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The effects of low dose rate irradiation and thermal aging on reactor structural alloys

T.R. Allen *, C.L. Trybus, J.I. Cole

Argonne National Laboratory-West, P.O. Box 2528, Idaho Falls, ID 83403-2528, USA

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Abstract

As part of the EBR-II reactor materials surveillance program, test samples of fifteen different alloys were placed into EBR-II in 1965. The surveillance (SURV) program was intended to determine property changes in reactor structural materials caused by irradiation and thermal aging. In this work, the effect of low dose rate (approximately 2×10^{-8} dpa/s) irradiation at 380–410°C and long term thermal aging at 371°C on the properties of 20% cold worked 304 stainless steel, 420 stainless steel, Inconel X750, 304/308 stainless weld material, and 17-4 PH steel are evaluated. Doses of up to 6.8 dpa and thermal aging to 2994 days did not significantly affect the density of these alloys. The strength of 304 SS, X750, 17-4 PH, and 304/308 weld material increased with irradiation. In contrast, the strength of 420 stainless steel decreased with irradiation. Irradiation decreased the impact energy in both Inconel X750 and 17-4 PH steel. Thermal aging decreased the impact energy in 17-4 PH steel and increased the impact energy in Inconel X750. Tensile property comparisons of 304 SURV samples with 304 samples irradiated in EBR-II at a higher dose rate show that the higher dose rate samples had greater increases in strength and greater losses in ductility. © 1999 Elsevier Science B.V. All rights reserved.

1. Introduction

As part of the EBR-II reactor materials surveillance (SURV) program [1–5], test samples of fifteen different alloys were placed into EBR-II in 1965, with the intention of determining microstructural and mechanical property changes in these alloys due to irradiation and thermal aging. A total of 10 identical SURV subassemblies (labeled SURV 1–SURV 10) were irradiated in the blanket region of the reactor core or thermally aged in the primary sodium tank storage basket. Subassemblies were then removed at intervals for testing and examination throughout the first 18 years of reactor operation. The samples experienced four possible environments: irradiated at 380–410°C in flowing sodium, irradiated at 380–410°C in a helium atmosphere, thermally aged at 371°C in a flowing sodium atmosphere, and thermally aged at 371°C in a helium atmosphere. The temperature of the irradiated samples was a function of the axial position in the core. The thermally aged samples were aged at the reactor inlet temperature. Six sample types were included in the surveillance program: hardness/density cylinders, tensile bars, impact specimens, bend test specimens, and springs. This work reports the changes in density, tensile properties, and impact strength for five Fe–Cr–Ni alloys irradiated at 380–410°C to fluences up to 3.2×10^{26} n/m^2 ($E > 0.1$ MeV) (a maximum dose of 14 dpa) and thermally aged at 371°C for 2994 days. The materials analyzed are 304 stainless steel, 420 stainless steel, nickel-base Inconel X750, 304/308 stainless steel weld material, and 17-4 PH steel. Samples from six irradiated subassemblies (SURV-1, SURV-2, SURV-3, SURV-4, SURV-5, and SURV-8) and one thermally aged subassembly (SURV-6) were analyzed. The 2994 days of thermal aging for SURV-6 occurred over the same time period as the irradiation of samples from SURV-4.

The peak displacement rate for the materials in the SURV subassemblies was approximately 2×10^{-8} dpa/s. This displacement rate is about two orders of magnitude

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* Corresponding author. Tel.: +1-208 533 7760; fax: +1-208 533 7863; e-mail: todd.allen@anlw.anl.gov

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lower than used in a typical accelerated reactor materials test but within the range of displacement rates experienced by commercial light-water reactor (LWR) core components [6]. While significant data on the mechanical properties of irradiated structural alloys exists in the literature (for a review see Refs. [7,8]), the data typically comes from high displacement rate experiments. Since some material's properties are known to be displacement rate dependent, the effect of low dose rate irradiation on mechanical properties needs to be determined. This work presents trends in room temperature mechanical properties from the low dose rate SURV experiments conducted in EBR-II. Additionally, the changes in mechanical properties caused by low dose rate irradiation are compared to the changes in mechanical properties caused by thermal aging to isolate temperature effects from radiation effects.

2. Experiment

Table 1 lists the bulk composition of the five alloys reported in this work. Samples from seven different subassemblies were examined. Six of the subassemblies were irradiated at temperatures ranging 380–410°C to peak fast fluences ($E > 0.1$ MeV) of 0.5×10^{25}, 1.3×10^{25}, 5.2×10^{25}, 9.3×10^{25}, 1.4×10^{26}, and 3.2×10^{26} n/m^2. A single subassembly was thermally aged at 371°C for 2994 days. The thermally aged samples were held at temperature for the same length of time that the samples from SURV-4 (9.3×10^{25} n/m^2) were irradiated. Doses were calculated from reactor fluence using NJOY flux-to-dpa cross sections. Although displacement rates varied along the length of each subassembly, the peak displacement rate was approximately 2×10^{-8} dpa/s. Fig. 1 presents the dimensions of the samples used in the SURV tests. The results from measurements on the hardness/corrosion cylinders, bend test bars, tensile specimens, and Izod impact specimens are discussed in this work. An Izod impact test differs from a Charpy impact test in that an Izod specimen is mounted vertically and multiple impacts can be made on a single sample (see Fig. 1).

