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EFFECT OF ELEVATED TEMPERATURE IRRADIATION ON THE STRENGTH AND DUCTILITY OF THE NICKEL-BASE ALLOY, HASTELLOY N

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ABSTRACT

The tensile properties of Hastelloy N have been determined after irradiation at 700°C to a dose level of 7×10^{20} nvt (E > 1 Mev) and 9×10^{20} nvt (thermal). The strength and ductility of the material were determined as functions of deformation temperature for the range of 20 to 900°C. These properties were also examined as functions of strain rate within the limits of 0.002 and 0.2 in./min for deformation temperatures of 500, 600, 700, and 800°C.

The stress-strain relationship is not affected by irradiation at 700°C. Ductility, as measured by the true uniform and fracture strains, is reduced for deformation temperatures of 500°C and above. The loss in ductility results in a reduction in the true tensile strength. This loss is more significant at test conditions resulting in intergranular failure, such as low strain rates at elevated temperature. Postirradiation annealing of the irradiated alloy does not result in improved ductility. These data are compatible with the experiments suggesting helium generation from the (n,α) reaction of boron as the cause of low ductility. PI annea

The low ductility of irradiated alloys in general is described in terms of the present knowledge of intergranular fracture. Means of improving the ductility are discussed.

INTRODUCTION

Nickel-base alloys and stainless steels are used extensively in nuclear reactors because of their resistance to corrosion, suitable mechanical properties, and fabricability. Because of the lack of consistent data on the strength and ductility of material irradiated at well-defined conditions, the design of reactor components is usually based on the mechanical properties of unirradiated material with appropriate safety factors. Investigations of the mechanical properties of irradiated material are needed to establish confidence in that element of safety.

Postirradiation tensile tests are considered useful tools for evaluation of irradiation damage. We have shown[1] reduction in ductility for irradiated stainless steel by using the tensile test technique. Hastelloy N has been irradiated at 700°C and the pre- and postirradiation tensile data compared to determine the irradiation effect. Tensile test temperature and strain rate are two variables markedly affecting the apparent strength and ductility of an alloy and both are considered in this investigation.

EXPERIMENTAL PROCEDURES AND TEST CONDITIONS

To evaluate the type and extent of irradiation damage at elevated temperature, Hastelloy N was irradiated at 700°C in the B-8 lattice position of the Oak Ridge Research Reactor (ORR) to an exposure of about 7×10^{20} nvt (E > 1 Mev) and 9×10^{20} nvt (thermal). The composition of Hastelloy N is as follows:

Element	Weight Percent
Mo	16.87
Cr	7.43
Fe	3.35
C	0.03
W	0.03
Si	0.60
Mn	0.55
V	0.26
P	0.001
S	0.006
Al	0.010
Ti	0.01
B	0.004
Co	0.07
Cu	0.02
Ni	Balance

Figure 1 shows a photograph of the irradiation rig. After irradiation the tensile samples (Fig. 2) are removed from the chamber and tested in an Instron tensile machine. The mechanical property data generated from these tests are then compared to data generated from unirradiated

[1]W. R. Martin and J. R. Weir, "The Effect of Irradiation Temperature on the Postirradiation Stress-Strain Behavior of Stainless Steel," paper presented at the ASTM Symposium on Flow and Fracture of Metals and Alloys in Nuclear Environments, Chicago, Ill., June 21–26, 1964.

material given a similar thermal history and deformed at identical conditions. The uniform elongation is the strain at maximum load. The true fracture strain is calculated using the formula,

$$\bar{\epsilon}_f = 2 \ \ell n \ \frac{D_o}{D_f} , \tag{1}$$

where

$\bar{\epsilon}_f$ = true fracture strain,
D_o = initial diameter, and
D_f = final diameter.

The true tensile strength is defined as the true stress at the ultimate engineering stress and is calculated as given by Dieter.[2]

[2]G. E. Dieter, Jr., Mechanical Metallurgy, p. 245, McGraw-Hill, New York, 1961.

