain boundaries. The mechanisms of defect-free channel formation are widely discussed in the literature, although conclusions on the full nature of the mechanisms remain inconclusive. For instance, channels formed in typical 300 series austenitic steels during deformation at relatively low temperatures may contain deformation twins [5] but also may be twin free; however, the influence of grain orientation in such cases was not studied in detail.

AISI 300-series austenitic steels are widely used in reactor materials in conventional nuclear power plants because of their outstanding mechanical formability, good high-temperature strength, and oxidation resistance [6]. However, these reactor materials are subjected to degradation due to a number of factors, including irradiation, stress, temperature, and coolant media. Among them, irradiation-assisted stress corrosion cracking (IASCC) is one of the most serious issues, and dislocation channeling was recently identified as a potential contributor to IASCC [2, 3]. It was found that stress-corrosion cracks began to form when the average channel height exceeded a critical value [3]. This finding is currently under further investigation as part of the Light-Water Reactor Sustainability Program (LWRS) of the Department of Energy [7].

Over the last decades, post-deformation analysis of the structure of deformed specimens has been the subject of many papers, and during that time, perfect examples of highly ordered and organized spatial structures were discovered [4, 8]. Several papers addressed the time evolution and dynamics of single channels [9, 10]. Much work has been done in trying to rationalize the formation and evolution of dislocation channels and their impact on the material performance [1, 2, 3, 11], but many aspects require additional attention, including the spatial distribution and evolution of the channels, origin sites, interaction with grain boundaries, etc. Some recently published papers demonstrate the complex, multi-scale character of deformation processes, which may include different phenomena like phase transformation, twinning, etc. Channel-grain boundary interaction may also be complex, leading to channel penetration or formation of a highly stressed area [11, 12]. During straining at room temperature, channel formation may be accompanied by specific phase transformations at the surface [13]. In addition to channel formation, areas with a high local strain level and significant local misorientation may form at the surface [14].

In the present work, steps are taken to holistically investigate channel dynamics during deformation of irradiated metallic polycrystals. By combining several analytical techniques, sites of channel appearance, spatial channel evolution, rate of channel propagation, and the fine structure of channels were investigated for austenitic alloys irradiated with neutrons up to 9.6 dpa.

2. Investigated Material and Experimental Methods

The materials used in this study were a high-purity commercial AISI 304 stainless steel (designated as SW-alloy) and 304-steel-based model alloy (designated as K-alloy); both materials were annealed prior to irradiation. The element composition and grain size are given in Table 1. Here, the alloy designations are retained since the alloys are referred to in other works as part of a cooperative research program on IASCC research [15] and were used in other studies [14, 16, 17]. During this program, more than 10 modified steels and alloys were produced, irradiated, and used across multiple institutions to characterize possible contributors to IASCC [18].

Specimens used in the present work were small 5×3.5 mm plates with a nominal thickness of 1.2 mm. The specimens were cut from the ends of non-deformed tensile bars that had been irradiated in the BOR-60 fast reactor at 593K at an average dose rate of $\sim 8 \times 10^{-7}$ dpa/s [18]. Stacking fault energy (SFE) was calculated using the equation provided in [19].

Prior to the deformation experiment, irradiated specimens were mechanically polished on both sides, removing ~ 200 μm of material from the surface using standard metallographic procedures. To get a defect-free surface, the specimens were then electropolished using a Struers unit with a standard A2 electrolyte. SW-alloy specimens were slightly etched with 10% oxalic acid at 6V DC to reveal grain structure and electropolished for 2 s to remove any etching products. Because of relatively small grain size (Table 1), the etching procedure was omitted for the K-alloy. The final thickness of the specimens was nominally 0.8 mm.

For electron backscatter diffraction (EBSD) and in situ deformation analysis, electropolishing may provide a better surface quality compared to colloidal silica polishing. For instance, Wynick and Boehlert [20] analyzed the effect of the specimen preparation method (mechanical polishing with alumina oxide and colloidal silica with or without subsequent electropolishing) on the quality of EBSD results. They suggested that mechanical polishing followed by electropolishing for a short time can produce better EBSD data (confidence indices and image quality) compared to mechanical polishing alone; removing the surface damage was more critical for EBSD than reduction of topography.

After specimen preparation, the flat irradiated specimens were subjected to a 4-point bend test using a specially designed bend assembly for sub-sized specimens (Figure 1). The span distance was 3.77 mm, with the diameter of the top and bottom rods being 0.4 and 1.23 mm, respectively. The assembly allowed deformation that produced the desired values of strain at the surface of the specimen.

Specimens were tested at room temperature on an MTS tensile screw-driven machine (model Insight 2-52; load capacity 2 kN). Mobile beam speed during bend tests was 0.1 mm/min. An Allied Vision Technology GX3300 digital camera was used to obtain images of the specimen surface before and after the bend experiment; the resolution was ~ 10 μm per pixel. VIC-2D commercial software and a custom program were used to calculate the strain field. The method, called optical extensometry or digital image correlation (DIC), is described in detail elsewhere [21, 22]. Using DIC, it was found that the final surface

strain values at the center of the specimens were 0.008 ± 0.002 (0.8%) and 0.006 ± 0.0015 (0.6%) for SW and K-alloys, respectively. The strain rate was estimated to be $\sim 10^{-4} \text{ s}^{-1}$ for both alloys.

Since tensile specimens were not available, yield stress values for the irradiated materials being investigated were defined using spherical indentation tests (an approach similar to [23] was used) and by populating a bend-tensile correlation curve for known materials with different strength levels. In the present work, the yield stress term corresponds to the conventional engineering definition, i.e. stress at which a 0.2% amount of macroscopic permanent deformation occurs ($\sigma_{0.2}$). The results (925 MPa for SW-alloy and 865 MPa for K-alloy) were confirmed by microhardness measurements using the correlation found in [24]. Thus, the expected inaccuracy in yield stress value is assumed to be below 10%. Note that the present work did not attempt any detailed discussion on property-property correlation [23, 24]; the only goal was to estimate yield stress level and conduct deformation experiments using objects of known histories and properties.

A Keyence VHX-1000 digital microscope running at 15 frames per second (fps) with a long focal lens was used to obtain images of the surface during the 4-point bend test experiments. The custom-built imaging system included a high-quality prism, which allowed in situ observation of the bottom surface of the specimen at a magnification of up to 1000 $\times$. There was no image shift during 4-point bending; only light dynamic focus correction was required during the experiment. Video recording obtained during the experiment was analyzed frame by frame using a custom program that allowed tracking of individual channels and analyzing their dynamics (propagation direction, rate, etc.).

The dimensions of the observed area were $\sim 400 \text{ }\mu\text{m}$ by $500 \text{ }\mu\text{m}$ ($\sim 0.20 \text{ mm}^2$) for SW-alloy and $\sim 250 \text{ }\mu\text{m}$ by $250 \text{ }\mu\text{m}$ ($\sim 0.06 \text{ mm}^2$) for K-alloy. The total number of grains in each region of interest was ~ 100 and ~ 150 grains for SW and K-alloys, respectively, with some of the grains containing multiple annealing twins. The higher number of grains within the smaller region is the result of the reduced grain size in the K-alloy. Grains located at the edge of the video frame were in most cases excluded from further evaluation. Small grains below a few microns were also omitted during analysis. For any in situ experiment, there always is some compromise between resolution and magnification on the one hand and observed area size and statistics on the other. In the present work, a decision was made to obtain more statistics (a hundred or so grains) at a moderate magnification in an attempt to balance these criteria.

