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TENSILE PROPERTIES OF HASTELLOY N WELDED AFTER IRRADIATION

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ABSTRACT

Fusion welds affecting 75% of the cross section were made in small tensile samples (0.125 in. in diameter) of Hastelloy N irradiated to thermal fluences up to 9.4×10^{20} neutrons/cm^2. All of the unirradiated samples and 67% of the irradiated samples were satisfactorily welded using a specialized technique developed for this program. Surface contamination is suspected to be the cause of the unsuccessful welds in the irradiated samples. The welded irradiated samples generally had as good tensile properties at 25 and 650°C as the irradiated base metal. The weld metal deformed appreciably at 650°C and made a significant contribution to the overall fracture strain. The fracture location in the irradiated samples tested at 650°C shifted from the weld metal to the base metal following the post-weld anneal of 8 hr at 870°C. The porosity which was observed near the fusion line of the irradiated samples probably results from transmuted helium bubbles, but this did not seem to affect the location of the fracture.

INTRODUCTION

The maintenance and repair of nuclear systems will frequently involve cutting and rewelding pipes and components that have been irradiated. The prospect of these repairs raises the obvious questions of how such welds can be made and what are their mechanical properties. It is this latter question that will be discussed in the present report. This report will also deal specifically with molten-salt reactors where the additional problem exists of removing residual fluoride salt or corrosion products. However, cleanliness will likely be a paramount problem in making remote welds in any reactor system.

The alloy studied is Hastelloy N, a nickel-based material developed specifically for use in molten-salt reactors.[1] The irradiated material studied had been exposed to the core of the Molten-Salt Reactor Experiment for long periods of time as a surveillance material for the reactor vessel. The welds made in this study were simple gas tungsten-arc fusion welds that melted about 75% of the cross-sectional area of a miniature (0.125 in. in diameter) tensile sample. Thus, the welds were made with very low heat input and minimal restraint, and the results can be used only qualitatively.

EXPERIMENTAL DETAILS

The heats of material involved in this study were air-melted and the chemical compositions are given in Table 1. These heats were used in fabricating the MSRE vessel; heat 5065 for the top and bottom heads and heat 5085 for the cylindrical shell.

Samples of these heats were placed in the various surveillance facilities of the MSRE.[2–4] These samples have the general configuration of a long rod 1/4 in. in diameter with periodic reduced sections 1 1/8 in. long and 1/8 in. in diameter. After the desired exposure, these rods can be segmented to obtain small tensile samples. The core surveillance assembly is located axially about 3.6 in. from the core center line where the thermal flux (< 0.876 Mev) is 4.1×10^{12} neutrons cm^{-2} sec^{-1} and the fast flux (> 1.22 Mev) is 1.0×10^{12} neutrons cm^{-2} sec^{-1}. The environment is a

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[1]W. D. Manly et al., "Metallurgical Problems in Molten Fluoride Systems," pp. 164–179 in Progr. Nucl. Energy Ser. IV 2, Pergamon, Oxford, 1960.

[2]W. H. Cook, Molten-Salt Reactor Program Semiann. Progr. Rept. Aug. 31, 1965, ORNL-3872, pp. 87–92.

[3]H. E. McCoy, An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — First Group, ORNL-TM-1997 (November 1967).

[4]H. E. McCoy, An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — Second Group, ORNL-TM-2359 (February 1969).

Table 1. Chemical Analysis of Surveillance Heats

Element	Content, wt %	
	Heat 5065	Heat 5085
Cr	7.2	7.3
Fe	3.9	3.5
Mo	16.5	16.7
C	0.065	0.052
Si	0.60	0.58
Co	0.08	0.15
W	0.04	0.07
Mn	0.55	0.67
V	0.22	0.20
P	0.004	0.0043
S	0.007	0.004
Al	0.01	0.02
Ti	0.01	< 0.01
Cu	0.01	0.01
B (ppm)	24, 37, 20, 10	38
O	0.0016	0.0093
N	0.011	0.013

molten fluoride salt, 65 LiF, 29.1 BeF_2, 5 ZrF_4, 0.9 UF_4 (mole %), at 650°C. There is a control facility in which the samples are exposed to static "fuel salt" containing depleted uranium. The temperature follows that of the MSRE. A second surveillance facility is located outside the core in a vertical position about 4.5 in. from the vessel. The temperature is also 650°C at this location and the thermal flux (< 0.876 Mev) is 1.0×10^{11} neutrons cm^{-2} sec^{-1} and the fast flux (> 1.22 Mev) is 1.6×10^{11} neutrons cm^{-2} sec^{-1}. The environment is nitrogen with 2 to 5% O_2, and the Hastelloy N samples have a thin oxide film after exposure.

In order to make fusion welds (no filler metal added) on the irradiated tensile specimens, it was necessary to design a special welding fixture that could be operated remotely in a hot cell. We aimed for a reasonable assurance of good penetration (high percentage of cross section of specimen to be weld metal) without specimen distortion. Figure 1 is a photograph of the welding fixture assembled for use in the hot cell. As can be seen, the fixture consists of a rigid stand, motor-driven chuck, specimen support, and a gas tungsten-arc welding

