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RADIATION HARDENING EFFECTS ON LOCALIZED DEFORMATION AND STRESS CORROSION CRACKING OF STAINLESS STEELS

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Abstract

Radiation hardening in austenitic stainless steels is shown to modify deformation characteristics and correlate well with an increased susceptibility to intergranular stress corrosion cracking (IGSCC). Available data on neutron-irradiated materials have been analyzed and correlations developed between fluence, yield strength and cracking susceptibility in high-temperature water environments. Large heat-to-heat differences in the critical fluence (0.2 to 2.5×10^{21} n/cm^2) for IGSCC are documented. In many cases, this variability is consistent with yield strength differences among irradiated materials. IGSCC correlates better to yield strength than to fluence for most heats suggesting a possible role of radiation-induced hardening (and microstructure) on cracking.

Microstructural evolution during proton and heavy-ion irradiation has been characterized in low-carbon 304SSs. Hardening results from a dispersion of dislocation loops in the matrix which increase in density and size with increasing irradiation dose. Scanning and transmission electron microscopy are used to examine dose, strain and temperature effects on subsequent deformation characteristics. This hardened microstructure produces inhomogeneous planar deformation within the matrix. Regularly spaced steps are created at the surface during deformation which increase in number with increasing macroscopic strain. Twinning is the dominant deformation mechanism at low temperature, while dislocation channeling is observed at 288°C. Deformation characteristics are discussed in terms of their potential impact on IGSCC behavior.

Introduction

Irradiation-assisted stress corrosion cracking (IASCC) remains an important concern in light-water reactor (LWR) core components. Despite an increase in research activity focussed on this phenomenon, there is no agreement how radiation exposure affects SCC susceptibility. Neutron irradiation is known to significantly alter material microstructure and microchemistry, accelerate kinetic processes, and produce changes in solution chemistry. These effects directly influence the local mechanics, reactivity and electrochemistry of the crack tip.

The development of a hardened microstructure may play a critical role in the intergranular (IG) SCC susceptibility of neutron-irradiated stainless steels. Yield strengths increase sharply over the same fluence range (0.5 to 2.0 X 10^{21} n/cm^2) where cracking susceptibility is observed. However, very little characterization of pertinent irradiated microstructures have been reported and no direct investigations of deformation behavior and SCC resistance have been performed.

The current work examines deformation characteristics and strength increases in irradiated austenitic stainless steels, and discusses their potential effects on IASCC. Available data from neutron-irradiation experiments have been compiled and analyzed to develop correlations between radiation hardening and cracking susceptibility. Radiation hardening is quantified by measurements of yield strength, while IGSCC susceptibility has been assessed by post-irradiation, slow-strain-rate (SSR) tests. Dislocation-defect interactions are examined in irradiated, low-carbon 304SSs after plastic deformation. Irradiation damage to levels between 1 and 5 dpa is produced by charged-particle bombardment at elevated temperatures. Scanning (SEM) and transmission (TEM) electron microscopy is used to characterize the influence of irradiation species (proton or heavy ion), temperature and dose on defect microstructures, and on subsequent deformation behavior.

Fluence and Radiation-Hardening Effects on IASCC

Recently, Bruemmer and Simonen[1] examined relationships among fluence, yield strength and SCC susceptibility. The most complete examples documenting neutron fluence effects on cracking were shown to be the SSR test results of Jacobs, et al.[2] and Kodama, et al.[3] on BWR-irradiated, commercial purity (CP) 304 SSs. Intergranular cracking was observed to increase sharply above a critical fluence level. Jacobs first detected cracking at a fluence of about 5×10^{20} n/cm^2, in good agreement with in-core, control blade sheath cracking.[4] Percent IG cracking in the SSR tests (strain rate of 3.7×10^{-7}/s, 32 ppm O_2) was used to indicate material susceptibility, and reached nearly 100% at a fluence of ~3×10^{21} n/cm^2. Kodama did not report significant IG cracking until a fluence $> 1.2 \times 10^{21}$ n/cm^2 and a fluence of ~6×10^{21} n/cm^2 was required to approach 100% IGSCC. Differences and similarities are documented in Figure 1 showing that the Kodama data appears to be simply

