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FERRITE FORMATION
IN
NEUTRON-IRRADIATED
TYPE 316 STAINLESS STEEL

AEC Research and Development Report

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NEUTRON-IRRADIATED
TYPE 316 STAINLESS STEEL

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ABSTRACT

Magnetic measurements were made on Type 316 stainless steel specimens that had been neutron irradiated to fluences of 1.8×10^{22} neutrons/cm² at 425°C and 3.5×10^{22} neutrons/cm² ($E > 0.1$ MeV) at 500 and 600°C. A significant increase of magnetization was observed for the irradiated specimens compared to the unirradiated specimens. The shape of the magnetization versus field curves showed that the irradiated specimens contained many small superparamagnetic particles. The magnetic particles are assumed to be the ferrite phase although other possibilities cannot be excluded. The amount and distribution of the magnetic phase varied with pre-irradiation and post-irradiation heat treatment. The maximum value of magnetization was equivalent to 3.6% ferrite in a specimen annealed 100 hr at 760°C before irradiation and 1 hr at 500°C after irradiation at 425°C.

I. INTRODUCTION

Austenitic stainless steels undergo a number of microstructural changes when irradiated to a relatively high neutron fluence in the temperature range 400 to 600°C. For example, there is the formation of a large number of small voids, the precipitation of $M_{23}C_6$ carbides and an unidentified phase, the reversion of ϵ martensite, and the formation of faulted and unfaulted dislocation loops and dislocation networks.^(1,2) Since the macroscopic swelling caused by void formation creates problems in the design of fast breeder reactors, this phenomenon has been studied extensively.

A number of laboratories have also studied void formation by irradiation with charged particles (protons, carbon ions, nickel ions) to simulate neutron irradiations.^(1,2) The main advantage of charged particle irradiation is that the time needed to accumulate significant damage is much less than is needed for neutron irradiation. Recently Keefer, Pard, Rhodes and Kramer reported the detection of ferrite in proton-irradiated Types 316 and 321 stainless steels.^(3,4) In those cases in which Keefer et al were able to observe ferrite by transmission electron microscopy, the ferrite generally was found to form in relatively large particles, 2μ diameter and larger. In 1955 Reynolds, Low and Sullivan reported increased magnetization values in neutron irradiated Type 347 stainless steel.⁽⁵⁾ However, ferrite formation has not been reported in recent investigations on neutron irradiated specimens. Also, electron microscope studies of specimens similar to the specimens used in the present study failed to find ferrite.⁽⁶⁾ Because of the possibility that the ferrite particles, if present in neutron irradiated specimens, might be much smaller than in the proton irradiated specimens, we decided to measure the magnetization of neutron irradiated specimens as a method of detecting ferrite.

II. EXPERIMENTAL PROCEDURE

Two different specimen configurations were used in this investigation. The first group of specimens were pieces cut from the grip ends of miniature tensile samples, as shown in Figure 1. The specimens were ~ 2 by 4 by 0.23 mm. To determine the magnetization, we cemented these samples inside small pieces of varnished fiber electrical insulating tubing having an OD of ~ 2.5 mm. This was done to facilitate transfer of the sample in the test coil. Prior to cutting these specimens, the tensile samples were tested to failure at various temperatures.⁽⁶⁾ Magnetic measurements were also made on similar pieces cut from unirradiated control specimens that had been tested at the same temperatures as the irradiated samples.

The tensile-test temperature proved to be an important variable in the subsequent magnetic measurements, and the time at test temperature is undoubtedly important. Each specimen was held at the test temperature for 1 hr prior to testing. After testing, the specimens were cooled to room temperature in a few minutes. All of the tensile tests were performed at a constant crosshead speed; thus, the total time at temperature varied from ~ 65 to 70 min, depending on the ductility of the specimen. As the grip ends are not deformed during testing, the temperature of the tensile test will henceforth be referred to as the "post-irradiation annealing temperature."

The second group of specimens were small cylinders, 3.8 mm diameter by 3.5 mm long, prepared for wet density measurement after irradiation. As these specimens were larger than the Group 1 specimens, they were not mounted in insulating tubing. These specimens were made from a different heat of Type 316 stainless steel than were the Group 1 specimens.

The composition of the alloys used in this study and the irradiation conditions are given in Table 1. Prior to irradiation, the samples were given one of the heat treatments listed in Table 2. A short description of the resulting pre-irradiation microstructure is also given. The Group 1 specimens received heat treatments A, B, C, or D; the Group 2 specimens received heat treatments B, E, or F.