Each alloy had a specific heat treatment and specified condition. The samples of 304 stainless were 20% cold-worked with a stress relief heat treatment of 468–496°C for 2 h following machining. The 304/308 weld material received a stress relief heat treatment of 468–496°C for 2 h following machining. No heat treatment of the 420 stainless steel was specified after receipt, but the hardness was required to be 40–45 Rockwell C. The high tensile and yield strengths and the low elongation of the unirradiated 420 indicate the 420 specimens underwent a low temperature temper (approximately 400–550°C). The X750 underwent a complicated heat treatment, putting the material in the equalized and aged (AH) condition. The first step was an anneal starting at 427°C and ramping to 1148°C at 167°C per hour. The samples were held at 1148°C for 4 h. Following rough machining, the samples were age hardened, starting at 427°C and ramping to 885°C at 167°C per hour. The samples were held at 885°C for 24 h. The final heat treatment started at 427°C and ramped to 704°C at 167°C per hour. The samples were held at 704°C for 20 h and then air cooled and ground to finish dimensions. The final hardness was required to be 37–40 Rockwell C. The 17-4 PH steel was hardened to 36–41 Rockwell C and stress relieved at 468–496°C for 2 h. The 304/308 tensile specimens consist of 304 stainless steel welded together with 308 stainless steel filler. The composition of the 308 weld rod metal was chosen to have a free ferrite content in the range of 4–10%, with a goal of approximately five percent free ferrite in the deposited weld.

Density was measured on all five alloys using an immersion density technique. To determine tensile properties, stress–strain curves were obtained at room temperature for all four alloys. Yield strength (0.5% offset), ultimate tensile strength, reduction in area, and uniform elongation were determined. Tensile tests were performed on multiple samples from each subassembly at a strain rate of 2×10^{-2} min^{-1}. Table 2 lists the number of tensile samples analyzed from each set of SURV subassemblies.

Impact energy was measured on Inconel X750 and 17-4 PH stainless steel at temperatures ranging from −24°C to 226°C. The subsized Izod impact specimens were tested using a Warner–Swazey Model BLI impact tester with a maximum impact energy of 22 J delivered at 3.5 m/s.

To determine the strengthening effect of fluence on the 304/308 weld material, bend test samples were tested at ambient temperature. The samples were supported on round pins on 2-in. centers, with the load applied perpendicularly to the center of the 3.75 × 0.424-in. face by a third round pin. Crosshead speed was 0.2 in./min. Maximum force of deflection was measured for each sample tested.

3. Results

There was no discernible differences of the sodium and helium atmospheres on the density, tensile, impact, or bend properties of any of the five alloys analyzed. Therefore, all of the data from a given subassembly is grouped together for analysis purposes, regardless of environment.

Sodium Compatibility. Weight change was measured in the samples of Inconel X750, 304 stainless steel, 420 stainless steel, and 17-4 PH steel exposed to sodium. The weight loss after 2994 days exposure for all four alloys was less than 0.001%. Metallographic examination did

Table 1
Composition of test samples (wt%)

Material	C	Cr	Cu	Fe	Mn	Mo	Ni	P	S	Si	Other	Sample type
X750	0.04	14.82	0.11	6.39	0.57		Balance		0.007	0.32	0.70 Al, 2.47 Ti, 0.84 Nb + Ta	Density
	0.03	14.7	0.09	6.52	0.55		Balance		0.007	0.27	0.78 Al, 2.54 Ti, 0.94 Nb + Ta	Tensile
420 stainless	0.36	13.47		Balance	0.38	0.02	0.22	0.018	0.010	0.42		Tensile
	0.33	13.47	0.10	Balance	0.40	0.03	0.25	0.013	0.007	0.38		Density
304 SS welded with	0.02	18.90	0.04	Balance	0.68	0.37	9.01	0.026	0.028	0.69		Tensile
308 SS filler	0.04	20.50		Balance	1.32		9.80			0.44		
304 stainless	0.08	18.38	0.18	Balance	0.89	0.21	10.00	0.018	0.020	0.68		
17-4PH	0.031	15.99	3.40	Balance	0.23		4.34	0.014	0.014	0.58	0.27 Nb, 0.02 Ta	Hardness
	0.046	16.04	3.48	Balance	0.31		4.16	0.018	0.019	0.69	0.25 Nb, 0.01 Ta	Tensile
	0.041	16.28	3.45	Balance	0.32		4.08	0.016	0.018	0.62	0.32 Nb, 0.02 Ta	Impact

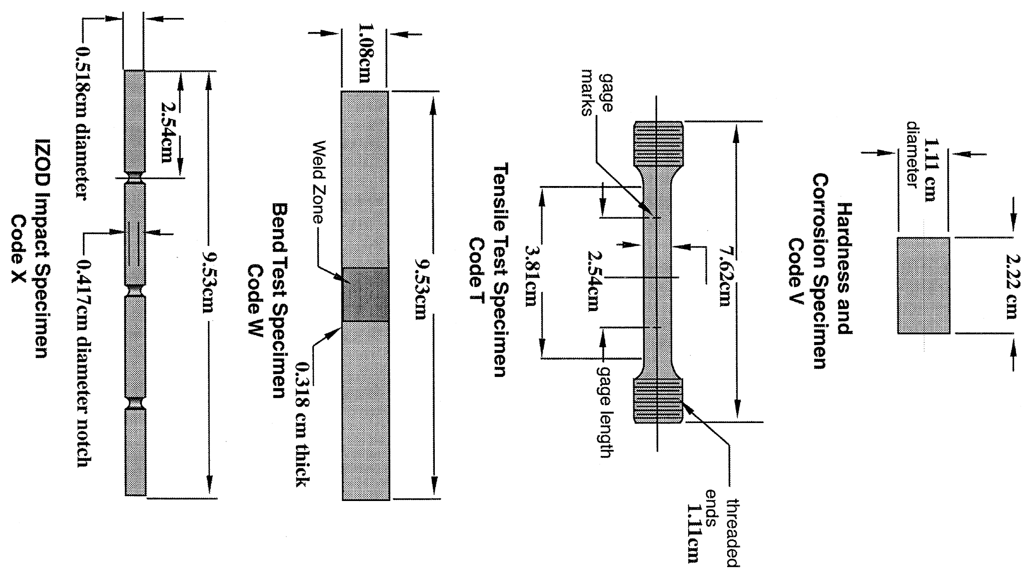

Fig. 1. Sample geometries for SURV test specimens.

