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**Irradiation Effects on Material Properties for  
304L Stainless Steel Base Metal and Welds**

***Final Report***

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## **Abstract**

A literature survey was conducted to understand the irradiation effects on the mechanical properties of 304L stainless steel, base, and weld metals. The objective was to identify the pieces of information that will allow for the assessment of suitability of data for the estimation of the end-of-life properties of CANDU calandria vessels after 60 years in service. The focus was on the magnitude of the fracture toughness of irradiated 304L stainless steel. Most of the data reported in the literature are for high irradiation and test temperatures that are conditions pertinent to fast reactors and light water reactors in which operating temperatures are above 350°C and 280°C, respectively. Based on the surveyed literature, one may infer that valid fracture toughness testing of 304 stainless steel specimens irradiated to dose of 4.1 dpa at temperatures 25 to 125°C, which are conditions that may be deemed close to those prevailing in the calandria, indicates that the heat affected zone (HAZ) of welds has a minimum fracture toughness  $K_{IC} \approx 108 \text{ MPa}\sqrt{\text{m}}$ . Fracture toughness tests not meeting all validity size requirements indicate that the HAZ may be the weakest part of the structure.

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## 1. Introduction

This report describes the work we performed in accordance to “Appendix A – statement of work” of the contract. We reviewed all papers on the list of references, which were provided by the Canadian Nuclear Safety Commission (CNSC), for content and applicability relative to base metal and welds under the temperature and irradiation conditions prevailing in the calandria vessel. We also performed a literature survey and found additional published papers that supplement data sets already used by the Canadian licensees. We studied the published data in an effort to draw conclusions concerning their adequacy in assessing the mechanical properties of the calandria vessel materials after 60 years of operation.

The CANada Deuterium-Uranium (CANDU) reactor is a type of nuclear power plant reactor designed to utilize natural uranium fuel in pressure tubes with heavy water as a coolant and moderator. Pressure tubes are enclosed within a large vessel called calandria. The calandria vessel is a horizontal, single-walled cylindrical vessel that contains heavy water moderator. Each end of the calandria is closed by a tubesheet. The heavy water inside the calandria is slightly pressurized and its temperature is controlled at about 60°C [1,36]. The calandria vessel components and tubesheets are made of 304L stainless steel, which is fabricated mainly by submerged arc welding process with 308L filler. During the reactor operation, all components including the calandria, the tubesheets, and welds experience high neutron doses and therefore accumulate irradiation damage.

Irradiation of steels by energetic particles creates microstructural defects by displacing lattice atoms from their original positions. Depending on the particle and its energy, the displacements may be either isolated, producing interstitial and vacancy pairs, or can be in the form of displacement cascades. In the latter case, interstitial and vacancy loops may form directly from the cascade event. Irradiation can cause compositional changes through processes such as radiation-induced segregation, thermally driven equilibrium and non-equilibrium segregation of alloying elements and impurities, dissolution and reformation of defects. Cumulatively, these processes can alter the mechanical properties as well as alter the susceptibility of the steel to other degradation processes such as irradiation-assisted stress corrosion cracking. Despite the accumulated knowledge about the consequence of the effects of irradiation on the properties of steels and other structural materials there remains a lack of correlation between irradiation and property change. Irradiation damage is usually measured in terms of displacement per atom (dpa) and can be calculated from the flux and the energy spectrum of neutrons. Prior to the use of dpa, which started in the mid-1970s, neutron fluence (total integrated flux) of fast neutron above a certain energy (typically 0.1 MeV or 1 MeV) was used as a measure to quantify radiation damage [2,3].

The maximum fluence (total time integrated flux) for tubesheets and welds inside the calandria vessel is expected to be about  $1 - 2 \times 10^{25}$  n/m<sup>2</sup> ( $E > 1$  MeV) after 60 years of reactor operation [1,36]. Exposing the base metal and welds to such a neutron irradiation fluence (1.5 – 3 dpa) increases the yield stress and decreases the ductility and fracture toughness of the material. The calandria vessel is one of the components of the CANDU reactor that is not easily accessible for non-destructive examination or testing after

installation. Since the calandria cannot be inspected, it should be designed to withstand the impact of irradiation damage with no required maintenance over a normal operating lifetime. In this report, we review the relevant literature in order to establish a basis for the estimation of the properties of the CANDU calandria vessel after 60 years of operation. Given that irradiation of austenitic stainless steels is a complex phenomenon whereby multiple factors such as environmental conditions can influence the mechanical properties, it is not possible to cover in this review all aspects of the irradiation effect by addressing the appropriate multi-dimensional parameter space. The review is focused on the effects of irradiation on the mechanical properties of austenitic stainless steels in a neutral environment, e.g. corrosion is not considered. The report is organized as follows: first we review the irradiation effects on fracture toughness or Charpy impact energy of 304 and 316 stainless steels. Second we present the results for the tensile properties of irradiated uniaxial tension specimens. Third we briefly summarize the microstructural evolution due to irradiation and discuss the remaining papers on the list supplied by the CNSC that are not relevant to the scope of the present work. Finally, we try to draw conclusions regarding the fracture toughness of the steels as fracture toughness is the key parameter that governs operation of the calandria vis-a-vis failure under load and irradiation.

## 2. Effect of irradiation on fracture resistance

The Charpy impact or the fracture toughness test is usually used to characterize the resistance of materials against fracture. The impact test is easy to conduct but it characterizes fracture resistance at high strain rates. It is mainly used to find the ductile-to-brittle transition temperature or as a screening test of the resistance of materials to fracture. On the other hand, although measurement of the fracture toughness through using cracked components is based on an elaborate protocol (ASTM E399-06 for linear elastic plane-strain fracture toughness; ASTM E1820-09 and ASTM E1737-96 for elastoplastic fracture toughness), fracture toughness characterizes mechanistically the material resistance to static loading.

Fracture toughness is the most appropriate property for design of mechanical components against fracture. Conservative fracture toughness estimates ( $K_{IC}$ ) are measured under small scale yielding conditions in plane strain.<sup>1</sup> To ensure such conditions that guarantee independence of the measured fracture toughness from specimen size, ASTM standard requires that all major specimen dimensions (crack length, uncracked ligament, and specimen thickness) be greater than  $2.5 \times (K_{IC}/\sigma_y)^2$ , where  $\sigma_y$  is the 0.2% offset yield stress of the material in tension. If the ASTM size requirement is not met, the test is not a valid  $K_{IC}$  test and a specimen with larger dimensions should be used for determining the fracture toughness. In the case of mild to medium strength steels with very large toughness, such conditions dictate the use of extremely large specimens, which are difficult to employ in irradiation experiments. Due to smaller size

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<sup>1</sup> The symbols  $K_{IC}$  and  $J_{IC}$  are used to denote fracture toughness estimates based on valid fracture testing. In the case of fracture toughness estimates from tests that do not meet the validity requirements, the symbols  $K_C$  and  $J_C$  are used.

requirements, elastoplastic fracture mechanics is used to measure the toughness ( $J_{IC}$ ) of such steels in terms of the  $J$ -Integral. The critical stress intensity factor  $K_{IC}$  can then be calculated from  $K_{IC} = \sqrt{(J_{IC} \times E / (1-\nu^2))}$ , where  $E$  is Young's modulus and  $\nu$  is Poisson's ratio. For 304 and 316 austenitic stainless steel,  $E = 193$  GPa and  $\nu = 0.29$ , and we assume that these moduli remain unchanged under irradiation. For the elastoplastic fracture mechanics approach, the requirement for valid test is that the major dimensions of the specimen should be greater than  $25 \times (J_{IC} / \sigma_y)$ . For example, an irradiated 304L stainless steel can have fracture toughness about  $K_{IC} = 200$  MPa $\sqrt{m}$  and yield stress about  $\sigma_y = 600$  MPa. In order to measure such a  $K_{IC}$  value, the ASTM standard requires that the dimensions of the fracture toughness specimen be greater than 27.7 cm. However, for a  $J_{IC}$  measurement, the specimen size requirement is reduced to 8 mm.

Data for the fracture resistance of irradiated austenitic stainless steels are rather limited [4]. Much of the data have been obtained at high irradiation and test temperatures under conditions pertinent to fast reactors in which operating temperatures are above 350°C. Since the mechanical properties of these steels depend on irradiation conditions and test temperature, extrapolation of information on mechanical properties from high temperature irradiation to low temperature irradiation conditions may not be legitimate, if at all possible.

Materials in terms of fracture resistance can be classified into three categories [71]. Category I includes materials with low fracture toughness, namely  $K_{IC} < 80$  MPa $\sqrt{m}$  ( $J_{IC} < 30$  kJ/m<sup>2</sup>). At these toughness levels, fracture can take place at or below yield stress loadings for relatively small flaw sizes. Category II includes materials with intermediate toughness  $J_{IC}$  in the range of 30 – 150 kJ/m<sup>2</sup> ( $K_{IC} = 80 – 180$  MPa $\sqrt{m}$ ). In these materials, fracture can occur by stable or unstable crack propagation close to yield stress loadings for components containing small to medium crack sizes. Category III includes high toughness materials with  $J_{IC} > 150$  kJ/m<sup>2</sup> ( $K_{IC} > 180$  MPa $\sqrt{m}$ ). Fracture in these materials generally takes place with stable tearing at stresses well above the yield stress.

Non-irradiated austenitic stainless base metals and welds are extremely resistant to fracture and usually fall into category III. The mean fracture toughness of 304 and 316 base metal is  $J_{IC} = 672$  kJ/m<sup>2</sup> ( $K_{IC} = 375$  MPa $\sqrt{m}$ ) at temperatures up to 125°C. The fracture toughness of welds greatly depends on welding process and less on composition. For example, for a given weld process, the 304 welds with 308 filler and 316 welds with 16Cr-8Ni-2Mo filler showed a similar toughness range indicating that fracture toughness is independent of filler metal. Among welds fabricated using different processes, gas tungsten arc welds (GTAW) have the highest toughness and submerged arc welds (SAW) the lowest. Mean  $J_{IC}$  for SAW is about 147 kJ/m<sup>2</sup> ( $K_{IC} \approx 175$  MPa $\sqrt{m}$ ) whereas this value for GTAW is 492 kJ/m<sup>2</sup> ( $K_{IC} \approx 320$  MPa $\sqrt{m}$ ). Examination of fracture toughness of heat affected zones (HAZ) and welds revealed that usually the HAZ are tougher than welds. Therefore, weld fusion zones are typically the weakest regions in welded joints [71].

In general, the toughness of neutron-irradiated austenitic stainless steels is significantly reduced such that irradiated stainless steels may fall into category II or even I. Figure 1 shows the effect of neutron dose on the fracture toughness  $J_{IC}$  of 304 and 316 stainless steel base metals irradiated in fast reactors at 350 – 450°C [27]. Below irradiation dose of 1 dpa, the loss of fracture toughness is very small, but it gets rapidly

larger with increasing dose in the range of 1 – 10 dpa. At doses above 10 dpa, the fracture toughness seems to asymptote to  $J_{IC} \approx 30 \text{ kJ/m}^2$  ( $K_{IC} \approx 80 \text{ MPa}\sqrt{\text{m}}$ ). Figure 2 shows the fracture toughness of 304 and 316 welds irradiated at 370 – 430°C as a function of irradiation dose [71]. As for the base metal, the fracture toughness of the welds, does not change significantly for doses up to 1 dpa. However, for doses beyond 1 dpa, the fracture toughness of the welds diminishes more rapidly than that of the base metal. For example, for annealed 304 plates and 308 welds irradiated at 427°C to doses in the range of 8.2 – 12 dpa and tested at the same temperature, the fracture toughness of irradiated 304 plates and 308 welds are  $J_{IC} = 100 \text{ kJ/m}^2$  ( $K_{IC} \approx 145 \text{ MPa}\sqrt{\text{m}}$ ) and  $J_{IC} = 30 \text{ kJ/m}^2$  ( $K_{IC} \approx 80 \text{ MPa}\sqrt{\text{m}}$ ), respectively [73]. With increasing dose, the weld toughness levels to minimum values similar to those observed for the irradiated base metal ( $J_{IC} \approx 10 - 30 \text{ kJ/m}^2$  or  $K_{IC} \approx 45 - 80 \text{ MPa}\sqrt{\text{m}}$ ) [71].