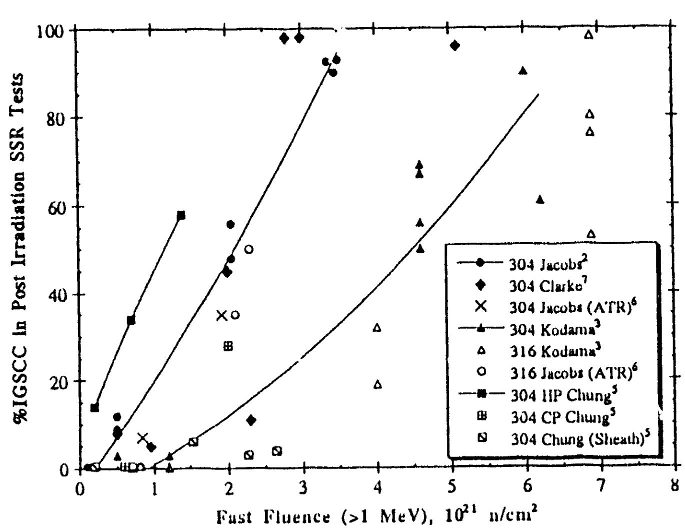

Figure 1. IGSCC Susceptibility of Irradiated Stainless Steels as a Function of Fast Neutron Fluence.

shifted to higher fluences. An accurate fluence to promote IGSCC is difficult to define, but is about 2×10^{21} n/cm^2. This 'critical' fluence is about 4 times higher than that found by Jacobs.

Significant differences in IASCC susceptibility were also seen among the three 304SS heats examined by Chung, et al.[5] Cracking was observed at a fluence as low as 0.2×10^{21} n/cm^2 for a high-purity (HP) 304SS, while a CP heat did not exhibit IGSCC even after a fluence of 2.5×10^{21} n/cm^2. Consistent with this heat-to-heat variability is the CP 304SS data of Clarke and Jacobs[7] and of Jacobs, et al.[8] showing isolated samples resistant to IGSCC at fluences above 2×10^{21} n/cm^2 while similar specimens failed by nearly 100% IGSCC. The comparison between the data of Kodama and that of Jacobs for 316 heats also shows an apparent difference in susceptibility. These differences in IGSCC response suggest that the "critical" fluence for susceptibility may vary by more than an order of magnitude among stainless steel heats.

Yield strength measurements[2,3,5-9] for various stainless steels are summarized as a function of irradiation dose in Figure 2. The 304SS (BWR) data of Jacobs[2] are the most complete and

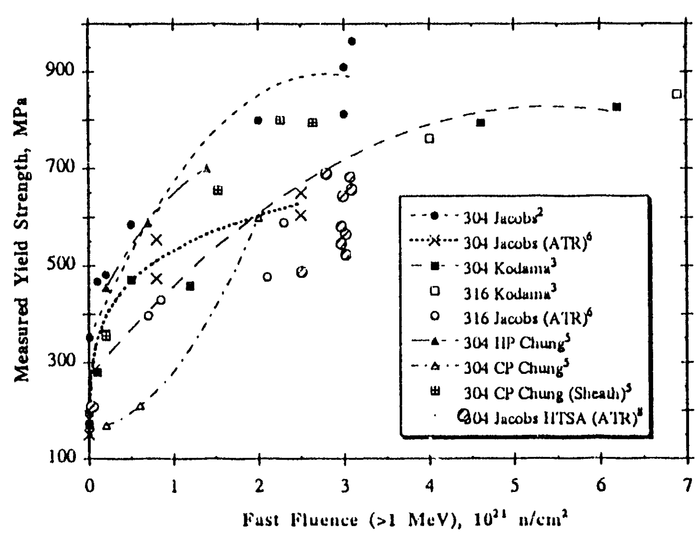

Figure 2. Measured Yield Strength for Neutron-Irradiated Stainless Steels as a Function of Fast Neutron Fluence.