Figure 1. Configuration of Specimen
Cut From Miniature Tensile Specimen for Magnetic Measurements
(Dimensions in mm)

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TABLE 1
COMPOSITION AND IRRADIATION CONDITIONS FOR TYPE 316
STAINLESS STEEL USED FOR MAGNETIC MEASUREMENTS
(wt%)

Specimen							
	C	Cr	Ni	Mo	Fe	Irradiation Temperature (°C)	Fluence (E > 0.1 Mev) (n/cm ²)
Group 1	0.06	17.3	13.6	2.3	Bal	425	1.8×10^{22}
Group 2	0.04	16.7	11.6	2.4	Bal	500*	3.5×10^{22}
						600*	3.5×10^{22}

*Nominal temperature

TABLE 2
PRE-IRRADIATION HEAT TREATMENTS

Designation	Treatment	Preirradiation Microstructure
A	1 hr at 980 °C	Solution annealed, no precipitates
B	1 hr at 980 °C + 8 hr at 760 °C	Carbides, primarily at grain boundaries
C	100 hr at 760 °C	Carbides on grain boundaries, carbides and sigma particles in matrix, mostly recrystallized
D	30 min at 1120 °C + 25% cold-rolled + 24 hr at 480 °C	No precipitates, cold work retained
E	30 min at 1120 °C + 25% cold-rolled	No precipitates, cold worked structure
F	Same as B + 22% cold-rolled	Carbides, primarily at grain boundaries; cold worked structure

Magnetization measurements were made by rapidly transferring the sample between two small test coils wound in series opposition. The change in magnetic flux in the coils generates a voltage proportional to the rate of change of flux. A Hewlett-Packard integrating digital voltmeter with a manual start was used to integrate the voltage over a time period of 1 sec. Since specimen transfer was completed in a short time compared to 1 sec, a reading proportional to the change in flux was obtained. The techniques used were similar to those described by Piercy.⁽⁷⁾ The magnetization was measured as a function of applied field by immersing the sample and test coil in the field of an electromagnet with 4-in. diameter pole pieces. The magnetic field of the electromagnet was variable from about 3×10^3 A/m to 7.9×10^5 A/m. Additional measurements at fields up to 2.4×10^6 A/m were made for some of the samples using a Varian V-3100 electromagnet with 6-in. pole pieces. A different pair of test coils was used for each group of specimens due to the different sample configurations.

For the first group of specimens, the integrating digital voltmeter readings, $\langle E \rangle_t$, were converted to relative magnetization by the equation

$$\frac{M}{M_{\text{Fe-Cr}}} = \frac{\langle E \rangle t}{\langle E \rangle_{\text{Ni}} t} \frac{A^{\text{Ni}}}{A} \frac{M_s^{\text{Ni}}}{M_s^{\text{Fe-Cr}}} = C \langle E \rangle t / A \quad \dots (1)$$

where M is magnetization of the specimen, $M_s^{\text{Fe-Cr}}$ is the saturation magnetization of an Fe - 18% Cr alloy, M_s^{Ni} is the saturation magnetization of nickel, $\langle E \rangle$ is the average voltage reading for an integration period, t , and $\langle E \rangle^{\text{Ni}}$ is the average voltage reading for a nickel specimen of approximately the same geometry as the stainless steel specimens. A is the cross-sectional area of the specimen, and A^{Ni} is the cross-sectional area of the nickel specimen. The ratio of $M_s^{\text{Ni}}/M_s^{\text{Fe-Cr}}$ was calculated from information in Bozorth showing that the saturation magnetization of Fe-Cr alloys varies linearly with chromium content.⁽⁸⁾ Also, the saturation magnetization of iron-nickel alloys shows no change from the pure iron value up to about 25 at.% Ni,⁽⁸⁾ and therefore, we assumed that nickel content would not affect the saturation magnetization values of the iron-chrome-nickel alloys.

Relative magnetization of the Group 2 samples was obtained in a similar manner. However, the factors on the right side of Equation 1 were obtained by comparing the average voltage readings of the Groups 1 and 2 control samples at a field of 2.4×10^6 A/m and forcing the left side of Equation 1 to be equal in both cases. Thus $M/M_s^{\text{Fe-Cr}} = K \langle E \rangle t$ for the cylindrical samples, where K is determined from the Group 1 control data.