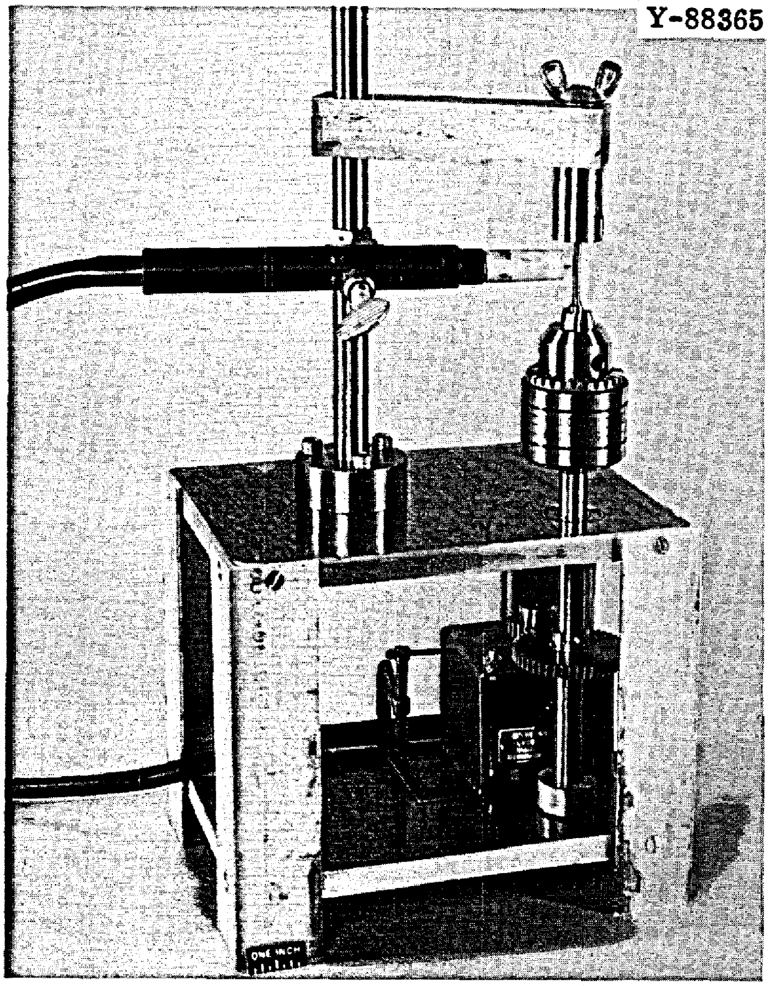

Fig. 1. Welding Equipment Developed for Making Remote Welds.

torch. The upper support has an internal curved surface that contacts the fillet radius of the tensile sample and keeps the sample aligned during welding. The torch was connected to a programmed welding power supply located outside the hot cell. The welding conditions were adjusted to obtain penetration of about 75% of the sample cross section.

All samples were abraded with 240-grit emery paper and cleaned with acetone before welding. We did the final abrasion on each sample with a clean piece of emery paper in an effort to minimize contamination.

The tensile tests were run on Instron Universal testing machines. The strain measurements were taken from the crosshead travel. The test environment was air in each case.

EXPERIMENTAL RESULTS

We welded 25 unirradiated samples both in the hot cell and in the laboratory and all welds visually appeared sound. We welded 15 irradiated samples; three welds were completely unsatisfactory and two others were very questionable due to surface cracks. Thus, 67% of the welded irradiated specimens were found to be sound by visual examination. The bad welds occurred randomly, and we suspected that cleanliness was our main problem in obtaining sound welds.

The results of tensile tests on base-metal samples are given in Table 2, and those for the welded samples are given in Table 3. Numerous variables are included, and care must be used in making comparisons. The changes in strength are not thought to be significant, and we shall discuss in some detail only the changes in the fracture characteristics.

A comparison of Groups 3, 4, and 5 in Table 2 shows that the fracture strain of the unirradiated base metal decreases with aging at 650°C. (Corrosion is very slight in the samples and the property changes are attributed entirely to thermal aging.[3,4]) The property changes are greater for heat 5085 than for heat 5065. Groups 1 and 2, Table 2, show that irradiation reduces the fracture strain with the magnitude of the change increasing with increasing fluence. The reduction in fracture strain in tests at 25°C is thought to be due to carbide precipitation, and samples 7982 and 7976, Group 2, Table 2, lend support to this hypothesis. The fracture strain at 25°C was only 32.8% in the as-irradiated condition, but improved to 48.3% after an anneal of 8 hr at 870°C. The reduction in the fracture strain at 650°C due to irradiation is even more dramatic. We attribute this reduction in fracture strain to the production of helium in the metal by the $^{10}B(n,\alpha)^{7}Li$ transmutation and have found that postirradiation annealing does not improve the properties at elevated temperatures.[5]

Comparison of the data for Group 3 in Tables 2 and 3 shows that welding decreases the fracture strain in the unirradiated condition and that

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[5] MSR Program Semiann. Progr. Rept. Feb. 28, 1966, ORNL-2936, p. 117.

Table 2. Tensile Properties of Base-Metal Samples

Heat Number	History	Sample Number	Test Temperature (°C)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
					Yield	Ultimate	Uniform	Total	
				Group 1					
5065	a	7915	25	0.05	51,700	109,300	41.4	41.5	34.1
5065	a	7913	650	0.002	40,400	46,300	3.2	3.4	6.0
5085	a	7888	25	0.05	52,300	95,000	28.7	28.9	20.0
5085	a	7886	650	0.002	35,000	42,400	4.5	5.0	13.1
				Group 2					
5065	b	7940	25	0.05	49,000	118,800	57.8	59.7	38.4
5065	b	7947	650	0.002	34,100	55,500	12.2	12.5	16.1
5085	b	7976	25	0.05	46,500	99,100	32.8	32.8	24.5
5085	b	7982[c]	25	0.05	46,700	119,000	48.2	48.3	34.2
5085	b	7965	650	0.002	31,300	49,900	11.1	11.6	18.6
				Group 3					
5065	d	1843	25	0.05	64,000	124,600	52.0	55.5	52.1
5065	d	280	650	0.002	46,300	75,400	22.8	24.0	28.1
5085	d	4295	25	0.05	51,500	120,800	52.3	53.1	42.2
5085	d	10,083	650	0.002	32,200	70,600	32.8	34.5	27.5
				Group 4					
5085	e	FC-3	25	0.05	45,500	111,200	46.8	46.8	31.5
5085	e	DC-25	650	0.002	31,500	62,500	22.8	24.3	27.2
				Group 5					
5065	f	10,215	25	0.05	60,900	126,700	46.5	47.4	39.3
5065	f	10,216	650	0.002	44,200	73,300	16.0	16.5	16.8

Table 2 (continued)

Heat Number	History	Sample Number	Test Temperature (°C)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
					Yield	Ultimate	Uniform	Total	
				Group 5 (continued)					
5085	f	10,166	25	0.05	53,900	115,900	38.4	38.6	29.7
5085	f	10,190	650	0.002	37,700	64,700	17.4	18.0	19.7

[a]Exposed to fuel salt in the core of the MSRE for 15,289 hr at 650°C to a thermal fluence of 9.4×10^{20} neutrons/cm^2.