are representative of the heats examined for IGSCC. A rapid rise in yield strength is observed with increasing fluence to about 900 MPa at 3×10^{21} n/cm^2 (~4 dpa). ATR irradiations tend to show lower strengths that reach about 70% of the BWR data. Several differences among the data sets can be detected. Although strength increases with dose, the BWR-irradiated 304 SSs that cracked at lower fluences tend to have higher strengths. For example, the IGSCC-susceptible HP heat of Chung exhibits yield strengths more than double the more resistant CP absorber tube material.

Yield strength measurements are compared to IGSCC results in Figure 3. Nearly all of the BWR-irradiated materials of Jacobs (304SS) and Kodama (304 and 316SS) fall very close to one another. Initial cracking in the SSR tests is seen only after the yield strength has increased to about 600 MPa (more than 3 times the typical value for annealed stainless steel). In

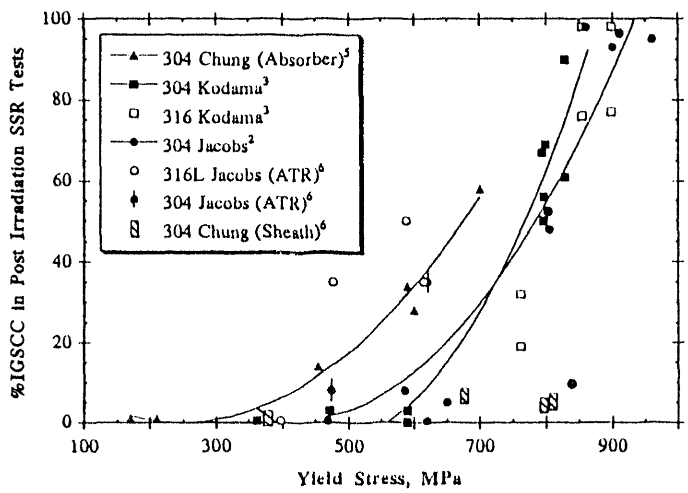

Figure 3. Comparison of Yield Strength to IGSCC for Neutron-Irradiated Stainless Steels.

addition, the two absorber tube heats (HP and CP) of Chung are now consistent with one another, reflecting the much higher strength and IG cracking measured for the HP material. However, the IGSCC versus strength curve is shifted to lower strengths with initial cracking observed at ~450 MPa even though the SSR test environment is less aggressive. Interestingly, the ATR-irradiated heats (304 and 316SS) also show cracking at lower strengths. One heat having very high strength and very little IGSCC is the CP 304SS sheath material of Chung. A large difference in the yield strengths required to promote IGSCC is evident between the sheath (800 MPa) and absorber (450 MPa) materials.

Many of the differences among data sets in Figure 1 are accounted for by comparing to the material yield strength in Figure 3. This indicates that the yield strength is a better measure of radiation damage than fluence. It also suggests that the radiation-induced hardening and microstructure may be playing a role in IASCC. Hardening alone does not appear to be sufficient to explain cracking susceptibility, but may act in combination with radiation-induced segregation.

Radiation-Induced Microstructures and Deformation

Deformation in Neutron-Irradiated Stainless Steels

Limited work has been conducted characterizing microstructural evolution during LWR irradiation with the majority of analysis performed for higher temperature and higher dose conditions.[10,11] The primary microstructural change during low-temperature (288°C), low-dose (<5 dpa) irradiation of stainless steels is the formation of small interstitial or vacancy dislocation loops. Typical densities appear to be on the order of 10^{16}cm^{-3} with sizes ranging up to ~20nm in diameter. Loop densities and sizes increase with dose, and Frank loops may eventually unfault to form a dislocation network. There was considerable effort 20 to 25 years ago to understand how dislocations interact with small loops and clusters produced by irradiation. In general, the small loops inhibit or retard dislocation motion thereby increasing the flow stress of the material (as documented in Figure 2).