III. RESULTS

The magnetization values for control samples were found to increase linearly with field and at the same rate for all samples in each group within experimental accuracy. A best fit to the experimental data for the control samples gives a permeability value of 1.003 which agrees with the permeability of annealed austenitic stainless steels given in the ASM Metals Handbook.⁽⁹⁾

All of the irradiated samples in Group 1, except those annealed at 700°C, showed an increase in magnetization compared to unirradiated samples at the same field. Differences in magnetization were observed for different post-irradiation annealing temperatures and different heat treatments. These results are summarized in Table 3 and in Figure 2, which show the relative magnetization value (not corrected for austenite contribution) at a field of 7.15×10^5 A/m vs post-irradiation annealing temperature. We estimate that the relative accuracy of the data is $\pm 5\%$. The largest source of error is in the measurement of cross-sectional area of the specimens. Three cases where duplicate specimens were measured showed agreement to $\pm 5\%$.

TABLE 3
UNCORRECTED VALUES OF RELATIVE MAGNETIZATION AT
 7.15×10^5 amp/m OF TYPE 316 STAINLESS STEEL
IRRADIATED IN EBR II AT 425°C TO A
FLUENCE OF 1.8×10^{22} n/cm²
(E > 0.1 Mev)

Heat Treatment	Post-irradiation Annealing Temperature (°C)				
	20	400	500	600	700
A	0.29	0.29	0.35	0.17	0.14
B	0.45	0.58	0.64	0.30	0.18
C	0.94	0.91	3.05	0.42	0.19
			2.80		
D	0.38	0.34	0.55	0.19	0.19

Control samples = 0.13

Figure 2. Relative Magnetization, %, at Field of 7.15 amp/meter Versus Post-Irradiation Annealing Temperature of Type 316 Stainless Steel, 1.8×10^{22} Neutrons/cm² ($E > 0.1$ MeV), 425°C Irradiation Temperature

These data show that heat treatment C has the highest magnetization value in every case followed by heat treatments B, D and A. In every case post-irradiation annealing at 500°C causes an increase in magnetization while annealing at 600°C causes a decrease in magnetization. Annealing at 700°C restores the magnetization to the pre-irradiation value. Post-irradiation annealing at 400°C produces essentially no change in magnetization value. This is expected since the irradiation temperature was 425°C.

Analysis of the data indicated that complete saturation of the ferrite was not achieved at a field of 7.15×10^5 A/m. Using these data as a guide, we chose specific samples for measurements at higher fields, up to 2.4×10^6 A/m. Because saturation was not achieved at the lower field, direct comparisons between the samples cannot be made. This is because the size of the ferrite particles strongly controls the shape of the magnetization curve. The larger the particles, the lower the field needed to produce saturation. This will be discussed further after the results of the higher field measurements have been presented.

Results of magnetization vs field measurements for the specimen with the largest amount of ferrite are shown in Figure 3. The data have been corrected for the austenite contribution and therefore represent the net magnetization due to ferrite. Measurements made with increasing field and decreasing field agree quite well except at low fields where there is some remanence for this particular specimen. Most of the specimens had no remanence. The error bars are standard deviations derived from 10 pairs of measurements at each field. Each pair of measurements consisted of a voltmeter reading obtained after transferring the sample in one direction followed by a second reading after transfer in the opposite direction, a procedure that cancels out voltmeter zero drift. Fairly large errors at low magnetization values are expected because actual voltmeter readings are only 5 to 10 times the least count of the voltmeter.

The ferrite particle size and number density can be determined from the experimental data using the analytical techniques developed by Becker⁽¹⁰⁾ and Cahn.⁽¹¹⁾ A magnetization vs inverse field curve is shown in Figure 4 for the same specimen whose magnetization vs field curve was given in Figure 3.

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Figure 3. Relative Magnetization Minus Relative Magnetization of Control Samples, %
Versus Magnetic Field for Neutron-Irradiated Type 316 Stainless Steel, Heat
Treatment C, Post-Irradiation Anneal 1 hr at 500°C, 1.8×10^{22}
Neutrons/cm² (E > 0.1 MeV), 425°C Irradiation Temperature

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Figure 4. Relative Magnetization Minus Relative Magnetization of Control Specimens, %, Versus Reciprocal Magnetic Field for Neutron Irradiated Type 316 Stainless Steel, Heat Treatment C, Post-Irradiation Anneal 1 hr at 500°C
1.8 x 10²² Neutrons/cm² (E > 0.1 MeV), 425°C
Irradiation Temperature