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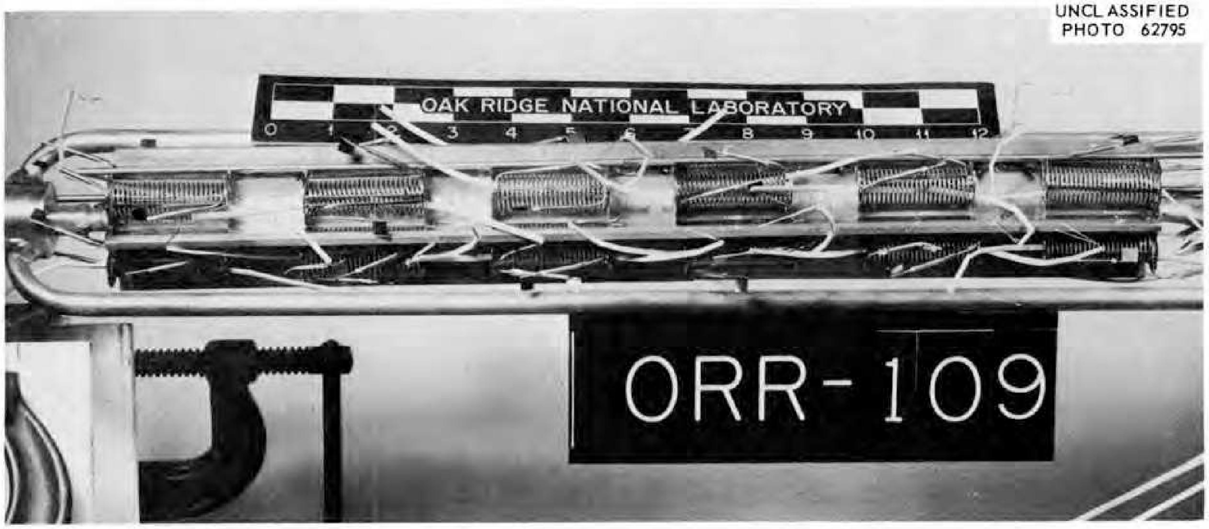

Fig. 1. Photograph of In-Reactor Irradiation Rig Showing Tensile Specimens in Furnaces.

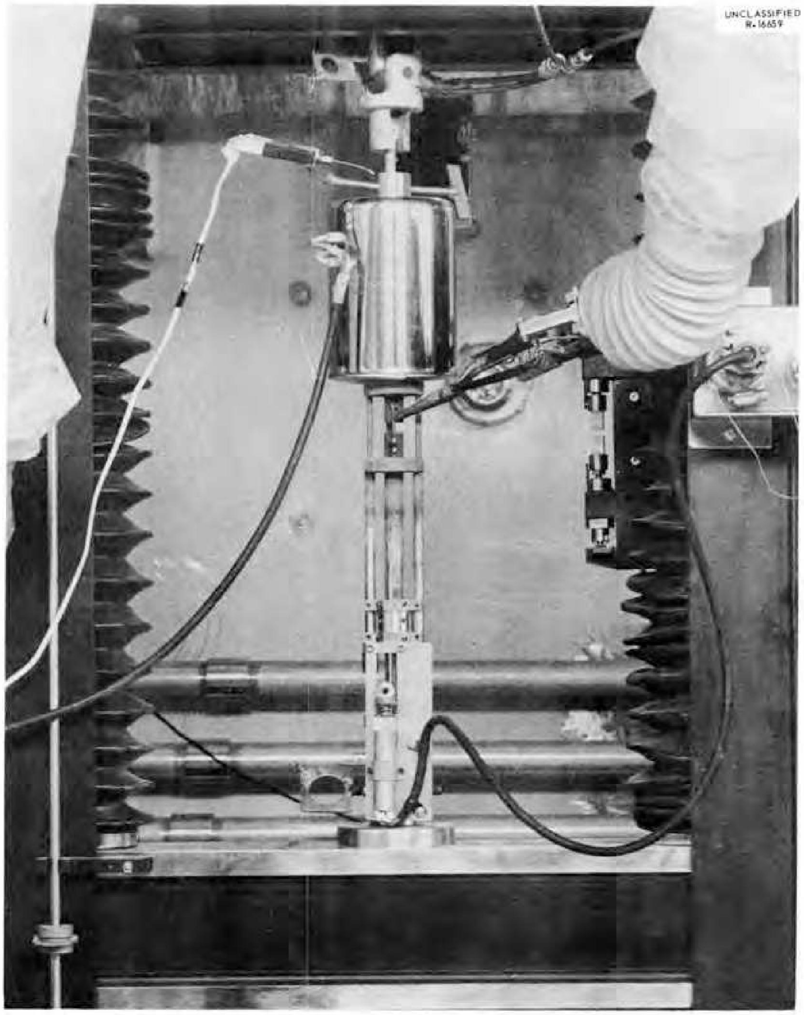

Fig. 2. Testing of Specimens in Instron Tensile Machine.