Commercial finite-element analysis (FEA) software (COMSOL Multiphysics 4.0) was used to evaluate stress distribution in the deformed specimens. Since the 4-point bend experiment involved just elastic and small plastic strains, the 2D model (Figure 1) was employed to conduct the necessary calculations. Young's modulus for both materials and for parts of the bend assembly (top and bottom rods) was accepted to be 200 GPa, with friction effects being neglected. The main goal of the simplified simulation was to calculate the stress value at the surface of the specimen as a function of load value; detailed simulation of the load-bend curve and analysis of plastic strain distribution were not included in this work.

Scanning electron microscopy (SEM) was performed using a JEOL JSM 6500F microscope with a field emission gun (FEG) coupled with an EBSD system. The accelerating voltage was 20 kV, and the working distance was 12 to 15 mm. EBSD maps were measured on a hexagonal grid with a step size of 0.1 to

2 μm . The main goal of the EBSD analysis was to get grain orientation data and obtain grain boundary network maps for the specimens. EBSD-based analysis prior to the deformation experiment was used to check the annealed conditions of the materials and confirm the absence of pre- and post-irradiation cold work. TSL OIM 6.0 software was used to conduct EBSD data processing including the calculations of elastic stiffness. In the present paper, the term “elastic stiffness” will be used as a synonym and will replace the term “Young’s modulus” in the discussion of single-grain behavior.

Elastic constants (C_{11} , C_{12} , C_{44}) were accepted to be the same for both alloys. Teklu et al. [25] studied single crystals of Fe-Cr-Ni alloys and found that C_{11} changed at ~ 1.06 GPa per 1% wt. Cr and only at 0.16 GPa per 1% wt. Ni. The role of other elements (Mn, Si) is weakly explored in the literature, especially for irradiated steels. Therefore, possible alloying effects on elastic constants were neglected and will not be discussed here.

Epoxy replicas of the deformed surface were prepared to characterize deformation relief and to measure channel height. These non-radiological specimens allowed for easier handling and transportation compared to the parent samples. The measurements were conducted using an Olympus OLS4000 laser confocal microscope outfitted with a 405 nm laser providing nominal 10 nm depth resolution (Z). Comparing channeling dynamics data obtained with an optical microscope and 3D relief data, it was established that the optic system running at 600–800 $\times$ allowed one to observe and reliably track only channels with heights greater than ~ 40 nm. Smaller channels, as a rule, were not resolved during in situ testing. Special approaches (usage of large well-polished single crystals [9, 26], special lighting and registration methods, or modern confocal microscopes) may increase the sensitivity to a few nanometers; however, this is not a trivial task considering the handling procedures necessary for irradiated materials and the available sample geometries. The limitation on the smallest observable channel height should be kept in mind during the data analysis below.

Non-irradiated archive SW-alloy was not available, but a new heat with similar properties (composition, grain size, etc.) was fabricated to serve as an analog to the SW irradiated material. Using archived K-alloy and a non-irradiated SW-alloy, SS-J3 specimens were produced to investigate the plastic behavior of the materials prior to irradiation; however, analysis of these data is beyond the scope of the present paper. Unfortunately, due to small slip line height, the in situ tests with non-irradiated materials were not successful enough to provide slip line dynamics data.

Samples for transmission electron microscopy (TEM) were prepared from specific sites of interest using standard focused ion beam (FIB) lift-out procedures. FIB was utilized instead of traditional electropolishing techniques to provide site-specific, near-surface investigations of the deformed specimens. A FEI Quanta 3D 200i DualBeam FIB was used to create the cross-sectional FIB lift-out specimens. Specimens were mounted to chevron posts and welded in two corners of the lamella to prevent twisting and bowing from internal stresses due to the deformation procedure while thinning to the final thickness. Low-angle, low-energy milling was completed after thinning the specimens to electron transparency to reduce potential damage induced by the FIB.

Thinned FIB specimens were investigated using a Philips Tecnai T20 TEM operated in TEM mode with an accelerating voltage of 200 kV. Local microstructure was investigated in each grain by tilting the

specimen to the $[011]$ zone axis and $g=3\bar{1}1$. This configuration generates a rel-rod streak between the 200 and $1\bar{1}1$ diffraction spots allowing for simultaneous imaging of radiation induced Frank loops and deformation twins in dark field imaging mode. Channel structure was further investigated using $[011]$ zone axis and $g=200$ to image defect structure in bright field mode.

3. Experimental Results and Discussion

3.1. General Characterization of the Areas Investigated

Prior to the deformation experiment, the selected areas were characterized using EBSD. The EBSD-acquired crystallographic information included grain size, orientation, and grain boundary type (Figure 2, Figure 3). In both materials, many grains were relatively large ($\sim 60\text{--}80\text{ }\mu\text{m}$, and even more in SW-alloy) and contained multiple annealing twins. Clusters of small grains ($\sim 5\text{--}10\text{ }\mu\text{m}$) were observed in the structure of K-alloy (Figure 4).

Schmid factor and elastic stiffness values were calculated for the selected straining direction. The fraction of “hard” grains with a Schmid factor less than 0.35 was about 6% for the SW-alloy and only $\sim 1.5\%$ for K-alloy. “Soft” grains with Schmid factor ≥ 0.4 composed $\sim 80\%$ of the region of interest in the SW-alloy and $\sim 93\%$ in the K-alloy. This difference is caused by some texturing of the investigated materials ($\sim 1.6\times\text{random}$ in SW and $\sim 2.1\times\text{random}$ in K) for the given straining direction. SW-alloy specimens had an increased fraction of hard (low Schmid factor) and stiff (high elastic stiffness) grains compared to K-alloy specimens (Figure 2, Figure 3). Since both the Schmid factor and elastic stiffness values are sensitive to grain orientation, grains with the same Schmid factor may have different elastic stiffness values and a different amount of elastic energy. Grain oriented close to $\langle 111 \rangle \parallel \text{TA}$ (tensile axis) will have the lowest Schmid factor and highest elastic stiffness parameters. For reference, such grains appear as blue grains in the IPF-map in Figure 3.

3.2. Channel Formation during Loading

Figure 4 shows the investigated areas for both alloys. One can see that multiple channels formed during deformation. The first 20 channels, formed during deformation, are marked as #1W–20W and #1K–20K for alloys SW and K, respectively. Figure 5 shows the relationship between acting stress and channel appearance kinetics.

As follows from the results, the first channel (#1W, Figure 4) appeared in SW-alloy at a stress level of $\sim 670\text{ MPa}$ ($\sim 72\%$ of the yield stress) based on in situ video stream data and FEA results. Several reports indicated that the triple junction points (TJPs) are considered to serve as preferential origin points to first observed surface channels at high strain. In the present work where low strains were investigated, the first observed channel location in the SW alloy was not associated with a TJP. The first observed channel appeared at the middle of the grain boundary (MGB). Furthermore, channels #2W–4W in the SW-alloy also appeared at MGBs far from TJPs, indicating a mechanism(s) exists within this specimen to provide channel initiation points far from a TJP. One such mechanism may be stress concentration due to specific grain configuration; this possibility will be discussed below.