Lucas [4] pointed out that the non-irradiated solution annealed (SA) stainless steels have high fracture toughness ( $K_{IC} > 200 \text{ MPa}\sqrt{\text{m}}$ ) but irradiation at high temperatures (290 – 430°C) and with increasing neutron dose decreases  $K_{IC}$  rapidly to the low value of 50  $\text{MPa}\sqrt{\text{m}}$ , which is attained at doses greater than about 10 dpa as shown schematically in Fig. 3. It is also reported by Lucas that welds have lower non-irradiated toughness and appear to embrittle more rapidly, but their toughness levels with increasing dose to minimum values similar to those observed for the irradiated SA alloys. The cold worked (CW) 316 steel irradiated in the temperature range of 370 – 430°C has on average a lower fracture toughness than the SA alloy in the non-irradiated condition ( $K_{IC} \approx 200 \text{ MPa}\sqrt{\text{m}}$ ). Irradiation appears to decrease the fracture toughness of CW steels, though at a lower rate, to an asymptotic value of 80  $\text{MPa}\sqrt{\text{m}}$  [4] at large doses.

Magnitudes of fracture toughness of 304 and 316 stainless steels irradiated and tested at 288°C under conditions for light water reactor (LWR) environment seem to fall within the scatter band for the data obtained at higher temperatures (Fig. 1) [27]. The materials were irradiated at 288°C to 0.3, 0.9, and  $2.0 \times 10^{25} \text{ n/m}^2$  ( $E > 1.0 \text{ MeV}$ ) equivalent to 0.45, 1.35, and 3.0 dpa, respectively. The fracture toughness magnitudes for the irradiated steels are  $J_{IC} = 507, 313, \text{ and } 188 \text{ kJ/m}^2$  ( $K_{IC} \approx 325, 255, \text{ and } 200 \text{ MPa}\sqrt{\text{m}}$ ), respectively. The fracture toughness was also measured for the HAZ of 304 welds with 308 fillers irradiated in LWR at about 290°C and tested at the same temperature. The reported fracture toughness for HAZ of SAW and shielded metal arc weld (SMAW) irradiated to dose 2.15 dpa are almost the same:  $J_{IC} > 100 \text{ kJ/m}^2$  ( $K_{IC} > 145 \text{ MPa}\sqrt{\text{m}}$ ) [74,75].

Irradiation and testing at higher temperatures ( $> 375^\circ\text{C}$ ) showed a similar decrease of fracture toughness that was sometimes accompanied by a change in the fracture mode. Despite the fracture mode change for such higher temperature irradiation ( $> 375^\circ\text{C}$ ), it has been suggested that the fracture toughness  $K_{IC}$  is proportional to the square root of the uniform elongation  $e_u$  in uniaxial tension multiplied by the flow stress  $\sigma_{flow}$  (average of the yield stress and the UTS) such that [41,59,66,67]

$$\frac{K_{IC}^{Irr}}{K_{IC}^{Nirr}} = \sqrt{\frac{e_u^{Irr} \sigma_{flow}^{Irr}}{e_u^{Nirr} \sigma_{flow}^{Nirr}}} \quad (1)$$



where the superscripts Irr and Nirr denote irradiated and non-irradiated material properties, respectively. However, there is no underlying mechanistic rationale for such a relationship. It has further been argued on the basis of limited fracture toughness data between 250 and 300°C and low doses (< 3 dpa) that the proportionality continues to hold at this temperature range as well [41]. Application of this proportionality relationship to stainless steels irradiated at higher doses (> 7 dpa) and in the temperature range of 250 – 300°C predicts fracture toughness less than 50 MPa√m. Again, this extrapolation relies on the relationship (1) between fracture toughness and tensile ductility, a relationship for which there is no underlying mechanistic basis as explained in the following.

Relationship (1) is obtained on the basis of a critical strain model for crack initiation [63]. This critical strain model states that  $K_{IC} = C\sqrt{\sigma_0 \varepsilon_f^* l_0^*}$  where  $C$  is a constant  $\sigma_0$  is the yield stress, and  $l_0^*$  is a characteristic distance ahead of the crack over which the plastic strain is higher than the critical fracture strain  $\varepsilon_f^*$ . This model is used to explain the fracture toughness of ductile materials failing by microvoid nucleation, growth and coalescence. In order to derive relationship (1), one needs to assume that  $\varepsilon_f^{*Irr} / \varepsilon_f^{*Nirr} \approx e_u^{Irr} / e_u^{Nirr}$  and that irradiation does not affect the constant  $C$  and the characteristic distance  $l_0^*$ . However, there is no fundamental mechanism supporting either of the assumptions. Irradiation changes the microstructure so it can affect  $l_0^*$ , and as for the scaling of the fracture strain, the material ahead of a crack tip experiences a triaxiality constraint that is extremely large in comparison to that developing in uniaxial tension. The stress triaxiality, a measure of constraint ahead a crack tip, is about 3 whereas in uniaxial stress deformation is 1/3. In order to show the effect of the triaxiality constraint on the deformation behavior, the result for a void growth simulation in a material subjected to different triaxiality levels is illustrated in Fig. 4. If one assumes that the coalescence takes place at certain amount of void growth, the corresponding macroscopic plastic strain under high constraint ( $T \approx 3$ ) and low constraint ( $T \approx 1/3$ , not shown) can differ by an order of magnitude.

One of the very few extensive investigations of low temperature irradiation of stainless steels is the study performed on the 1950's vintage, rolled and welded stainless steel pipe for which the effect of irradiation on mechanical properties of the base metal, welds, and heat affected zone was quantified [5,17,18,19,21,70]. The material was 304 type stainless steel welded by metal inert gas process with 308 filler. The average ferrite level for welds was between 10% and 15%, which is relatively high for stainless steel welds. Tensile tests on the non-irradiated materials showed no orientation dependency. Charpy test and fracture toughness results, however, demonstrated strong dependence on the orientation of the specimens [17,18]. The value of Charpy energy and fracture toughness of the base material and the HAZ for L-C orientation specimens (specimen along the axial direction of the pipe with a crack growing along a circumferential direction of the pipe, see Fig. 5) were higher than those for specimens with C-L orientation (crack growing along the axial direction). The orientation dependency was attributed to the presence of  $\delta$ -ferrite stringers parallel to the pipe axis (rolling direction). The HAZ specimens in the C-L orientation had the lowest energy absorption and fracture

toughness among all non-irradiated specimens. The minimum value reported for HAZ fracture toughness before irradiation was about  $180 \text{ MPa}\sqrt{\text{m}}$  (not a valid fracture toughness test though). The fact that no orientation dependence was observed in tensile tests but strong orientation dependence is the case of toughness tests demonstrates—in addition to the reasons mentioned in the preceding paragraphs—that *one should avoid using the tensile ductility of a material to infer its fracture resistance  $K_{IC}$ .*

The toughness of the 1950's vintage stainless steels irradiated at temperatures between  $25^\circ\text{C}$  and  $125^\circ\text{C}$  to doses from 0.065 to 2.1 dpa was also measured [18,19]. Irradiation reduced the energy of the Charpy test and fracture toughness of the base, the weld, and the HAZ materials, regardless of orientation and test temperature. Irradiation did not change the orientation dependency, that is, the C-L orientation specimens had lower values of Charpy energy and fracture toughness than specimens in the L-C orientation (see Fig. 5). The largest reduction in fracture toughness  $J_C$  was for the HAZ material, 53%, and the smallest reduction was for the base material, 39% [18,19]. This lowest fracture toughness,  $K_{IC} \approx 125 \text{ MPa}\sqrt{\text{m}}$  (reduction by about 30%), for the irradiated HAZ specimen at  $125^\circ\text{C}$  was for the C-L orientation. Similarly, the lowest fracture toughness for the irradiated base material was  $K_C \approx 169 \text{ MPa}\sqrt{\text{m}}$  (reduction by about 22%). On average, the Charpy impact energy was reduced by 40% for welds and HAZ [5,21]. Most of the fracture toughness data were obtained using specimens that did not satisfy the thickness requirement for valid fracture toughness determination. However, the reported fracture toughness for the HAZ specimen in the C-L orientation was a valid one.

Toughness measurements for the same 1950's vintage stainless steels irradiated to higher doses were presented recently in [70]. The HAZ specimens with C-L orientation tested and irradiated at  $125^\circ\text{C}$  to dose 4.4 dpa had fracture toughness values of  $K_{IC} \approx 137 \text{ MPa}\sqrt{\text{m}}$  which showed no further reduction with increasing irradiation dose from 2 dpa to 4.4 dpa. This was also the case for the base metal. The HAZ samples irradiated at  $25^\circ\text{C}$  to 4.1 dpa and tested at  $25^\circ\text{C}$  had slightly lower fracture toughness ( $K_{IC} \approx 108 \text{ MPa}\sqrt{\text{m}}$ ). This shows that for the HAZ of the 1950's vintage type steel, increasing the irradiation/test temperature increased the fracture toughness. Nevertheless, the smallest fracture toughness for the welded joints irradiated to about 4 dpa was greater than  $100 \text{ MPa}\sqrt{\text{m}}$  which means that they fall into category II of fracture toughness.

The amount of  $\delta$ -ferrite affects the mechanical properties of the irradiated welds in austenitic stainless steels [58]. The presence of  $\delta$ -ferrite in the welds is necessary to prevent solidification cracking during fabrication. Figure 6 shows the result of impact tests on 18Cr-10Ni austenitic stainless steel welds with various amounts of  $\delta$ -ferrite content irradiated at  $70^\circ\text{C}$  [58]. The impact energy reduction for welds with  $\delta$ -ferrite content less than 7% irradiated to  $3 \times 10^{24} \text{ n/m}^2$  (0.45 dpa) was less than 50%. However, welds with higher ferrite content (10 – 12%) exhibited higher reductions in impact energy despite the fact that they had been exposed to lower doses. Irradiation also increased the ductile to brittle transition temperature. It was concluded that the 18Cr-10Ni weld with  $\delta$ -ferrite content less than 7% and irradiated up to dose of  $10^{25} \text{ n/m}^2$  ( $\sim 1.49$  dpa) at temperatures less than  $500^\circ\text{C}$  had satisfactory impact energy resistance [58]. In particular, irradiation at  $70^\circ\text{C}$  reduced the impact energy by no more than approximately 50% (Fig. 6) for testing temperatures above  $20^\circ\text{C}$ .

Low temperature irradiation studies were also driven by the need to investigate materials for the International Thermonuclear Experimental Reactor (ITER) in which the proposed operating temperature range for the fusion reactor structure is in the range 100 – 250°C [20,48,60,65,77]. In one study, the effects of neutron irradiation on fracture toughness of different 316 stainless steels (ERH, JPCA, and J316) were investigated [48,60,65,77]. The temperature at the crack tip of fracture toughness specimens during irradiation was calculated to be either 65 – 125°C (nominally 90°C) or 250 – 300°C (nominally 250°C) and the specimens were irradiated up to 3 dpa ( $3.7 \times 10^{25}$  n/m<sup>2</sup>,  $E > 0.1$  MeV). The helium generation for all test specimens was about 50 appm (10-20 appm He/dpa). The specimens were in SA, CW, or welded conditions. The ERH was welded using 16-8-2 filler metal by gas tungsten arc welding and the JPCA specimens were welded with filler wires with similar composition to the base metal by gas tungsten arc welding and electron beam welding.

The effect of irradiation and test temperature on fracture toughness of 316 SA steels is shown in Fig. 7 [48]. The non-irradiated SA steels were found to have high fracture toughness,  $K_C > 300$  MPa√m. The magnitudes of  $K_C$  for J316, ERH, and JPCA irradiated at 90°C to 3 dpa and tested at 90°C were about 370, 340, and 240 MPa√m, respectively. The fracture toughness of the CW steels before and after irradiation were typically 75 to 100 MPa√m lower than for SA materials with the exception of JPCA irradiated and tested at 250°C for which the fracture toughness of CW and SA specimens were identical [60,65]. All welded samples were very tough before and after irradiation. GTAW of JPCA had almost the same toughness as the SA JPCA after irradiation and testing at 90°C ( $K_C \approx 240$  MPa√m) while electron beam welds (EBW) of JPCA demonstrated even a higher toughness as shown in Fig. 8. These result demonstrates that the welding process can greatly affect the fracture toughness of the joints [69,71]. In general, the fracture toughness  $K_C$  of steels irradiated to 3 dpa decreased with increasing irradiation/test-temperature, however, all specimens exhibited high fracture toughness even at 250°C. This suggests that the fracture toughness obtained at higher irradiation/test-temperatures may provide a conservative estimate for the toughness at lower temperatures up to 3 dpa. It should be noted that almost all of the fracture toughness data were obtained using specimens that did not satisfy all the size requirements to constitute a valid test.

In another study of ITER materials, the change in mechanical properties of 304 in SA condition irradiated to doses up to  $1.2 \times 10^{26}$  n/m<sup>2</sup> ( $E > 1$  MeV) at 50°C was investigated [20]. As shown in Fig. 9, the fracture toughness decreased with dose to  $J_C = 100$  kJ/m<sup>2</sup> ( $K_C \approx 150$  MPa√m) for 304SA tested at room temperature after irradiation to dose of  $10^{26}$  n/m<sup>2</sup>. Again the specimens did not meet all the size requirements for the fracture toughness tests to be valid.