The high field magnetization values shown in Figure 4 fall on a straight line that intercepts the ordinate at a value $100 M_O/M_S^{\text{Fe-Cr}}$, the saturation magnetization or, in this case, the total percent ferrite of the specimen, and intercepts the abscissa at $1/H_h$ where H_h is defined by this intercept. According to Cahn,⁽¹¹⁾ the number of particles per unit volume is given by

$$N = M_O H_h / kT, \quad \dots(2)$$

and average volume is given by

$$\bar{V} = 1/N(M_O/M_S^{\text{Fe-Cr}}) \quad \dots(3)$$

with a standard deviation of the particle size distribution of

$$\sigma_V = \bar{V} \left[(3 H_h / H_l) - 1 \right]^{1/2} \quad \dots(4)$$

H_l is defined as the magnetization at which the initial slope of the M/M_S vs H curve intersects the value of $M_O/M_S^{\text{Fe-Cr}}$ (see Figure 3).

Experimentally determined values for the various specimens are listed in Table 4. Particle sizes and volumes shown in the table have probable errors of about $\pm 20\%$. Values of $\bar{d}$ were calculated assuming the particles to be spherical. As expected, the saturation values shown in Table 4 for the Group 1 specimens are larger for all of the specimens than corresponding magnetization values at 7.15×10^5 A/m given in Table 3. However, the relative rankings of the various specimens as to amount of ferrite remain unchanged. Also, comparing the specimens annealed at 500°C with those annealed at 400°C , we see that the major effect of annealing at 500°C is to increase average volume, the particle density being relatively unaffected. For the Group 2 specimens irradiated at 500°C , the heat treatment B specimens showed average particle volumes about three times the particle volumes of samples given the other heat treatments in Group 2 and also all the Group 1 specimens, except heat treatment C specimens annealed at 500°C .

It should be noted that, although the sample given treatment B and irradiated at 500°C had the same percentage ferrite as the sample with treatment B, but irradiated at 425°C and annealed at 500°C , a direct comparison cannot be made because the fluence was lower in the former. Irradiation at 500°C produces fewer, but larger, particles than irradiation to a lower fluence at 425°C .

TABLE 4
CALCULATED FERRITE PARTICLE DISTRIBUTION PARAMETERS

Heat Treatment	Temperature (°C)		$10^6/H_h$ (M/A)	$100 M_o/M_s^{Fe-Cr}$ (% Ferrite)	N $10^{24}/M^3$	$\bar{V}$ $10^{-27} M^3$	$\bar{d}$ $10^{-10} M$	$M_{1.44}/M_o$	
	Irradiation	Anneal							
Group 1	[Neutron Fluence: 1.8×10^{22} neutrons/cm ² (E > 0.1 MeV)]								
	A	425	400	1.0	0.48	2.1	2.3	16	0.19
	A	425	500	1.6	0.58	1.5	4.0	20	0.11
	B	425	400	2.1	0.79	1.6	4.8	21	0.30
	B	425	500	2.8	0.84	1.5	5.8	22	0.43
	C	425	400	1.9	1.3	2.9	4.4	20	0.57
	C	425	500	3.6	3.6	4.3	8.4	25	0.64
	D	425	400	1.3	0.59	2.0	2.9	16	0.17
D	425	500	2.1	0.82	1.7	4.9	21	0.16	
Group 2	[Neutron Fluence: 3.5×10^{22} neutrons/cm ² (E > 0.1 MeV)]								
	B	500	—	6.1	0.82	0.58	14.1	30	0.72
	B	600	—	1.3	0.19	0.63	3.0	18	0.16
	E	500	—	1.9	0.70	1.5	4.4	20	0.19
	E	600	—	0.8	0.08	0.43	1.9	15	0
	F	500	—	2.2	0.74	1.4	5.1	21	0.29
	F	600	—	0.8	0.08	0.43	1.9	15	0.18

The standard deviation of the particle size distribution can in principle be obtained from Equation 4. However, our low field data are not good enough to allow use of this equation with any degree of confidence, and we have therefore used an alternate procedure to give an indication of the spread of particle sizes. If the particles act as superparamagnetic particles, the magnetization should be given by the Langevin function.

$$\frac{M}{M} = \coth \left[\frac{M_s^{\text{Fe-Cr}} HV}{kT} \right] - \left[\frac{kT}{M_s^{\text{Fe-Cr}} HV} \right] \quad \dots (5)$$

In Equation 5, H is the field and V is the volume of the superparamagnetic particles.