As the stress level increased to ≥ 800 MPa, channels in the SW-alloy began to appear at or near TJPs, as shown in channels #6W–8W (Figure 4). The number of channels as a function of stress increased steadily up to ≥ 800 MPa (Fig. 4) followed by a jump in channels due to the contribution of TJP initiation sites.

In K-alloy, the first channel (#1K) appeared at a lower stress level (~ 570 MPa or $\sim 65\%$ of yield stress) compared to SW-alloy (Figure 5). TJPs served as a channel source more often than the MGB locations in the K-alloy compared to the SW-alloy, as shown in Figure 4. In the SW-alloy, only 20 channels were observed to form inside the area below the yield stress level. In the K-alloy, ~ 60 channels were observed to form below the yield stress level (Figure 4), but only the first 20 were tracked in detail due to the complexity of the analysis. Note, the yield stress levels are those defined in Section 2 (925 MPa for SW-alloy and 865 MPa for K-alloy).

Figure 6 illustrates the appearance of a dislocation channel at the MGB in the K-alloy. One can see the channel (channel #15K in Figure 4) became visible in image #1 and propagated from the grain boundary to the middle of the grain in $\leq 1/15$ s. After that the channel reached an opposite grain boundary in image #2, taking $\sim 2/15$ s to cross the grain; its origin point may be traced exactly using EBSD-acquired grain boundary network data.

Kamaya et al. [27] conducted an FEA analysis of a polycrystalline aggregate consisting of 100 randomly oriented grains and determined that TJPs are not necessarily the points with the highest stress levels and that a maximum stress may be observed at a MGB. In the present work, it is difficult to define an exact reason for the formation of channels at MGB in both alloys. The difference in the fraction of initiation points of each type may be attributed to the different stacking fault energy (SFE) for these materials or may be caused by the smaller fraction of stiff grains in the K-alloy compared to the SW-alloy.

For both alloys, some delay was observed between the formation of first several channels and the remaining ones (Figure 5). For instance, channels #1–#3 in both alloys appeared relatively quickly, one by one, and the others formed only after an additional increase in stress. Most probably, initial channels formed at some specific sites where local grain configuration led to a high concentration of local stress, as discussed previously. The locations where channels appear (e.g., TJP or MGB) can depend on local grain configurations, and a detailed explanation of each case requires a micromechanics approach which combines EBSD grain orientation data with FEA [4, 27].

In the K-alloy, early channel formation was not observed in the clusters of small grains (Figure 4), and even at the final strain level (0.6%), the number of channels inside such clusters was limited. The first iteration may be explained by the well-known Hall-Petch relationship [28] – the cluster of small grains may have a higher yield stress than the surrounding volume of the material with regular grains.

Channel formation below yield stress was observed in many studies. For instance, Edwards et al. [29] conducted a detailed investigation of copper irradiated with neutrons and deformed at different strain levels. Defect-free channels were observed in the copper samples loaded at 270 MPa or 84% of macroscopic yield stress value (~ 320 MPa) [29]. Here, the formation of channels on the surface of the specimen was observed at $\sim 72\%$ for the SW-alloy and $\sim 65\%$ for the K-alloy.

The formation of channels below the yield stress might be controlled by local grain configuration near the channels. For example, the first channel appeared in a soft grain (one with high Schmid factor), which was neighboring a hard (low Schmid factor) grain, as shown in Figure 2 and Figure 3. Many initial channels in the SW-alloy also appeared in soft grains that shared a grain boundary with hard, stiff grains. The role of neighboring grains was analyzed by Sauzay [30] for a group of hexagonal grains of random crystallographic orientation; it was shown that depending on the orientation of neighboring grains, acting stress in a single grain may vary by approximately $\pm 18\%$, which means the possibility of plastic strain at $\sim 80\%$ yield stress, a value close to that observed experimentally in the literature and in this study when experimental uncertainties are taken into account.

An interesting question is what is the lowest stress at which one can observe the formation of channels? As noted above, the observation method used in the present work (visible light microscopy) had a clear resolution limitation. Channels with a height less than ~ 40 nm (roughly 150–300 passed dislocations) could not be recognized and tracked. Hence, dislocation motion and structure evolution may take place below the stress level reported previously if techniques with improved resolution limits are used.

Using FEA for modeling random polycrystalline aggregate with grains obeying anisotropic elasticity, Kamaya et al. [27] found that the maximum stress near grain boundary in the polycrystalline aggregate may be ~ 1.5 times greater than the applied external stress. This result means that the plastic strain in some grains and especially at the surface may be initiated at 66% of the yield stress. Such a result suggests that channel formation could have occurred at stress values lower than those stated here, but this cannot be verified using the techniques applied in the present work. Also, since FEA results are sensitive to the element size, the exact numerical values should be used carefully.

Additionally, it should be noted that the results discussed above were obtained analytically, by FEA, or experimentally for material loaded in a “harmless,” neutral, noncorrosive environment. The formation of earlier channels may be influenced by strain rate and surface and environmental conditions. Penetration of diffusing species, like hydrogen, also may influence stress distribution and early plastic flow. In particular, hydrogen accumulation may significantly impact deformation localization kinetics. Aubert et al. [31] have shown that ~ 135 wt. ppm of hydrogen increased the average slip height by a factor of 2 in polycrystalline non-irradiated 316L steel. At the same time, for ion-irradiated 304 steel, Miura et al. [32] found that slow-strain-rate ($\sim 10^{-7} \text{ s}^{-1}$) tensile testing in a hydrogen atmosphere will decrease the step height by factor of ~ 2 .

Using Gumbel statistics, it was suggested [27] that the maximum stress at the surface may be ~ 2.22 times larger than the applied stress. Thus, signs of plastic flow at the surface may be expected at 45% of the yield stress. Recently this expectation was confirmed experimentally; Fukuya et al. [33] investigated local strain distribution in tensile-deformed specimens of 73 dpa irradiated SUS316 stainless steel. Using EBSD, a clear change in local misorientation value was observed near grain boundaries after load at about half the yield stress. Hence, based on the results discussed above, one should expect that channeling will be initiated at about half the yield stress value.

3.3. The Role of Elastic Stiffness

As was noted above, many early channels (i.e., channels that appeared at stress levels below engineering yield stress) in the SW-alloy appeared in the grains located close to stiff grains with a high elastic stiffness value. Only ~7% of the grains in the SW-alloy had elastic stiffness value of 230 GPa or more, but four of the first channels to appear occurred in grains which neighbored such stiff grains (Figure 4). Thus, the role of elastic stiffness may be more important than previously thought, and analysis of the elastic stiffness parameter may be a suitable addition to the Schmid and Taylor factors.

A first-order approximation of the role of elastic stiffness can be determined by defining stiff grains as “hard inclusions.” For instance, Lasko et al. [34] investigated stress fields around hard inclusions using FEA; it was established that increased stress areas are located at the ends of the inclusion along the straining direction and decreased stress levels areas are located at the side.