Effects of irradiation at low temperatures on mechanical properties of stainless steels were also examined for the Accelerator Production of Tritium (APT) project [23,24]. The structural materials in some part of the APT systems are irradiated by high energy protons and neutrons (up to 1 GeV) in the temperature range 50 – 160°C. Since the energy of the particles is much higher than for particles in fission reactors, the amount of hydrogen and helium produced per dpa in the APT is significantly higher than the amounts produced in fission reactors. The effect of irradiation on fracture toughness of

304L and 316L under high energy proton and neutron irradiation condition (800 MeV) with maximum fluence of  $4.5 \times 10^{25}$  proton/m<sup>2</sup> (12 dpa, 10000 appm H, 1000 appm He in 304L) was investigated. The irradiation temperatures were between 50 and 160°C and the specimens were tested at the same temperature as they had been irradiated. The sub-size disk-shaped compact tension specimen was employed for fracture toughness measurements. Figure 10 shows the effect of irradiation with high energy protons and neutrons on the fracture toughness of 304L and 316L stainless steels [23]. With increasing dose, the toughness  $K_C$  leveled to a low plateau of about 100 MPa√m at 3-4 dpa for 304L steel whereas for the 316L steel, the toughness leveled to about 60-70 MPa√m. The fracture toughness of irradiated 304L and 316L steels in the APT project leveled with dose to about the same values as for the irradiated steels in fission reactors (see Figs. 1 and 3). But it appears that due to high hydrogen and helium production, the fracture toughness as found in the APT project decreases with dose at a faster rate. It should be mentioned that the tests were not valid tests according to the ASTM standard for size requirements. However, the reported values were claimed to be conservative since the fracture toughness of non-irradiated 304L estimated from the sub-size compact tension specimens was less than that from a valid fracture toughness test. The valid fracture toughness for non-irradiated 304L steel is  $K_{IC} = 360$  MPa√m [71]. Examination of the fracture surfaces revealed ductile dimples. During crack growth experiments with 316L steels irradiated to doses greater than 4.5 dpa, short periods of unstable crack growth were observed [24]. The loss of resistance to stable crack growth was attributed to formation of weak localized zones by irradiation.

Chopra and Shack [74] plotted the fracture toughness data reported in the literature for austenitic stainless steels irradiated in fast reactors and LWR as a function of irradiation dose as shown in Fig. 11. The irradiation temperatures ranged from 90°C to 427°C and the test temperatures were from room temperature to 427°C. A fracture toughness trend curve that bounds the data for  $J_{IC}$  from below is also plotted on the same figure. For doses less than 5 dpa, the curve can be expressed as  $J_{IC} = 9 + 120e^{-0.6 dpa}$  (Fig. 11). McKinley *et al.* [76] carried out a similar interpolation study using the data for high temperature irradiation and came up with a threshold fit represented by  $K_{IC} = 38 + 175e^{-0.3 dpa}$  for the entire range of irradiation dose.

### 3. Effect of irradiation on tensile properties

Unlike fracture toughness tests, performing tensile tests is much easier and more data are available in the literature for tensile properties of neutron irradiated austenitic stainless steels. Irradiation increases yield stress and ultimate tensile strength, and decreases ductility and work hardening capacity of stainless steels. The key parameters that affect these properties are irradiation temperature, test temperature, and fluence. In each of the paragraphs below, the type of the material whose behavior is reported is underlined.

The study of the effect of neutron irradiation on tensile properties of 304 stainless steel base plate and welds goes back to the early 1960s [11,12,13]. The tensile samples made of 304 base plates and welds with 308 filler were irradiated at temperatures below 100°C in high purity water with maximum fluence about  $8 \times 10^{24}$  n/m<sup>2</sup> ( $E > 1.0$  MeV) (1.2

dpa) [11,12]. The tensile tests were done at room temperature. Before irradiation the ultimate tensile strength was 84 ksi (~580 MPa) for 304 base metal, weld, and HAZ. After irradiation, the ultimate tensile strength was increased to 104 ksi (~720 MPa) and again it was almost the same for base metal, weld, and HAZ. The effect of irradiation on yield stress and ductility was higher for weld metal. Weld metal showed higher yield stress and lower ductility than HAZ and base metal after irradiation. The yield stress of the non-irradiated base metal was 35 ksi (240MPa) and increased to 91 ksi (630 MPa) after irradiation. For the weld metal, the yield stress was increased from 57 ksi (390 MPa) to 100 ksi (690 MPa) by irradiation. With regard to elongation, it decreases with fluence (Fig. 6, [11]): from about 65, 55, 35% for non-irradiated material to about 42, 38, 17% respectively for base plate, HAZ, and weld material. These data are close to the values reported for tensile properties of the 1950's vintage type 304 stainless steel weldment components [5,17,18,19].

The effect of low temperature irradiation (<100°C) on tensile properties of austenitic stainless steel base metal has been investigated extensively. Irvin and Bement [14] presented the effect of test temperature (<800°C) on tensile tests for irradiated and non-irradiated 304 stainless steel plates. The materials were either annealed or 25% CW before being irradiated at 60°C in water. In the test temperature range below 300°C, yield stress, ultimate strength, elongation and reduction of area for non-irradiated and irradiated materials decreased gradually with increasing test temperature. Irradiation increased the yield stress and ultimate tensile strength (Fig. 12) and reduced the ductility as shown in Fig. 13. The effect of higher temperature irradiation (290°C) on the elongation of 304 steel is shown in Fig. 14. It can be concluded from Figs. 13 and 14 that the ductility of the 304 steel irradiated and tested at 60°C is higher than that of 304 steel irradiated and tested at 290°C with the same dose. Bloom *et al.* [15] examined the effect of irradiation temperature on tensile properties of 304 steel at room temperature. Annealed tensile samples were irradiated to a fluence of  $7 \times 10^{24}$  n/m<sup>2</sup> ( $E > 1$  MeV) (~1 dpa) at temperatures between 93 and 454°C. The yield stress and ultimate strength of samples tested at room temperature exhibited a maximum at irradiation temperature of about 150°C as shown in Fig. 15. Higgy and Hammad [16] investigated the effect of irradiation fluence on tensile properties of annealed 304 type stainless steel. The samples were irradiated to maximum fluence of  $1.43 \times 10^{24}$  n/m<sup>2</sup> ( $E > 1$  MeV) (0.21 dpa) while they were in contact with cooling water at 50°C and subsequently tested at room temperature. A distinct yield point was observed for samples irradiated at fluences higher than  $1.3 \times 10^{23}$  n/m<sup>2</sup> ( $E > 1$  MeV) (0.019 dpa) instead of smooth yielding, which was the case for non-irradiated samples or those that were exposed to lower neutron fluence. The 304 type steel irradiated to  $1.43 \times 10^{24}$  n/m<sup>2</sup> ( $E > 1$  MeV) showed yield stress increase by 150%, ultimate strength increase by 16%, and uniform elongation decrease by 38% (Fig. 16). The material properties leveled with irradiation at about  $5 \times 10^{23}$  n/m<sup>2</sup> (0.075 dpa)

In the test temperature range between room temperature and 300°C, the ductility of austenitic stainless steels reduces by increasing the test temperature. Microstructure and deformation mechanisms studies of irradiated 304L [64], and 316L SA and CW [61,62] stainless steels showed that twinning is an important deformation mechanism at rapid strain rates and at low temperatures. However, at higher temperature and slower strain rates, dislocation channeling is favored. Since the stacking fault energy increases with

temperature, it was concluded that twinning is not possible at temperatures greater than 200°C. This was interpreted as the cause of minimum ductility at higher temperatures.

Matsui *et al.* [20] investigated the change in tensile properties of irradiated type 304 and 316 austenitic stainless steels as a function of irradiation dose. The materials were irradiated to doses ranged from  $5 \times 10^{24}$  to  $1.2 \times 10^{26}$  n/m<sup>2</sup> ( $E > 1$  MeV) (0.75 – 17.9 dpa) at 50°C. Figure 17 shows that irradiation increased the proof stress of SA 304 and 316 steel specimens from about 200 to about 700 MPa. However, the proof stress of CW 304 steel changed from about 750 MPa to about 850 MPa. For 304 SA steel, the proof stress saturated at fluence greater than  $1.0 \times 10^{26}$  n/m<sup>2</sup>, while the saturation dose for 304CW was smaller than  $6.0 \times 10^{24}$  n/m<sup>2</sup>. Figure 17 also shows that the irradiation effect on the ultimate tensile strength is smaller than that on the proof stress [20].

Figure 18 shows the effect of low temperature neutron irradiation on the tensile stress-strain behavior of 316 SA stainless steel [10,48]. Irradiation caused a small yield drop followed by work hardening. Low temperature irradiation and testing (60°C) increased the yield stress from about 300 MPa to about 700MPa at 7 dpa. From 7 to 19 dpa, the ultimate strength remained almost constant at about 750MPa while the yield stress increased slightly. Uniform elongation reduced with dose to 20% at 19 dpa. For specimens irradiated and tested at 200°C, the deformation behavior was similar to that for specimens tested at 60°C, but was characterized with a larger reduction in uniform elongation. Irradiation of specimens at 300°C eliminated material hardening after an initial yield drop and the uniform elongation dropped to 0.3%.

The effect of irradiation on the tensile properties of 316 austenitic stainless steel (SA and CW conditions) and 16Cr-8Ni-2Mo weldment was reported by Wiffen and Maziasz [45]. The materials were irradiated at 55°C in contact with reactor cooling water to fluences in the range of  $0.5 - 1.35 \times 10^{26}$  n/m<sup>2</sup> ( $E > 0.1$  MeV) which produced 4.5 – 10.8 dpa and 180 – 520 appm He. Uniaxial tension tests at 35°C for the materials irradiated at 55°C showed that the yield stress for the SA steels was increased to about 810 MPa from 250 MPa, the yield stress for non-irradiated steels. The uniform and total elongations reduced to half of that for non-irradiated materials; however, the uniform elongation was still higher than 20%. For CW steels, irradiation increased the yield stress from about 800 MPa to 930 MPa, i.e. 130 MPa increase in yield stress. But the uniform elongation was reduced to 0.4% by irradiation. The yield stress of the weldment was close to the average of CW and SA 316 type steels. As for the uniform elongation, it was reduced by a factor of two after irradiation (from about 14% to about 7%) [45].

The tensile properties of annealed 304L and 316L austenitic stainless steels after irradiation with high energy protons and spallation neutrons (peaking at 800 MeV with associated generation of 100 appm/dpa of He and 1000 appm/dpa of H) were also explored [22,23]. The specimens were irradiated to dose levels ranging from 0.01 to 12 dpa over the temperature range 30 – 150°C. The tensile tests were performed using miniature tensile specimens at 20, 50, 80 and 164°C, which approximately were also the irradiation temperatures. As shown in Fig. 19, the yield stress for both steel types increased rapidly for doses less than 0.1 dpa followed by gradual increase up to 4 dpa, after which it seems to have saturated to more than twice the non-irradiated values. The uniform elongation for non-irradiated specimens ranged from 40% to 60%. Irradiation

caused the uniform elongation to sharply decrease to less than 20% at 0.01 dpa and then gradually decrease with dose up to 3 dpa, after which it abruptly dropped to less than 1%. Despite the differences in the uniform elongation among the irradiated specimens, the fracture surface of all specimens had a ductile appearance with localized features.

Grossbeck and coworkers [57,68] reviewed the effect of irradiation on tensile properties of austenitic stainless steels irradiated over a range of temperature and dose conditions pertaining to fusion reactors in which profuse helium production also occurred. Most of the data were for high temperature irradiation and/or irradiated materials in CW condition. It was concluded that while helium production affects the tensile properties of the irradiated stainless steel, it has a small effect at low irradiation/test-temperatures since helium embrittlement is a high temperature phenomenon. In fact, the researchers state that the variation of properties from one heat of steel to another was sometimes higher than the effect of helium concentration alone. Grossbeck and coworkers also investigated the effect of temperature on irradiation creep which is plastic deformation in the presence of stress and irradiation. It was found that irradiation creep at 60°C is equal to or larger than that at higher temperatures. This seems to have a beneficial effect on the fracture resistance of materials during irradiation since it can relieve local crack tip stresses at existing flaws and prevent possible catastrophic crack propagation.

Lucas [4,41] reviewed the effect of irradiation on SA austenitic stainless steels (mainly 316LN steel) at temperatures less than 300°C. Similar to the case of 304 stainless steel (see Fig. 16), the yield stress of the irradiated 316 steel increased and the work hardening rate decreased with irradiation dose. Figure 20 shows the yield stress of irradiated and non-irradiated annealed 316 steel as a function of irradiation/test-temperature. The yield stress increased with temperature and reached its maximum at irradiation doses about 7 – 10 dpa at irradiation/test-temperature range of 250 – 270°C. The irradiation also degraded the uniform elongation.