A principal phenomenon that results from the interaction of small loops and moving dislocations is "dislocation channeling." In the channeling process, the initial dislocation annihilates and/or combines with the defects on the slip plane during glide. Subsequent dislocations will tend to glide along this same path, clearing out additional defects resulting in a channel free of defects. Such channels have been seen in a wide variety of materials, but primarily in pure metals.[12] Channeling tends to be restricted to those materials where the damage is in the form of a high loop density as opposed to a dislocation network.

Deformation characteristics in irradiated austenitic stainless steels may be somewhat unique due to their very low stacking fault energy. The stacking fault energy will be very important since it determines the propensity for cross slip and ease of jog formation, both of which are critical during dislocation-loop interaction and channel formation. Some early work reported deformation occurred in bands in irradiated and deformed 304SS indicating possible channel formation.[13] Inhomogeneous deformation of this type has been recently documented by Gorynin, et al.[14] in a stainless steel irradiated at 300°C to a fluence of 1×10^{21} n/cm^2 (E>1 MeV). Planar deformation was also observed by Suzuki, et al.[15] during in-situ deformation of proton-irradiated 304SS at room temperature. However, defect-free channels were not produced during deformation. Some other early work on deformation of an irradiated Cu-Al alloy with a very low stacking fault energy, however, indicated that channels were difficult to form.[16]

The evidence is very limited therefore as to whether dislocation channels actually form in stainless steels. If channels do not form, there must be some other deformation mechanism to account for the ductility that is observed. To better understand the mechanisms for hardening in the irradiated stainless steel, high-energy particle irradiation is being used to create a microstructure that is similar to that produced by neutron irradiation. The effect of a wide range of parameters such as dose, irradiation temperature, and material composition on deformation microstructure can, be efficiently studied using ion-irradiation techniques. Preliminary results examining deformation characteristics at 288°C in proton-irradiated material, and in nickel-ion irradiated material at room temperature, are described in the following sections.

Deformation in Proton-Irradiated 304SS

Experimental

High-purity, very low carbon 304SS (Fe-20.7Cr-8.9Ni-1.1Mn-0.09Si-0.003C) was cold rolled and heat treated in argon at 850°C for one hour to achieve a recrystallized grain structure with a grain size of ~10 μm. TEM and tensile samples were irradiated to 1 dpa with 3.4 MeV protons at 400°C. Following irradiation, the tensile samples were strained in tension to 3 and 9% total elongation at 288°C in argon. The post-deformation surface of the tensile specimens was characterized by scanning electron microscopy (SEM), while characterization of microstructural evolution during deformation was performed using a Philips EM400T transmission electron microscope (TEM). In addition, surfaces of tensile samples tested in 288°C water environments for SCC resistance[17,18] were also examined.

Observations

Deformed samples exhibit well-defined, and widely spaced, steps on the surface within individual grains. Steps are clearly visible even optically, and stand out as ledges in the SEM as shown in Figure 4(a). These steps corresponded to the presence of dislocation channels on <111> slip planes identified by TEM examination, Figure 4(b). The dislocation channels consist of multiple parallel planes in which one dislocation after another travels across the breadth of the grain to the opposite grain boundary. Dislocation pile-ups are observed at the boundaries without obvious slip transfer through the boundary. Defects and dislocation loops are no longer visible within many of the channels as illustrated in Figure 5, but isolated dislocations can be seen along the side walls of a channel.