[b]Exposed to MSRE cell environment of N_2–2 to 5% O_2 for 20,789 hr at 650°C to a thermal fluence of 2.6×10^{19} neutrons/cm^2.

[c]Given a postirradiation anneal of 8 hr at 870°C.

[d]Unirradiated, annealed 2 hr at 900°C.

[e]Unirradiated, annealed 2 hr at 900°C, exposed to static barren "fuel" salt for 4800 hr at 650°C.

[f]Unirradiated, annealed 2 hr at 900°C, exposed to static barren "fuel" salt for 15,289 hr at 650°C.

Table 3. Tensile Properties of Welded Samples

Heat Number	History	Sample Number	Post-weld Anneal	Test Temperature (°C)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)	Location of Failure
						Yield	Ultimate	Uniform	Total		
					Group 1						
5065	a	7899	b	25	0.05	56,600	92,200	15.2	15.4	16.0	c
5065	a	7898	b	650	0.002	40,700	55,200	7.5	7.6	8.6	d
5085	a	7872	b	25	0.05	52,900	105,700	33.3	33.6	25.2	d,e
5085	a	7870	none	650	0.002	36,900	45,400	4.4	5.4	9.5	d,e
5085	a	7871	b	650	0.002	38,000	52,300	7.5	9.3	2.4	d
					Group 2						
5065	f	7959	b	25	0.05	52,700	55,300	2.5	4.3	19.6	c
5065	f	7957	none	650	0.002	35,400	48,800	6.1	6.8	19.2	c
5065	f	7958	b	650	0.002	32,700	55,100	10.9	11.3	10.8	d
5085	f	7992	none	25	0.05	48,600	104,200	40.2	40.4	33.2	d
5085	f	7990	b	25	0.05	47,300	112,800	40.5	40.8	26.8	d,e
5085	f	7994	none	650	0.002	33,100	55,700	12.2	12.9	13.1	c
5085	f	7991	b	650	0.002	32,700	62,300	18.2	18.6	13.9	d
					Group 3						
5065	g	4158	b	25	0.05	63,700	138,700	43.2	43.4	30.3	c
5065	g	4155	none	650	0.002	37,500	59,300	9.7	10.4	15.6	c
5065	g	4162	b	650	0.002	43,200	80,300	19.5	20.1	16.0	c
5085	g	10,086	b	25	0.05	49,800	105,900	29.9	30.0	15.7	c
5085	g	10,085	none	650	0.002	35,400	61,500	12.7	13.7	10.7	c
5085	g	10,087	b	650	0.002	30,400	70,600	33.3	34.5	18.0	c

Table 3 (continued)

Heat Number	History	Sample Number	Post-weld Anneal	Test Temperature (°C)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)	Location of Failure
						Yield	Ultimate	Uniform	Total		
					Group 4						
5085	h	10,082	none	25	0.05	53,200	121,600	57.0	60.8	18.1	d
5085	h	10,081	none	650	0.002	29,400	56,100	14.5	15.5	14.0	c
					Group 5						
5085	i	9010	none	650	0.002	33,700	55,700	10.1	10.7	12.8	c

[a]Exposed to fuel salt in core of MSRE for 15,289 hr at 650°C to a thermal fluence of 9.4×10^{20} neutrons/cm^2; welded in cell.

[b]Eight hours at 870°C.

[c]Weld metal.

[d]Base metal.

[e]Exceptions to the general fracture trend.

[f]Exposed to MSRE cell environment of N_2–2 to 5% O_2 for 20,789 hr at 650°C to a thermal fluence of 2.6×10^{19} neutrons/cm^2; welded in cell.

[g]Unirradiated, welded outside cell.

[h]Unirradiated, welded in cell.

[i]Exposed to static barren "fuel" salt for 4800 hr at 650°C; welded in cell.

the fractures were located in the weld metal for the conditions investigated. Group 4, Table 3, involves unirradiated samples welded in the hot cell. Sample 10,081 is a duplicate of 10,085 prepared outside the hot cell and attests to the reproducibility of the welding technique. Sample 10,082 was not given a postweld anneal before testing at 25°C as was sample 10,086 and the location of fracture changed from the weld metal to the base metal. Sample 9010, Group 5, Table 3, had been exposed to fluoride salt for 4800 hr at 650°C, and its good properties show that no basic problem prevents welding components that have been exposed to salts.

The samples in Groups 1 and 2, Table 3, were welded after irradiation. These samples generally have lower fracture strains than their unirradiated counterparts shown in Groups 3, 4, and 5, Table 3. The fracture strains for heat 5085 tested at 25°C are an exception, since they are about equal for unirradiated and irradiated welds. The fracture strains for samples from heat 5065 which were irradiated, welded, and tested at 25°C are quite low (samples 7899 and 7959, Groups 1 and 2, Table 3).

A comparison of the properties of the irradiated base metal, Groups 1 and 2, Table 2, with those of the samples irradiated and welded, Groups 1 and 2, Table 3, shows that the welds generally have as high a fracture strain as did irradiated base metal. The poor properties of heat 5065 at 25°C after welding are again an exception to this generalization. The fracture strain of samples irradiated, welded, annealed 8 hr at 870°C, and tested at 650°C is higher than for the comparable irradiated base metal sample. Note that the fracture location in the irradiated sample shifts from the weld metal to the base metal following the postweld anneal of 8 hr at 870°C. This is in contrast to the unirradiated welds where fracture occurred in the weld metal of both as-welded and postweld annealed samples.