On the list of papers provided by the CNSC, there was a number of papers reporting tensile properties of irradiated 304 stainless steels not related to conditions/scope of this project. These papers can be briefly summarized as follows: Pokor, *et al.* [46,47] and Conermann, *et al.* [50] reported the effect of irradiation at temperature range around 300°C on yield stress and work hardening behaviour of SA 304L and CW 316. Morisawa *et al.* [53] studied the effect of hydrogen charging/discharging on tensile properties of irradiated SA 304 stainless steel for specimens irradiated at temperature 288°C.

#### **4. Microstructure of irradiated austenitic stainless steels**

Neutron irradiation produces significant microstructural changes, which bring about the macroscopic changes reported in the preceding sections. The effect of irradiation on the microstructural evolution is briefly outlined in this section.

Irradiation temperature has a pronounced effect on the microstructural evolution of materials. Maziasz [7] presented a review of the data for radiation induced microstructural evolution from fission reactor irradiation experiments of 300 series

austenitic stainless steel and identified the nature of the dominant microstructure as a function of irradiation temperature. The microstructure of austenitic stainless steels irradiated at temperatures below 300°C is characterized by a radiation-produced dislocation structure. This structure consists mainly of extremely small features along with larger interstitial Frank loops. The small features, usually called “black dots,” are mainly interstitial loops or vacancy clusters with diameter of 2 – 4 nm. Frank loops are interstitial loops that can become as large as 50 nm in size at high irradiation doses. The density of “black dots” is independent of temperature but the density of larger Frank loop increases with temperature [7,10]. Near 300°C, the microstructure of irradiated stainless steels changes from one dominated by small cluster and dislocation loops to one with larger loops, network dislocation, and bubbles or voids as shown in Fig. 21 [32]. Thus, 300°C can be regarded as the transition temperature between two different microstructural evolution regimes in irradiated stainless steels. At irradiation temperatures above 300°C, “black dot” damage is mostly recovered and depending on the temperature and irradiation dose, is replaced by voids or precipitates. Radiation-produced cavities, precipitates, and segregation are high temperature phenomena and they are not usually observed under low temperature irradiation conditions [33]. Even under high energy (500 – 800 MeV) mixed proton and spallation neutron irradiation at 30-80°C, the microstructure of 316L stainless steel exhibited no bubble or void formation at 10.3 dpa with a resolution limit of 1 nm, although the amount of helium retained in the specimen increased at a rate of about 180 appm/dpa [42]. In fact, the microstructure was predominantly comprised of small loops (black dots) and larger faulted Frank loops smaller than 6 nm in size at 0.7 dpa, which grew to average size of 50 nm at 10.3 dpa. An exception to this picture is the observation of very small cavities with diameter of about 2.5 nm in 304L SA irradiated to 0.5 dpa at 120°C [6].

There are only limited studies of irradiation effects on the microstructure of welds [9,25]. Chi *et al.* [25] investigated the microstructure of irradiated bcc  $\delta$ -ferrite and fcc austenite matrix to understand the effect of  $\delta$ -ferrite on Vickers micro-hardness of ion-irradiated austenitic stainless steel welds with a base metal of 304 steel with 308L or 308L/309L fillers. The specimens were irradiated with iron ions ( $\text{Fe}^{+4}$ ) at 60°C. The Vickers micro-hardness measurements suggest that irradiated bcc  $\delta$ -ferrite was hardened 1.5 times more than fcc austenite matrix. The microstructures of  $\delta$ -ferrite and austenite matrix before irradiation did not show any large differences in TEM, but after irradiation the defects in the austenite matrix were larger with higher density than those in the  $\delta$ -ferrite. Bae *et al.* [9] studied the microstructure of heat-affected zone in SUS304 stainless steels after irradiation. The material was electron irradiated at 400°C up to 5.4 dpa, which resulted in void formation, swelling, and radiation induced segregation. These are all defects characteristic of high temperature irradiation.

## 5. Remaining papers

On the list of references provided by CNSC, there are some papers which are not relevant to the scope of this project. These are papers that describe phenomena observed at high temperature irradiation of stainless steel or loading or environmental conditions



not relevant to the calandria operation. Recall that the calandria vessel is in contact with heavy water at 60°C.

Irradiation assisted stress corrosion cracking (IASCC) was the main subject in references [8,29,31,32,33,40,49,50,54,55,56]. IASCC is a phenomenon whereby an irradiated material fails through intergranular cracking with very little energy dissipation. It can occur during irradiation of materials in contact with high temperature water or vapor as in core components of light water reactors. Irradiation can change the composition of some elements such as chromium at grain boundaries due to radiation-induced segregation (RIS) [2]. Although IASCC is well documented, there remains no direct correlation of the susceptibility of a grain boundary to cracking to either the loss of chromium or existence of a defect hardened matrix. It is posited that the defect-free channels produced by the mobile dislocations plays an as yet undetermined role in causing IASCC. 316 stainless steel irradiated at 60°C and tested at the same temperature with the slow strain rate technique failed in a ductile manner indicating that IASCC may not be an issue at low temperature irradiation [54]. Therefore, IASCC may not be relevant under the calandria conditions. Similarly, void swelling [43,44] may also be irrelevant because it, too, is a high temperature phenomenon.

Another subject that may not be relevant to the calandria conditions is the effect of irradiation on fatigue crack propagation that was the focus of the investigations in references [34,35]. Lastly, development of welding methods for repairing irradiated stainless steels [30], corrosion of steels after irradiation [37], intergranular stress corrosion cracking of non-irradiated sensitized 304 stainless steel [38], effect of irradiation on Young's modulus [26], hydrogen embrittlement [28,52] are also subjects beyond the scope of this project.

## 6. Conclusions

The effect of neutron irradiation on material properties of 304 and 316 austenitic stainless steel base plates, HAZ, and welds was reviewed. The review was carried out from a perspective of relevance to conditions prevailing in the calandria vessel of the CANDU reactors. It is well known that the temperature at which the steels are irradiated has a profound effect on the nature of defect types and their densities created in the steels during irradiation and subsequently on mechanical properties. Therefore, the focus was mainly on low temperature irradiation.

- Fracture toughness tests on 304 stainless steels showed that the HAZ of welds can be the weakest point of the welded structures. Irradiation of those structures at dose of 4.1 dpa and low temperatures (25 to 125°C) reduces the HAZ fracture toughness to  $K_{IC} \approx 108 \text{ MPa}\sqrt{\text{m}}$  (valid fracture toughness testing) and base metal fracture toughness to about 180  $\text{MPa}\sqrt{\text{m}}$  (ASTM requirement for specimen size was not met).
- For the 316 SA stainless steel irradiated at 90°C to 3 dpa and tested at 90°C, values of fracture toughness greater than 240  $\text{MPa}\sqrt{\text{m}}$  have been reported. As the irradiation and testing temperatures increase to 250°C, the fracture toughness decreases. For welds, the process of welding affects the fracture toughness.

Nevertheless, the 316 welds showed toughness values similar to those for 316 SA base plates. The toughness of 316 SA and welds was found using specimens that did not meet all size requirements. Similarly, from tests not meeting all size requirements,  $K_{IC} \approx 150 \text{ MPa}\sqrt{\text{m}}$  for the 304 SA stainless steel irradiated to doses up to  $1.2 \times 10^{26} \text{ n/m}^2$  ( $E > 1 \text{ MeV}$ ) at  $50^\circ\text{C}$  was reported. Under high energy proton and neutron irradiation conditions at temperatures ranging from  $50$  and  $160^\circ\text{C}$  (up to  $12 \text{ dpa}$ ), minimum fracture toughness values of about  $100 \text{ MPa}\sqrt{\text{m}}$  for 304L and  $60 - 70 \text{ MPa}\sqrt{\text{m}}$  for the 316L steel were reported, but not all specimen size requirements were met during testing.

- The impact energy reduction for welds with  $\delta$ -ferrite content less than 7% irradiated to  $3 \times 10^{24} \text{ n/m}^2$  was less than 50%. However, welds with higher ferrite contents (10 – 12%) exhibited higher reductions in impact energy despite the fact that they had been exposed to lower doses.
- Correlation between fracture toughness and uniaxial tension ductility in the literature is not supported mechanistically or experimentally.

## 7. Proposed research

The review of the published literature shows that although fracture toughness data are available for the irradiated 304 and 316 stainless steel base plates and welds, the underlying mechanistic understanding of fracture in these materials is lacking. Through mechanistic understanding of irradiation degradation in austenitic stainless steel, we can develop a fracture model with predictive capability. Irradiation conditions influence the microstructure of the irradiated materials and depending on the irradiation and loading conditions, materials may fail by different modes. For instance, the synergism or competition between microvoid growth/coalescence and shear localization in bringing about fracture is not well understood. Modern experimental fracture analysis techniques such as focused ion beam extraction of samples underneath the fracture surfaces in conjunction with micromechanics analysis can help reveal and quantify the operating mechanisms. The goal would be to develop and validate a lifetime prediction methodology for failure of the base metal and welds exposed to neutron irradiation.

The limited low temperature fracture toughness data indicate that high irradiation/testing temperature data are conservative estimates for properties at low temperatures. Hence, if one can confidently verify that the fracture toughness decreases with increasing irradiation/testing temperature, one can then utilize the high temperature data for conservative design and assessment at low temperature/irradiation conditions. Specifically, for the Calandria operating conditions, the weldment of 304 steel needs to be tested at different irradiation/testing temperature at  $3 \text{ dpa}$  to confirm the conservativeness of the high temperature data. The difference between low and high temperature data for irradiated toughness may be related to different deformation/fracture mechanisms which again should be understood and modeled.

## 8. Appendix: Post-presentation discussion

After completion of the research and submission of the draft report, in accordance with the contract, the results were presented in a seminar held in Ottawa, Canada on September 21, 2012 before an audience including CNSC staff, staff from Canadian utilities, and service providers for the Canadian utilities. The following questions and responses are based on the question and answer session which immediately followed the above-mentioned seminar:

### *Question*

Fracture toughness data of 304SA steel in reference [20] (Figure 9 in this report) shows larger variability at 4 dpa than at other doses. Does this suggest that there is something unusual in the response at intermediate doses or is this scatter in the data? (Same question as phrased by Blair Carroll: Based upon available information is it possible to conclude that the variability in fracture toughness increases at higher dpa levels for austenitic stainless steels?)

### *Response*

It should be remembered that the data shown in Fig. 9 were obtained through testing that did not meet the specimen size requirement of the ASTM standard. Therefore, the data may not reflect accurate fracture toughness values, and as such inferences regarding variability may be baseless. At any rate, even though the variability is high at 4.5 dpa, the corresponding minimum fracture toughness value is about  $200 \text{ MPa}\sqrt{\text{m}}$  which is indeed a high toughness level.

### *Question*

Blair Carroll: Is it valid to compare fracture toughness test results for materials irradiated at different MeV levels?

Nikos Christodoulou: Different test conditions, different flux spectra, different damage mechanisms, how do you make the comparison between the different irradiation conditions. The trend that you see is that the fracture toughness decreases with increasing dpa and test temperature. This might be the best conclusion that you can make.

### *Response*

The main factors known to affect irradiation damage are dose expressed in dpa and irradiation temperature. Testing temperature also influences the measured fracture toughness. The dose expressed in dpa is calculated from neutron flux and neutron energy spectra. Therefore, we feel that one may use results from tests at MeV levels so long as these tests are interpreted [57] in terms of these three main factors, i.e. irradiation dose, irradiation temperature, and test temperature.

*Question*

Blair Carroll: Has environment (i.e. chemistry of water in contact with material) been considered in any of the investigations of the effects of irradiation on fracture toughness? Could the environment affect the fracture toughness?

*Response*

The effect of environment has been addressed by studies on irradiation assisted stress corrosion cracking (IASCC) in which irradiated materials fail by intergranular cracking. IASCC is associated with high temperature irradiation as mentioned in Section 5. The effect of the environment was usually investigated by using the slow strain rate technique [e.g. 49,50,54-56]. Specifically, a 316 stainless steel specimen irradiated and tested for stress corrosion in oxygenated pure water at low temperature (60°C) failed in a ductile manner [54] showing that IASCC may not be an issue at low temperature and therefore, may not be relevant under the calandria conditions. There are limited data on the effect of BWR environment on fracture toughness and J-R curve for stainless steel welds which show that the environment does not have a major effect [74]. It has been proposed that for BWR environment there is a threshold for IASCC of ~1 dpa and a larger dose, ~3 dpa, for PWR environment.

*Question*

Nikos Christodoulou on fracture toughness effect of temperature: Fracture toughness should increase with temperature at least in zirconium. Why is this trend not seen in the stainless steel?

*Response*

Fracture toughness decreases with increasing irradiation and test temperature for austenitic stainless steel [48,65]. This may be related to the microstructure stability as a function of temperature.