Such a stress concentration scheme is a plausible explanation for why channels prefer to form near stiff grains (e.g., channels #1W and #4W in the SW-alloy). Under external load, areas of higher stress form at the ends of the grain along the straining direction and areas of lower stress appear at the sides of the stiff grain. The size of the areas subjected to stress concentration is comparable to the size of a stiff grain, allowing one to explain the origin points of channels #3W, #9W, #14W, #18W, and #19W in the SW-alloy (Figure 4), which are in close spatial relation to stiff grains.

In the K-alloy, the fraction of the stiff grains (>210 GPa) was less than that in the SW alloy (~15% vs. ~30%), as shown in Figure 2 and Figure 3. Moreover, most of the stiff grains were concentrated in a small grain clusters (Figure 4); therefore, the role of stiff grains in the K-alloy was not as pronounced as in the SW-alloy. Nevertheless, the role of elastic stiffness may be revealed by analyzing the local grain configurations. For instance, channel #1K in the K-alloy appeared in a large grain (> 40 μm) with a moderate stiffness value of ~180–210 GPa. This grain belonged to a chain of stiff grains oriented in the straining direction. This “stiff” chain was located between two groups of relatively soft grains (stiffness <150 GPa). One may expect that such a configuration would lead to a degree of overstress in the middle grain chain where channel #1K formed. Similar conclusions can be made for channel #2K. This channel appeared very close to a large grain with low (120–150 GPa) elastic stiffness.

The role of grain elastic stiffness and local grain configuration on mechanical behavior was analyzed by Sauzay and Jourdan [35]. It was shown that the neighbor grain configuration leading to the highest stress concentration in the middle grains corresponds to stiff neighbor grains aligned with the tensile axis but soft neighbor grains located perpendicularly to this axis.

Figure 7 shows the relationship between elastic stiffness and Schmid factor for both alloys. All of the data points are located along an arc-like line, which can be attributed to a strong relationship between the crystalline orientation of the grain and its elastic and plastic parameters. Channels below the yield stress (“earlier channels”) were observed only in grains with a Schmid factor greater than ~0.42.

However, in the SW-alloy a grain must have an elastic stiffness greater than ~100 GPa to allow the formation of channels at stresses close to the yield stress level. For the K-alloy, two pronounced clusters

were observed in Figure 7. The largest cluster includes not the softest grains but grains with high elastic stiffness (180 GPa or more). Such analysis supports the belief that in addition to the high Schmid factor, the formation of early channels requires a critical level of elastic stiffness. Given this, such conditions cannot be considered as a steadfast rule as earlier analysis showed that local grain configurations and neighboring grains also influence channeling in a particular grain. For example, Evrard and Sauzay [36] concluded that the effect of neighboring grains should be especially visible at low stress levels for which crystalline elasticity is dominant. Also, it may be speculated that Schmid factor and elastic stiffness, taken separately, are not enough to explain strain localization and predict the formation of earlier channels; a new metric may be required. For instance, an “effective Schmid factor” concept was offered in [30] to consider deformation processes in hard and stiff grains.

3.4. Twinning in Dislocation Defect-free Channels

Defect-free channels in irradiated austenitic steel or Fe-Cr-Ni alloys may have a complex structure. In addition, the channels may contain deformation twins or be twin free [5]. In general, twinning is an expected deformation mechanism for 300-series steels strained at room temperature [5]; as the temperature increases, twinning may be suppressed due to an increase in SFE. Twinning is usually not considered for slow-strain-rate tensile experiments (SSRT) in corrosion environment due to the relatively high temperature ($\sim 300^\circ\text{C}$) and small strain rate ($\sim 10^{-7} \text{ s}^{-1}$). However, while investigating irradiated 304 steel, Onchi et al. [37] observed twins in the specimens deformed at 290°C at $3.5 \times 10^{-7} \text{ s}^{-1}$ in an inert gas environment. Thus, twinning cannot be directly ruled out for high-temperature environments and may contribute to dislocation channel–grain boundary interactions.

Deformation twinning is known to be sensitive to grain orientation [38]; for instance, no twins were observed during tension in grains oriented close to $\langle 101 \rangle \parallel \text{TA}$ in 300-series steel [39] and Cr-Mn steel [38, 40]. However, the effect of grain orientation on twinning has been seldom explored for irradiated materials; it is possible that high acting stress can eliminate or reduce the effect of grain orientation and that deformation twins can form even in unfavorably oriented grains.

To analyze the influence of grain orientation on twinning in irradiated steel, the EBSD map obtained for the deformed SW-alloy specimen (Figure 2) was used to select several locations for detailed TEM investigations as the nature of the channels could not be accurately investigated using scanning electron microscopy (SEM)–EBSD. The SW-alloy was selected for this analysis since twinning was expected to be more pronounced in the SW-alloy than in the K-alloy due to the lower SFE. Locations were selected to provide information on channels in grains of different orientation with a low ($\langle 111 \rangle \parallel \text{TA}$, ~ 0.3), medium ($\langle 101 \rangle \parallel \text{TA}$, $\langle 001 \rangle \parallel \text{TA}$ ~ 0.40), and high (~ 0.5) Schmid factor.

For the selected locations, TEM showed expected radiation-induced Frank loops on $\{111\}$ planes with accompanying channels. The size and number density of the Frank loops were not investigated as they have been previously reported for this alloy system [41]. Bright field and dark field imaging of the specimens revealed a complex nature with several samples revealing twinning within the channel on the $\{111\}$ planes, as shown in Figure 8. Images in Figure 8 were taken between 1 and $4 \mu\text{m}$ from the free surface.

The analysis of the effect of grain orientation (Figure 9) demonstrated deformation twins present in grains oriented close to the $\langle 111 \rangle \parallel \text{TA}$ ($[111]$ -corner of the unit triangle) and in the grains oriented close to the Schmid factor maximum (#1). No twins inside of the dislocation channels were observed in grains oriented close to $\langle 001 \rangle \parallel \text{TA}$ and $\langle 101 \rangle \parallel \text{TA}$ (#2, #3 in Figure 9). A larger dataset was not collected due to the inefficiency of FIB techniques in providing high-throughput sample preparation, especially when large-grained materials are being investigated, such as the SW-alloy. Nevertheless, these results allow one to assume that, at least for the small strain region, twinning in irradiated stainless steel is sensitive to grain orientation.

Figure 8(c) shows an interesting case in which a dislocation channel penetrated the twin boundary. The top grain, G_1 , in Figure 8 (c) shows twinning (with the specimen tilted to highlight the twinning nature), while the lower grain oriented close to $\langle 101 \rangle \parallel \text{TA}$ (#3 in Figure 9) showed no twinning. Caution must be taken as the lower grain was not tilted for twinning diffraction, but a diffraction pattern of a selected area when the specimen was rotated on the $[011]$ zone axis showed no twinning reflections. The orientation of the twinning grain to the straining direction was unknown as it was not on the surface of the specimen and therefore not captured by EBSD analysis.

The behavior of the material may be different at higher strain levels, but the limited strain level achieved in the present work did not provide sufficient information for this to be confirmed. Non-irradiated steel of similar composition was analyzed in [39] using EBSD; no twinning was reported for $\langle 001 \rangle \parallel \text{TA}$ -grains even after straining at $\epsilon \sim 0.6$ and more. Also, since twinning is very sensitive to SFE [42], material composition variation could very well influence twinning after neutron irradiation; for 300-series steels, this possibility requires additional investigations.