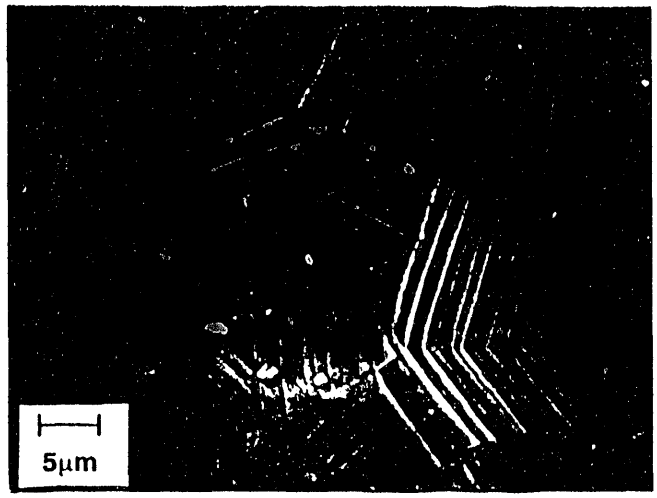

Figure 4(a). Surface appearance of proton-irradiated 304L SS after 9% strain at 288°C.

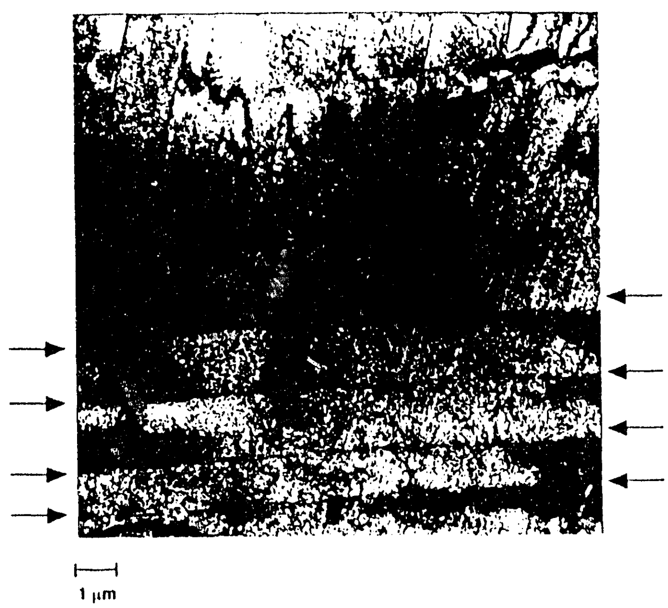

Figure 4(b). TEM microstructure of proton irradiated 304L SS after 9% strain at 288°C.

Each grain has one or two directions in which channeling has occurred, and at the intersections when two slip systems are active, cross slip is readily apparent. Cross slip is not visible in the 3% sample, though in some grains a second slip system is visible. Increasing the bulk deformation from 3 to 9% increases the density and width of channels and reduces their

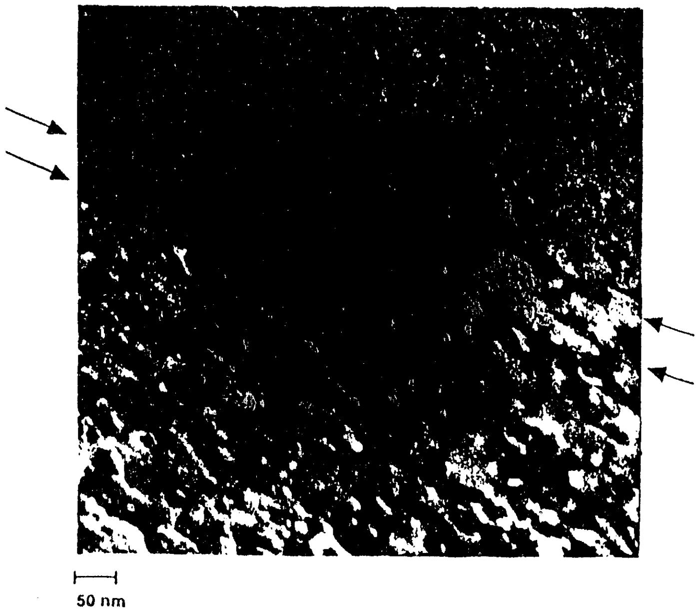

Figure 5. Dislocation channel in proton-irradiated 304L SS, deformed 9% at 288°C.

