OAK RIDGE NATIONAL LABORATORY
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ELEVATED-TEMPERATURE MECHANICAL PROPERTIES OF WELDS IN A Ni-Mo-Cr-Fe ALLOY

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ABSTRACT

A non-age-hardenable, high-strength alloy was developed at the Oak Ridge National Laboratory for use as the primary containment material for the Molten Salt Reactor Experiment (MSRE). The alloy was tailored to give good strength, ductility and corrosion resistance to molten fluoride salts in the 1100 to 1500°F temperature range. The work reported here concerns the elevated-temperature mechanical properties of welds made in this alloy, known as INOR-8, as represented by several heats of MSRE-grade material.

Tensile tests on transverse weld samples in the as-welded and annealed conditions show a good combination of strength and ductility at temperatures ranging from 70 to 1800°F. Tensile properties of these weld samples compare favorably with those of the base metal. Stress relieving at 1600°F for 2 hr results in a lowering of the tensile yield strength. Creep-rupture tests at 1100, 1300 and 1500°F on these same type specimens show significant improvement in strength and ductility at 1300°F following a hydrogen atmosphere stress relief. Both as-welded and stress-relieved creep-rupture behavior was as good as the base metal behavior. The nil-ductility temperature, as determined by

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simulated heat-affected zone thermal cycle tests, was found to be 2300°F. Reasonable recovery of mechanical properties follows a simulated welding cycle with a 2300°F maximum temperature.

INTRODUCTION

In order to fully realize the potential of nuclear reactor systems utilizing molten fluoride salts as fuel, the structural materials must possess adequate combinations of strength, ductility, and corrosion resistance at temperatures in the 1100–1500°F temperature range. The non-age-hardenable nickel-molybdenum-chromium-iron alloy, designated as INOR-8 [Ni–17 Mo–7 Cr–5 Fe (wt %)] is such an alloy and was the culmination of a comprehensive alloy development and evaluation program carried out at the Oak Ridge National Laboratory.[1–3] It is now commercially available** and is the primary containment material for the Molten Salt Reactor Experiment which achieved criticality on June 1, 1965, at Oak Ridge, Tennessee. A vital part of the overall welding study on INOR-8 was the determination of the elevated-temperature mechanical properties of welds. This report summarizes the findings on the mechanical behavior of welds in some of the actual heats of material used in the MSRE.

MATERIALS, TESTING PROCEDURE AND EXPERIMENTAL RESULTS

Transverse samples machined from 1-in.-thick welds, made under highly restrained conditions were used in the testing. These butt welds were fabricated in the manner illustrated in Fig. 1 and described in earlier work.[3] The transverse tensile specimens used were of the

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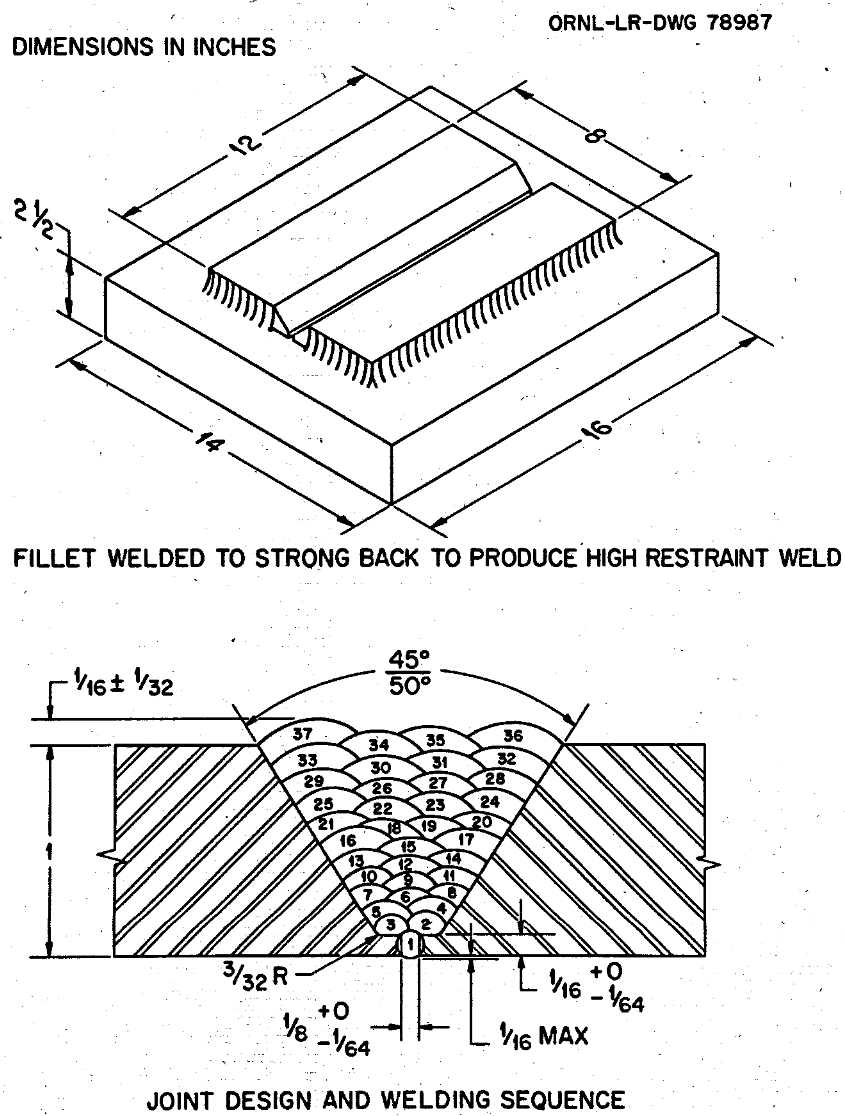

Fig. 1 — The INOR-8 high-restraint weldability test specimen used to provide samples for the mechanical properties study.

design shown in Fig. 2. The manual gas tungsten arc-welding process was used to fabricate the joints; the filler metal used was of the same nominal composition as the base metal. The INOR-8 material used in this study was taken from the stock purchased for construction of the MSRE. The heat numbers and the tests to which each heat of INOR-8 was subjected are tabulated below.

Heat Number	Type of Test
5055	(Weld Metal)
5057	Creep-stress relieved, hydrogen
5060	Creep-as-welded
5062	Tensile-as-welded
5064	Tensile-stress relieved, argon
5067	Creep-stress relieved, hydrogen
5068	Creep-stress relieved, argon
5069	Creep-as-welded
5070	Tensile-stress relieved, hydrogen
5071	Tensile-as-welded
5072	Creep-stress relieved, hydrogen
5073	Tensile-stress relieved, hydrogen
5074	Tensile-as-welded; creep-stress relieved, argon
5075	Tensile-stress relieved, argon; Creep-as-welded
5081	Tensile-stress relieved, argon
5083	Creep-as-welded
5089	Creep-stress relieved, hydrogen
5090	(Weld Metal)

The chemical analyses of these heats are presented in Table 1. Note that the filler metal used to fabricate the weld specimens was taken from heat numbers 5055 and 5090.