INFLUENCE OF DEFORMATION TEMPERATURE

The data for Hastelloy N strained at a rate of 2% per min are given in Table 1 for irradiated and unirradiated material as a function of postirradiation deformation temperature. The 0.2% offset yield stress was not significantly affected by irradiation. The deviations in the true tensile strength are believed to be insignificant except for deformation temperatures of 500°C and above. The true tensile stress was reduced about 50% at 700°C. Ductility of the alloy, as measured by the true uniform and fracture strains, ϵ, was affected at deformation temperatures of 500°C and above. The true fracture strain of the unirradiated

Table 1. Tensile Strength and Ductility of Irradiated and Unirradiated Hastelloy N

Deformation Temperature, °C	Stress, psi				Ductility, %			
	Yield Strength		True Tensile Strength		True Uniform Strain		True Fracture Strain	
	Irrad.	Unirrad.	Irrad.	Unirrad.	Irrad.	Unirrad.	Irrad.	Unirrad.
	$\times 10^3$	$\times 10^3$	$\times 10^3$	$\times 10^3$				
Room temperature	46.3	45.5	168.6	166.5	42.3	40.6	42.5	39.0
100	43.9	43.9	159.5	161.0	40.1	40.3	44.6	37.2
200	38.4	40.7	150.6	157.5	40.3	41.9	42.5	50.7
300	36.0	40.7	154.3	147.0	42.2	37.9	44.6	41.4
400	35.0	40.7	146.9	153.0	40.2	39.3	42.5	46.9
500	35.8	35.8	129.5	144.0	35.3	42.4		
600	32.5	36.2	82.4	109.0	11.8	26.7	21.9	31.6
700	31.0	34.1	53.4	102.8	8.0	30.8	11.6	42.1
800	28.5	30.9	38.4	59.9	3.7	12.2	6.9	86.6

material exhibits a minimum at elevated temperature. This minimum is typical for the alloy and is reflected in the reduction of area and total elongation measurements normally reported. However, no ductility minimum is observed for the irradiated alloy, and the ductility as measured by either uniform or fracture strain decreases with increasing deformation temperature.

STRAIN RATE SENSITIVITY AT ELEVATED TEMPERATURE

The strength and ductility of the irradiated and unirradiated alloy are given in Table 2 as functions of strain rate for deformation temperatures of 500°C and above. The ductility decreases with decreasing strain rate. The ductility of the irradiated material is particularly low at 800°C and 0.2% per min strain rate. The effects of irradiation on the uniform and fracture strains, shown in Fig. 3., are approximately equivalent in magnitude. However, the effect on uniform strains appears to saturate, whereas the magnitude of the irradiation effect on the fracture strains continues to increase with increasing deformation temperature.