*Question*

Blair Carroll: Is the variability in the fracture tests data with respect to dpa at the low levels worth exploring?

*Response*

Usually the fracture toughness of irradiated austenitic stainless steel to low level dose is very high and therefore it is very difficult to satisfy the ASTM size requirements. Perhaps, this is the reason for the observed variability.

*Question*

From the audience: Did you find any studies suggesting that up to 4 dpa the failure is intergranular?

### *Response*

We did not see anything in the literature showing intergranular cracking of austenitic stainless steel irradiated up to 4 dpa at low temperature. For example, the fracture surfaces of irradiated 316L and 304L stainless steels at low temperature (50 – 160°C) up to 9 dpa doses under spallation environment had a ductile appearance [23]. In another study [48], tensile samples of 316 stainless steels irradiated to 19 dpa at temperatures up to 400 °C failed in ductile tearing mode even though in some cases the strain to necking was less than 1%. Fracture toughness samples of irradiated 316 stainless steels irradiated to 3 dpa at 90°C and 250°C exhibited ductile failure. However, it has been reported [29] that heavily irradiated austenitic stainless steel under PWR conditions and very slow strain rate deformation,  $10^{-5}$  and  $10^{-7}$  s<sup>-1</sup>, can fail by intergranular cracking.

### *Question*

Given that there is a large range of data from previous studies, how do you rationalize the data that was obtained prior to 1990? What is the impact of specimen size on real components?

### *Response*

We assessed all data in this report by examining whether they were obtained through valid fracture toughness testing regardless of the year they were obtained. If the validity requirement is not met, there is no similitude between the stress and deformation fields in the specimen and the real component.

### *Question*

From the audience: From laboratory tests to real materials. How to transfer the data reliably? Are the mechanisms in lab test samples the same as in in-situ conditions?

### *Response*

If the laboratory test is valid according to the ASTM standards, the estimated fracture toughness is a conservative assessment of the toughness of the real-life component. This assessment reflects plane strain conditions which are known to be associated with a high triaxiality constraint and therefore with a low fracture toughness.

### *Question*

From the audience: Linkage of hardness to fracture toughness. Is there any?

### *Response*

Hardness is an indirect measure of the yield strength. Hence it relates to fracture toughness only qualitatively, that is, the higher the strength the lower the toughness.

*Question*

From the audience: Relationship between ductility and fracture toughness, relationship between elongation and fracture toughness.

*Response*

There have been attempts to relate uniaxial tension ductility to fracture toughness, but such a relationship lacks any underlying physical basis. Please see discussion in the report after Eq. (1).

*Question*

From the audience: How much helium in the matrix is present in any of the tests of fracture toughness? Where is the He and what was the mechanism of failure?

*Response*

There have been cases in the literature where fracture toughness of austenitic stainless steels was measured in the presence of helium [e.g., 23,24,48,60,65,77]. For the ITER case with 10 to 20 appm He/dpa, it was estimated that the fracture toughness was greater than  $200 \text{ MPa}\sqrt{\text{m}}$  [48,60,65,77]. In the case of high energy proton and neutron irradiation of 304L and 316L (up to 12dpa) with 1000 appm He and 10000 appm H, it seems that there is no marked helium and hydrogen effect up to 3 – 4 dpa. Helium embrittlement in general is a high temperature effect and associated with weakened grain boundaries. Regarding the amount of helium present in the individual specimens used for the measurement of the fracture toughness, we provide the relevant information in the main body of the report.

*Question*

From the audience: Calandria does not experience the same dose uniformly, there are region experiencing a higher dpa level. How to map the distribution of the dpa over the calandria especially in the area of welds?

*Response*

We considered a worst case scenario in which the maximum fluence for tubesheets and welds would be about  $1 - 2 \times 10^{25} \text{ n/m}^2$  ( $E > 1 \text{ MeV}$ ) or 1.5 – 3 dpa, after 60 years of reactor operation [1,36].

*Question*

From the audience: Hydrogen influence on the fracture toughness of irradiated metals. What is the influence of hydrogen embrittlement?

*Response*

Irradiation induced defects act as hydrogen traps in austenitic stainless steels. Irradiated steels showed higher loss of ductility in the presence of hydrogen [29]. However, hydrogen embrittlement was not specifically reviewed in relation to irradiation because it was not in the scope of this project.

*Question*

From the audience: in Fig. 14, is there a reason for the trough going to the left and then to the right at 290°C?

*Response*

Reference [14] provides no explanation.

*Question*

From the audience: In Fig. 19, provide an explanation for the abrupt decrease of the uniform elongation at 3 dpa.

*Response*

It is likely that this uniaxial ductility drop is a result of hydrogen/helium embrittlement.

*Question*

In a work hardening material, deformation does not localize: This is not really right as there is work hardening within the channels, macroscopic test not sufficiently sensitive to detect this work hardening which occurs within a relatively small volume.

*Response*

In a specimen deforming homogeneously under uniaxial tension, there is no macroscopic shear localization if the material has a stress-strain relationship with a positive slope (work-hardening). In the case of 3-D deformation, onset of macroscopically observed plastic instability depends on the boundary value problem specifics and hence, one cannot make any general predictions regarding shear flow localization. Hence the observation of internal soft channels that eventually harden is a possible one.

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<sup>2</sup> References 1-58 are the list of references in the order that was provided by CNSC. The rest are the relevant papers or documents we found in the open literature. With regard to Ref. [34], the title and the citation were each from two different papers with the same lead author; the title of the reference was corrected. The other paper similar to Ref. [34] is:

P. Shahinian, H.E. Watson, H.H. Smith, 1973, “Effect of Neutron Irradiation on Fatigue Crack propagation in Types 304 and 316 Stainless Steels at High Temperatures”, *Effects of Radiation on Substructure and Mechanical Properties of Metals and Alloys*, ASTM STP 529, pp. 493-508.



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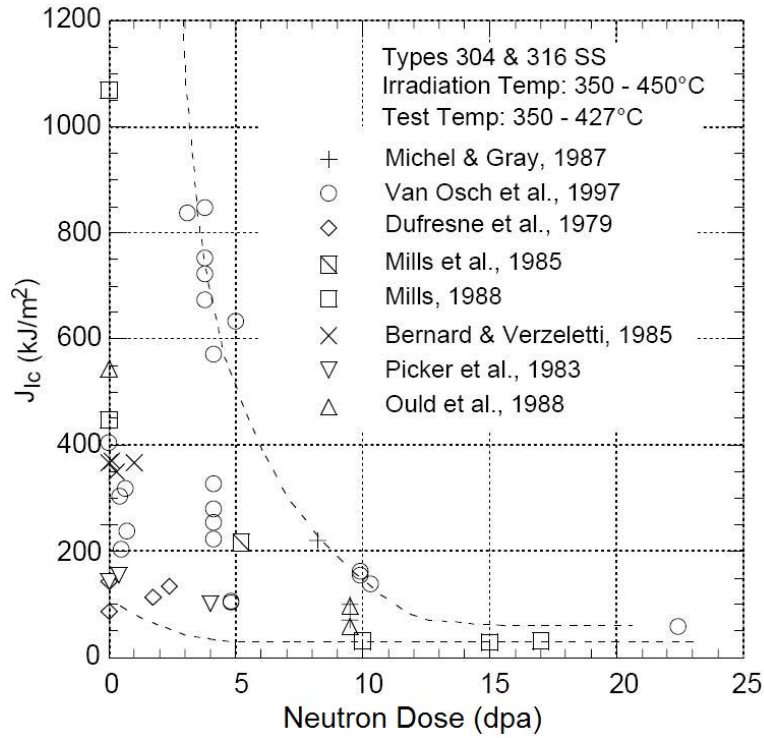
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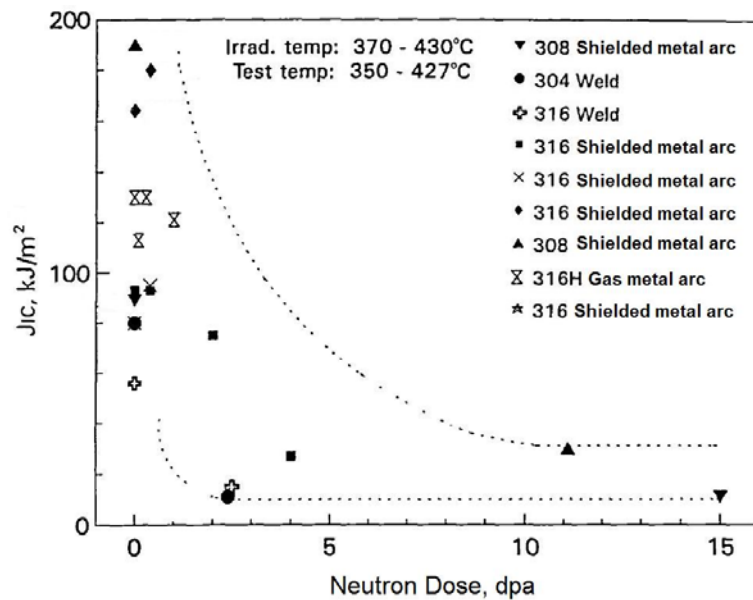
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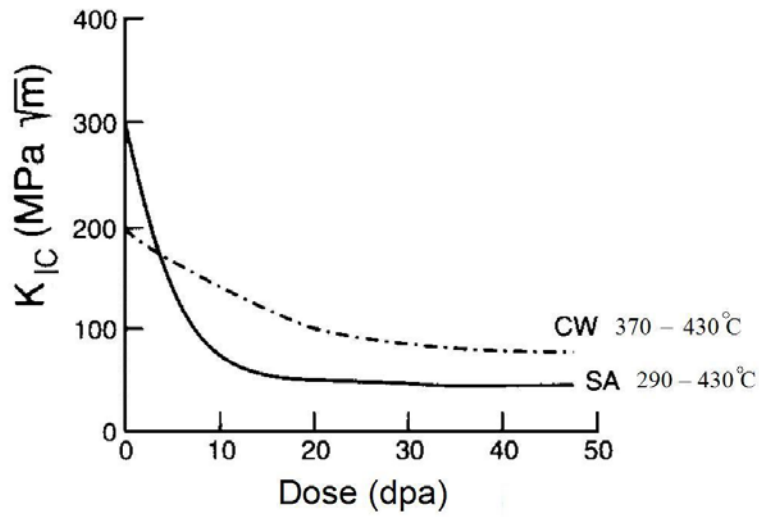
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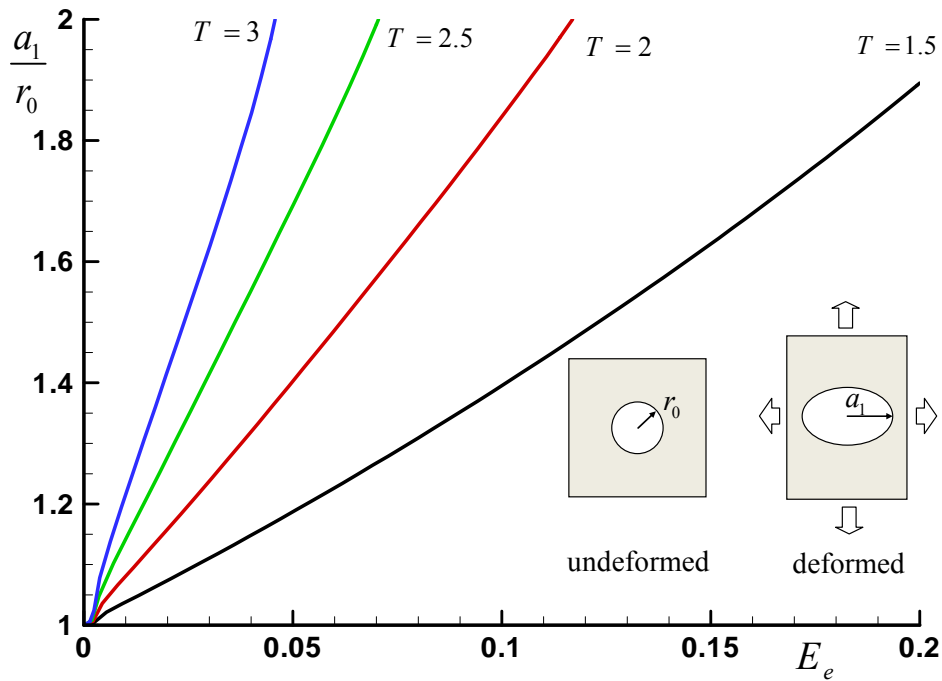
**Figure 1.** Fracture toughness  $J_{IC}$  as a function of neutron dose for 304 and 316 stainless steels base metal [27].