spacing. Isolated stacking faults are observed in the matrix between major channels for both samples. Adjacent to these channels on <111> planes are bands of ϵ martensite. The spacing and location of these bands of martensite suggest that it may result from the transformation of a heavily deformed region at the edges of the dislocation channels. Whether this transformation occurred during 288°C straining or during low-temperature TEM specimen preparation has yet to be determined.

Slip steps were also seen on the surfaces of SCC samples, irradiated at either 400° or at 200°C, and tested in 288°C water environments. The slip steps are more prominent in samples taken to higher strains before failure (up to ~20%), but the spacing of these steps did not increase significantly above that for the 9% sample. Thus, dislocation channeling appears to be the primary deformation mode prior to crack initiation and during crack propagation. This particular 304SS heat is highly susceptible to IGSCC after proton irradiation at 400 or 200°C even though radiation-induced segregation is limited.[17,18] This implies that a radiation hardening mechanism may be important in the onset of SCC.

Discussion

Defect-free regions are observed within the matrix deformation bands indicating that dislocation channeling does occur in 304SS under these irradiation and deformation conditions. Contrary to the low-temperature results of Suzuki et al,[15] interstitial loops have apparently been unfaulted, converted to mobile perfect loops and eliminated to form the defect-free channels. Current experiments examine a much more dense irradiation microstructure with smaller (>10X) loop sizes, and employ a deformation temperature that is 265°C greater than Suzuki's experiments. Since the irradiation microstructure and deformation temperature used in this work are similar to LWR conditions, it appears likely that dislocation channeling is the primary deformation mode during SSR-SCC tests of neutron-irradiated SSs described in Figure 3.

Low-Temperature Deformation in Heavy-Ion-Irradiated 304LSS

Experimental

Commercial purity 304LSS (Fe-18.6Cr-8.9Ni-1.8Mn-0.46Si-0.016C-0.083N) was prepared both as TEM samples and as miniature tensile samples approximately 17-mm long with a gage width of 3 mm. Heavily cold worked samples were heat treated in vacuum at 900°C for two hours to achieve a recrystallized grain structure with a grain size of ~30 μm. TEM and tensile samples were irradiated to a total dose of 2 or 5 dpa with 5 MeV Ni^{++} ions at 500°C. Following irradiation, the tensile samples were strained in tension to 5 and 10% total elongation at 23°C. The deformation specimens were then characterized by SEM and TEM techniques as described for the proton-irradiated material.

Observations

Heavy-ion irradiation to 5 dpa produced a dislocation loop density of approximately 3×10^{16} cm^{-3}. Dislocations were also present as loop tangles and short segments. A bimodal size distribution of loops was observed, with a significant density of small loops (less than 5 nm) and a high density of loops between 10 and 20 nm in diameter. The larger loops show black-white contrast under dynamic diffracting conditions, while the small loops (and defect clusters) exhibit "black spot" contrast.

SEM observations of the deformed surface of samples irradiated to 5 dpa show a high density of steps on the surface as illustrated in Figure 6. These steps change orientation across grain and twin boundaries, and have reasonably uniform spacings within individual grains. Actual spacings between steps varied from grain to grain. A primary difference between the 5 and 10% deformed samples is in the spacings of the steps. Step spacing after 5% deformation ranged from about 1 to 5 μm, approximately twice that for the 10% sample with spacings from 0.3 to 2 μm.