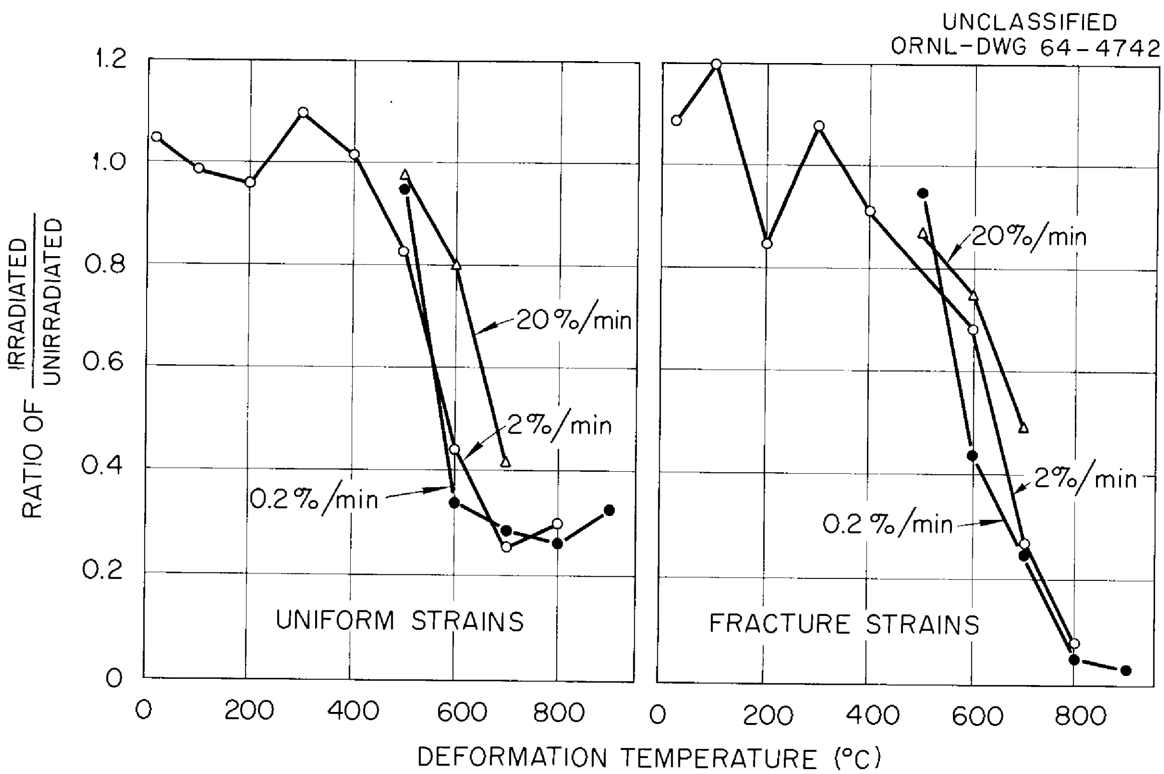

Fig. 3. Effect of Irradiation on Ductility of Hastelloy N as a Function of Strain Rate and Deformation Temperature.

Table 2. Strain Rate Sensitivity of Irradiated and Unirradiated Hastelloy N

Deformation Temperature, °C	Strain Rate, per min	Stress, psi				Ductility, %			
		Yield Strength		True Tensile Strength		True Uniform Strain		True Fracture Strain	
		Irrad.	Unirrad.	Irrad.	Unirrad.	Irrad.	Unirrad.	Irrad.	Unirrad.
		× 10³	× 10³	× 10³	× 10³				
500	0.2	32.7	34.9	136.1	145.6	41.2	42.3	46.6	53.4
500	0.02	35.8	35.8	129.5	144.0	35.3	42.4		51.4
500	0.002	34.4	37.4	122.5	131.5	32.3	33.3	36.5	34.2
600	0.2	32.9	34.1	112.7	134.4	31.2	38.9	36.5	48.7
600	0.02	32.5	36.2	82.4	109.0	17.7	26.7	21.9	31.6
600	0.002	34.2	34.6	63.1	106.0	10.3	29.9	13.2	29.7
700	0.2	30.5	30.9	66.8	106.5	13.5	32.2	19.2	39.2
700	0.02	31.0	34.1	53.4	102.8	8.0	30.8	11.6	42.1
700	0.002	32.1	33.7	47.0	80.5	5.6	20.0	7.8	29.0
800	0.2	29.3	29.3	45.7	79.8	6.7		11.6	
800	0.02	28.5	30.9	38.4	59.9	3.7	12.2	6.9	86.6
800	0.002	29.3	32.5	32.2	42.9	1.8	6.5	4.9	93.7

POSTIRRADIATION ANNEALING

The effect of postirradiation heat treatment of the alloy at the recommended solid solution temperature of 1175°C for 1 hr is shown in Table 3. The elevated temperature ductility of the irradiated alloy is not improved by the heat treatment, thereby indicating the thermal stability of the configuration causing the reduced ductility. Since the heat treatment results in resolution of carbide precipitates, any influence of irradiation on the precipitation of these carbides is not responsible for the observed ductility reduction.

Table 3. Effect of Postirradiation Heat Treatment of Irradiated Hastelloy N at 1175°C for 0.5 hr

(Tested at a strain rate of 0.002% per min)

Deformation Temperature, °C	Condition	Stress, psi		Ductility, %	
		0.2% Offset Yield	True Tensile	True Uniform	True Fracture
		$\times 10^3$	$\times 10^3$		
700	Unirradiated	33.7	80.5	20.0	29.0
700	Irradiated	32.1	47.0	5.6	7.8
700	Irradiated plus postirradiation heat treatment	30.5	44.2	6.3	6.9
900	Unirradiated	21.8	22.2	1.2	70.0
900	Irradiated	23.2	23.2	< 0.4	< 0.4
900	Irradiated plus postirradiation heat treatment	23.5	23.5	< 0.6	< 0.7

DISCUSSION

The results indicate that the effect of irradiation at elevated temperature on the strength and ductility of Hastelloy N is qualitatively the same as that reported for stainless steel.[3,4] The stress-strain relationship at elevated temperature is not affected by irradiation at 600°C and above. This is in contrast to irradiation below 600°C, where the stress-strain relationship is affected by irradiation,[3] as shown in Fig. 4.