3.5. Grain Orientation and Number of Active Slip Systems in a Single Grain

The number of active slip planes in individual grains was analyzed using optic and SEM images of the deformed surface. The criterion used to make this determination was that at least one dislocation channel must be clearly visible in an individual grain at a magnification of $600\times$ for the slip plane to be considered active. The analysis (Table 2) revealed that one slip plane appeared to be active in $\sim 50\%$ of the grains, two slip planes in $\sim 30\%$ of the grains, and three in $\sim 3\%$. A significant fraction of grains ($\sim 19\%$ in K and $\sim 9\%$ in SW) did not demonstrate visible channels. Grains without visible slip planes, as a rule, were smaller in size than their neighbors; these grains may contain channels with heights less than 40 nm. Also, some grains, which did not produce visible channels, could contain channels that were parallel to the specimen surface and thus invisible during optic or SEM observations.

Similar results (53, 37, 3, and 7%, respectively) were obtained by West [43] for 316L stainless steel irradiated by protons at 7 dpa and strained at a low strain rate to 5% at 400°C in a corrosion environment. It is interesting that different materials and irradiation and test conditions (strain rate and level, test, temperature, environment) did not significantly change the fractions of grains with a different number of the active slip planes when compared to the present work.

The number of active slip planes was also evaluated as a function of grain orientation to strain direction, and the results are shown in Figure 10. The SW-alloy grains oriented close to the $[111]$ corner of the unit

triangle have two active slip planes with high probability. The probability of one slip plane being visible is much higher for grains near the center and near the [101] corner. Grains near the [001] corner may have up to three active slip planes. It was expected that grains with a higher Schmid factor, located at the center of the unit triangle, will demonstrate more active slip planes; however, many such grains demonstrated just one active slip system. Grains without visible, active channels were distributed along the [001]-[111] line. It was expected that grains oriented close to $\langle 111 \rangle$ ||TA would have no slip lines at small strain since the Schmid factor for these grains is low. However, many such grains have one or two active slip systems, probably caused by channels in the neighbor grains.

In the K-alloy, grain orientation role was less pronounced except for grains oriented close to [001]-corner, which sometimes demonstrated three active slip planes. Note that the [111]-area in K-alloy was relatively depopulated due to some texturing (Figure 3).

3.6. Spatial Organization of the Channels in Single Grain

Many researchers noted perfect spatial organization of the channels and their uniform or almost uniform spatial distribution at the surface [3, 29] and volume [4]. For example, Tucker et al. [8] and Sauzay et al. [4] observed regular rectangular patterns in irradiated and deformed niobium [8] and 304 steel [4]. However, the dynamics of structure evolution and channel formation was not investigated in detail because of the use of ex situ analysis. In the present work, the number of channels in a single grain was usually small, one to three, because of the limited strain range. Nevertheless, some grains demonstrated a large number of channels and provided direct in situ data on the spatial organization of the channels.

Figure 11 shows the sequence of channel appearance in a typical grain. For the SW-alloy, one can see that channel #1 formed in this particular grain at a TJP which served as stress concentrator (this channel corresponds to the channel #10W in Figure 4). Note that channel nomenclature in the following analysis pertains to those labeled in Figure 11 unless otherwise stated. After that, channel #2 (or #14W in Figure 4) appeared approximately in the middle between channel #1 and the opposite grain boundary (A, B in Fig. 7). All following channels formed at the middle or one-third of the way between existing channels of the previous generation (ratios $\sim 1:1$ or $\sim 1:2$, as shown in Figure 11).

It is interesting to note that channel #3 in this SW-alloy grain appeared at a TJP and did not satisfy both $1:1$ and $1:2$ ratios. After passing $\sim 5 \mu\text{m}$, this channel demonstrated a double turn and crossed the grain approximately at the middle between #2 and opposite grain boundary. As the strain level increased, smaller channels formed and spacing (distance between channels) decreased. Finally, two small channels (#8 and #9) appeared from an opposite grain boundary, moved in the back direction, but did not reach the other grain boundary since deformation was interrupted. These channels demonstrated $\sim 1:2$ and $1:1$ spatial distribution ratios.

The K-alloy behaved in a manner similar to the SW alloy (Figure 11): after channels #1 and #2 appeared in the grain, channel #3 appeared one-third of the way between them. Channel #4 appeared at a slight variance to the perfect $1:1$ ratio. In general, the spatial organization is less pronounced in the K-alloy compared to the SW-alloy, which may result for a number of reasons, including smaller grain size, a larger number of twin boundaries and TJPs, or the effects of alloying.

The grain shape strongly influenced the appearance and degree of the spatial order. This effect appeared most clearly and was most pronounced in elongated grains whose long axis was at a 45 degree angle to the strain direction, as in Figure 11a. The channel ordering phenomenon seemed to be limited in the grains whose long axis was near parallel to the strain direction; however, grain orientation was not a key parameter influencing this phenomenon. In the SW-alloy, pronounced spatial organization was observed in different grains and did not seem to be obstructed even in relatively hard and stiff grains oriented close to $\langle 111 \rangle$ ||TA.

The spatial distribution phenomenon is also sensitive to the complexity of the grain and its neighbors; the more complex the grain shape (more TJPs and internal twins), the less pronounced the spatial organization of the channels appeared to be. Figure 12 shows a grain of complex shape with multiple internal twins and low-angle boundaries (see also Figure 4). In this grain, channels #1, 2, 3, and 5 formed at stress concentrators like TJPs and therefore did not obey the spatial distribution rule described above. Channel #4 appeared at approximately one-third of the way between the twin boundary and channel #1; however, the ratio is not perfect. Channel #6 demonstrated very interesting behavior that started at about one-third of the way between channels #1 and #4 but later made a turn and followed the $\sim 1:2$ ratio.

The histogram in Figure 13 shows the position of the next channel relative to the channels of the previous generation. In this figure, the value of “0.5” means an exact middle position (1:1 ratio), and “0.33” means a position one-third of the way between existing channels (1:2 ratio). In some cases, if a channel appeared between an existing channel and grain boundary, the distances could not be measured properly, so all such cases were excluded from the histogram. In other words, Figure 13 represents only data related to the interaction of channels. An exception was made for twin boundaries that were parallel to the channel being measured.

In the case of the present work, the statistical information was limited because of the small strain level and low density of the channels. Many grains contained just a few (three to five) channels, many of which were related to stress concentrators. Also, more fine channels, invisible with the used optic system, may exist in the structure, and it is not clear whether they followed the spatial distribution rule described above or not. Such fine channels may be revealed using post-test SEM, but the sequence of these appearances cannot be retrieved. Nevertheless, data shown in Figure 13 allow one to conclude that the ratios 1:1 and 1:2 are most often observed. It is interesting that a peak in the range of 0.35–0.40 does not correspond to an exact 1:2 ratio (0.33...) and is in better agreement with the “golden ratio” (1:1.618... or ~ 0.38). Only few exceptions were found (see point at the 0.15–0.20 range).

One of the driving forces of the spatial organization effect may be a back stress from dislocation pile-ups. Byun and Hashimoto [44] discussed this aspect and offered a relationship which allowed for estimation of the back stress level at a potential origin point, depending on the distance from existing pile-ups. In the simplest case, a minimum back stress level will be at the middle between two existing channels [44]. Thus, the results listed in [44] allow one to explain the frequent appearance of a new channel exactly between two existing ones. At the same time, the origin of the second spatial peak (~ 0.33) is not explained by the analysis conducted in [44] and hence another mechanism could be occurring.