Because of material fabrication schedules, it was necessary to do the welding operations on plate which had the final rolling operation performed normal to the welding direction. This schedule caused the materials' inherent stringer line to be located in the plate-thickness direction and parallel to the fusion line. A typical cross section of

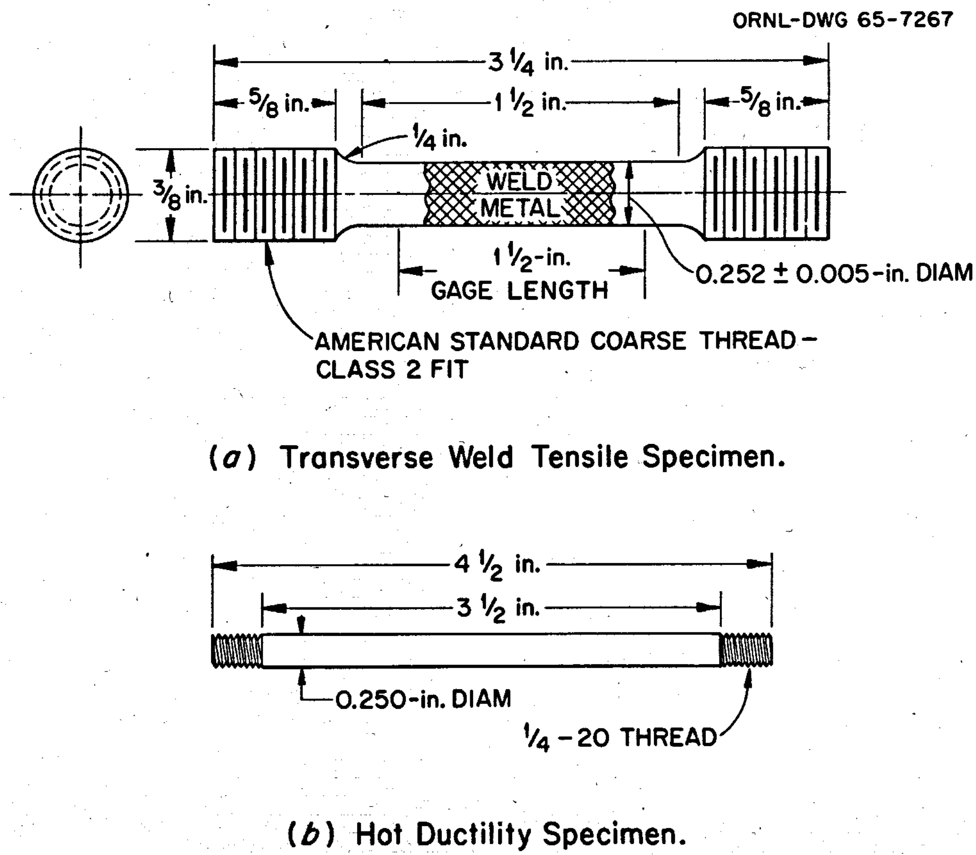

Fig. 2 – Transverse tensile specimen used for high-restraint INOR-8 weld evaluations.

Table 1. Chemical Analysis of the INOR-8 Heats

Heat Number	Analysis (wt %)[a]													
	Mo	Cr	Fe	C	Si	Al	Ti	B	Co	V	Mn	W	P	S
5055	16.20	7.86	3.76	0.06	0.61	0.06	0.02	0.005	0.10	0.21	0.69	0.03	0.006	0.008
5057	16.80	7.47	3.50	0.05	0.52	0.01	0.01	0.005	0.09	0.23	0.58	0.02	0.001	0.006
5060	16.20	6.92	4.03	0.07	0.56	0.01	0.01	0.004	0.02	0.26	0.46	0.04	0.001	0.001
5062	16.17	7.45	3.78	0.04	0.58	0.01	0.01	0.005	0.08	0.24	0.63	0.08	0.001	0.006
5064	16.37	7.90	3.59	0.07	0.69	0.01	0.01	0.005	0.06	0.29	0.59	0.03	0.001	0.006
5067	17.07	7.23	4.20	0.06	0.43	0.01	0.01	0.004	0.09	0.30	0.60	0.06	0.001	0.006
5068	16.50	6.45	4.11	0.05	0.58	0.01	0.01	0.004	0.09	0.27	0.45	0.07	0.003	0.008
5069	16.22	6.42	3.93	0.07	0.56	0.01	0.01	0.006	0.12	0.23	0.52	0.06	0.003	0.009
5070	16.06	6.62	3.93	0.06	0.60				0.04	0.22	0.64	0.03	0.001	0.008
5071	16.22	7.21	4.35	0.07	0.63	0.01	0.01	0.004	0.07	0.20	0.65	0.02	0.001	0.008
5072	15.50	7.03	4.06	0.06	0.57	0.01	0.02	0.005	0.09	0.27	0.57	0.04	0.001	0.012
5073	16.21	6.77	3.90	0.05	0.60	0.01	0.01	0.006	0.07	0.34	0.50	0.03	0.004	0.008
5074	16.17	6.77	3.70	0.07	0.62	0.01	0.01	0.007	0.07	0.24	0.51	0.02	0.001	0.006

Heat Number	Analysis (wt %)[a]													
	Mo	Cr	Fe	C	Si	Al	Ti	B	Co	V	Mn	W	P	S
5075	16.42	6.76	4.08	0.06	0.57	0.03	0.01	0.001	0.06	0.28	0.49	0.04	0.003	0.005
5081	16.90	7.74	3.56	0.06	0.60	0.01	0.01	0.005	0.07	0.27	0.57	0.04	0.003	0.006
5083	17.03	7.51	3.80	0.05	0.52	0.01	0.01	0.005	0.10	0.28	0.60	0.05	0.001	0.006
5089	16.69	6.78	3.81	0.07	0.36	0.01	0.01	0.004	0.14	0.38	0.34	0.05	0.011	0.012
5090	16.22	7.59	4.03	0.05	0.56	0.01	0.01	0.005	0.12	0.39	0.58	0.04	0.001	0.008

[a]Balance nickel.

the weld fusion line area is shown in Fig. 3. This view illustrates the orientation of the stringer formation with respect to the fusion line and its perpendicular orientation with respect to the welding direction and specimen axis.

The transverse weld specimens contained weld metal, heat-affected zones, and base metal and were tested at elevated temperatures in standard tensile tests (0.03 min^{-1}) and in creep tests. Elevated-temperature tensile tests were performed between 600 and 1800°F at 200°F intervals, and creep testing was done at 1100, 1300 and 1500°F. The specimens were tested in both the as-welded and stress-relieved conditions, with stress relieving being performed in both argon and hydrogen atmospheres (2 hr at 1600°F). Samples were also machined from the as-received base metal for the determination of elevated-temperature hot ductility after being subjected to simulated heat-affected zone thermal cycles.

The room- and elevated-temperature tensile tests were run in air using a standard 12,000-lb capacity hydraulic testing machine at a crosshead speed of 0.05 in./min or a strain rate of 3.33%/min. Stress-strain relationships were obtained using load cell-deflectometer outputs. Elevated-temperature tests were performed using a clamshell-type furnace, in which specimens were allowed a 1/2-hr equilibration period to reach the test temperature. The results of these tests for the as-welded and stress-relieved conditions are presented in Tables 2 and 3 for test temperatures between room temperature and 1800°F.

The creep tests were run in air using standard lever arm testing machines, and strain data were obtained through dial-gage extensometers

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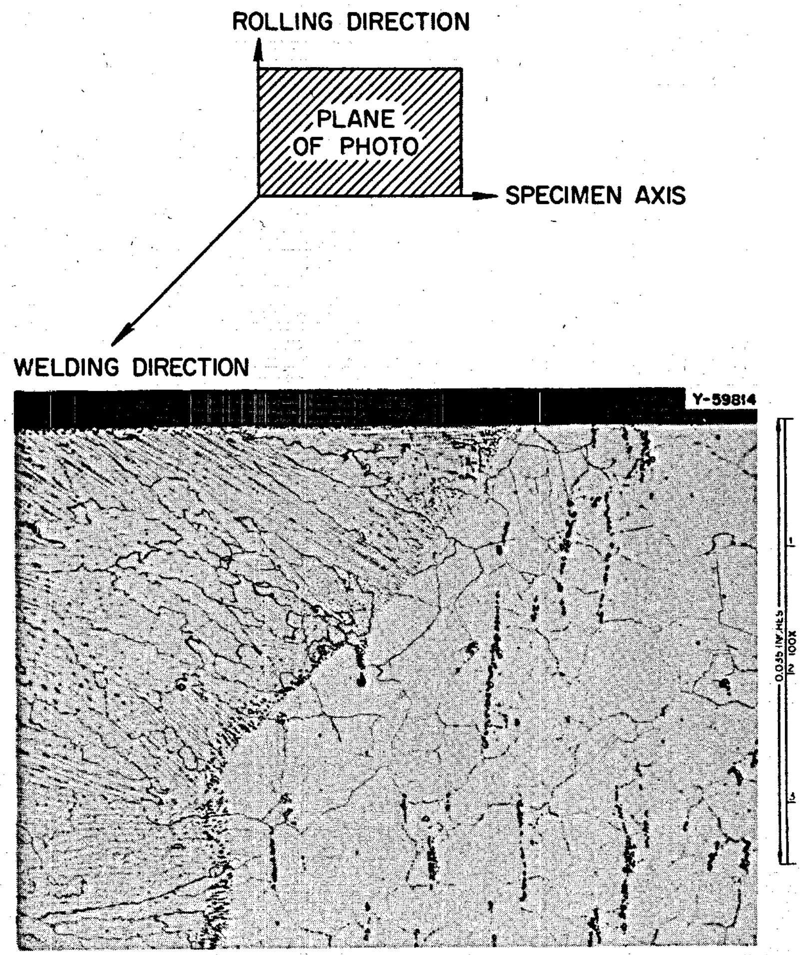