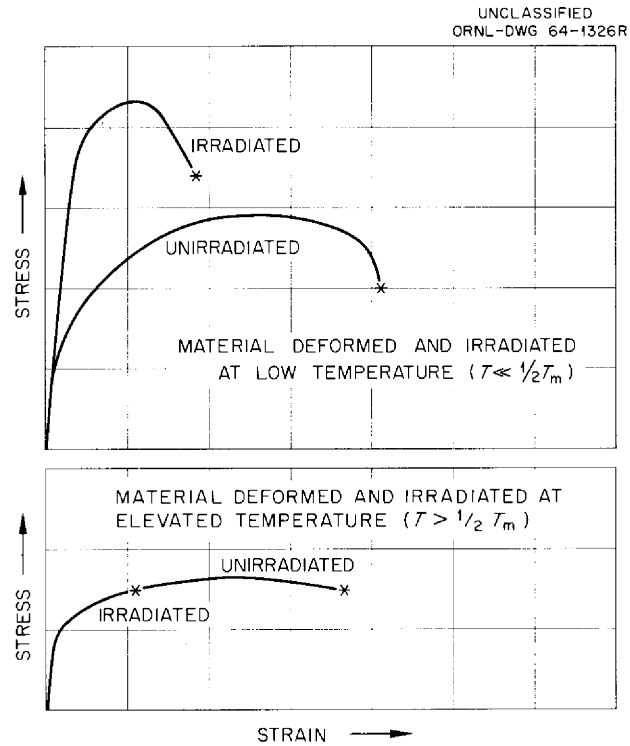

Fig. 4. Effect of Irradiation on the Stress-Strain Curves.

The defects introduced by fast neutrons annealed during irradiation at the elevated temperatures. The reduction in tensile strength at elevated temperatures is a result of the inability of the irradiated alloy to strain plastically. The fracture at a reduced strain therefore decreases the true tensile strength, and the magnitude of the reduction increases as the test conditions are altered to increase the strain-hardening coefficient (i.e., increased strain rates). If the alloy is irradiated at elevated temperature, the reduction in ductility occurs only for deformation at elevated temperatures. Metallographic examination by the

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[3]W. R. Martin and J. R. Weir, "The Effect of Irradiation Temperature on the Postirradiation Stress-Strain Behavior of Stainless Steel," paper presented at the ASTM Symposium on Flow and Fracture of Metals and Alloys in Nuclear Environments, Chicago, Ill., June 21–26, 1964.

[4]W. R. Martin and J. R. Weir, Nature **202**, 997 (1964).

light microscope shows that the deformation temperature at which the irradiated alloy becomes embrittled is associated with the transition from transgranular to the intergranular mode of fracture. When the irradiated and unirradiated specimens fracture transgranularly, no loss of ductility is observed (Fig. 5). Figure 6 shows that grain boundary