Several of the samples were examined metallographically. The fracture of an unirradiated welded sample is shown in Fig. 2. This sample was tested at 25°C without postweld annealing, and fracture occurred in the base metal. The weld area has a larger diameter, indicating that

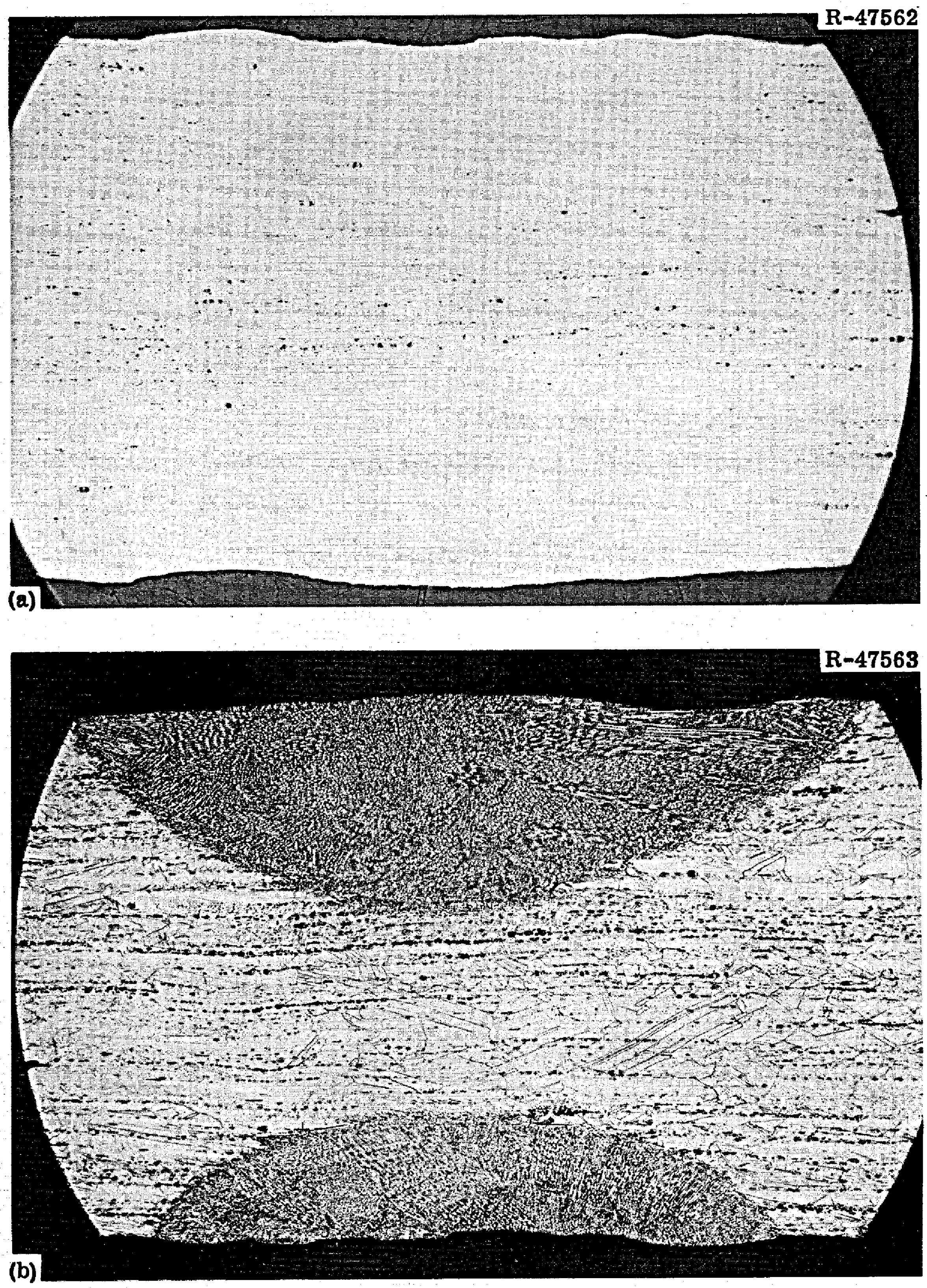

Fig. 2. Photomicrographs of Sample 10,082. Heat 5085, Unirradiated, welded in the hot cell and tested at 25°C. Fracture occurred in the base metal. (a) As polished. (b) Etchant: glyceria regia. 35×.

it is stronger than the base metal under these test conditions. The fracture of an unirradiated weld sample is shown in Fig. 3. The fracture is across the weld zone and the base metal fracture has both trans- and intergranular sections. There is also some porosity in the weld metal. The fracture of an unirradiated welded sample tested at 650°C is shown in Fig. 4. This sample had been exposed to molten salt for 4800 hr at 650°C, and the weld looks very sound with only a little porosity. The intercellular cracks in the weld metal indicate that the weld metal did deform.

The fracture of an irradiated sample that fractured as it was removed from the welding fixture is shown in Fig. 5. There is some porosity near the fusion line and some within the weld metal. The microstructure of another sample that was welded after irradiation is shown in Fig. 6. This sample was tested at 650°C, and the fracture was intergranular and located in the base metal. Again, there is a large amount of porosity near the fusion line and in the weld metal. Much of the porosity near the fusion line is associated with the carbide stringers that are present. Because of the similar chemical behavior of carbon and boron, it is quite reasonable to suspect that these stringers of carbides would also be enriched in boron. Transmission electron microscopy of this material shows that helium bubbles are present in this material (Fig. 7), and the heating may allow enough diffusion to occur near the fusion line for the bubbles to agglomerate.

DISCUSSION OF RESULTS

These tests have shown that the fracture strain of Hastelloy N in tensile tests at 25 and 650°C decreases with long exposure at 650°C. Neutron irradiation causes an even more dramatic decrease in the fracture strain. We fused about 75% of the cross section of both unirradiated and irradiated samples. Welding alone caused rather large decreases in the fracture strain of unirradiated samples. These samples responded much as we had noted earlier in another study[6] involving

[6]H. E. McCoy and D. A. Canonico, "Preirradiation and Postirradiation Mechanical Properties of Hastelloy N Welds," Welding J. (N.Y.) 48(5), 203-s–211-s (May 1969).