not indicate any significant changes in any of these four alloys.

Density. Densities as a function of fast fluence and thermal aging time are listed in Table 3 for the five alloys examined. For SURV 1–6, neither irradiation or thermal aging had a significant effect on density. The largest change in density for SURV 1–6 was a 0.4% decrease in 304 stainless steel from SURV-5.

Strength. Yield strength and ultimate tensile strength are plotted as a function of the square root of dose in Figs. 2 and 3, respectively. Average changes in strength for irradiated (SURV-4) and thermally aged (SURV-6) samples of 304 stainless, 420 stainless, Inconel X750, and 304/308 weld metal are listed in Tables 4–7, respectively.

The yield strength and ultimate tensile strength of 304 stainless, Inconel X750, and 304/308 stainless weld

Table 2
Distribution of tensile samples analyzed

Experiment	304 stainless	420 stainless	Inconel X750	304/308 stainless	17-4 PH
Control	3	3	3	3	3
SURV-1	7	8	8	8	8
SURV-3	2	2	2	2	–
SURV-4	4	4	4	4	–
SURV-5	4	4	4	4	–
SURV-6	3	4	4	4	–

Table 3
Density (g/cm^3) for SURV alloys as a function of subassembly peak fluence

Alloy	Control	1.3×10^{25} n/m^2, $E > 0.1$ MeV, SURV-2	5.2×10^{25} n/m^2, $E > 0.1$ MeV, SURV-3	9.3×10^{25} n/m^2, $E > 0.1$ MeV, SURV-4	1.4×10^{26} n/m^2, $E > 0.1$ MeV, SURV-5	2994 days at 371°C SURV-6
Inconel X-750	8.267	8.284	8.271	8.258	8.259	8.257
304 SS	7.935	7.936	7.926	7.906	7.905	7.925
420 SS	7.698	7.703	7.700	7.689	7.692	7.689
17-4 PH	7.750	7.749	7.745	7.736	7.737	7.731

material all increase with irradiation dose while the yield and ultimate tensile strength of 420 stainless decrease with dose. Thermal aging to 2994 days increases the yield and ultimate strength of 304 stainless steel by amounts comparable to the increase caused by 2 dpa of irradiation. Thermal aging to 2994 days increases the yield strength of X750 slightly (about an 8% increase), but has no effect on the ultimate tensile strength. Thermal aging to 2994 days causes a slight increase on the yield strength of 420 stainless, but decreases the ultimate tensile strength of 420 stainless by about 8%. The yield and ultimate tensile strength of the 304/308 weld material are not significantly affected by the thermal aging. The ratio of ultimate tensile strength to yield strength as a function of square root of dpa is plotted in Fig. 4. For all four alloys at low dose, the ratio decreases with irradiation dose. The decrease is largest for the 304/308 weld material and smallest for the 304 and 420 stainless steels. At higher dose, the ratio increases slightly for 304 stainless steel and Inconel X750.

Tensile properties for 17-4 PH tensile samples were measured for SURV-1, but are not available for longer irradiation times because the samples from SURV-3 broke at the grips and no further tensile tests on irradiated 17-4 PH were attempted. Table 8 lists the unirradiated tensile properties along with the tensile

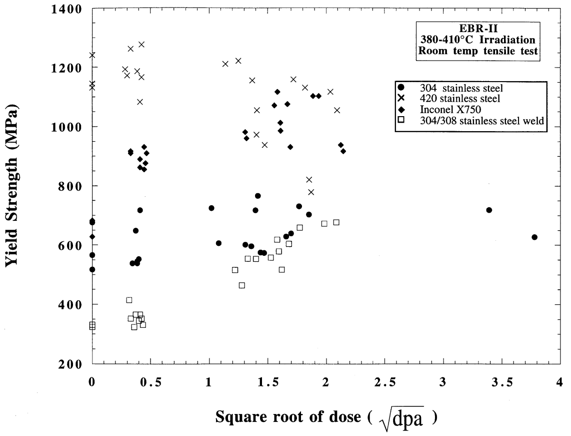

Fig. 2. Yield stress as a function of square root of dose.

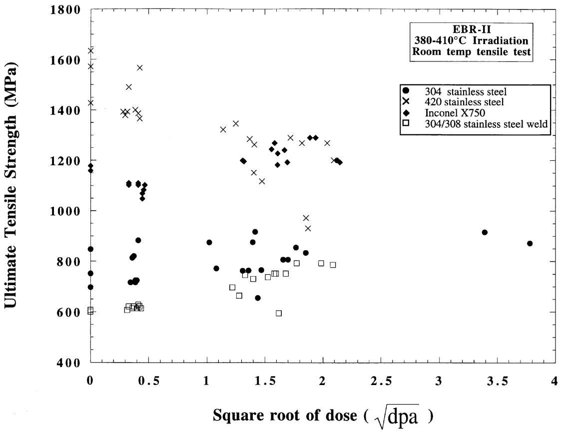

Fig. 3. Ultimate tensile strength as a function of square root of dose.