Another driving force of the spatial organization may be considered. Sauzay et al. [4] considered a channel to be a soft thin layer located between two bulk and hard halves of an initial parent grain. In other words, a parent grain after channel appearance may be considered as two independent grains and may lead to the redistribution of stress and the appearance of secondary stress concentration areas at some specific locations. The approach used in [4] is not contradictory to the dislocation pile-up theory mentioned above; both approaches may be used.

Analyzing dislocation back stress, Mughrabi [45] discussed the stress level between two dislocation walls in non-irradiated deformed metal. The stress was measured directly via microscopy to determine the radii of curvature of dislocations (see [45] for detail). It was shown that the minimum back stress level is in the middle between two dislocation walls; however, the second weakly pronounced local minimum may be present at ~ 0.25 .

Neuhauser and Rodloff [26] investigated slip band development in irradiated copper single crystals using high-speed (up to 10,000 frames per second) video recording. It was demonstrated that channeling in single-crystal copper had a complex structure hierarchy: rough slip band clusters appeared on the surface of a single crystal with ~ 0.5 mm spacing. Each cluster consisted of several slip bands, and finally, each slip band included numerous very fine slip lamellas. In the case of the present work (deformed polycrystalline austenitic steel), just one structure level exists: single channels (slip bands in terms of [26]); the slip lamellas were not observed. It is interesting that for the case of the irradiated copper, slip bands in clusters demonstrated a spatial distribution similar to the present work (mainly 1:1 ratio); however, this specific aspect was not discussed in [26].

Ananthakrishna [46] summarized and analyzed current theoretical and experimental approaches to the investigation of dislocation collective behavior; it was shown that to describe highly organized structures in the deformed metallic materials, one may use a fractal dimension parameter. Such a parameter may show a connection between previous and the next generations of structure elements. For instance, Kleiser et al. [47] reported a fractal dimension of 0.5 in the scale 0.06–2 μm for deformed copper. The fractal dimension value of 0.5 means the next generation of structure elements will appear at the middle between existing ones. The fractal analysis approach may be very interesting and fruitful, but it is beyond the scope of the present work.

Also, since back stress may play a key role, it is important to investigate the spatial distribution of dislocations in the channel; however, a large dataset for such analysis does not exist. In particular, Kacher et al. [48] conducted in situ TEM deformation experiments using stainless steel samples irradiated with ions. Channel evolution and growth were observed in situ including spatial distribution of dislocations. As expected, dislocation density at the head of the growing channel was higher than that in the grain body behind the channel head.

Thus, as discussed above, channel appearance point in most cases is not random; channel appearance sequence may be considered as a highly ordered process, which depends on material parameters (composition, SFE), orientation of the grain of interest, and its neighbors.

3.7. The Duration of Channel Operation

How long a dislocation channel can operate to support plastic deformation remains an open question. There is no generally accepted point of view. Most studies focus on post-deformation analysis of the structure and cannot provide any information on the dynamics. In situ TEM experiments provide dynamics information on dislocation-defect interaction but are limited in strain range and deal with 2D objects. To get a detailed answer, one needs to conduct in situ experiments to observe and precisely measure the change in channel height and the evolution of channel profiles at the surface. Such techniques require complex experimental configurations.

First steps were taken here to determine what, if any value, in situ tracking using simple light microscopy could be used. This was completed by evaluating the change in contrast of channels as a function of elapse time. For instance, the visual contrast of the channel, shown in (pointed by black arrow), changed significantly from image #2 to #3 without changes in the lighting conditions. Thus, at first inspection it can be determined the channel operated for a few seconds after it reached the opposite grain boundary and until its activity was blocked by pile-up back stress.

Channel evolution time may be obtained from channel height measurements taken at different strain levels. It was an attractive and obvious idea to use optic in situ data to track the evolution of channel height. However, no correlation was observed between channel brightness in the optic light image and channel height in the laser confocal image. Channels with close height values demonstrated significant contrast and brightness variations depending on the light conditions (light source position, angle, etc.).

Nevertheless, it was observed that the brightness of many channels changed during the in situ experiment, whereas the brightness of other elements visible in the image (inclusions, grain boundaries) remained the same (Figure 6). Thus, channel brightness change could not be caused by a change in focus or lighting conditions. It was possible to conclude that the evaluated channel was operating at least during a few tens of seconds. Given this, the technique employed here only provided limited detailed insight into channel operation. It is suggested that future experiments employ techniques capable of tracking the channel site in situ such as the application of in situ laser confocal light microscopy.

3.8. Channel Propagation Rate

A frame-by-frame analysis of the video stream showed that fresh channels moved across grains at a relatively high rate. In ~30% of the cases, channels appeared and reached the opposite grain boundaries in less than 1/15 s (the time interval between frames). For these cases, the channel growth rate was estimated to be $\geq 500 \mu\text{m/s}$. In other cases, the channel movement across the grain could be visible at 2–4 frames (grow rate $\sim 100\text{--}300 \mu\text{m/s}$, see Figure 6).

For simple grains (i.e., grains without annealing twins or low-angle boundaries), grain size or distance from the origin point did not influence the propagation rate. Even in grains that were large enough (80–100 μm), channels moved at approximately the same rate when the distance to the channel origin point increased. There was no pronounced difference between the investigated materials. Also, no clear

relationship between grain orientation (i.e., twinned/twin-free channels), Schmid factor, or elastic stiffness and channel grow rate was established.

For complex grains (with internal twins or low-angle boundaries), the channel propagation rate, as a rule, decreased after the channel interacted with and moved through a twin boundary. Figure 14 demonstrates the dynamics of channel propagation through a complex grain. Once the channel appeared (point #1), it reached the A-point in 1/15 s or faster (propagation rate 150 $\mu\text{m/s}$ or more), but it took three frames (3/15 s) to reach the B-point, which corresponded to the propagation rate of $\sim 80 \mu\text{m/s}$. The rate at the BC-path was about 1 $\mu\text{m/s}$ instead of this is soft twin plate with a Schmid factor about 0.45 (Figure 2). The rest of the path (CD, DE, etc.) channel moved at an average rate of $\sim 3\text{--}7 \mu\text{m/s}$.

This channel (which is a channel #1W in Figure 4) passed six twin boundaries, reached an opposite high-angle boundary, and tended to pass it. The interaction of channels with grain boundaries is a very interesting topic [10, 12]; however, detailed analysis of channel-grain boundary interactions was beyond the scope of this study.

Interestingly, after reaching D and E points, the channel seemed to stop for $\sim 2\text{--}4$ s before penetration through the twin boundary. This time was required probably to accumulate enough dislocations at the pile-up to pass the twin boundary. In most cases, channels slowed after passing a twin boundary or low-angle grain boundary. This effect may also be seen in Figure 11 for the SW-alloy. Channels penetrated the twin boundary relatively easily but did not reach another grain boundary until the end of the experiment.