TEM examination of deformed samples revealed bands extending across the grains corresponding in spacing to the steps identified on the surface. The bands lie parallel to the

Figure 6. Surface appearance of ion-irradiated 304L SS after 10% strain at room temperature.

Figure 7(a). TEM microstructures of ion-irradiated 304L SS after 10% strain at room temperature.

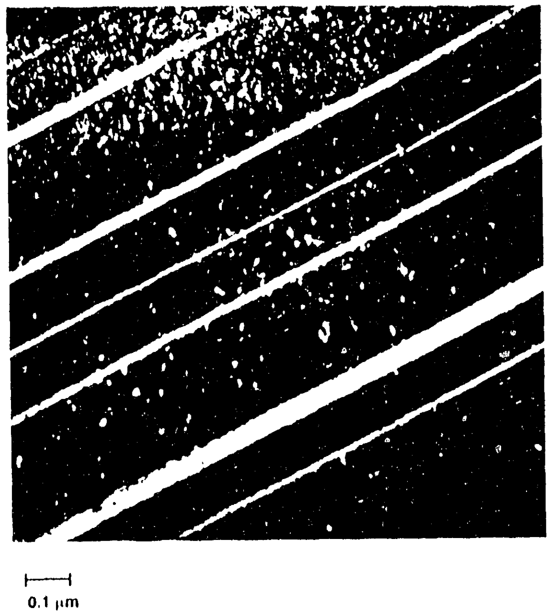

Figure 7(b). Deformation twins in ion-irradiated 304L SS, deformed 10% at room temperature.

slip planes and generally appear on more than one slip plane in an individual grain as shown in Figure 7. Some of the bands in the 5% deformed sample (Figure 7a) show through-thickness fringe contrast similar to extended stacking faults suggesting a planar nature. Many thicker bands do not exhibit this contrast and have been identified as deformation twins through diffraction spot and dark field analysis. Isolated stacking faults were also observed in the matrix between the bands. Planar faults were no longer evident as deformation was increased to 10%. All of the bands in this sample had finite thickness and were indexed as deformation twins on <111> planes. Twins were uniformly spaced in most grains and ranged in width up to ~20nm as illustrated in Figure 7(b). There also appear to be localized regions of ϵ martensite in the high-strain microstructure which may be formed during sample preparation.

Discussion

The twins observed in the 10% deformed material appear to originate from the narrow slip bands or channels seen in the 5% deformed material. Because of the inability of faulted dislocations to form extensive jogs, the dislocations are confined to the initial glide plane. The interaction of the faulted glide dislocations with the irradiation defects could produce some very effective dislocation locks, particularly at low temperature. Dislocation pile-ups would be expected with an increase in the localized stress. A point will be reached where it may be easier to nucleate a twin than to continue dislocation glide. The situation is analogous to nucleating twins in heavily deformed, unirradiated metals. Unlike dislocation channeling, twinning does not remove the loop damage on the twin plane. As shown by Venables,[17] a twin intersecting a perfect dislocation in the matrix converts it to a perfect dislocation in the twinned matrix. Twin dislocations do not become jogged by the intersection. The mechanism by which slip dislocations interact and absorb irradiation defects would not be applicable to twinning dislocations. Twin growth can be slowed and eventually stopped through the elastic interaction with the small dislocation loops, hence only narrow twins are observed. This is the general case for low stacking fault energy alloys where twin nucleation is easy, but growth is restricted due to the inhibition of the surrounding dislocations. Only fine deformation twins are commonly observed in these alloys.

The observation of channels with finite and observable width in the proton-irradiated material as compared to the nickel-ion irradiated SS could be due solely to the higher deformation temperature (288° versus 23°C). The temperature at which vacancy mobility becomes important in stainless steel is near 300°C. Increased thermal activation coupled with a lower strain rate should allow dislocations to move out of the glide plane forming wider channels. The lack of twins at 288°C could then be due to an inability to reach the critical stress necessary for twin nucleation. It is possible that e martensite is a more favorable transformation than twinning at the elevated temperature, but it must be established precisely when the martensite forms. Martensite can be formed in heavily deformed SSs by low-temperature exposure, but it is not known whether the -40°C temperature used during TEM specimen preparation is sufficient in this case.