Table 4
Mechanical properties of 304 stainless steel

Experiment	Average dose (dpa)	Aging time (days)	Average UTS (MPa)	Average yield strength (MPa)	Average reduction in area (%)	Average uniform elongation (%)
Control	0	0	766	586	65	40
SURV-4	2.0	2994	807	655	70	45
SURV-6	–	2994	834	690	72	45

Table 5
Mechanical properties of 420 stainless steel

Experiment	Average dose (dpa)	Aging time (days)	Average UTS (MPa)	Average yield strength (MPa)	Average reduction in area (%)	Average uniform elongation (%)
Control	0	0	1545	1172	36	6
SURV-4	2.6	2994	1207	1048	47	12
SURV-6	–	2994	1428	1214	45	12

Table 6
Mechanical properties of Inconel X750

Experiment	Average dose (dpa)	Aging time (days)	Average UTS (MPa)	Average yield strength (MPa)	Average reduction in area (%)	Average uniform elongation (%)
Control	0	0	1172	662	24	24
SURV-4	2.7	2994	1234	1028	26	13
SURV-6	–	2994	1159	717	28	25

Table 7
Mechanical properties of 304/308 weld material

Experiment	Average dose (dpa)	Aging time (days)	Average UTS (MPa)	Average yield strength (MPa)	Average reduction in area (%)	Average uniform elongation (%)
Control	0	0	607	324	59	38
SURV-4	2.5	2994	710	566	51	19
SURV-6	–	2994	628	303	67	31

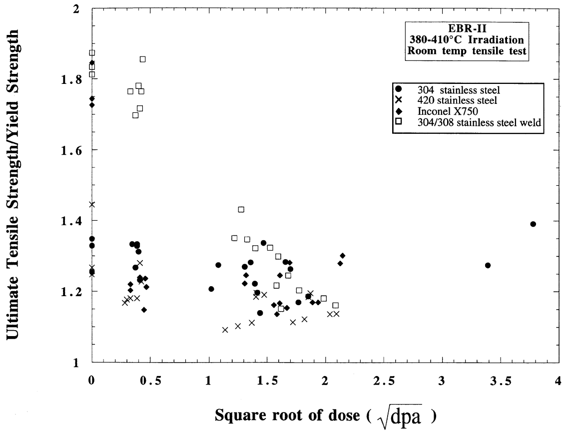

Fig. 4. Ratio of ultimate tensile stress to yield stress as a function of square root of dose.

properties from SURV-1 for 17-4 PH steel. The small irradiation dose (0.1 dpa) and/or aging time led to significant hardening.

Reduction in area. The relationship between reduction in area and dose is shown in Fig. 5. The changes in reduction in area for irradiated (SURV-4) and thermally aged (SURV-6) samples are listed in Tables 4–7. The reduction in area increases slightly with irradiation dose for 304 and 420 stainless steel, decreases slightly for the 304/308 weld material, and is relatively constant for Inconel X750. Thermal aging causes the reduction in area to increase slightly in 304 stainless, 420 stainless, and 304/308 weld material, while having little effect in Inconel X750.

Uniform elongation. Changes in uniform elongation as a function of dose can be seen in Fig. 6. The changes in uniform elongation for irradiated (SURV-4) and thermally aged (SURV-6) samples are listed in Tables 4–7. Irradiation causes the uniform elongation to increase

Table 8
Mechanical properties of 17-4 PH steel

Experiment	Average dose (dpa)	Aging time (days)	Average UTS (MPa)	Average yield strength (MPa)	Average reduction in area (%)	Average uniform elongation (%)
Control	0	0	174	168	53	17
SURV-1	0.1	1021	213	204	36	11

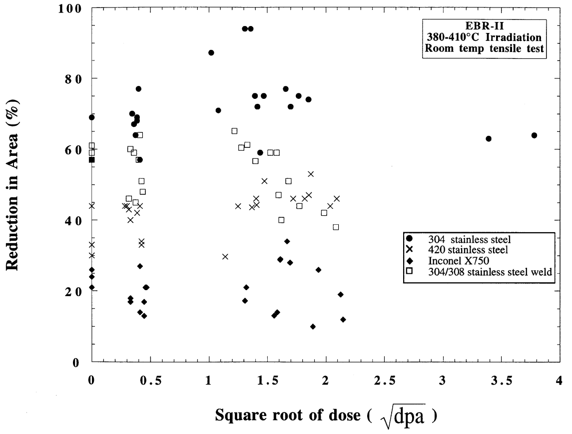

Fig. 5. Reduction in area as a function of square root of dose.