Fig. 3 — Typical fusion-line area of the 1-in. thick restrained weld used to provide specimens for the mechanical property study. Etchant: CrO_3, HCl, H_2O. 100×.

Table 2. Short-Time Tension Tests of Reactor Grade INOR-8 in As-Welded Condition

Test Temperature (°F)	Yield Strength 0.2% Offset (psi)			Tensile Strength (psi)			Elongation in 1 1/2 in. (%)		
	Heat 5062	Heat 5071	Heat 5074	Heat 5062	Heat 5071	Heat 5074	Heat 5062	Heat 5071	Heat 5074
Room	63,100	67,200	66,000	105,700	108,800	91,500	31.5[a]	27.5[a]	10.0
600	54,000	58,400	52,100	93,900	95,500	84,300	26.5[a]	27.5[a]	19.0
800	54,300	51,900	52,600	92,100	91,400	82,300	29.0[a]	29.5	13.5
1000	46,400	50,700	47,100	84,100	83,800	79,800	24.0[a]	26.5[a]	16.5
1200	43,300	49,500	47,600	73,300	73,400	66,800	17.5[a]	17.5[a]	7.5
1400	42,400	46,600	42,100	61,400	58,600	59,600	11.0[a]	8.5[a]	8.5[a]
1600	36,400	37,700	36,400	38,700	38,400	38,200	10.0[a]	9.5[a]	8.5[a]
1800	21,100	21,900	20,800	21,800	22,400	21,200	17.5[a]	22.0[a]	13.5

[a] Failed in weld metal.

Table 3. Short-Time Tension Tests of Reactor Grade INOR-8 in Stress-Relieved Condition

Test Temperature (°F)	Yield Strength 0.2% Offset (psi)			Tensile Strength (psi)			Elongation in 1 1/2 in. (%)		
	Heat 5070[a]	Heat 5073[a]	b	Heat 5070[a]	Heat 5073[a]	b	Heat 5070[a]	Heat 5073[a]	b
Room	46,000		56,500	97,300		102,500	21.0		26.5[c]
600	45,400	43,400	43,400	90,600	90,400	91,800	25.5	25.0[c]	29.0[c]
800	48,400	39,900	43,500	85,500	87,200	89,000	26.0	27.5	28.5[c]
1000	39,500	40,400	40,000	85,500	85,300	84,000	32.0	33.0[c]	30.5[c]
1200	39,500	39,300	40,000	75,800	70,400	74,500	28.5[c]	20.5	28.0
1400	36,800	37,000	40,000	62,100	57,800	61,000	14.0[c]	14.0[c]	14.5
1600	34,500	35,000	35,500	39,600	40,300	39,500	17.5[c]	10.0[c]	11.0
1800	21,500	21,800	21,200	21,900	22,100	21,700	9.0	19.5	28.5[c]

[a]Stress relieved 2 hr at 1600°F, hydrogen.

[b]Averaged values of Heats 5064, 5075, and 5081 stress relieved 2 hr at 1600°F, argon.

[c]Failed in weld metal.

attached to the specimen grips. A detailed tabulation of the average values of the creep-rupture test results are given in Table 4.

All heat treatments prior to testing were carried out in either an argon or hydrogen atmosphere. The specimens were cooled from the annealing temperature by pulling them from the hot zone into the water-cooled end of the furnace muffle. Thermocouples, which were attached to the specimens during the heat treatment, recorded an average cooling rate down to 500°F of approximately 250°F/min. The thermal treatment used, 2 hr at 1600°F, is the standard stress relieving treatment specified for INOR-8 material.

A convenient method for measuring overall weldability is a hot ductility test, which has been developed by Nippes and Savage of Rensselaer Polytechnic Institute.[4] This test synthetically reproduces, in a laboratory specimen, the time-temperature cycle experienced by any selected point in the heat-affected zone of an arc weld. Samples were taken from all the heats tabulated previously (p. 4) and subjected to this hot ductility test. The results of these tests will be treated in the following section.

Table 4. Average Results of Elevated-Temperature Creep Tests on INOR-8 Transverse Weld Specimens

Test Temperature (°F)	Applied Stress (psi)	Time to Rupture (hr)			Elongation (%)			Minimum Creep Rate (hr^{-1})		
		As Welded[a]	Stress Relieved[b] H_2	Stress Relieved[b] Ar	As Welded[a]	Stress Relieved[b] H_2	Stress Relieved[b] Ar	As Welded[a]	Stress Relieved[b] H_2	Stress Relieved[b] Ar
1100	74,000	1.3	1.7		14.1	13.0		2.6×10^{-3}	1.3×10^{-2}	
1100	54,000	197.8	188.3		2.5	8.2		2.3×10^{-5}	1.0×10^{-5}	
1100	49,000	308.4	570.5		2.2	5.3		1.4×10^{-5}	3.2×10^{-5}	
1300	45,000	3.7	6.4	5.5	3.9	8.2	5.4	4.9×10^{-3}	7.4×10^{-3}	7.0×10^{-3}
1300	24,000	158.4	337.8	185.4	3.7	8.8	7.4	1.2×10^{-4}	1.6×10^{-4}	2.2×10^{-4}
1300	20,000	472.3	936.7	452.2	4.6	10.8	3.7	3.5×10^{-5}	6.6×10^{-5}	4.4×10^{-5}
1500	22,000	12.7	12.1		16.9	20.9		5.8×10^{-3}	7.0×10^{-3}	
1500	13,000	172.1	117.5		14.4	9.8		4.8×10^{-4}	4.4×10^{-4}	
1500	10,000	446.9	314.5		8.3	8.1		9.8×10^{-5}	2.0×10^{-4}	

[a]Data from tests on heats tabulated on p. 4 of this report.

[b]Stress relieved at 1600°F, 2 hr in atmosphere specified.

DISCUSSION AND EVALUATION OF RESULTS

Tensile Tests

The average tensile properties of specimens in the as-welded condition (heats 5062, 5071 and 5074), hydrogen stress-relieved condition (heats 5070 and 5073), and argon stress-relieved condition (heats 5064, 5075 and 5081) are presented in Figs. 4, 5 and 6. These figures show ultimate tensile strength, 0.2% offset yield strength and elongation vs temperature. The scatter bands for wrought metal shown in the figures were taken from earlier work.[5]

A qualification on the comparisons between transverse weld sample properties and wrought metal properties should be made at this point. Figure 7 illustrates specimen and stringer orientations which were encountered in these two classes of specimens. Note that, as previously mentioned, the material fabrication schedules resulted in the stringers of the transverse weld samples being oriented normal to the stress direction. This stress direction is the y-direction in Fig. 7. The wrought material investigation[5] done previously did not include specimens cut parallel to the y-direction. The fact that these plates were not thick enough to yield reasonably large specimens in the y-direction plus the assumption of symmetry of properties about the z-axis led to the omission.