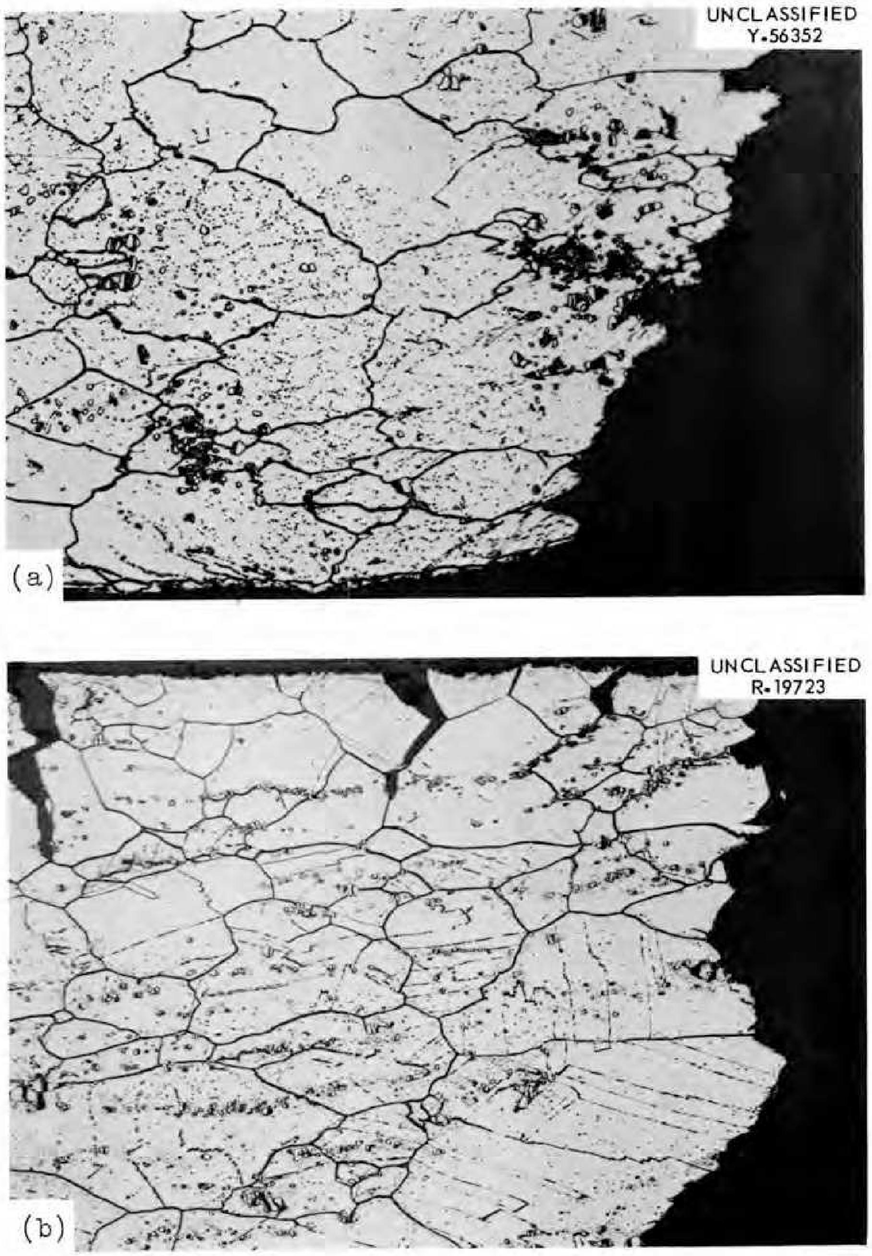


Fig. 5. Comparison of the Fracture of Irradiated and Unirradiated Hastelloy N at Approximately 35% Strain. Tested at a strain rate of 0.2% per min and 500°C. Etchant: aqua regia. 100×. (a) Unirradiated Hastelloy N shows no grain boundary failure at 46.6% strain and specimen rupture by a transgranular mode. (b) Irradiated alloy shows intergranular surface cracks but specimen failure by a transgranular mode.

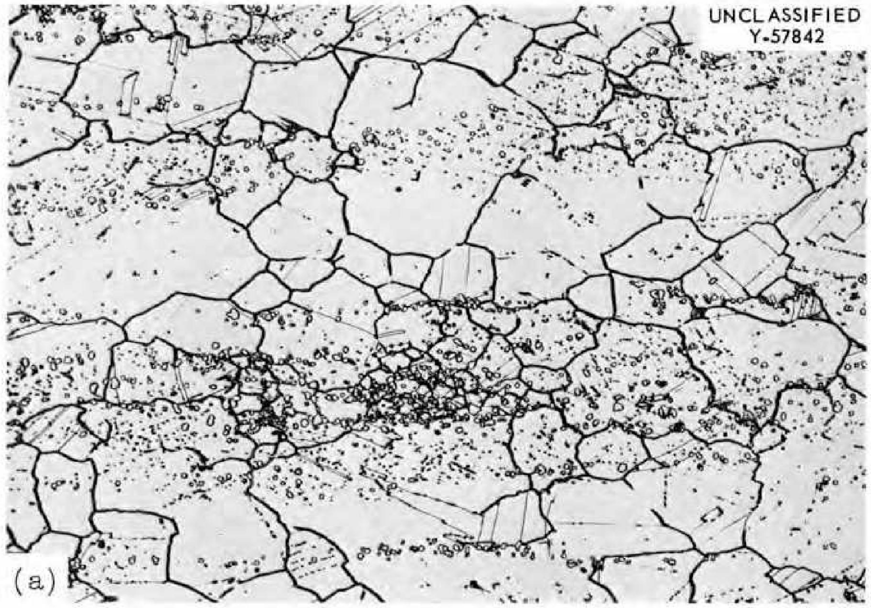
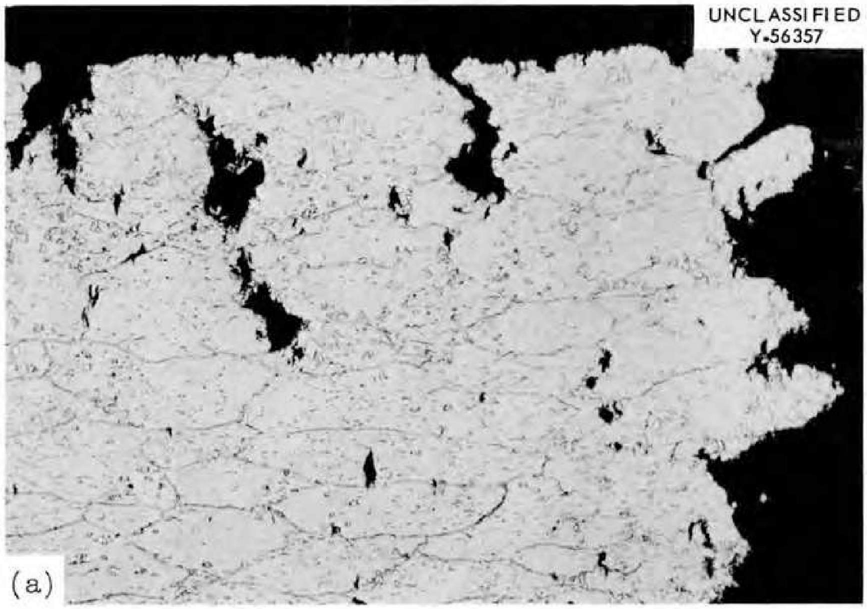