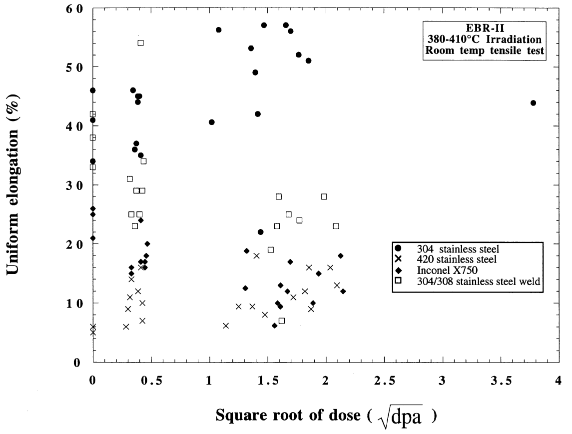

Fig. 6. Uniform elongation as a function of square root of dose.

in 304 stainless, nearly double in 420 stainless steel, decrease in Inconel X750, and decrease in the 304/308 weld material. Thermal aging causes the uniform elongation to remain fairly constant in 304 stainless steel, double in 420 stainless steel, remain constant in Inconel X750, and decrease slightly in the 304/308 weld material. The accuracy of the elongation measurements in the 304/308 weld material are less reliable because many of the samples broke at or outside the gauge marks. The failure was therefore in the parent material and not the weld.

Uniform elongation is plotted versus $1-\sigma_y/\sigma_u$ (where σ_y is the yield strength and σ_u is the ultimate tensile strength) in Fig. 7. For 304 stainless, Inconel X750, and the 304/308 weld material, as the yield strength approaches the ultimate tensile strength (as $1-\sigma_y/\sigma_u$ decreases), the uniform elongation decreases. For 420 stainless steel, as the yield strength approaches the ultimate tensile strength (as $1-\sigma_y/\sigma_u$ decreases), the uniform elongation increases very slightly.

Metallography. To assist in comparing the effects of radiation and thermal aging, samples SURV-4 and SURV-6 were etched and photographs at 500 times magnification were taken. Optical metallography did not reveal anything to explain differences between irradiation and thermal aging.

Impact data. In addition to tensile measurements, the impact strength of Inconel X750 was measured at temperatures ranging from −24°C to 226°C. The impact energy for Inconel X750 as a function of temperature is shown in Fig. 8. Increasing radiation dose causes the impact strength to decrease, while thermal aging causes an increase. The impact energy for 17-4 PH steel as a function of temperature is shown in Fig. 9. Increasing radiation dose and thermal aging both cause the impact strength to decrease.

Bend test data. Many of the 304/308 tensile specimens broke outside the gauge marks in the parent 304 material rather than in the weld material. Therefore, the mechanical property of the weld itself was not measured.

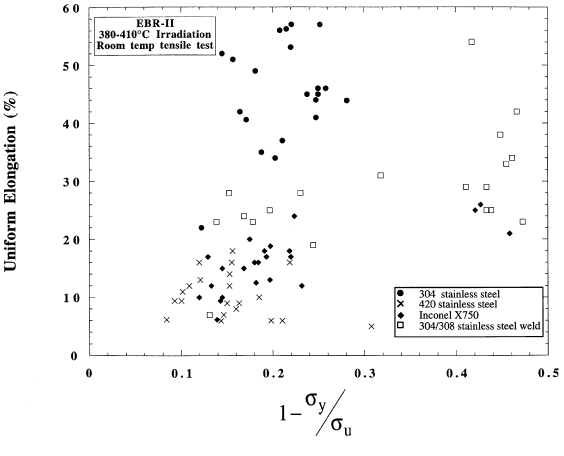

Fig. 7. Uniform elongation as a function of strength ratios.

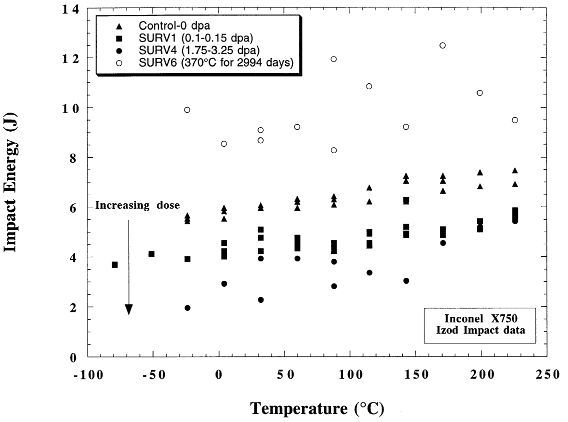

Fig. 8. Impact energy of Inconnel X750 as a function of temperature.

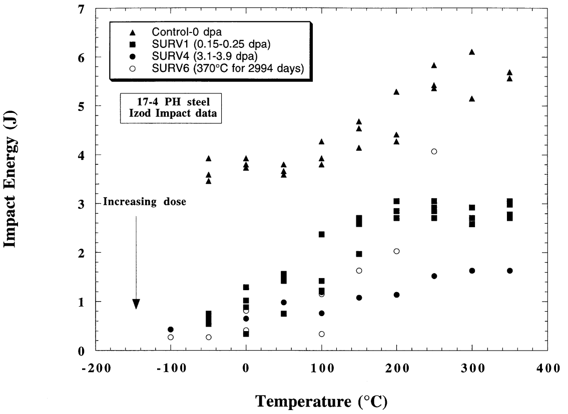

Fig. 9. Impact energy for 17-4 PH steel as a function of temperature.

As a secondary method of determining weld strength, bend tests were performed. The results from control samples SURV-4 and SURV-6 are presented in Table 9. Irradiation (SURV-4) increases the maximum force during deflection by 33% while thermal aging (SURV-6) has little effect.

4. Discussion

The mechanical properties of each of the five alloys have a different response to irradiation and thermal aging. The changes in mechanical properties of each alloy will be discussed and possible mechanisms will be presented. Because no samples were examined using transmission electron microscopy and because the sample material from SURV-1 to SURV-6 is no longer available for examination, microstructural causes for the changes in mechanical properties are presented as a best hypothesis.