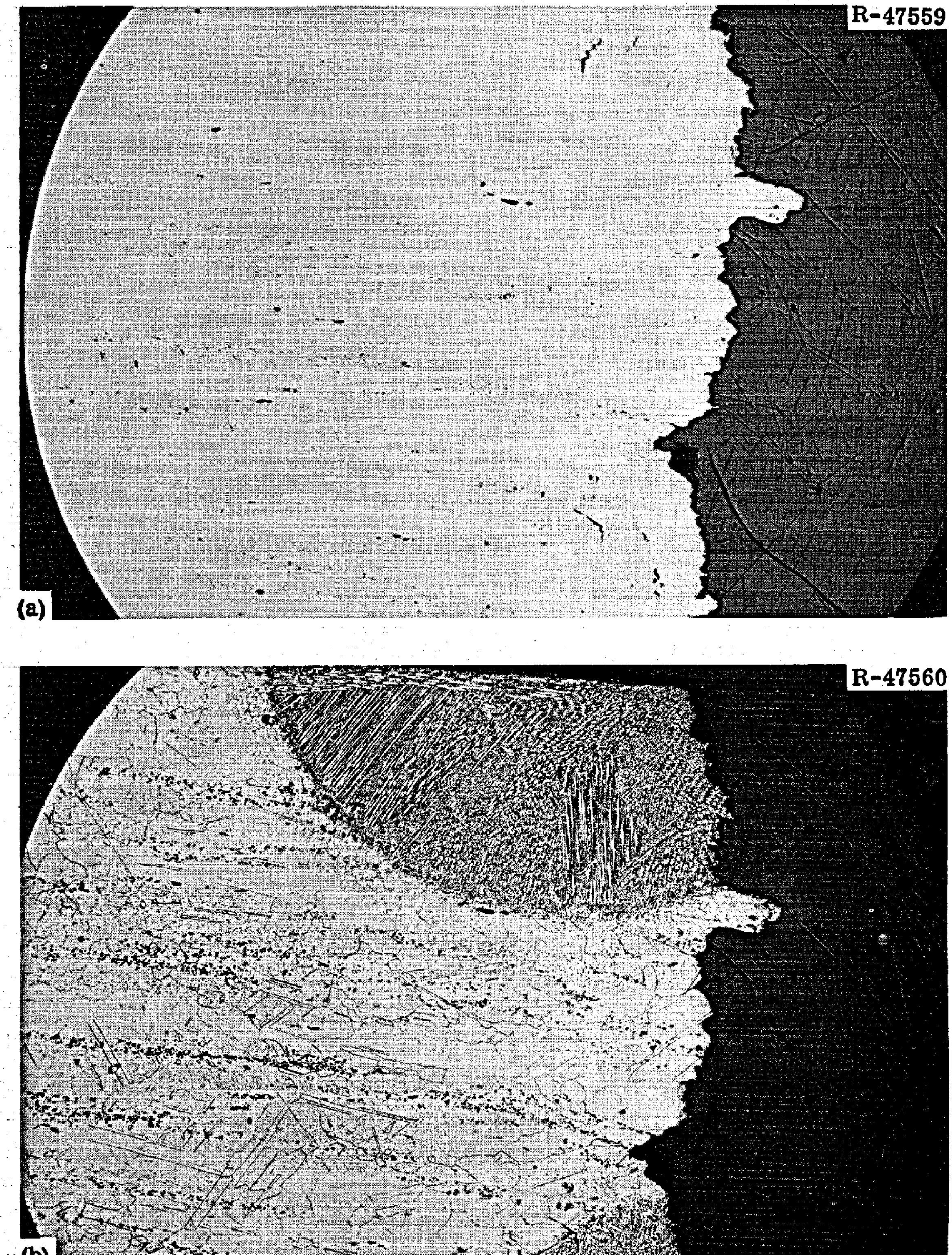

Fig. 3. Photomicrograph of Sample 10,081. Heat 5085, Unirradiated, Welded in Hot Cell, Tested at 650°C. Fracture occurred in the weld metal. (a) As polished. (b) Etchant: glyceria regia. 35X.