Only slight variations in ultimate strengths were observed between the as-welded and stress-relieved tests; although, at the lower testing temperatures these strengths were less than those for the wrought metal. In the case of the yield strengths noticeable differences existed between the as-welded samples and stress-relieved samples. As-welded specimen yield strengths were consistently higher than those of

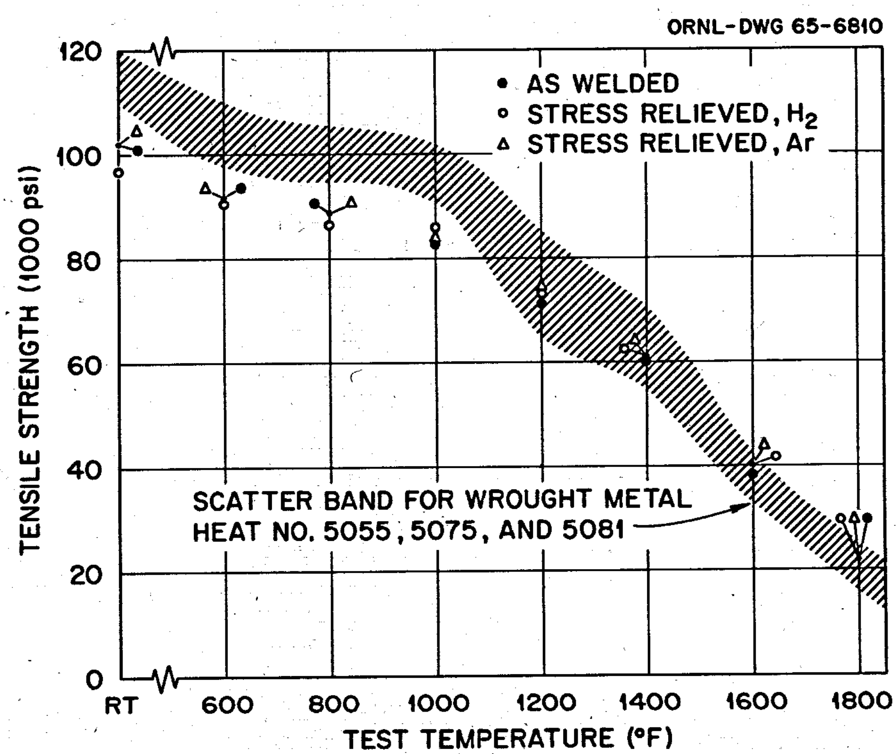

Fig. 4 — Ultimate tensile strength of MSRE INOR-8 transverse weld specimens.

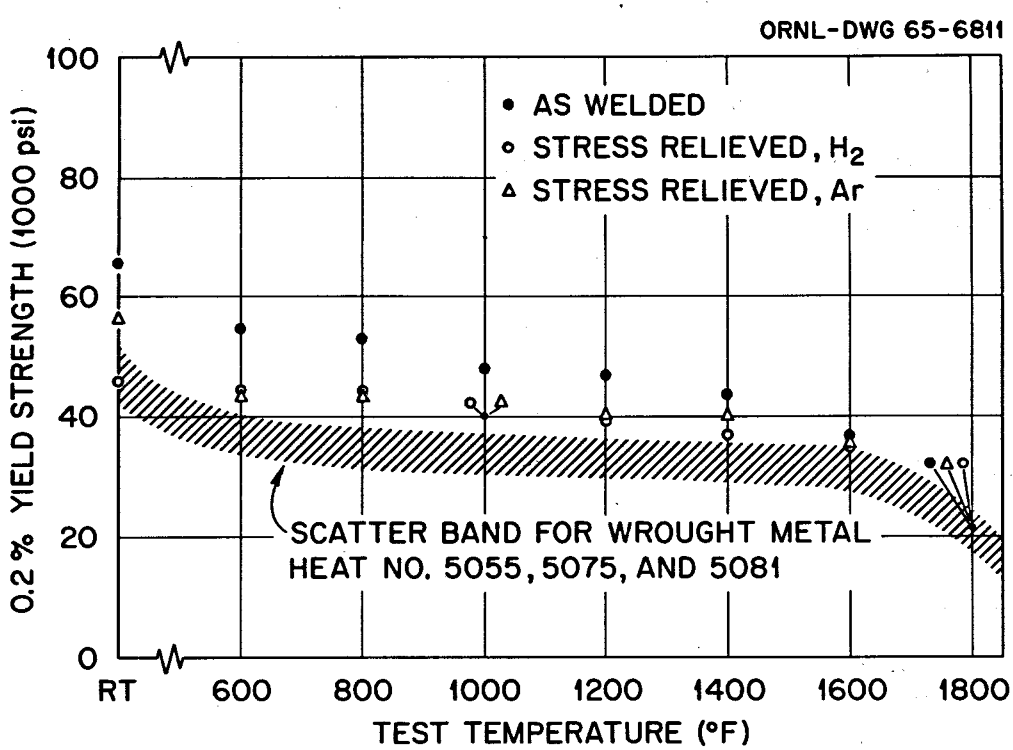

Fig. 5 — Yield strength (0.2%) of MSRE INOR-8 transverse weld specimens.

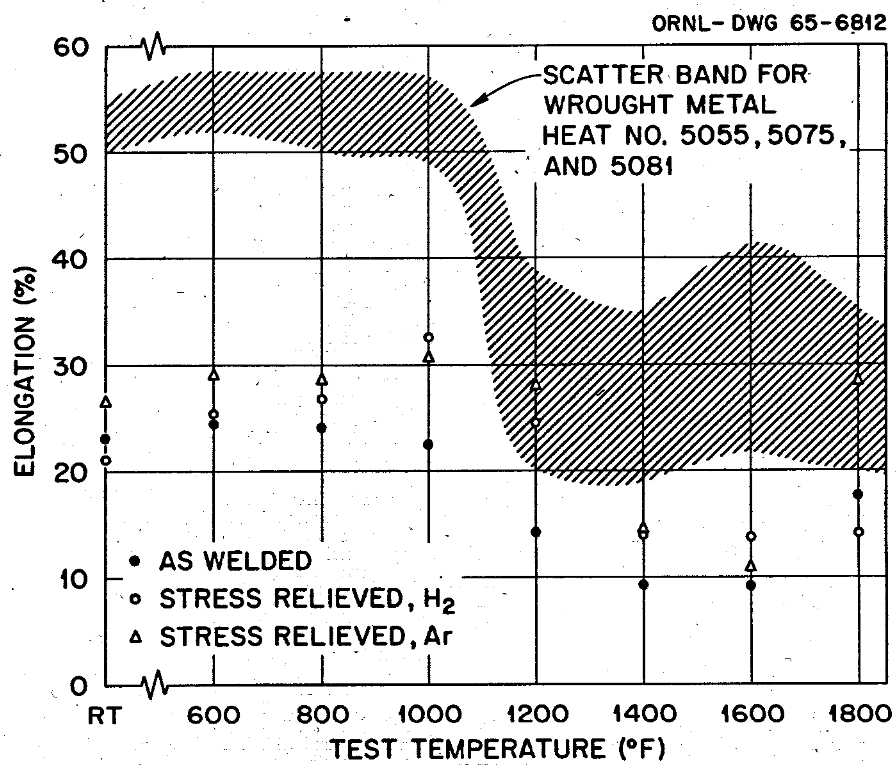

Fig. 6 — Ductility of MSRE INOR-8 transverse weld specimens.