**Figure 2.** Fracture toughness  $J_{IC}$  as a function of neutron dose for 304 and 316 stainless steels welds [71].

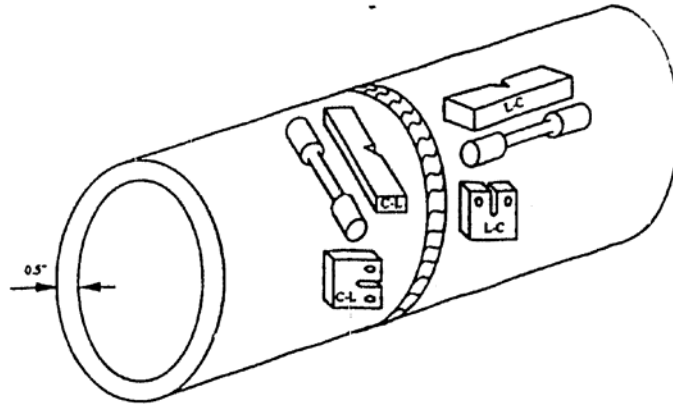


**Figure 3.** General trends of fracture toughness  $K_{IC}$  for cold worked (CW) and solution annealed (SA) austenitic stainless steels as a function of neutron dose [4].

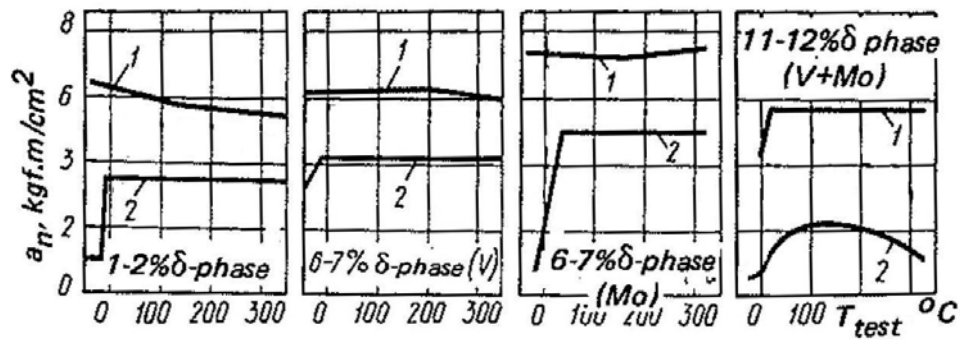


**Figure 4.** Unit cell results of normalized void size vs. macroscopic effective strain  $E_e$  at various applied macroscopic stress triaxialities  $T$ .

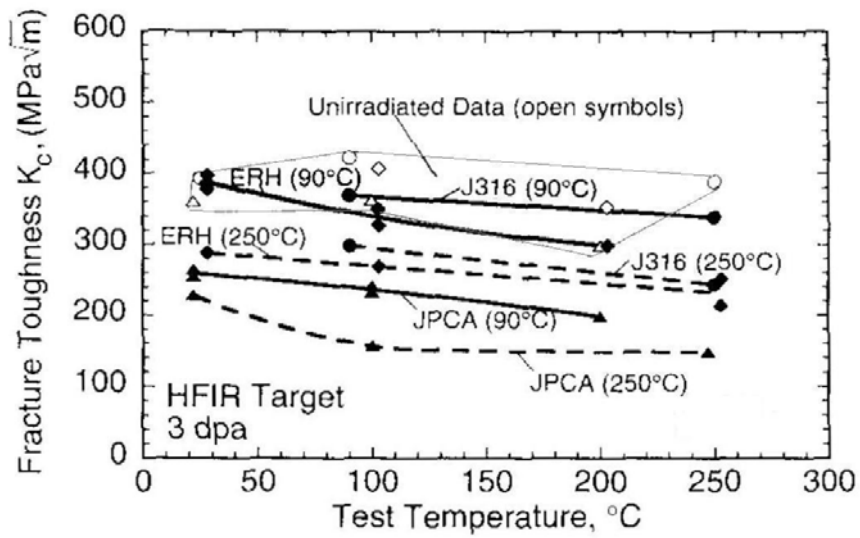




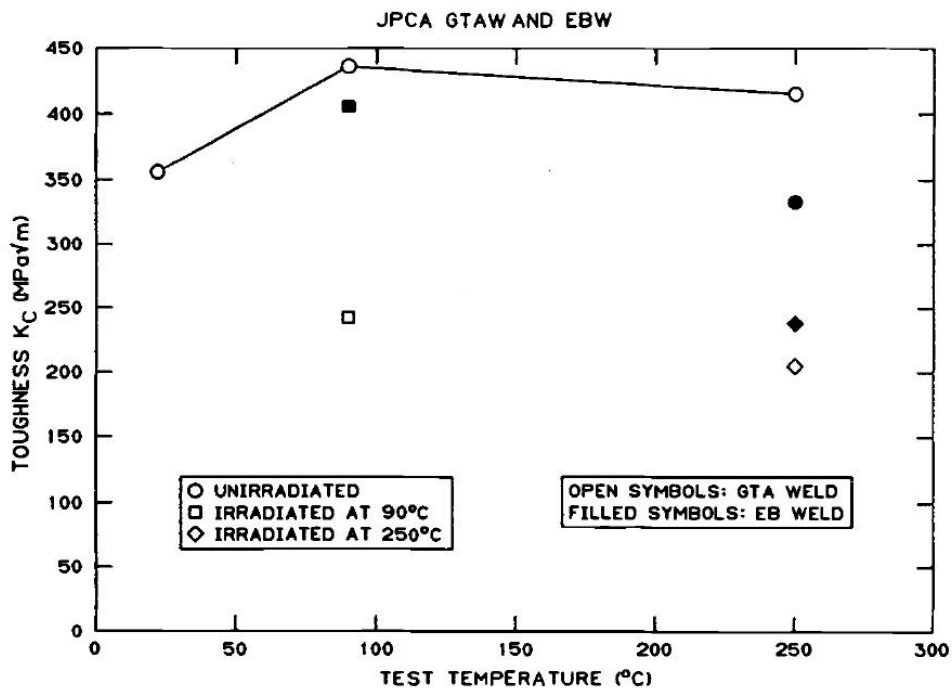
**Figure 5.** Schematic illustration of specimen orientation [17].



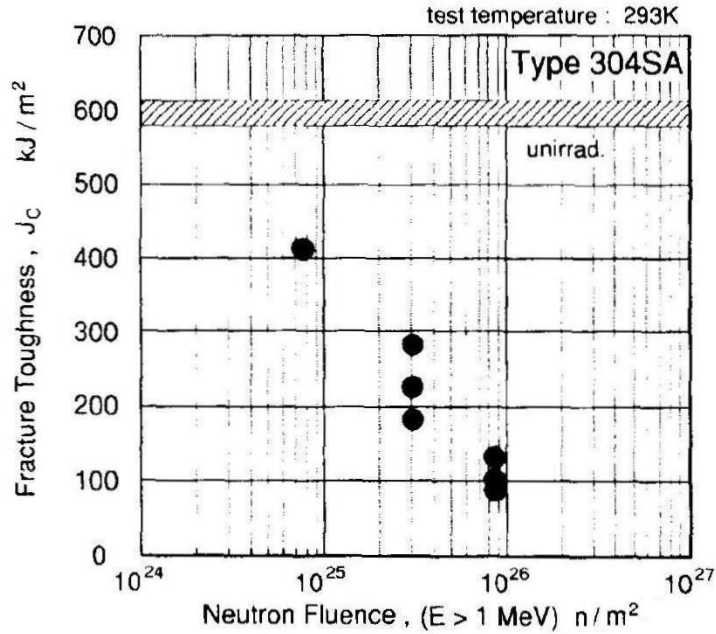
**Figure 6.** Effects of ferrite content ( $\delta$ -phase) and alloying elements on the impact toughness of non-irradiated (1) and irradiated (2) welds in 18Cr-10Ni stainless steel. The welds were exposed to fluence of  $3 \times 10^{24}$  n/m<sup>2</sup> at 70°C [58].



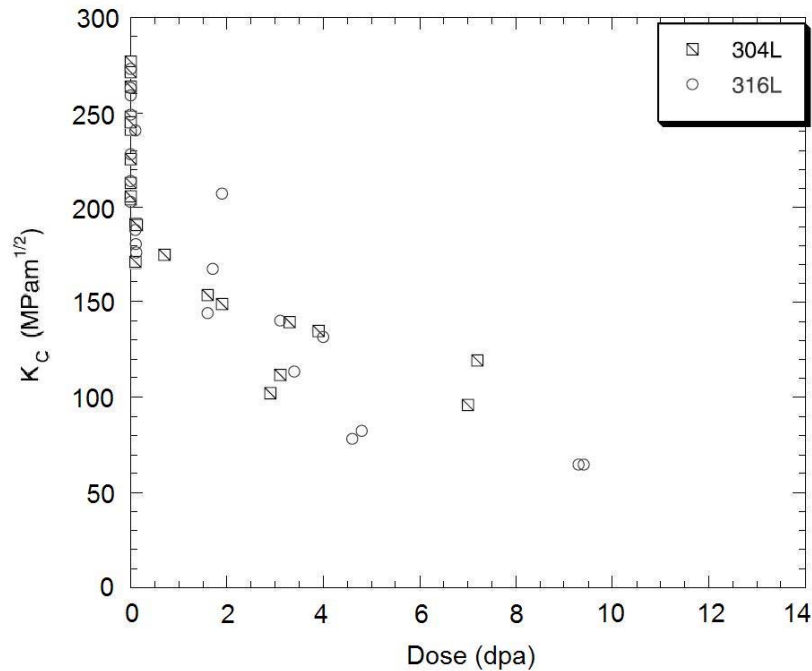
**Figure 7.** Variation of fracture toughness as a function of test temperature for specimens irradiated to dose of 3.0 dpa at different temperatures [48]. The specimen dimensions did not satisfy all the size requirements for valid fracture toughness testing.



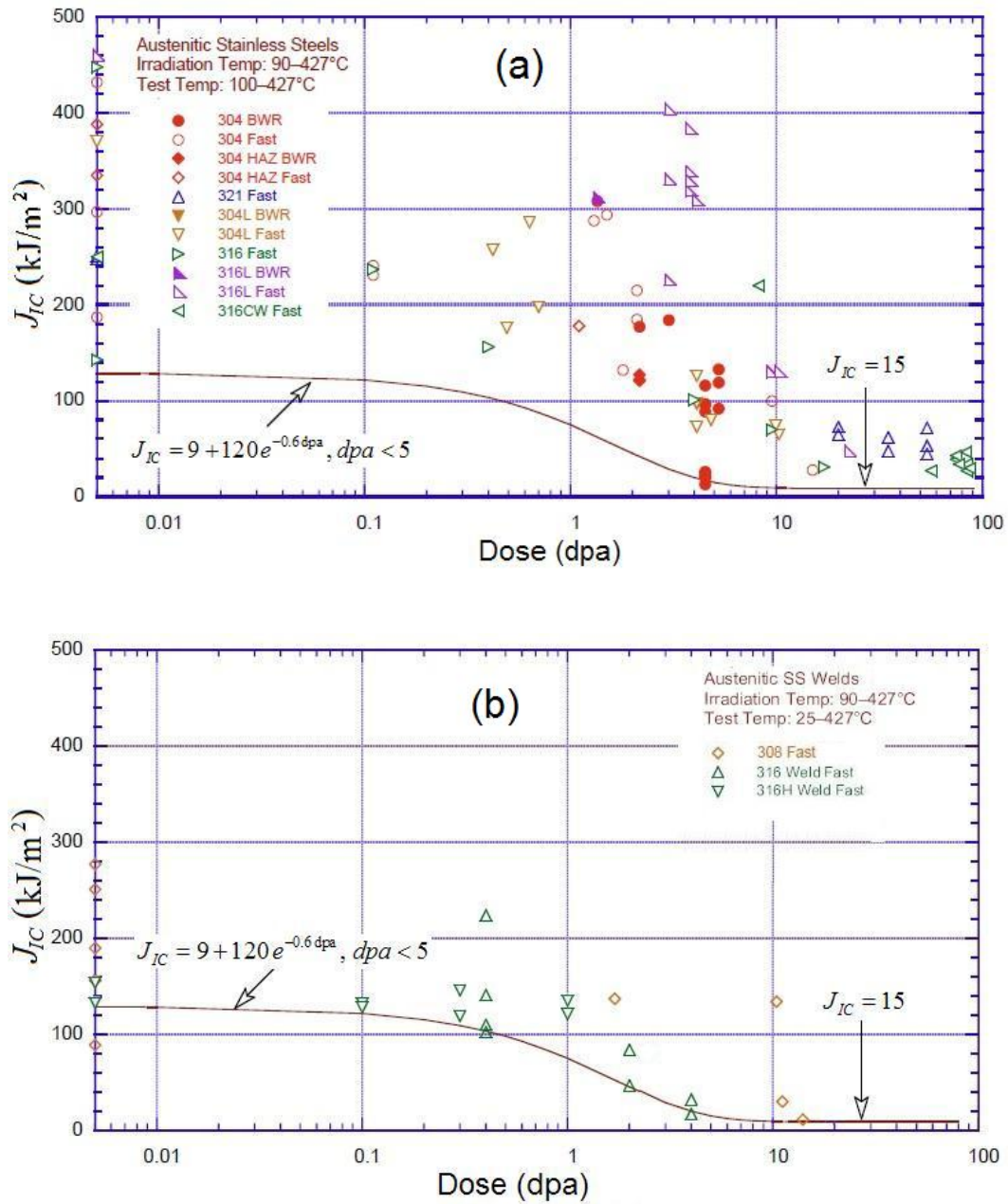
**Figure 8.** Fracture toughness for the gas tungsten arc welds (GTAW) and electron beam welds (EBW) of JPCA steels [65].