Swelling. Irradiation and thermal aging at 371°C had little effect on the density of any of the five alloys studied for doses up to 6.8 dpa and aging times of 2994 days. Therefore, dimensional instability is not a problem for these doses and thermal aging times. These findings are consistent with similar observations of high dose rate 316 stainless samples as summarized by Maziasz [9] who suggests that the large cavity formation which causes swelling is not expected below about 400°C for doses less than 7 dpa.

304 stainless steel. The 304 stainless steel went into the test assemblies with only a stress relief heat treatment and the initial high yield strength (586 MPa) corresponds to residual cold work. Both the low dose irradiation and the thermal aging cause the 304 stainless to harden while attaining greater uniform elongation prior to failure. A similar increase in yield and ultimate tensile strength, with a corresponding increase in uniform elongation at low dose was reported by Fish et al. [10] for 20% cold worked 316 stainless steel irradiated in EBR-II and tested at 371°C.

The possible effect of dose rate on the mechanical properties of 304 stainless steel can be examined by comparing the data from the SURV samples to the data of Fish and Hunter [11]. Fish and Hunter also examined the room temperature tensile properties of 304 stainless steel irradiated in EBR-II at 371°C, but in their study, the flux was a factor of seven higher than that of the SURV subassemblies while the strain rate was the same for both test sets. Yield and ultimate tensile strength for the current study and the Fish and Hunter study are plotted in Figs. 10 and 11 as a function of dose. Additionally, the reduction in area and uniform elongation are plotted in Figs. 12 and 13. The fluence data of Fish and Hunter have been converted to dpa using an EBR-II thumbrule of 1 dpa $= 2 \times 10^{25}$ n/m^2 ($E > 0.1$ MeV).

Mechanical behavior between the two studies is different. For the SURV 304 samples, the yield strength has reached a peak of around 700 MPa, while the yield strength of 304 in the Fish study plateaus around 900 MPa. Similarly, the ultimate tensile strength of the 304

Table 9
Bend test properties of 304/308 weld material

	Control sample	SURV-4	SURV-6
Range of maximum load (N) [a]	1175–1255	1544–1682	1139–1233

[a] Maximum load occurred at approximately 1 cm of crosshead travel.

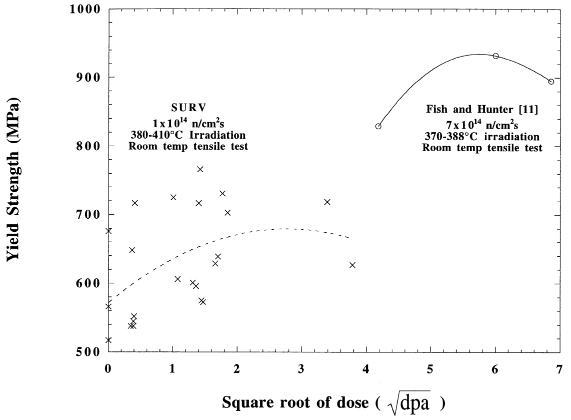

Fig. 10. Yield strength as a function of fluence for 304 stainless steel irradiated in EBR-II.

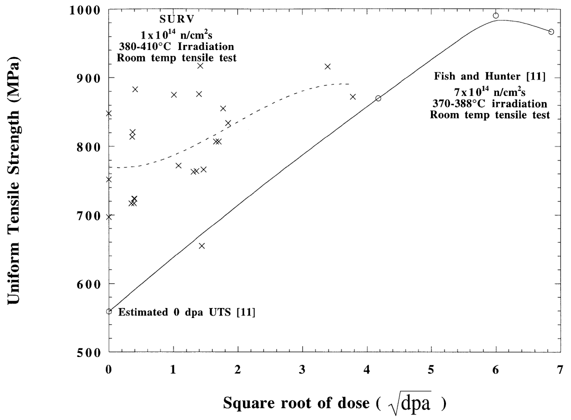

Fig. 11. Uniform tensile strength as a function of fluence for 304 stainless steel irradiated in EBR-II.

irradiated in the Fish study plateaus at about 975 MPa while the ultimate tensile strength of SURV specimens is peaking around 900 MPa. The reduction in area for the SURV subassemblies is relatively constant with dose up to 16 dpa while the reduction in area decreases sharply for the Fish and Hunter data. Finally, the uniform elongation also differs between the two data sets. For the SURV subassemblies, the uniform elongation increases at low fluence and then decreases. As mentioned previously, this increase and decrease in uniform elongation at low fluence was also seen by Fish et al. [10] in 316 stainless steel and is plotted in Fig. 13 for comparison. In summarizing Figs. 10–13, the SURV 304 stainless steel irradiated at a lower dose rate is more ductile and has lower strength than the 304 irradiated at a higher dose rate in the Fish and Hunter study. A similar dose rate effect was seen in 304 stainless steel irradiated in EBR-II at 371°C and tested at 371°C [12,13]. The lower dose rate samples had a lower yield strength and greater uniform strain.

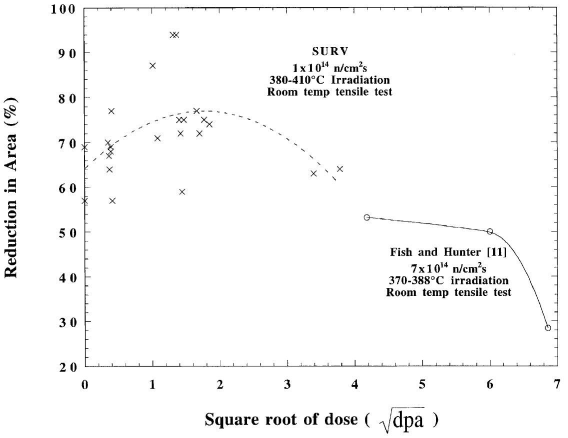

Fig. 12. Reduction in area as a function of fluence for 304 stainless steel irradiated in EBR-II.