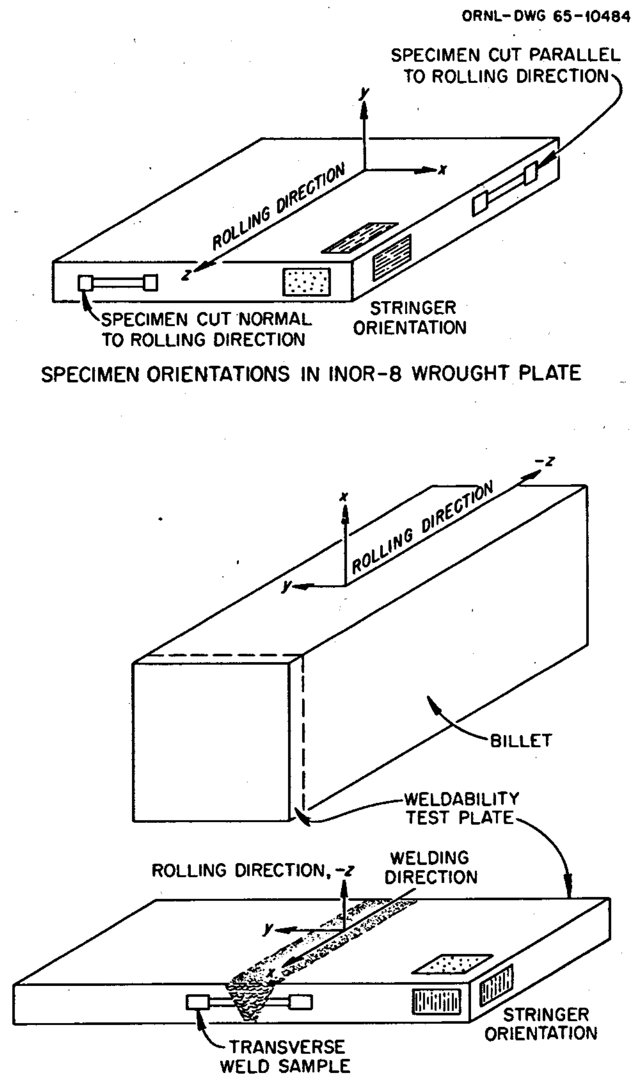

Fig. 7 — Sketch showing orientations of stringers and specimens for wrought metal and transverse weld samples.

specimens tested after stress relieving; no difference in annealing atmosphere was observed except at room temperature. Yield strengths for transverse specimens in all conditions were generally higher than the wrought metal yield strengths.

The total elongation data, presented in Tables 2 and 3 and depicted graphically in Fig. 6, show that the overall ductility of welds is less than that observed in the wrought metal. However, this difference is probably due to the specimen orientation, as previously explained. Stress relieving produces a general improvement of this property at all test temperatures, with argon providing a slightly better atmosphere than hydrogen.

The macrographs of Figs. 8, 9, and 10 illustrate the variations in fracture location with each heat and with test temperature. The as-welded tests in heat 5074 of Fig. 8 exhibit failures in the base metal, except at 1400 and 1600°C, with low ductilities (Table 2) as compared with the weld metal failures in heat 5062 of Fig. 9. The variation of fracture locations with temperature for the stress-relieved heat 5070 (Fig. 10) is typical of specimens tested in this condition.

On the basis of the data presented above on the ultimate and yield strengths the assumption of symmetry in strength properties seems justified. The ductility data, however, and in particular the ductility of those specimens which fractured predominantly in the base metal, suggest that the strain-at-fracture properties of INOR-8 are not symmetrical about the z axis.

Metallographic examination was performed on one heat tested in the as-welded condition (5074) and one heat in the stress-relieved condition (5073). The micrographs shown in Fig. 11A and 11B for the 1800°F tests

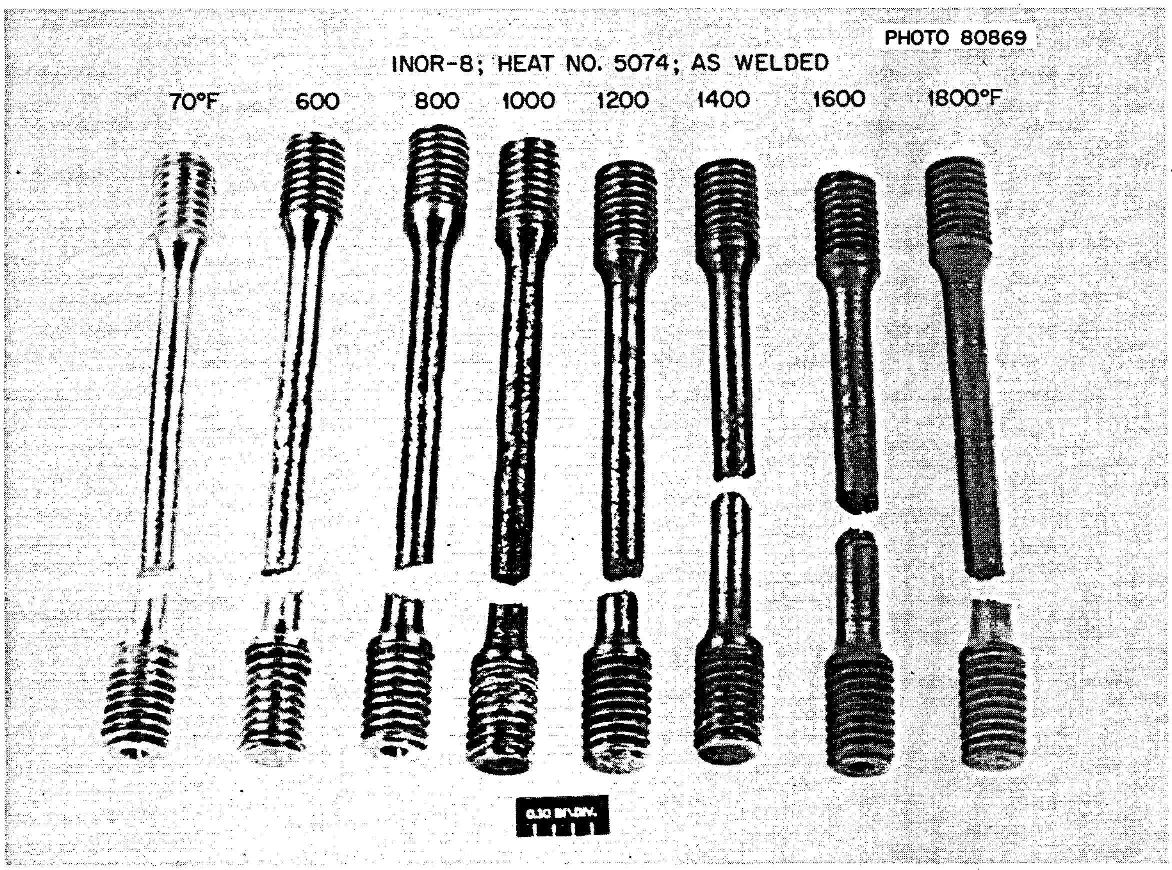

Fig. 8 — Tensile tests in INOR-8 heat 5074 (as welded) showing location of fracture.