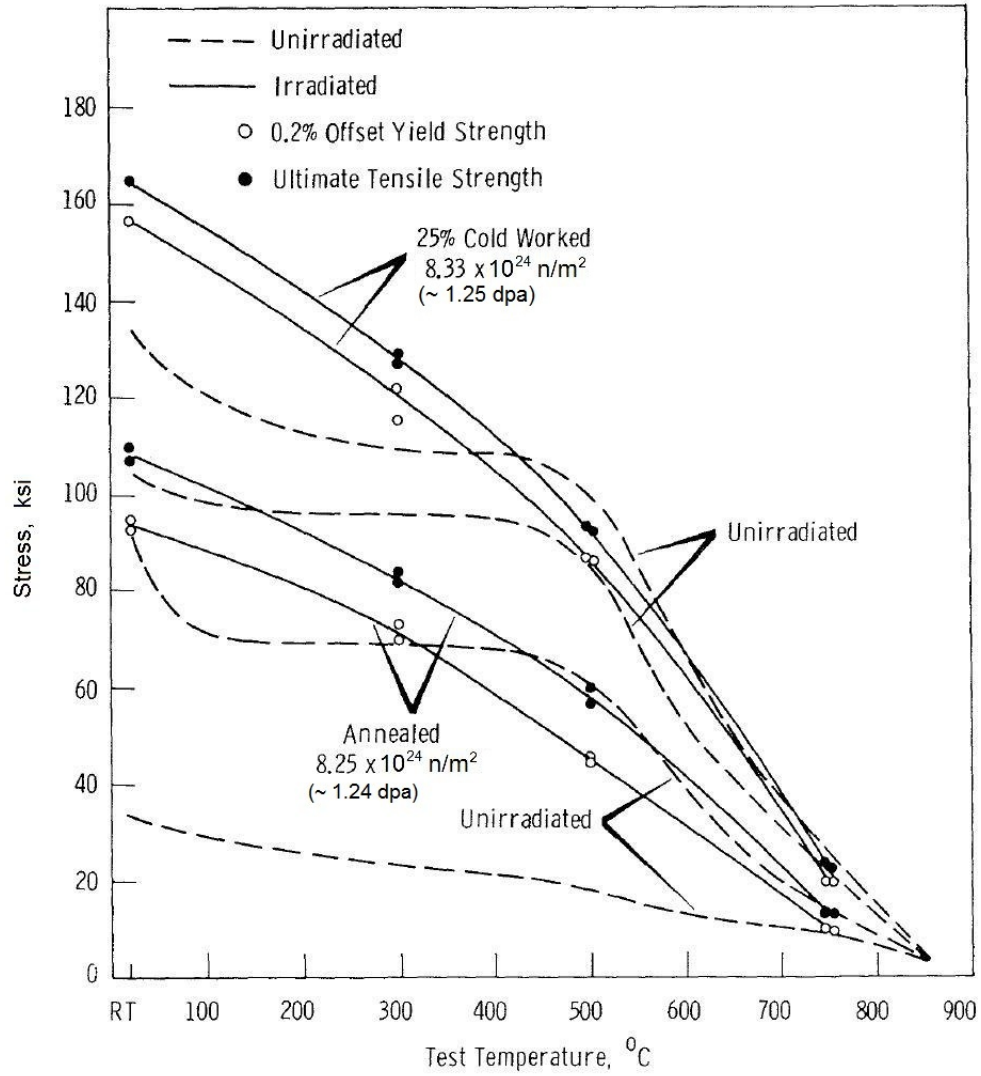
**Figure 9.** Fracture toughness  $J_C$  of 304SA as a function of dose at  $50^\circ\text{C}$  [20]. The specimen dimensions did not satisfy all the size requirements for valid fracture toughness testing.



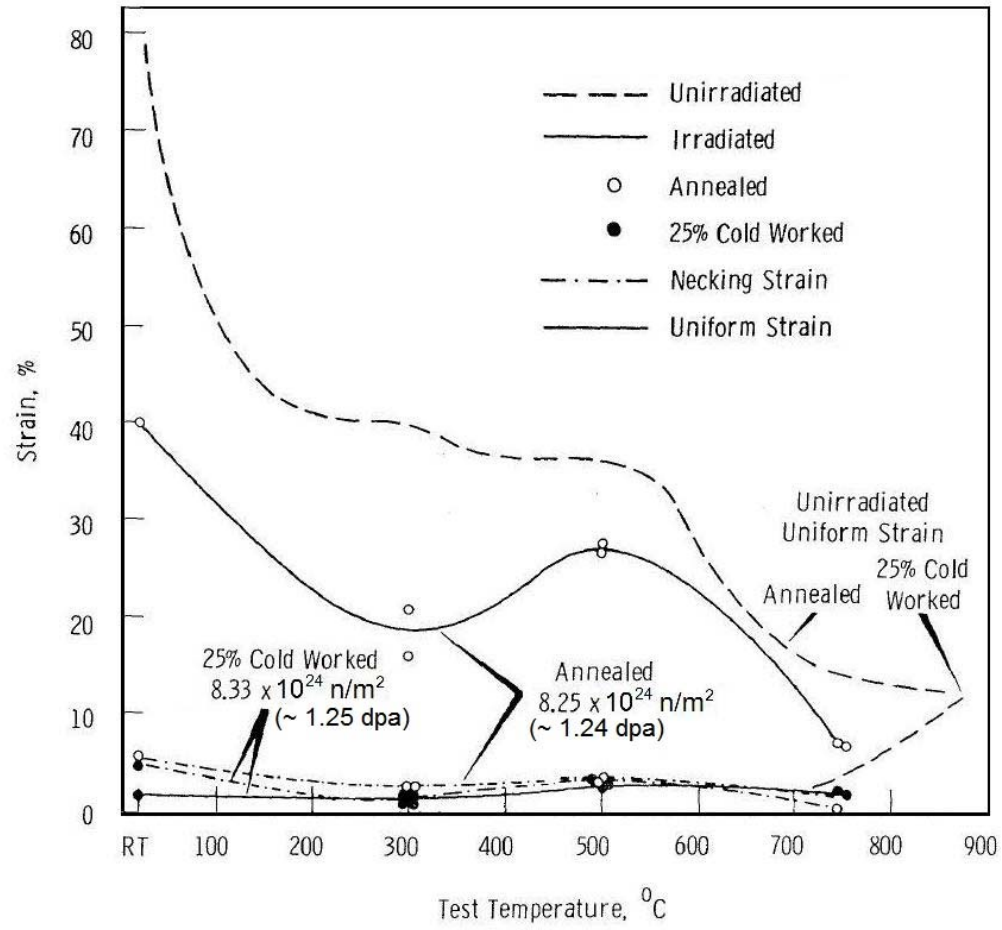
**Figure 10.** Variation of fracture toughness with dose for annealed 304L and 316L after irradiation with high energy particles (800 MeV). The irradiation and test temperatures were the same and in the range of  $50\text{-}160^\circ\text{C}$ . The size requirements for valid fracture toughness test were not satisfied [23].



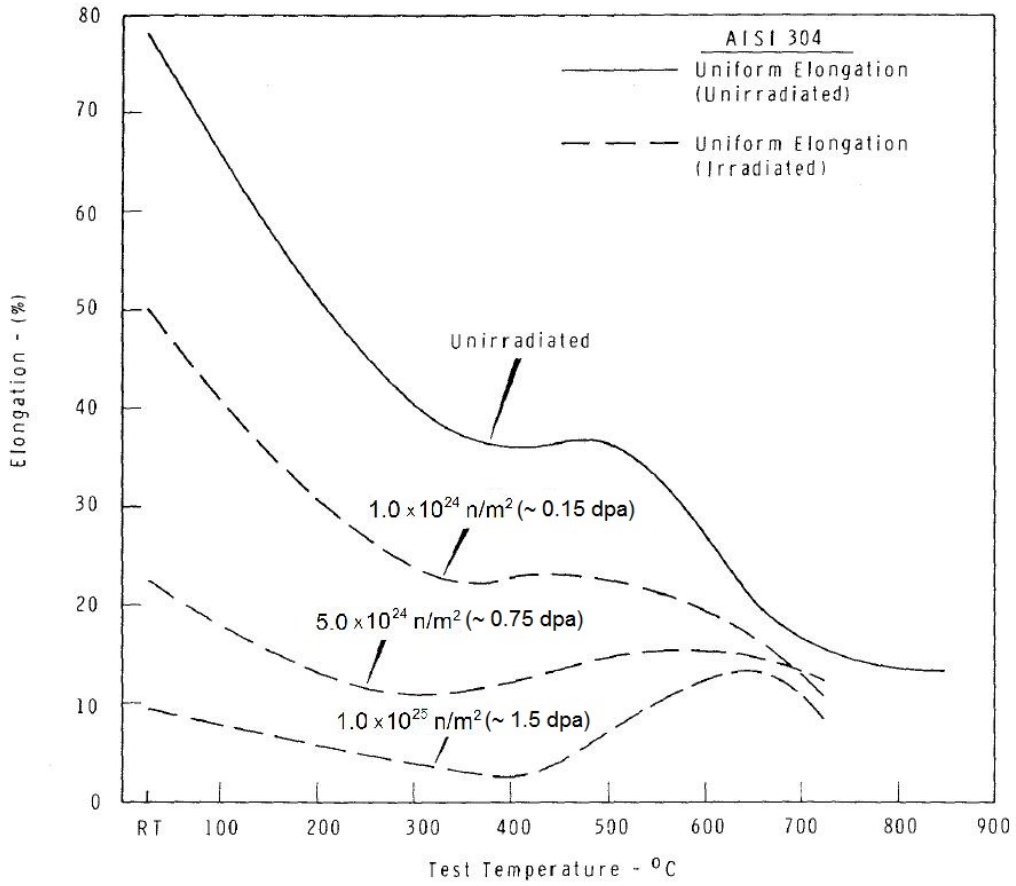
**Figure 11.** Variation of fracture toughness  $J_{IC}$  as a function of dose for 304 and 316 stainless steel (a) base plates and (b) welds in the literature [74].