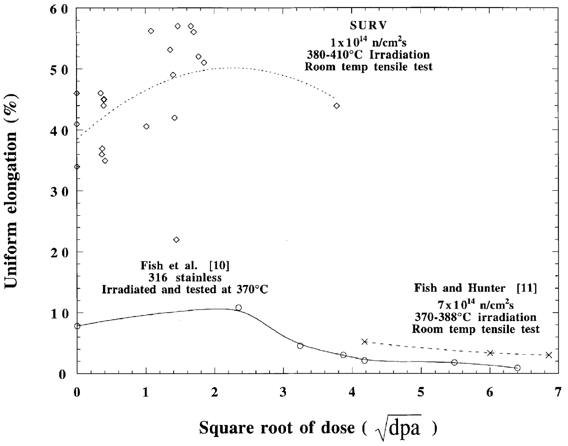

Fig. 13. Uniform elongation as a function of fluence for 304 stainless steel irradiated in EBR-II.

The SURV samples were irradiated at a slightly higher temperature than the samples from the Fish study. To conclude that the difference in mechanical behavior is primarily due to dose rate, the effect of temperature must be eliminated. A detailed study of the effect of temperature on the yield strength of 316 stainless can be used to estimate if a temperature difference is responsible for the yield strength difference in the 304 samples. Fish et al. [10] showed that the difference in yield strength of 316 stainless steel between samples irradiated and tested at 427°C and those irradiated and tested at 371°C was about 100 MPa. The SURV samples were irradiated between 380 °C and 410°C. Using the 316 data as a guide, the yield strength of the SURV 304 samples should be about 15–70 MPa less than yield strength of samples from the study of Fish et al. The actual difference is about 200 MPa. Therefore, the lower dose rate of the SURV samples appears to cause a measurably lower yield strength. Similarly, Grossbeck [15] derived empirical relationships for the yield strength as a function of temperature in austenitic steels. His expression for the yield strength in stainless steel alloys irradiated to doses between 10 and 15 dpa is

$$\mathrm{YS} = 1025\left\{1 - \exp\left[-\left(\frac{665 - T}{120}\right)\right]\right\} - 235. \tag{1}$$

Using this expression, the expected difference in yield strength between 371°C and 410°C is 35 Mpa, which is much smaller than the difference in yield strength between the data of Fish and the SURV data.

Although the comparison of the two experiments indicates a possible dose rate difference, the comparison is not conclusive. Three limitations prevent a firm conclusion. First, the doses to which the samples were irradiated do not overlap. The highest dose analyzed from the SURV samples is 14 dpa while the lowest dose from the Fish and Hunter study was around 18 dpa. Therefore, any comparison between the two sets of data requires an extrapolation of the trends in the SURV samples. Second, the two sets of samples did not come from the same heat of steel and did not have the same initial amount of cold work. Fish and Hunter estimated the 0 dpa UTS to be much lower than that measured in the SURV samples. Finally, because of the large amount of scatter in the measurements from the SURV samples, predicting the trends in the mechanical properties to higher doses is difficult. In this case, the trend lines for the SURV data on Figs. 10–13 are best fit polynomials which may not describe the behavior at higher dose. While the data indicates a difference may exist due to dose rate, a better controlled experiment is needed to make conclusive statements.

The similar properties in the irradiated and thermally aged 304 stainless steel may be explained in terms of possible microstructure changes. An increase in strength and uniform elongation of the irradiated (SURV-4) and thermally aged (SURV-6) samples suggests that long-term thermal aging results in phase instability of the 304 stainless steel, particularly because the cold-worked microstructure can enhance nucleation of second phase particles. Radiation-induced phases have been observed to form at higher dose/temperature regimes [9] and many of these phases (for instance $M_{23}C_6$, M_6C, Laves, σ and χ) are identical to phases known to form thermally but at higher temperatures. These same phases, although not normally observed under thermal aging, may have formed during the very long term, low temperature thermal aging and contribute to the increased strength and uniform elongation.

420 stainless steel. Irradiation causes a significant loss in yield strength and ultimate tensile strength in 420 stainless steel, with a corresponding increase in reduction in area and increase in uniform elongation. The loss of strength in the irradiated 420 is similar to the loss of strength expected when a martensitic steel is held at temperatures of 600°C and above [14]. This loss of strength may be attributed to a change in the carbide structure to a coarser distribution of $M_{23}C_6$ and M_7C_3 precipitate [20]. The loss of strength may also be related to a transformation of martensite to a tempered martensite. In the irradiated 420, the influx of point defects may act to reduce martensite stability and accelerate transformation to ferrite. Long term thermal aging at 371°C also decreases strength and while improving ductility of 420 stainless, but at a significantly slower rate than the irradiation. This lower temperature decrease in hardening may be related to an increase in the number of cementite precipitates, with a decrease in the amount of carbon in solid solution [20].