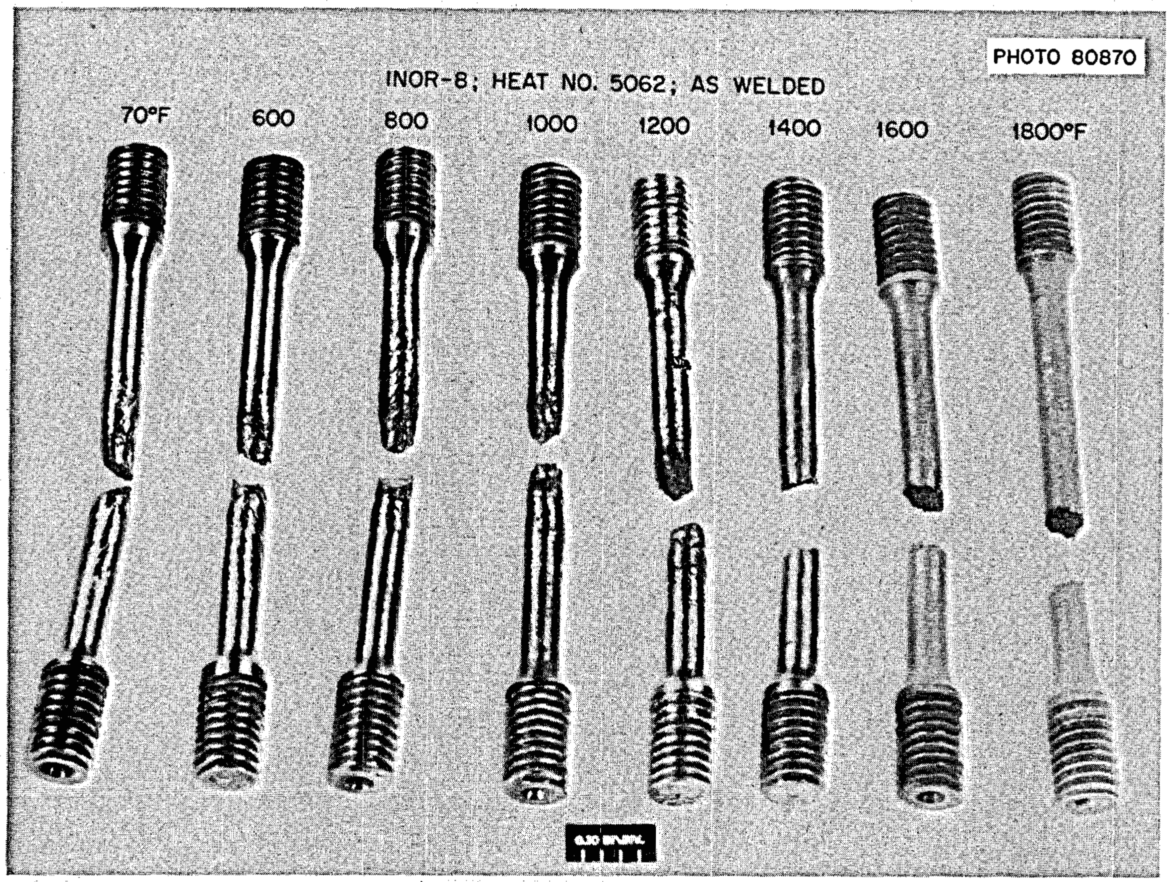

Fig. 9 – Tensile tests in INOR-8 heat 5062 (as welded) showing location of fracture.

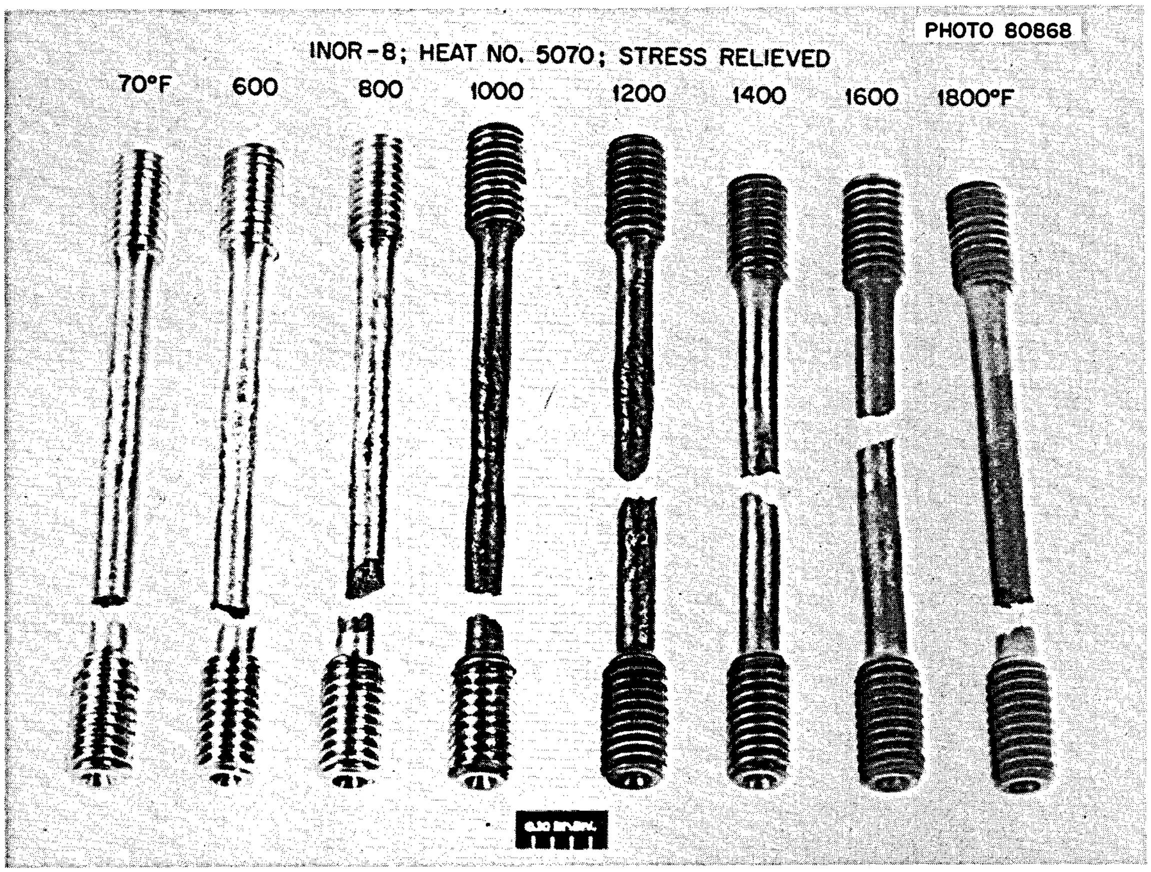

Fig. 10 – Tensile tests in INOR-8 heat 5070 (stress relieved 2 hr at 1600°F in hydrogen) showing location of fracture.