Published data for comparison purposes are limited, especially for irradiated steels. Some data are available on the irradiated copper; for instance, Rodloff and Neuhauser [9] used high-speed cinematography to study slip band development in neutron-irradiated copper single crystals. At a strain rate of $\sim 10^{-5} \text{ s}^{-1}$, the channel propagation rate in copper was found to vary from ~ 50 to $500 \mu\text{m/s}$. In general, these data are consistent with the results of the present paper (a few to $500 \mu\text{m/s}$); however, different material, irradiation conditions, and acting stress make a direct comparison difficult. Also, it is unclear how strain rate may influence the rate of channel propagation. It is difficult to expect a direct relationship since dislocation slipping in particular is a relaxation-driven process.

Conclusions

In the present work, an attempt was made to investigate the deformation localization as a complex, multi-scale phenomenon. Dislocation channel evolution was investigated in situ during straining of AISI 304 austenitic stainless steel and model 304-based high-nickel alloy irradiated with neutrons. The research was focused on the analysis of meso-scale level, on behavior of grains and group of grains. By combining several analytical techniques (high-resolution optical microscopy, laser confocal microscopy, SEM-EBSD, and TEM), the deformation processes at meso-level, the appearance and propagation of dislocation channels and surface structure evolution were studied in detail. The following conclusions may be drawn from this study:

- Dislocation channels were detected to appear at stress level 65%–70% of engineering yield stress (σ_y). Dislocation channels appeared not only at TJPs but also at the middle of grain boundaries. With stress increase, channel number in the K-alloy increased (model high-nickel alloy, high SFE) more quickly than in the SW-alloy (commercial 304 steel, low SFE).
- The elastic stiffness parameter (Young's modulus of a single grain) played an important role in the appearance of channels below the yield stress. In the SW-alloy with a high fraction of stiff grains, the initial channels appeared in soft grains (with a high Schmid factor) located close to stiff grains (with high elastic stiffness). In the K-alloy, with a small fraction of stiff grains, initial channels formed at specific grain configurations, where large soft grains caused overstress of their more stiff neighbors.
- Spatial organization phenomenon was observed and analyzed. It was demonstrated that the next-generation channels often appeared at specific positions between existing channels, and ratios 1:1 or 1:2 were the most common. It was suggested that the phenomenon may be caused by back stress from dislocation pile-ups.
- Fine structure of channels was analyzed using FIB-(S)TEM for grains of different orientation. It was shown that in 304 steels (SW-alloy) at a strain level of 0.8% the deformation twins presented inside channels in the grains oriented close to $\langle 111 \rangle // TA$, but not in $\langle 001 \rangle // TA$ nor $\langle 101 \rangle // TA$ grains.

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References

1. A. Arsenlis, M. Rhee, G. Hommes, R. Cook, J. Marian, *Acta Materialia* 60 (2012) 3748–3757.
2. G.S. Was, D. Farkas, I.M. Robertson, *Curr. Opin. Solid State Mater. Sci.* 16 (2012) 134–142.
3. Z. Jiao, G.S. Was, *J. Nucl. Mater.* 408 (2011) 246–256.
4. M. Sauzay, K. Bavard, W. Karlsen, *J. Nucl. Mater.* 406 (2010) 152–165.
5. T.S. Byun, E.H. Lee, J.D. Hunn, Plastic deformation in 316LN stainless steel – characterization of deformation microstructures, *J. Nucl. Mater.* 321 (2003) 29–39.
6. J.T. Busby, *J. Nucl. Mater.* 392 (2009) 301–306.
7. “Light Water Reactor Sustainability Program, Integrated Program Plan,” INL/EXT-11-23452, April, 2014.
8. R. P. Tucker, M. S. Wechsler, S. M. Orr, *J. Applied Physics*, 40 (1969) 400–408.
9. R. Rodloff, H. Neuhauser, *Phys. Stat. Sol. (a)* 49, 445 (1978) 445–454.
10. J. Kacher, I.M. Robertson, *Acta Materialia* 60 (2012) 6657–6672.

11. E.A. West, M.D. McMurtrey, Z. Jiao, G.S. Was, *Met. Mat. Trans A*, 43A (2012), 136-146.
12. M.D. McMurtrey, G.S. Was, L. Patrick, D. Farkas, *Materials Science and Engineering A* 528 (2011) 3730–3740.
13. M.N. Gussev, K.G. Field, J.T. Busby, *J.Nucl.Mater.* 446 (2014) 187–192.
14. K.G. Field, M.N. Gussev, J.T. Busby, *J.Nucl.Mater.* 452 (2014) 500–508.
15. P. Scott, *Materials Reliability Program: A Review of the Cooperative Irradiation Assisted Stress Corrosion Cracking Research Program (MRP-98)*, EPRI Report 1002807, 2003.
16. M.N. Gussev, J.T. Busby, L. Tan, F.A. Garner, *J.Nucl.Mater.* 448 (2014) 294–300.
17. Z. Jiao, G.S. Was, *J. Nucl. Mater.* 407 (2010) 34–43.
18. J.P. Massoud, P. Dubuisson, P. Scott, V.K. Chamardine, *CIR II Program: Description of the Boris 6 and 7 Experiments in the BOR-60 Fast Breeder Reactor*, EPRI Report 1011787, 2005.
19. Dai Qi-Xun, Wang An-Dong, *Chinese Physics*, 11(2002), 596–600.
20. G.L. Wynick, C.J. Boehlert, *Materials Characterization* 55 (2005) 190– 202.
21. K. Suzuki, S. Jitsukawa, N. Okubo, F. Takada, *J. Nucl. Eng. Design* 240 (2010) 1290–1305.
22. B. Pan, K. Qian, H. Xie, A. Asundi, *Meas. Sci. Technol.* 20 (2009) 1–17.
23. F. M. Haggag, R. K. Nanstad, J. T. Hutton, D. L. Thomas, R. L. Swain, “Use of Automated Ball Indentation to Measure Flow Properties and Estimate Fracture Toughness in Metallic Materials,” *Applications of Automation Technology to Fatigue and Fracture Testing, ASTM 1092*, A. A. Braun, N. E. Ashbaugh, and F. M. Smith, Eds., American Society for Testing and Materials, Philadelphia, 1990, pp. 188-208.
24. J.T. Busby, M.C. Hash, G.S. Was, *J. Nucl. Mater.* 336 (2005) 267–278.
25. A. Teklu, H. Ledbetter, S. Kim, L.A. Boatner, M. McGuire, V. Keppens, *Metallurgical and Materials Transactions*, 35A (2004) 3149 – 3154.
26. H. Neuhauser, R. Rodloff, *Acta Metallurgica* 22, (1974) 375–384.
27. M. Kamaya, Y. Kawamura, T. Kitamura, *Int. J. Solids and Structures* 44 (2007) 3267–3277.
28. R.W. Armstrong, *Materials Transactions*, 55 (2014) 2-12.
29. D.J. Edwards, B.N. Singh, J.B. Bilde-Sorensen, *J. Nucl. Mater.* 342 (2005) 164–178.
30. M. Sauzay, *Acta Materialia* 55 (2007) 1193-1202.
31. I. Aubert, J.-M. Olive, N. Saintier, *Materials Science and Engineering A* 527 (2010) 5858–5866.
32. T. Miura, K. Fujii, H.Nishioka, K. Fukuya, *J. Nucl. Mater.* 442 (2013) S735-S739.
33. K Fukuya, H. Nishioka, K. Fujii, T. Miura, Y. Kitsunai, *J. Nucl. Mater.* 432 (2013) 67–71
34. G.V. Lasko, Y.Y. Deryugin, S. Schmauder, D. Saraev, *Theor. Appl. Fract. Mec.* 34 (2000) 93–100.
35. M. Sauzay, T. Jourdan, *Int.J.Fracture*, 141 (2006) 431-446.
36. P. Evrard, M. Sauzay, *J. Nucl. Mater.* 405 (2010) 83–94.
37. T. Onchi, K. Dohi, N. Soneda, J.R. Cowan, R.J. Scowen, M.L. Castano, *J.Nucl.Mater.* 320 (2003) 194–208.
38. T.H. Lee, C.S. Oh, S.J. Kim, S. Takaki, *Acta Materialia*. 55 (2007) 3649–3662.
39. M.N. Gussev, J.T. Busby, T.S. Byun, C.M. Parish, *Materials Science & Engineering A* 588(2013)299–307.
40. I. Gutierrez-Urrutia, S. Zaefferer, D. Raabe, *Mater. Sci. Eng. A* 527 (2010) 3552–3560.
41. K.J. Stephenson, G.S. Was, *J.Nucl.Mater.* 444 (2014) 331–341.
42. T.S. Byun, *Acta Materialia* 51 (2003) 3063–3071.
43. E.A. West, Influence of local stress and strain on intergranular cracking of 316L stainless steel in supercritical water, Ph.D. Thesis, The University of Michigan, 2010.
44. T.S. Byun, N. Hashimoto, *J. Nucl. Mater.* 354 (2006) 123–130.
45. H. Mughrabi, *Acta Metallurgica* 31 (1983) issue 9, 1367–1379.
46. G. Ananthakrishna, *Physics Reports* 440 (2007) 113 – 259.
47. T. Kleiser, M. Bocek, *Z. Metalkde* 77 (1986) 587.