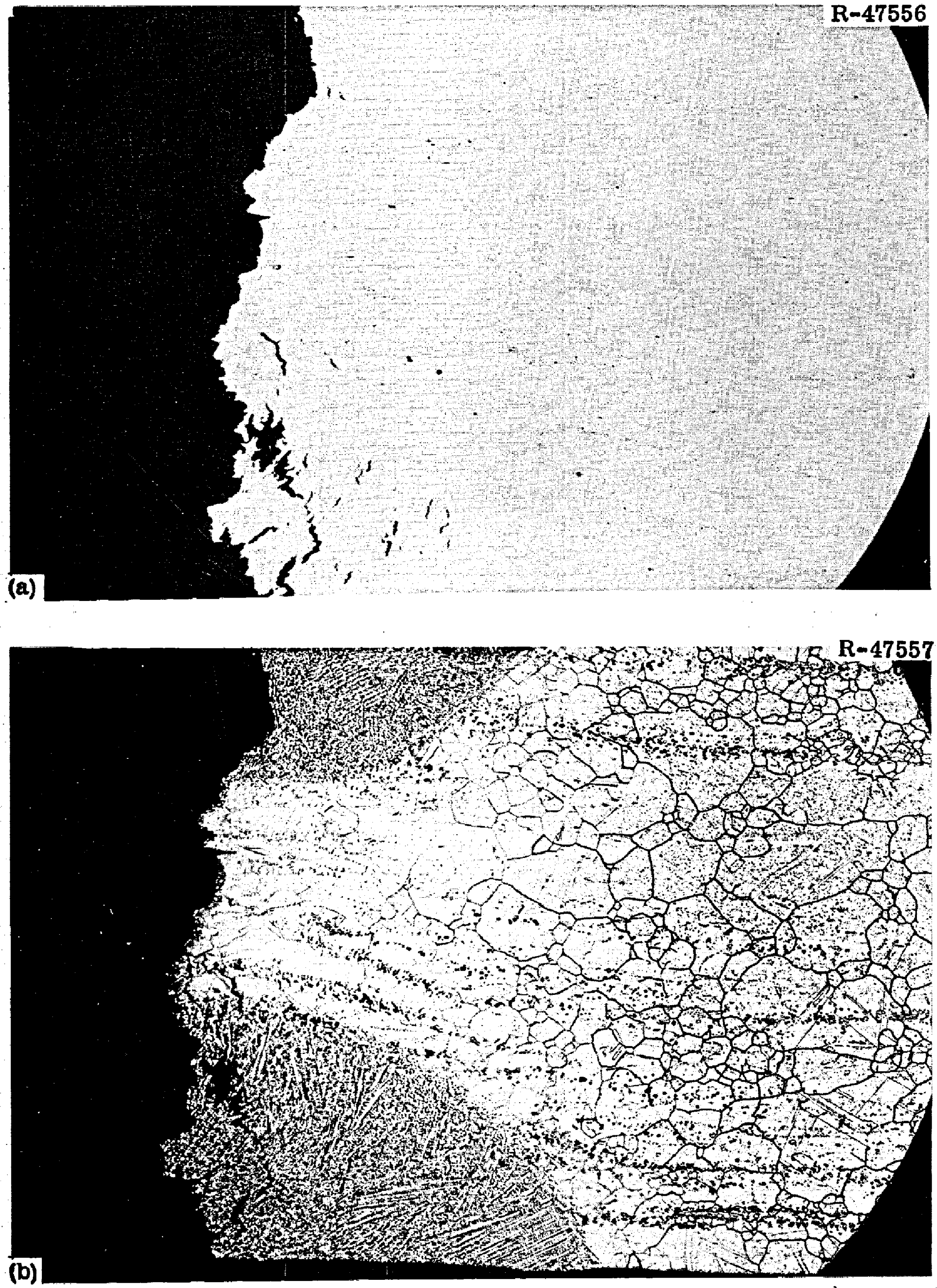

Fig. 4. Photomicrograph of Sample 9010. Heat 5085, exposed to static barren "fuel" salt for 4800 hr at 650°C, welded in hot cell, tested at 650°C. Fracture occurred in the weld metal. (a) As polished. (b) Etchant: glyceria regia. 35×.

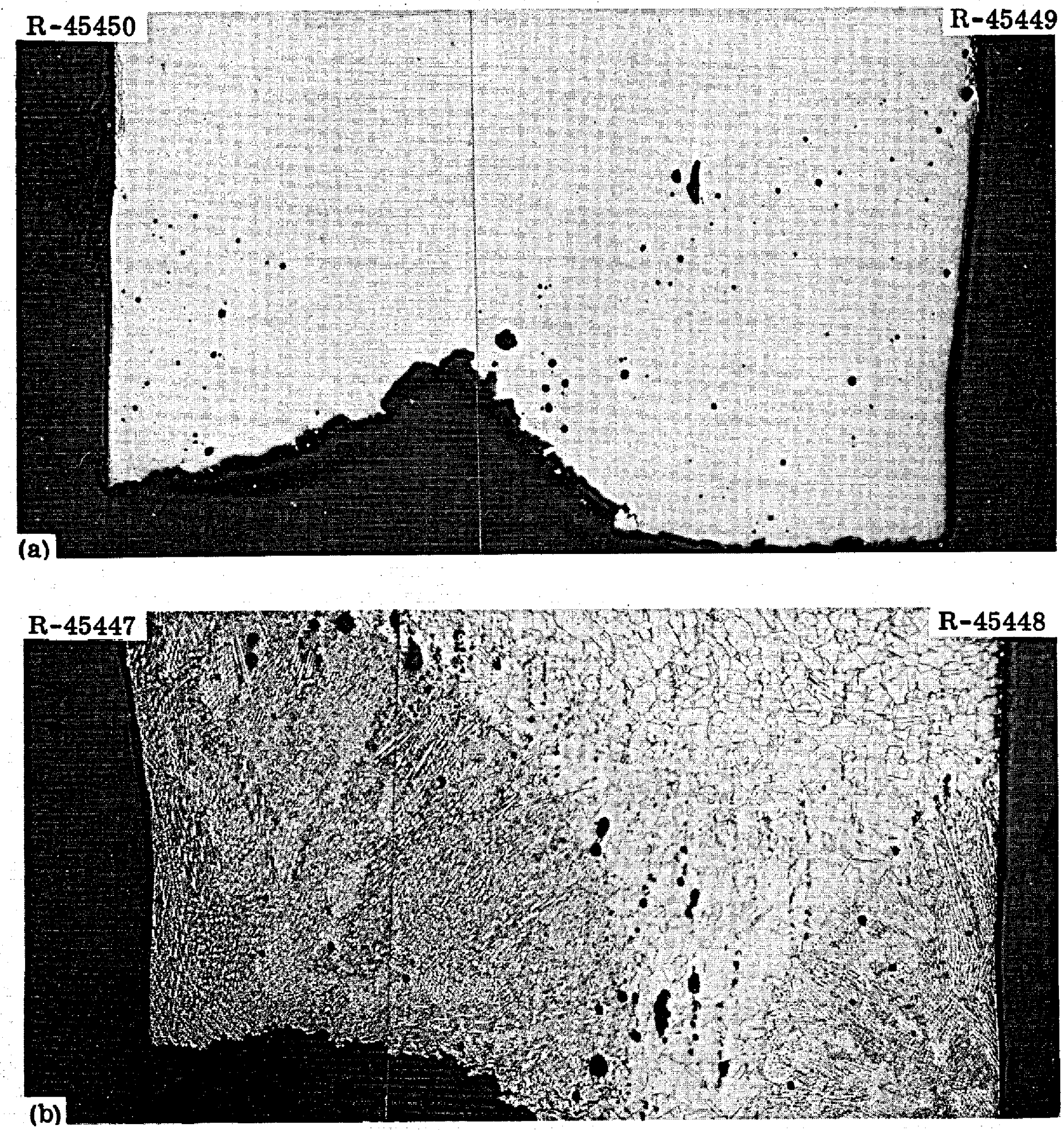

Fig. 5. Photomicrographs of Sample 7897. Heat 5065, irradiated for 15,289 hr in fuel salt in the MSRE at 650°C. Thermal fluence was 9.4×10^{20} neutrons/cm^2, welded in hot cell, and broke while removing from weld fixture. (a) As polished, (b) etchant: aqua regia. 35X.