Figure 5 shows calculated values of M/M_o using Equation 5 with $M_s^{\text{Fe-Cr}} = 1.778 \times 10^7 \text{ erg-m/A-m}^3$, $H = 1.44 \times 10^5 \text{ A/m}$, $T = 300^\circ\text{K}$, and assuming spherical particles. The important thing to note is that large particles produce near saturation values of M/M_o at this low field. For particles in the range of $15 \text{ to } 30 \times 10^{-10} \text{ m}$ in diameter, the calculated values of M/M_o are 0.03 to 0.28. Experimentally determined values of M/M_o at a field of $1.44 \times 10^5 \text{ A/m}$ are listed in Table 4 under $M_{1.44}/M_o$. In most cases the experimentally determined values are greater than the calculated values for the same diameter. Some deviation is to be expected. However, the large discrepancies between the experimental and calculated values for samples given Treatments B and C indicate a large range of particle sizes compared to the experimentally calculated average diameter.

Figure 5. Calculated Ratio $M_{1.44}/M_0$ Versus Particle Diameter

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IV. DISCUSSION OF RESULTS

In the preceding sections we have made the assumption that the increased magnetization of the irradiated specimens compared to the unirradiated specimens could be related to the presence of ferrite. Although magnetization measurements are commonly used to determine the amount of ferrite in austenitic stainless steels,^(13, 14) the circumstances under which ferrite usually forms are well known and the existence of the ferrite phase has been verified by x-ray or electron diffraction in these cases. Neutron irradiated samples corresponding to each pre-irradiation heat treatment have been examined by electron microscopy and no precipitates were found that could account for the increased magnetization.⁽⁶⁾ However, these samples contain many small carbide precipitates, voids, faulted loops and unfaulted loops. Thus, identification of small particles of the size indicated by the magnetization measurements is very difficult. The fact that ferrite has been observed to form in proton irradiated Type 316 stainless steel in the temperature range 500 to 600°C supports the idea that ferrite should form in neutron irradiated stainless steel since the same basic effects are produced by both types of irradiation. In proton irradiated Type 316 stainless steel there is evidence that the amount of ferrite increases with increased fluence. Ferrite was observed by electron diffraction in a foil in which about 20 dpa* had been created at 500°C.⁽³⁾ Ferrite was not observed in a foil in which about 1 dpa had been created. However, the sample from which this latter foils was taken was weakly magnetic due to a region of higher damage and, consequently, higher ferrite transformation.

For the neutron irradiated samples, we calculated the number of displacements per atom as about 12 and thus, the magnetic effects observed are in qualitative agreement with the proton irradiation results. However, there is qualitative disagreement between proton irradiated and neutron irradiated samples at 600°C. Proton irradiated samples showed a greater tendency for ferrite formation at 600°C than at 500°C,⁽³⁾ while the neutron irradiated samples (Group 2) showed the opposite tendency (Table 4).

*The number of dpa are calculated according to the recommendations of D. G. Doran, Trans ANS 17, 207 (1973)

A comparison of the present results with the earlier magnetic measurements on Type 347 stainless steel shows that different mechanisms of ferrite formation must be involved in the two cases. Reynolds et al⁽⁵⁾ found that the amount of ferrite formed during irradiation was proportional to the amount of ferrite formed before irradiation by cold work. In the present case there was no evidence for formation of ferrite in the cold-worked specimens prior to irradiation and the cold-worked specimen showed less ferrite formation during irradiation than some of the annealed specimens. In the present case the large number of ferrite particles indicates that irradiation aids the nucleation of ferrite.

Basically, there are two different mechanisms for formation of ferrite in austenitic stainless steels, namely: nucleation and growth of ferrite of different composition than the parent austenite and martensitic transformation of austenite to ferrite of the same composition. Irradiation can influence both of these mechanisms.^(15, 16) Due to the limited data at present, we are unable to determine which of the above mechanisms is operative in our samples. Further study will, hopefully, lead to clearer understanding of the processes.

V. SUMMARY

- 1) Measurements of a neutron irradiated Type 316 stainless steel have shown increases in magnetization that are evidently related to formation of ferrite particles.
- 2) The ferrite particles are small enough to show superparamagnetic behavior and thus, their size and number distribution can be determined by analysis of the magnetization curve. The small size of the ferrite particles explains the fact that they have not been observed by electron microscopy in neutron irradiated specimens.
- 3) Pre-irradiation heat treatment and post-irradiation heat treatment influence the ferrite formation.

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