Fig. 6. Comparison of Irradiated and Unirradiated Hastelloy N at 29% Strain. No fractures are observed in unirradiated sample tested at 700°C and 0.2% per min strain rate. Etchant: aqua regia. 100×. (a) Unirradiated. (b) Irradiated.

cracks are found in the irradiated material at smaller strains than in the unirradiated alloy when observed at 100× using the light microscope. These cracks propagate along the boundary rather than widen in the irradiated material and cause complete specimen rupture at a considerably reduced strain, as shown in Fig. 7. Therefore, it would appear that both the nucleation and propagation of cracks are affected.



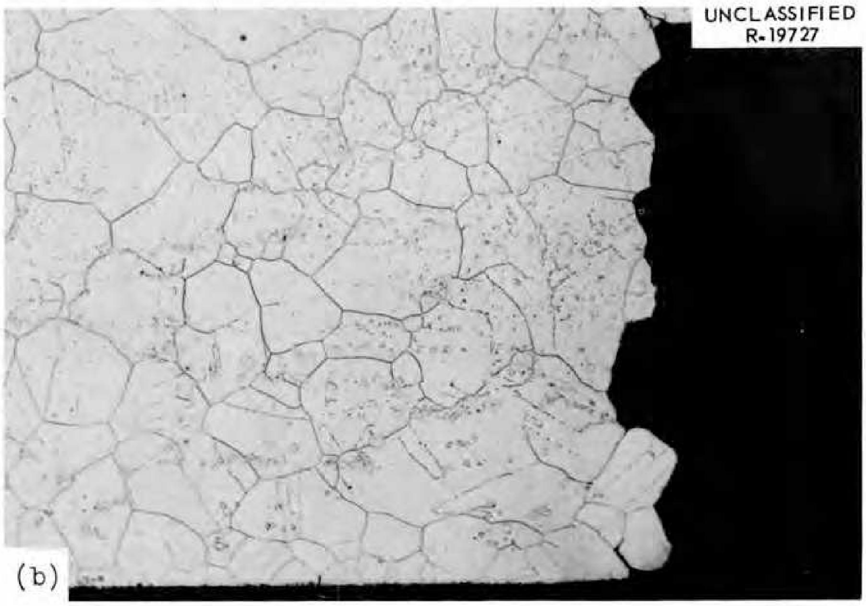


Fig. 7. Comparison of the Grain Boundary Cracks at Fracture for Unirradiated and Irradiated Alloy Strain at 900°C and at a Strain Rate of 0.2% per min. Etchant: aqua regia. 100×. (a) Unirradiated alloy fracture at 70% strain. (b) Irradiated alloy fracture at approximately 0.4% strain.

Thus, the influence of irradiation at elevated temperature is one affecting only the fracture of grain boundaries. Intergranular fracture is classified into two types:

1. wedge type that originate at triple points,

2. cavity type in which small cavities are nucleated along grain boundaries.

The intergranular tensile test fractures observed for the irradiated and unirradiated alloys have been of the wedge type. These type cracks are formed on boundaries transverse, and on occasion oblique, to the direction of the stress applied to the bulk specimen. Although many questions need to be answered about intergranular wedge-type fracture, it is generally believed[5] that a prerequisite is grain boundary sliding. Localized deformation along the boundaries results in stress concentrations that nucleate fracture if not dissipated by boundary migration or recrystallization. Any mechanism proposed for the reduced ductility of irradiated alloys must be one that does not increase the effectiveness of the boundaries as dislocation barriers. Grain boundary sliding could be affected by irradiation either reducing the magnitude of boundary deformation that an aggregate can accommodate before fracture or by increasing the rate of boundary sliding. Measurement of grain boundary deformation, perhaps by the Rachinger[6] method, needs to be determined as a function of stress for irradiated and unirradiated alloys. Certainly, the reduced ductility could be related to factors[7] governing the rate of extension of the cracks. Electron fractography is required to elucidate this area.

The elevated temperature embrittlement has been reported by Roberts and Harries[8] to be related to thermal rather than fast neutrons. Slow neutrons, apart from scattering, show four types of capture reactions:

1. emission of gamma radiation (n,γ),
2. ejection of an alpha particle (n,α),
3. ejection of a proton (n,p), and
4. fission (n,f).