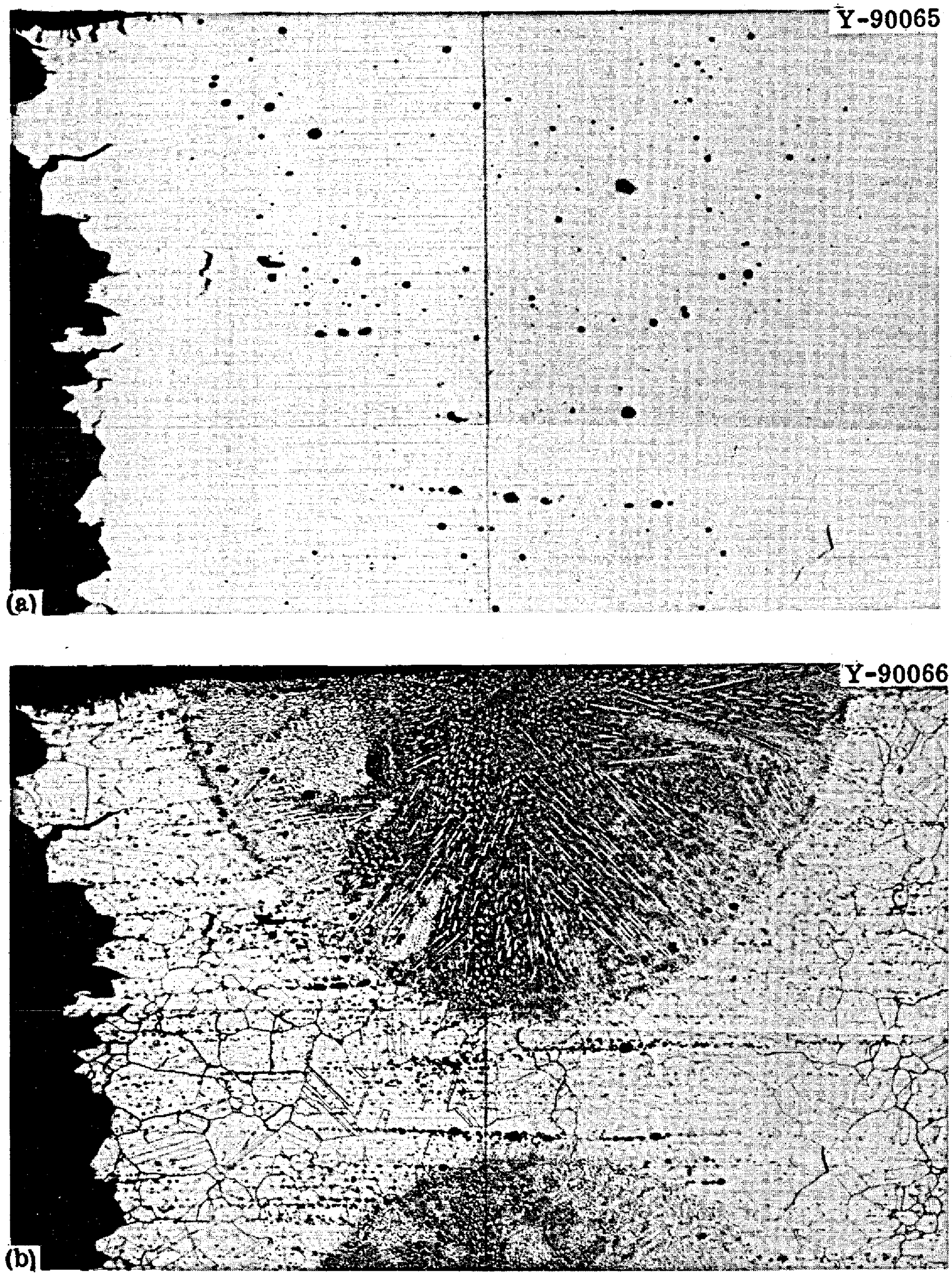

Fig. 6. Photomicrographs of Sample 7870. Heat 5085, irradiated for 15,289 hr in fuel salt in the MSRE at 650°C to a thermal fluence of 9.4×10^{20} neutrons/cm^2, welded in a hot cell, and tested at 650°C. Fracture occurred in the base metal. (a) As polished, (b) etchant: aqua regia. 35×.

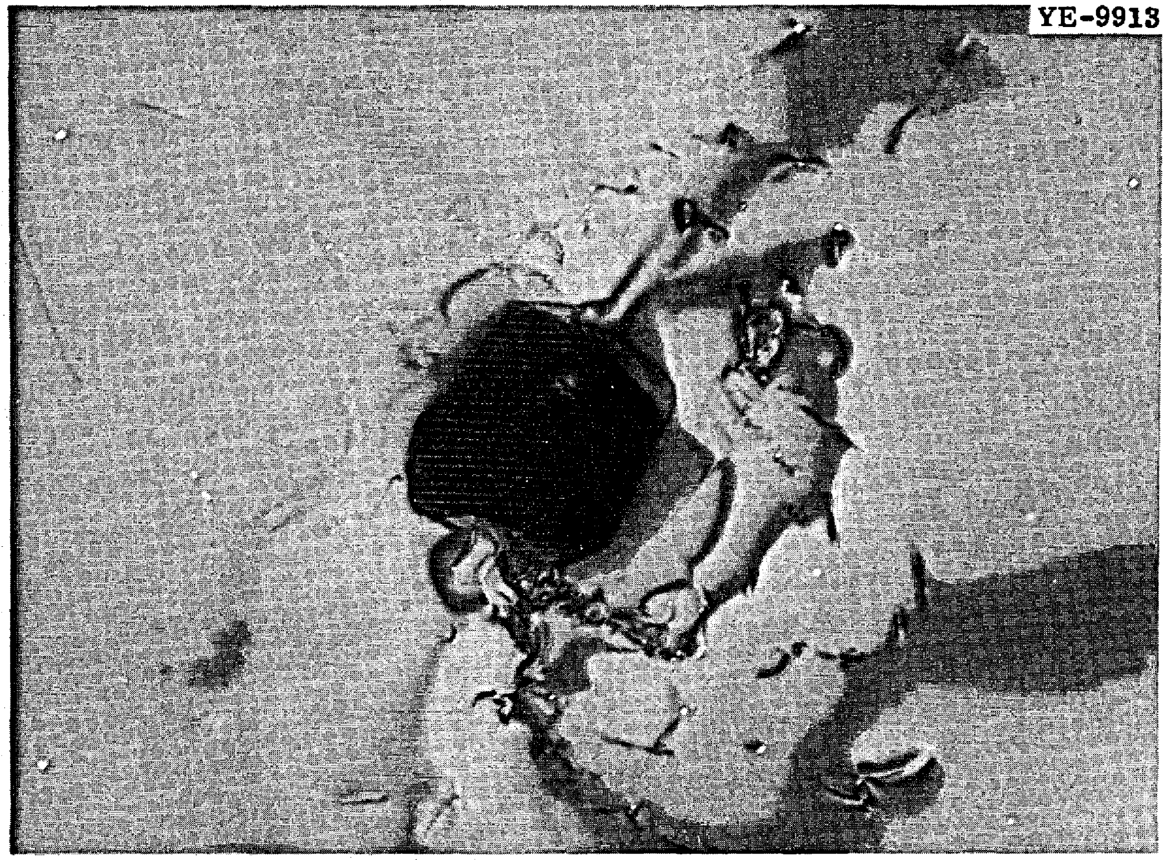

Fig. 7. Transmission Electron Micrograph of Hastelloy N (Heat 5085) Irradiated in the MSRE to a Thermal Fluence of 9.4×10^{20} neutrons/cm^2 at 650°C. 25,000×.