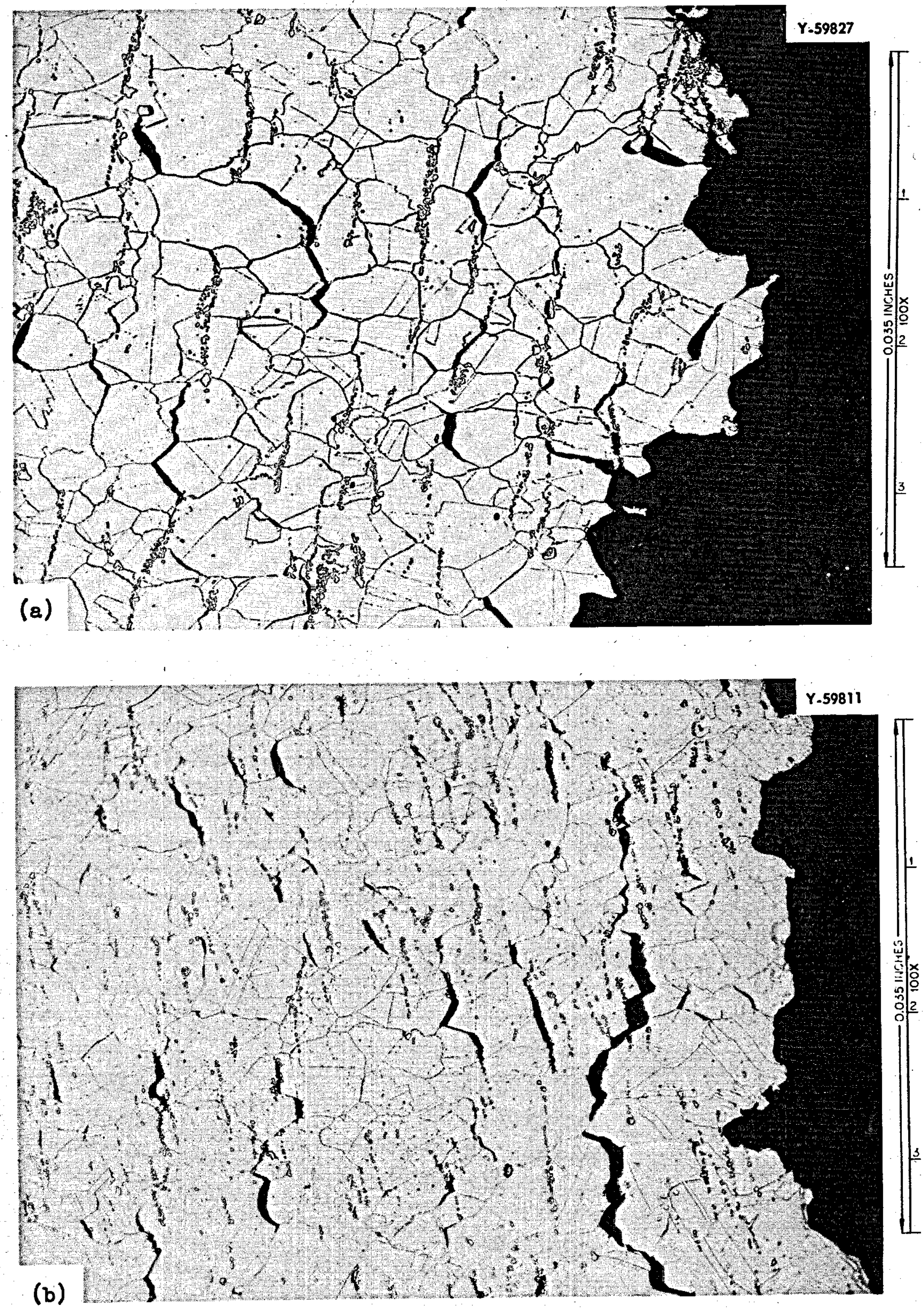

Fig. 11 – Short-time tensile test failures of INOR-8 material tested at 1800°F in air. A. Heat 5074, as welded; B. Heat 5073, stress relieved 2 hr at 1600°F in hydrogen. Etchant: CrO_3, HCl, H_2O. 100X.

are representative of the base metal failures of all the tensile tests. All failures, whether in the base metal or weld metal, were generally intergranular. As noted by McCoy,[6] most of the small base metal cracks appear to have been initiated by cracks in the precipitates. Here, as in McCoy's work, the intergranular cracks are predominantly normal to the applied stress with their lengths comparable to the grain diameter. As can be seen in Fig. 11, the cracks are parallel to and exist primarily in the stringer line.

Creep-Rupture Tests

Creep-rupture data for specimens in the as-welded and argon or hydrogen stress-relieved conditions are presented graphically in Figs. 12–15. Average values of rupture time vs applied stress at 1100, 1300 and 1500°F are presented in Fig. 12. These studies indicate that as-welded specimens possess stress-rupture strengths equivalent to those of the base metal at all test temperatures. Stress relieving, using a hydrogen atmosphere, created a significant improvement in the creep properties of samples tested at 1300°F. A factor of two improvement was noted in both the time-to-rupture and the total strain properties when this treatment was employed (see Table 4). The creep-rupture tests on stress-relieved samples at 1100 and 1500°F showed little or no improvement over as-welded properties. The curves of Fig. 13 illustrate the stress-temperature relationships necessary to cause rupture in 10, 100 and 500 hr for these composite specimens. The improvement over the as-welded strength at 1300°F by stress relieving in hydrogen at 1600°F for 2 hr is vividly shown in this figure.

The minimum creep rate for tests on as-welded and stress-relieved specimens, shown in Figs. 14 and 15, respectively, was generally observed

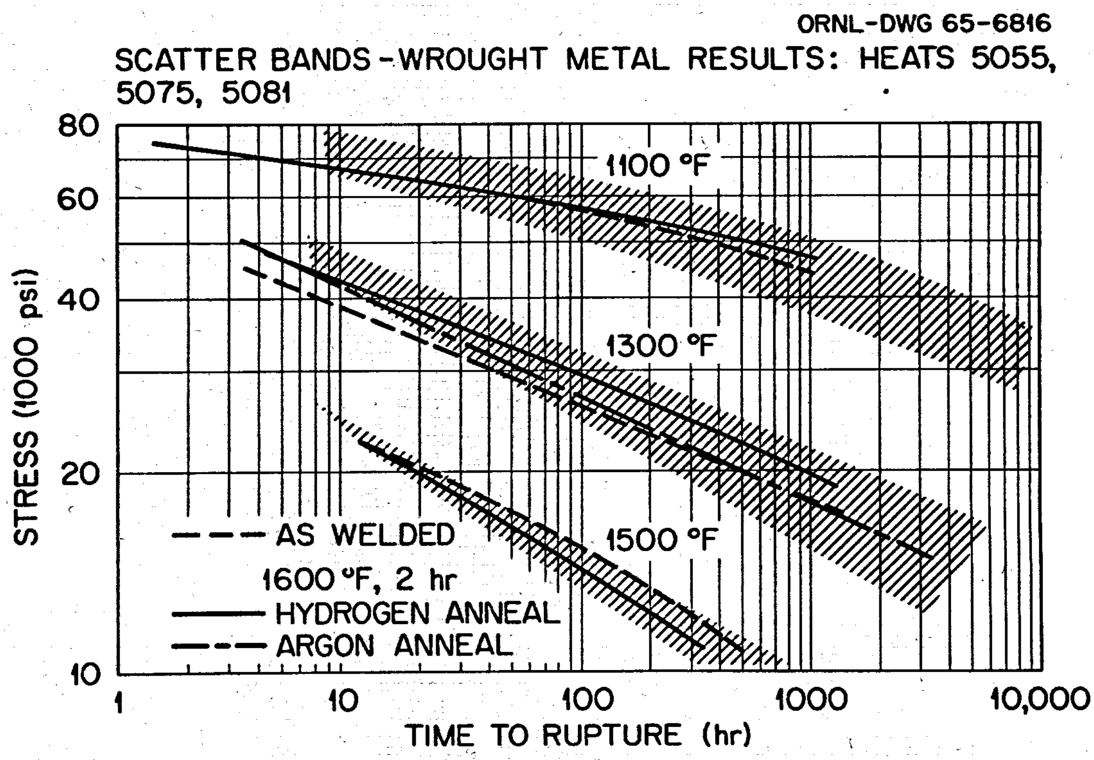

Fig. 12. Average creep-rupture data for transverse weld specimens of MSRE INOR-8 in the as-welded and argon and hydrogen stress-relieved condition.

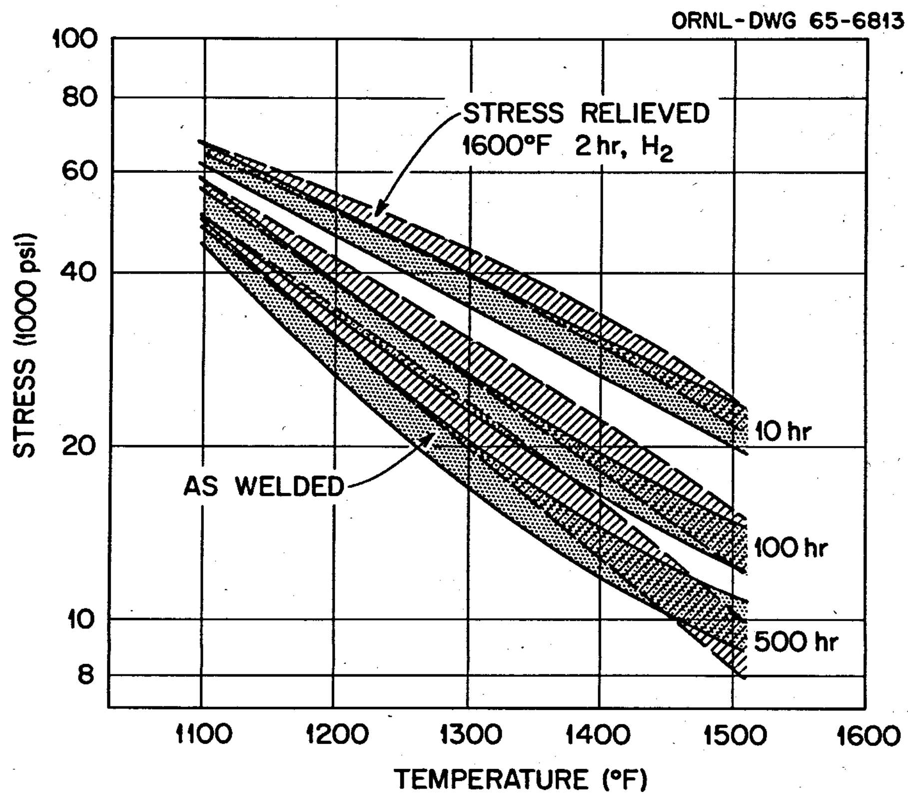

Fig. 13 — Creep-rupture data for transverse weld specimens of MSRE INOR-8 in the as-welded and hydrogen stress-relieved conditions.