Of the four reactions, the (n,α) reactions appears the most likely to cause the embrittlement. This reaction, producing helium localized at the grain boundary, could affect the nucleation of cracks at the grain

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[5]J. R. Low, The Fracture of Metals, p. 61, MacMillan and Company, New York, 1963.

[6]W. A. Rachinger, J. Inst. Metals 81, 33 (1952).

[7]D. McLean, Grain Boundaries in Metals, p. 335, Clarendon Press, Oxford, 1957.

[8]A. C. Roberts and D. R. Harries, Nature 200, 773 (1963).

boundary and, conceivably, crack propagation. The thermal stability of the defect, as indicated by the postirradiation heat treatment data, is compatible with the hypothesis of helium generation. The thermal stability of this defect is in sharp contrast with data by Bailey[9] and others[10] that show that the low-temperature (< 500°C) damage caused by fast neutrons can be annealed at 400 to 980°C. However, the concentration of the species necessary for (n,α) reaction is in the parts per million range for stainless steel and nickel-base alloys. Boron-10 and lithium-6 are two such species, given by Barnes,[11] that generate large specific volumes of gas. If the boron is concentrated at the grain boundaries, the atomic fraction of the helium produced can be large even with the average concentration of ^{10}B in the 1- to 10-ppm range. Studies by other investigators[12] indicate boron segregation in the grain boundaries of austenitic alloys. The grain boundary concentration of helium necessary to cause embrittlement is unknown although it can be estimated from the beryllium irradiations[13] to be $< 0.7 \times 10^{-4}$.

Studies[14,15] to examine the influence of boron content on the properties of irradiated alloys show complex relationships between boron content and ductility for the alloy deformed at elevated temperature. However, these studies are complicated by the fact that boron for the concentrations investigated influences the strength and ductility of

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[9] R. E. Bailey and M. A. Silliman, Symposium on Radiation Effects of Materials, vol. 3, ASTM Special Technical Publication 223, 1958.

[10] W. E. Murr and F. R. Shober, Annealing Studies on Irradiated Type 347 Stainless Steel, BMI-1621 (March 1963).

[11] R. S. Barnes and G. W. Greenwood, Proc. U. N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1958 5, 481 (1958).

[12] C. Crussard, J. Plateau, and G. Henry, Proceedings of the Joint International Conference on Creep, pp. 1–91, vol. 1, Institution of Mechanical Engineers, London, 1963.

[13] J. R. Weir, Proceedings of the International Conference on the Metallurgy of Beryllium, London, 1961, pp. 395–409, Chapman & Hall, London, 1963.

[14] N. Hinkle, W. E. Brundage, and J. C. Zukas, Solid State Div. Ann. Progr. Rept. Aug. 31, 1960, ORNL-3017, p. 120.

[15] High-Temperature Materials Program Progr. Rept., Jan. 24, 1964, GEMP No. 31, Part A, p. 30.

the unirradiated alloy. The influence of boron on the solubility of carbon, alteration of precipitate distribution, and grain size is well documented.[12,16,17]

Of course, the foregoing discussion is dependent on the embrittlement being related to thermal neutrons and helium generation. The data generated, to date, is indirect evidence. However, if the ductility is a result of helium generation, the problem of embrittlement of irradiated materials deformed at elevated temperature will have to be solved by removing the species undergoing (n,α) reaction from the grain boundaries. Several approaches are available:

1. The average concentration may be lowered by electron-beam melting.

2. Distribute the boron within the grain as stable precipitates, such as borides and combination precipitation of $Fe_{23}(BC)_6$. Helium atoms and/or bubbles may be trapped at the precipitate, thereby greatly reducing the concentration of helium bubbles in the grain boundary.

3. Lower the concentration at the boundary by increasing the number of boundaries on which the specie precipitates or segregates.

The characteristics of the damge caused by irradiation at elevated temperature are as follows:

1. Yield stress and tensile strength are not affected.

2. Ductility of material is reduced.

3. The reduction in ductility is noted by large decreases in the uniform and fracture strain and by small decreases in the true tensile and fracture stresses.

4. The reduction in ductility is more significant at test conditions resulting in intergranular failure, such as low strain rates at elevated temperature.

5. Heat treatments that anneal the damage causing the low-temperature irradiation effect do not improve the ductility at elevated temperature.

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[16] V. V. Levitin, Phys. Metals Metallog. **11**, 67 (1961).

[17] R. F. Decker, J. P. Rowe, and J. W. Freeman, Boron and Zirconium from Crucible Refractories in a Complex Heat Resistant Alloy, NACA-1392 (1958).

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