welds in large plates of Hastelloy N. Some of the irradiated samples were welded and these were found to have fracture strains at least as high as those observed for the irradiated base metal. (Heat 5065 tested at 25°C is an exception and its ductility was very low after welding.) Our previous work had involved some samples that were welded and then irradiated.[7] Most of our samples that were welded after irradiation had higher fracture strains at 650°C than the samples in our previous study that were welded before irradiation. This is probably due to the drastic redistribution of helium that occurs when the metal is fused. Most of the helium should be lost from the weld metal, and this exhibits

[7]H. E. McCoy and D. A. Canonico, "Preirradiation and Postirradiation Mechanical Properties of Hastelloy N Welds," Welding J. (N.Y.) 48(5), 203-s–211-s (May 1969).

more ductility at high temperatures than the irradiated base metal where the helium is thought to be associated with grain boundaries. Thus, the weld metal will strain and make a significant contribution to the total strain.

A rather consistent pattern evolves for the location of the fracture in welded samples. In both irradiated and unirradiated samples tested at 25°C, the fracture occurs in the base metal in as-welded samples and shifts to the weld metal after a postweld heat treatment of 8 hr at 870°C. The weld metal in the as-deposited form is stronger at 25°C (Fig. 2) and does not deform as much as the base metal. After annealing, the weld metal softens and fracture occurs in the weld metal. This observation does not indicate anything about the relative ductilities of the weld and base metals, since the sample geometry allows the weaker material to deform without any deformation occurring in the stronger material. Thus, the as-deposited weld metal is strong at 25°C, but may be extremely brittle. At 650°C unirradiated welded samples failed in the weld metal in the as-welded and heat-treated conditions. The irradiated samples failed in the weld metal when tested in the as-welded condition and the fracture shifted to the base metal after annealing for 8 hr at 870°C. Metallographic studies indicate that the weld metal and the base metal both deform when tested at 650°C. Thus, the location of the fracture is likely governed by crack propagation. Cracks can propagate in unirradiated welds more easily in the weld metal than in the base metal and fracture occurs in the weld metal. Cracks seem to propagate very easily through irradiated base metal and the postweld annealed weld metal has better resistance to crack propagation. Thus, irradiated welded samples fail in the weld metal in the as-welded condition and in the base metal after annealing.

Two metallographic features in the irradiated welds deserve some comment; the porosity in the weld metal and the porosity near the fusion line (Figs. 5 and 6). The voids in the weld metal are thought to be related to superficial surface films on the samples before welding or are characteristics of the particular heats of material involved.

The porosity near the fusion lines is associated preferentially with the carbide stringers. We have noted that these stringers are high in silicon,[8] and that melting starts in these areas when the alloy is heated to about 1400°C (ref. 9). Thus, the supposition that these void areas result from localized melting would seem reasonable were it not for the observation that unirradiated welded samples do not contain this porosity (Figs. 2, 3, and 4). The possibility that they are large agglomerates of helium that form during welding must at least be considered. A shell of material 0.037 cm (~ 0.015 in.) thick around the fusion zone would contain about 0.2 cm^3 of transmuted helium at atmospheric pressure and 1400°C. If this helium were distributed as small bubbles 0.005 cm (~ 0.002 in.) in diameter, there would be about 2×10^6 bubbles present in this small volume. Thus, it seems likely that the porosity near the fusion line is actually helium bubbles. There is no evidence that either type of porosity influenced the location of the fracture.

SUMMARY

Our studies have shown that fusion welds can be made in irradiated Hastelloy N after exposure to fluoride salts. The rather meager statistics indicate that acceptable welds are not obtained as frequently in the irradiated material as in the unirradiated samples.

Samples that had been irradiated and welded were found generally to have as good tensile fracture strain at 25 and 650°C as the base metal. Welded samples that were given a postweld anneal of 8 hr at 870°C were even more ductile than the irradiated base metal. At 25°C both unirradiated and irradiated welds failed in the base metal in the as-welded condition and in the weld metal after annealing 8 hr at 870°C.

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[8] R. E. Gehlbach and H. E. McCoy, Jr., "Phase Instability in Hastelloy N," pp. 346–366 in International Symposium on Structural Stability in Superalloys, Seven Springs, Pennsylvania, September 4–6, 1968, Vol. II. Available from Dr. John Radavich, AIME High-Temperature Alloys Committee, Micromet Laboratories, West Lafayette, Indiana.

[9] H. E. McCoy, Influence of Several Metallurgical Variables on the Tensile Properties of Hastelloy N, ORNL-3661 (August 1964).

At a test temperature of 650°C, the unirradiated welds failed in the weld metal in the as-welded and heat-treated condition. The samples irradiated and welded failed in the weld metal in the as-welded condition and in the base metal after annealing.

The weld metal in all samples contained minor porosity that likely reflects the welding characteristics of this alloy under the welding parameters that we used. The irradiated samples had a large amount of porosity associated with the carbide stringers near the fusion line. We feel that this porosity resulted from the agglomeration of small transmutation-produced helium bubbles during the welding.

The data are not sufficient to draw a meaningful conclusion about the weldability of irradiated reactor components of Hastelloy N; the samples were too small, the heat input too low, and the degree of restraint too low. The observation that the fused weld metal will deform readily at 650°C is encouraging since this indicates that the weld metal might deform small amounts to relieve stresses between relatively large and brittle components or pipe segments. The observed porosity near the fusion line means that this area will be weakened. Welds in large sections will be required to determine whether the composite joint of weld metal and fusion zone has acceptable properties.

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