48. J. Kacher, G.S. Liu, I.M. Robertson, *Micron* 43 (2012) 1099–1107.

Table 1. Damage dose, stacking fault energy, element composition (wt.%)*, and average grain size for investigated materials

Alloy	Type	Dose, dpa	SFE, mJ/m ²	Mn	Si	Cr	Ni	Mo	C	N	Grain size, μm
K	Model alloy	9.6	53	1.00	0.03	18.21	25.08	0.02	0.02	0.0005	24
SW	Commercial 304 steel	4.4	22	1.07	0.24	18.42	10.45	n/d	0.022	0.025	67

* In both alloys: P<0.01%; S<0.01%; Ti < 0.02; Nb < 0.005.

Table 2. Fraction of grains with different number of active slip planes

Alloy	1 slip plane	2 slip planes	3 slip planes	No active planes
K	40%	38%	3%	19%
SW	60%	28%	3%	9%

Figure 1. Scheme of 4-point bend test (dimensions are in mm) and corresponding 2D finite-element model mesh used to determine stress and strain.

Figure 2. Irradiated SW-alloy. IPF-map, IPF coloration parallel to the tensile direction (a), Schmid factor (b), and elastic stiffness (GPa) (c), maps and texture plot (d) for the area of interest. Elastic stiffness map was calculated using the following constants: $C_{11} = 197$ GPa; $C_{12} = 125$ GPa; $C_{44} = 122$ GPa. #1S–#3S: initial dislocation channel formation directions.

Figure 3. K-alloy. IPF-map, IPF coloration parallel to the tensile direction (a), Schmid factor (b), elastic stiffness ($C_{11} = 197$ GPa; $C_{12} = 125$ GPa; $C_{44} = 122$ GPa) (c), maps and texture plot (d) for the area of interest. "Soft chain" marks a group of grains with relatively low elastic stiffness.

Figure 4. Direct optic image of the deformed surface superposed with grain boundary network defined by pre-strain EBSD (see also Fig. 2). Random high-angle grain boundaries (RHAB) – black; Random low-angle grain-boundaries (RLAB) – blue; twin (Σ) boundaries– red; CSL (Σ , $\Sigma 27$) – yellow. White arrows #1–#20 indicate the origin points and the propagation directions of the first 20 dislocation channels. For SW-alloy, the elastic stiffness values with respect to straining direction are shown for three stiff grains.

For K-alloy, dash ovals highlight clusters of small grains.

Figure 5. Integral number of channels in the field of view (Fig. 3) for the investigated alloys as function of stress. Filled symbols indicate cases where the channel origin points were located at the middle of the grain boundary and as a rule close to stiff grain (>210 MPa); open symbols indicate channels that started at triple junction points. Channel #8 in K-alloy marked by cross since this channel came into the area from an external (unobservable) source; this channel cannot be classified.

Figure 6. Appearance of channel at the middle of the grain boundary. K-alloy. Very weak channel became visible in image #1 (black arrow). Channel propagation direction shown by dash white arrow. To improve channel visibility, contrast was adjusted for the images in the left column. Grain boundary coloring scheme is the same as in Figure 4. Time interval between frames is 1/15 sec. Comparison of images #2 and #3 demonstrates that channel operated during some time as its contrast changed significantly. Dashed circles show visual details whose brightness remained the same.

Figure 7. Elastic stiffness vs. Schmid factor for grains with and without channels. Only the first 20 channels to form in the field of view are shown.

Figure 8. Microstructure of grains with dislocation channel. SW-alloy. a) Grain oriented close to $\langle 111 \rangle || \text{TA}$ (tensile axis) (see Figure 9 for detail). b) Grain with orientation close to $\langle 001 \rangle || \text{TA}$. White arrows show dislocation channel. White dash circle shows diffraction spot related to deformation twin. c) Channel (black arrow) penetrated Σ -grain boundary. Channel has internal twins in G_1 grain, but G_2 -portion is twin free (see diffraction image). Note, all samples were tilted in TEM to near the $[011]$ zone axis orientation to provide diffraction contrast images of deformation twins.

Figure 9. Orientation of deformed grains which contain defect-free channels with (filled circles) and without (empty circles) deformation twins. IPF parallel to the tensile direction ($[010]$).

Figure 10. Influence of grain orientation on number of active visible slip systems in K- and SW-alloys. Blue dot – 1; black triangle – 2; red filled circle – 3; and open circle – none. IPF parallel to the tensile direction ([010]).

Figure 11. Dislocation channels in a typical grain: a) optic image, 600 $\times$. Arrows #1 – #9 show origin point and propagation direction of the channels. Black lines show RHAB, thin solid blue lines show RLAB, red lines show twin Σ GBs, and yellow line shows Σ high-order twin boundary. b) scheme.

Figure 12. Channels formation sequence in a grain of complex shape (SW-alloy). Grain “as is” (left, optical image with high angle light used) and with marked grain boundaries (right). See Figure 4 for the color scheme. Black arrows indicate origin point and propagation direction of dislocation channels #1–#8; the numeration corresponds to the order of channel appearance. Channels in annealing twins are not numbered.

Figure 13. SW-alloy. Position of the next-generation channels relative to existing ones $D/(A+B)$, where $D=\min(A,B)$; see Figure 11.

Figure 14. Channel propagation rate in a complex grain. Twin boundaries are marked by dash lines (refer to Figure 4 for detail on grain boundary types).