Implications on IASCC Susceptibility

Radiation hardening in austenitic SSs results from the formation of small interstitial loops which effectively impede dislocation source operation and dislocation motion through the matrix. Bulk plasticity is limited to localized deformation within bands as illustrated by the charged-particle irradiations. The low stacking fault energy in 304SS makes the process of dislocation channeling difficult and promotes extensive twinning at low temperatures. A primary question is how this inhomogeneous deformation characteristics may influence IG failure. Manahan, et al.[20] has shown that IG cracking can be induced in highly irradiated SSs without an environmental component if the test strain rate is slow enough. However, it is well established that oxidizing high-temperature water environments are required for IG cracking at low-to-moderate fluences ($<3\times10^{21}$ n/cm^2).[3,20] Therefore, IASCC susceptibility may be a precursor to a more general IG embrittlement susceptibility in irradiated SSs.

Slip planarity has long been identified as detrimental to SCC and hydrogen embrittlement resistance by promoting dislocation pileups and high local stresses. Since grain boundary ledges are an excellent stress concentration site and a primary dislocation source, these interfacial regions can achieve very high stresses. Under tensile loading, several options are available at the boundary to accommodate these high stresses including emission of dislocations which must eliminate, bypass or cut through matrix defects to form slip bands, creation of deformation twins, or boundary cracking. Deformation studies on irradiated copper indicated that dislocation channels form during plastic deformation, but reach a saturation width and density at relatively small strains.[21] In other words, dislocation channels may have a finite life before pile-ups are created to shut down the source and there exists a minimum spacing between these sources. This behavior is similar to that observed in the irradiated 304SSs examined in this study. As available channels for plasticity are eliminated, local stresses may exceed the grain boundary cohesive energy and promote IG crack advance. The environmental effect on this cracking could result from sharpening the IG crack tip by dissolution (increases local stress) and by supplying corrosion-induced hydrogen to the boundary region (decreases interfacial cohesive energy). Both of these processes will be sensitive to the radiation-induced grain boundary composition.

Intergranular cracking may also be promoted by localized plasticity within or near the boundary plane. Since the grain boundary is a sink for radiation-induced defects, a narrow region (on the order of nanometers) surrounding the interface will be free of dislocation loops. Grain boundary sliding and active slip on <111> planes within this narrow zone may occur and could be enhanced by the presence of hydrogen. This process is more likely to occur in-core where continuous migration of vacancies and interstitials to grain boundaries will increase local diffusivities and dislocation mobilities. Localized plasticity of this type may act in concert with a decohesion mechanism to prompt IASCC. In both cases, hydrogen may play an important role. A more detailed interfacial characterization is necessary to determine how grain boundaries deform in irradiated SSs and to elucidate dynamic irradiation and deformation effects on dislocation activity.

Conclusions

Radiation hardening in austenitic SSs is shown to modify deformation characteristics and correlate well with an increased susceptibility to intergranular stress corrosion cracking (IGSCC). Large heat-to-heat differences in the critical fluence (0.2 to 2.5×10^{21} n/cm^2) for IGSCC are found to be consistent with yield strength differences among irradiated materials. IGSCC correlated better to yield strength than to fluence for most heats, suggesting a possible role of radiation-induced hardening (and microstructure) on cracking. Radiation-hardened microstructures produced by charged particle irradiation are shown to promote inhomogeneous planar deformation during plastic straining at 23 or 288°C. Regularly spaced steps are created at the surface during deformation which increase in number with increasing macroscopic strain. Twinning is the dominant deformation mechanism at low temperature, while dislocation channeling is observed at elevated temperature.

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