Inconel X750. Alloy X750 is used primarily for its high strength and corrosion resistance although it can be susceptible to stress corrosion cracking in a light water reactor environment [16]. In this study, however, exposure to an aggressive versus non-aggressive environment (sodium vs. helium) at relatively low temperature failed to induce a measurable difference in tensile properties. In both cases, the yield strength and ultimate tensile strength increase and the uniform elongation decreases with dose. The increase in strength reaches a plateau by approximately 2.5 dpa. This is consistent with classic radiation hardening caused by the development and evolution of irradiation-produced defects. In contrast, thermal aging has little effect on X750 tensile properties. The impact strength of X750 decreases with radiation and increases with thermal aging. The formation of a radiation-induced microstructure leads to a more brittle material while the thermal aging produces a recovery to a more ductile state. Kenik [17] has found the presence of small dislocation loops (5–25 nm in diameter) and isolated cavities (1.5–8 nm in diameter) in alloy X750 irradiated at 360°C to a fluence of 2.3×10^{24} n/m^2 ($E > 1$ MeV) or approximately 0.4 dpa. In terms of effectiveness as a strengthener, the small dislocation loops should be the most potent strengtheners while the small cavities will act as only moderate strengtheners [7]. Absence of swelling in the X750 SURV samples suggest that cavity formation is minimal.

304/308 stainless steel weld material. For the 304/308 weld material, the yield strength and ultimate tensile strength increase and the uniform elongation and reduction in area decrease with irradiation. Elongation measurements in the 304/308 weld material are less accurate because many of the samples broke at or outside the gauge marks. The failure was therefore in the parent material and not the weld. The 304/308 tensile samples, with a lower unirradiated yield strength, harden more rapidly than the 304 stainless steel. Thermal aging had little effect on the 304/308 weld samples.

Because the tensile properties were determined in a large part by the parent 304 material, the effect of radiation or thermal aging on the weld material is not definitive. On the other hand, the bend test measurements provide a more direct measurement of the effect of irradiation and thermal aging on the weld material. The bend test measurements indicate that radiation hardens the weld material while thermal aging has little effect, similar to the tensile results. The parent material and the weld material change in similar fashion in response to radiation and thermal aging.

Other work has found similar effects of irradiation and thermal aging on 304/308 weld material. The increases in room temperature yield strength and ultimate tensile strength are similar to those reported by Sindelar et al. [18] on 304/308 weld material irradiated to exposures between 0.1 and 4×10^{25} n/m^2 ($E > 0.1$ MeV) at temperatures less than 130°C. The data reported were an average for the entire dose range. Sindelar reported a yield strength increase of 275 MPa (from an initial value of 370 MPa) and an ultimate tensile strength increase of 115 MPa (from an initial value of 609 MPa). The tensile properties of the weld material and the base metal were quite similar.

Alexander and Nanstad [19] found that thermal aging of 308 weld material for 50,000 h at 343°C had little effect on tensile properties, with no increase in yield strength and only a small increase in ultimate tensile strength. These results were true for ferrite concentrations up to 12%. Although the tensile properties were not effected by thermal aging, Charpy impact tests showed an increase in transition temperature and decrease in upper shelf energy. The cracking susceptibility was related to the ferrite phase, which hardened with aging. Yielding and plastic flow were accommodated in the austenite phase which did not harden with aging, explaining the lack of change in tensile measurements.

17-4 PH steel. The impact energy of 17-4 decreased with both irradiation and thermal aging. Lowering the aging temperature is known to decrease the Charpy impact energy in 17-4 PH steel and increase the yield and ultimate tensile strength [20]. Anthony [21] has shown that prolonged exposure of 17-4 PH steel in the temperature range of 371–510°C causes four microstructural changes: (1) continued tempering of the martensitically transformed matrix, (2) continued overaging of the primary (Ni_3Cu) precipitate, (3) gradual ferrite to austenite transformation within the matrix, and (4) the embrittling precipitation of the alpha prime phase, a chromium-rich ferrite. In this study, both irradiation and thermal aging make the material more brittle, with no significant difference between irradiation and thermal aging on the impact behavior. The neutron exposure appears to be of less importance than the temperature at which the material is held. The chromium-rich alpha prime phase described by Anthony may be responsible for the embrittlement in both the irradiated and thermally aged SURV samples.

5. Conclusions

The effect of low dose rate irradiation and thermal aging at 371°C has been investigated for 304 stainless steel, 420 martensitic steel, Inconel X750, 17-4 PH steel, and 304/308 stainless steel weld material. Both the irradiated and thermally aged 304 stainless steel had small increases in strength and ductility, most likely due to second phase precipitation and annealing of cold work. The 304 tensile data was compared to tensile data from 304 stainless steel irradiated in EBR-II at the same temperature, but at a higher dose rate. The samples irradiated at a higher dose rate were stronger and less ductile material, but lack of overlap in dose and significant experimental scatter do not allow for a conclusive determination on the effect of dose rate. Both the irradiated and thermally aged 420 martensitic steel lost strength and gained ductility. The transformation occurred more quickly in the irradiated steel. Inconel X750 and 304/308 weld material undergo classic irradiation hardening, increasing strength and decreasing ductility. Thermal aging had little effect on either Inconel X750 or the 304/308 weld material. 17-4 PH steel becomes very brittle upon irradiation or thermal aging. The irradiation and thermal aging of 17-4 PH may create a distribution of chromium-rich precipitates that cause the loss of ductility.

Interpretation of the mechanical property measurements is complicated by a lack of microstructural data. Samples from experiments run to higher dose (up to 20 dpa) and longer thermal aging time (6525 days) remain from SURV-7 to SURV-10. The mechanical properties of these high dose samples need to be correlated with microstructure to better define the mechanisms of change under irradiation and thermal aging.

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