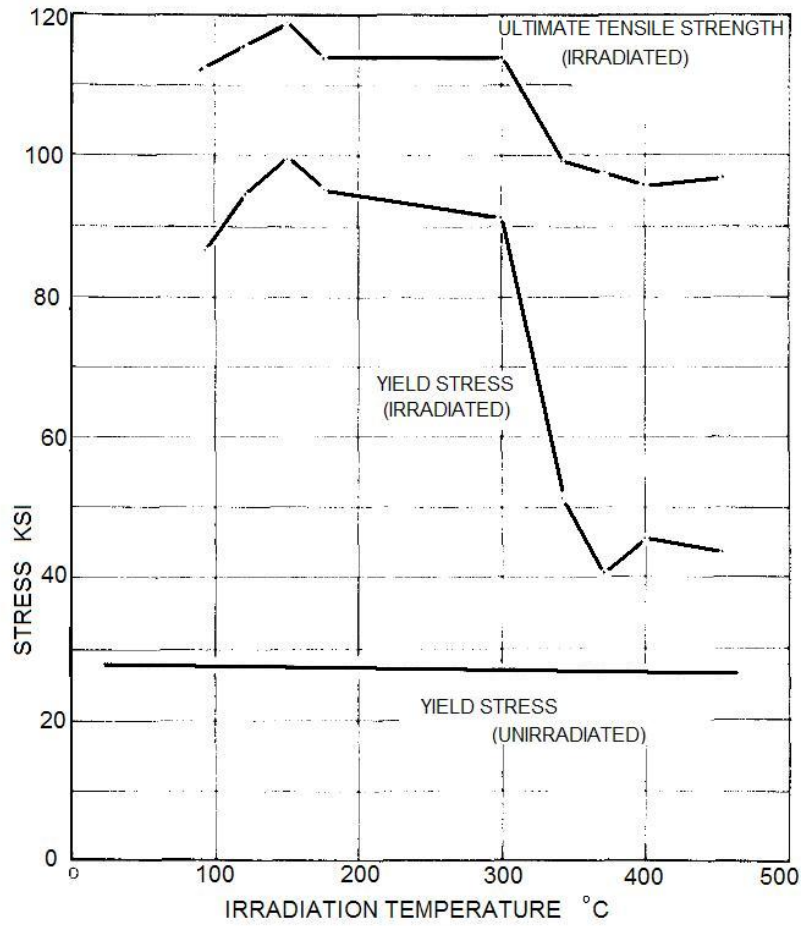
**Figure 12.** The effect of test temperature on yield stress and ultimate tensile strength of annealed and 25% cold worked 304 stainless steel irradiated at 60°C [14]. (Fluence is reported for neutrons with  $E > 1$  MeV)



**Figure 13.** The effect of test temperature on ductility of annealed and 25% cold worked 304 stainless steel irradiated at 60°C [14]. (Fluence is reported for neutrons with  $E > 1$  MeV)

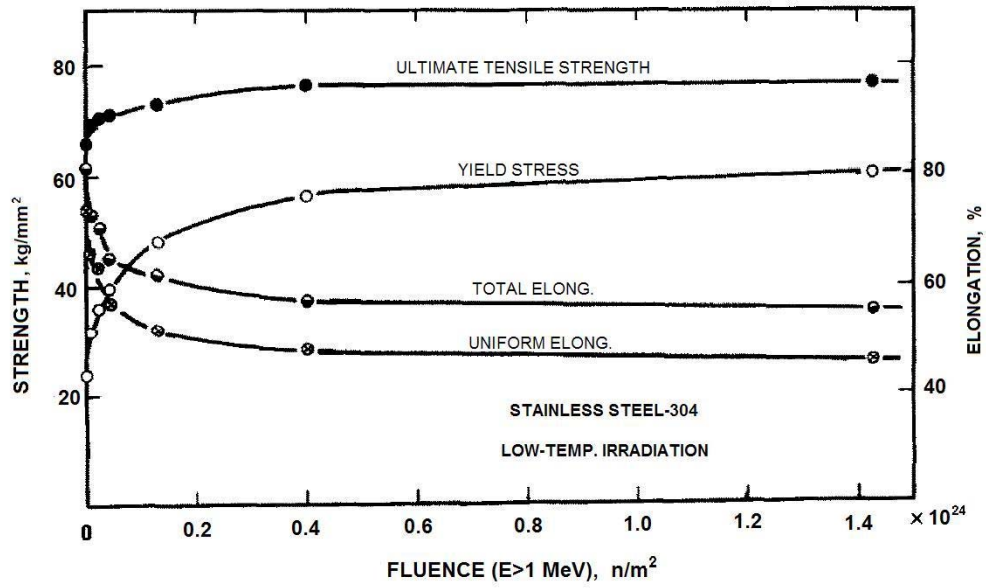


**Figure 14.** The effect of test temperature on uniform elongation of annealed 304 stainless steel irradiated at 290°C [14]. (Fluence is reported for neutrons with  $E > 1$  MeV)

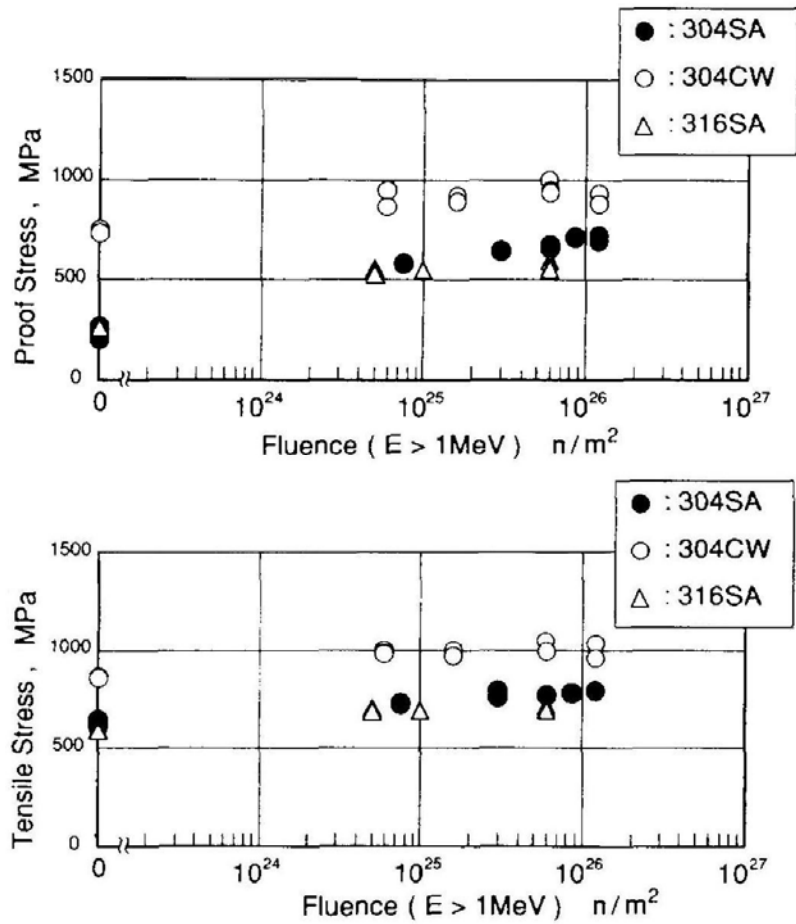


**Figure 15.** The effect of irradiation temperature on yield stress and ultimate tensile strength of annealed 304 stainless steel tested at room temperature. The irradiation fluence was  $7 \times 10^{24}$  n/m<sup>2</sup> ( $E > 1$  MeV) [15].

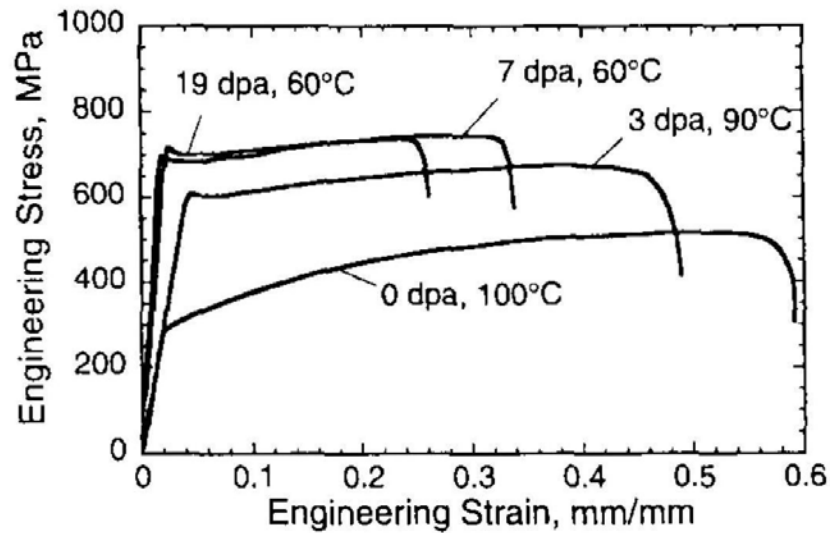




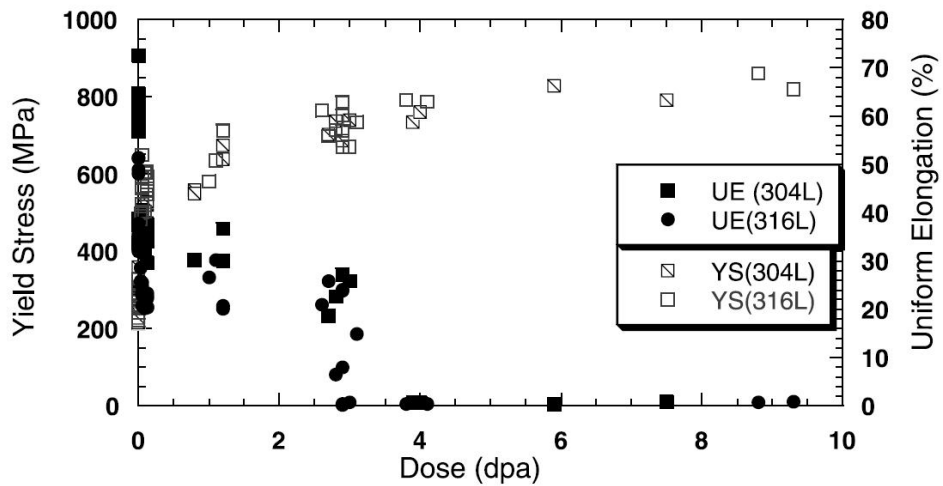
**Figure 16.** The effect of fluence on room temperature tensile properties of 304 stainless steel irradiated at 50°C [16].



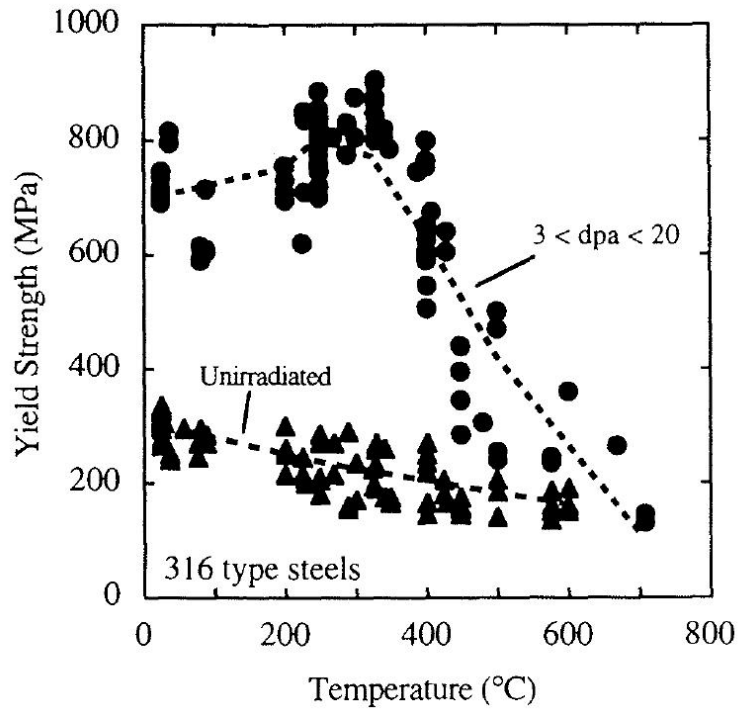
**Figure 17.** The effect of irradiation at 50°C on proof stress and ultimate tensile strength of type 304 steel in solution annealed (SA) and cold worked (CW) conditions and solution annealed type 316 stainless steels as a function of fluence [20].



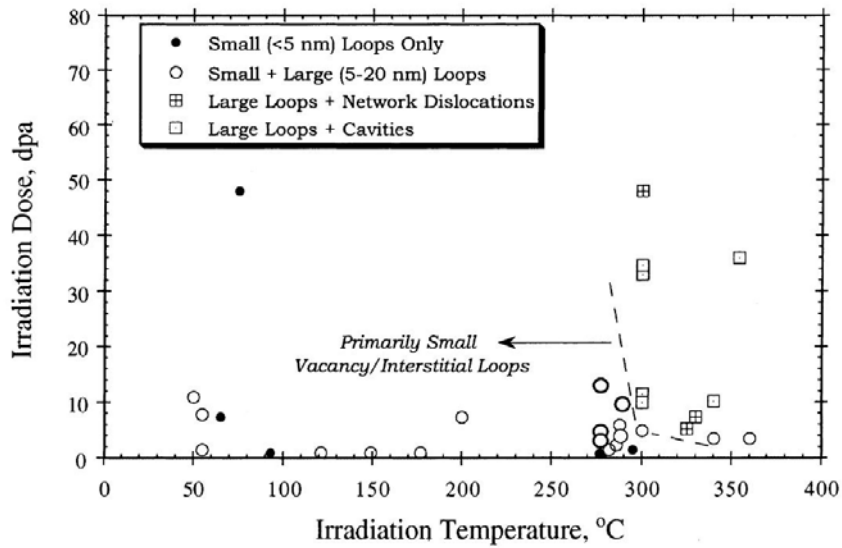
**Figure 18.** Typical stress-strain curves for solution annealed 316 steel specimens irradiated and tested at temperatures below 100°C [48].



**Figure 19.** Effect of irradiation dose on uniform elongation (UE) and yield stress (YS) for 304L and 316L stainless steels in a high energy proton and spallation neutron environment. The irradiation and test temperatures were between 50 and 160°C [23].



**Figure 20.** Effect of irradiation/test-temperature on yield stress of irradiated solution annealed 316 stainless steels [41].



**Figure 21.** Reported defect structures in 300-series stainless steels as a function of irradiation temperature and dose [32].