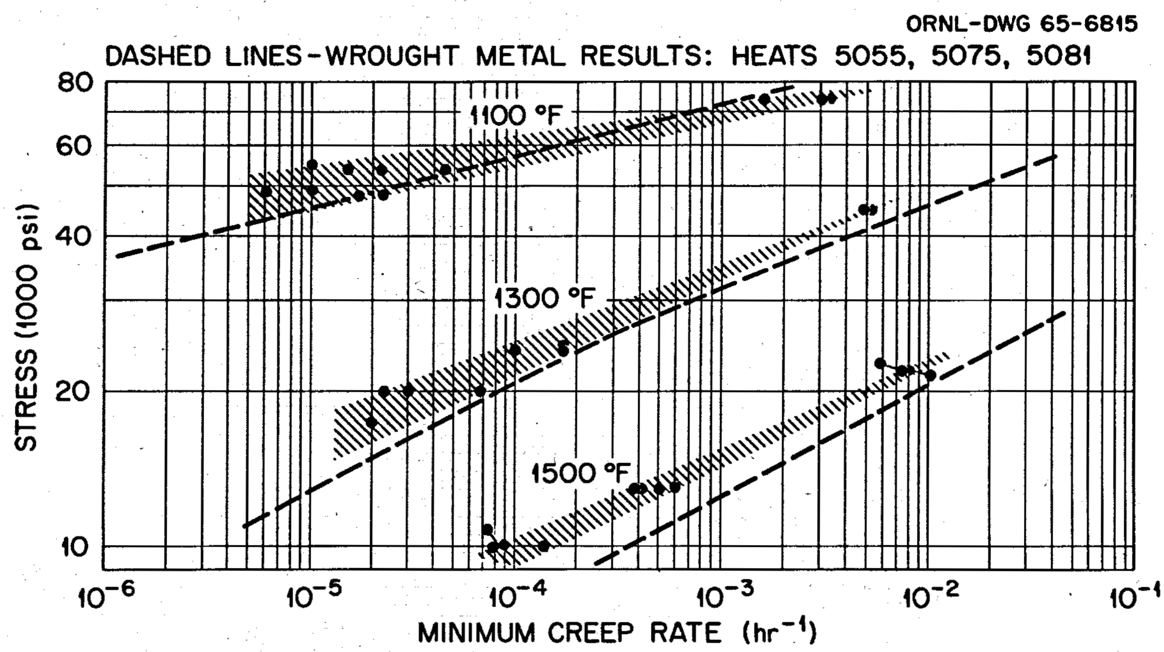

Fig. 14 – Minimum creep-rate data for as-welded transverse weld specimens in MSRE INOR-8 heats 5060, 5069, 5075 and 5083.

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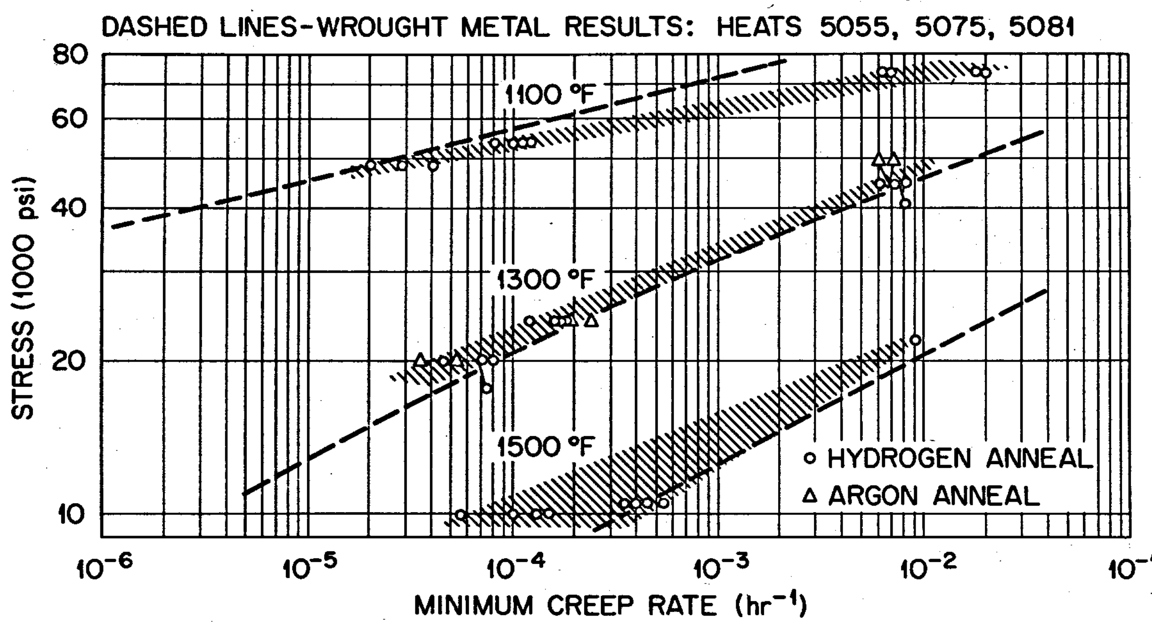

Fig. 15 — Minimum creep-rate data for stress-relieved transverse weld specimens in MSRE INOR-8. Heats 5057, 5067, 5072 and 5083, annealed in hydrogen, and heats 5068 and 5074, annealed in argon, were held for 2 hr at 1600°F.

to be slightly lower than that found for wrought metal at all test temperatures and stresses. One exception to this general observation was noted for stress-relieved specimens tested at 1100°F.

Metallographic examination of several creep-rupture specimens revealed that stress relieving had the general effect of shifting the rupture location from the base metal to the weld metal. The fracture of the as-welded specimen in Fig. 16 shows a typical intergranular failure in base metal, with associated grain-boundary cracking normal to the applied stress. The appearance of the base metal cracks is very similar to those discussed above for the short-time tensile tests. The weld metal failure of Fig. 16 is typical of creep test fractures observed in stress-relieved specimens. Some fractures of specimens in this condition were observed to occur along the weld fusion line.

Hot Ductility Tests

Hot ductility experiments using synthetic heat-affected zone specimens revealed the elevated-temperature nil-ductility point for this material to be 2300°F as shown in Fig. 17. The area of the heat-affected zone which would experience this maximum peak temperature corresponds to a plane calculated to be 0.032 in. from the weld fusion line. The curves of this figure indicate that reasonable recovery of the mechanical properties after heating to the nil-ductility temperature was exhibited, although some damage occurred as a result of the welding thermal cycle. Since no attempt was made to identify each sample with a heat number, the observed behavior is considered to be typical of MSRE reactor grade INOR-8.

As shown in Fig. 18, no gross grain boundary liquation has occurred; however, numerous individual precipitate particles are present which retard

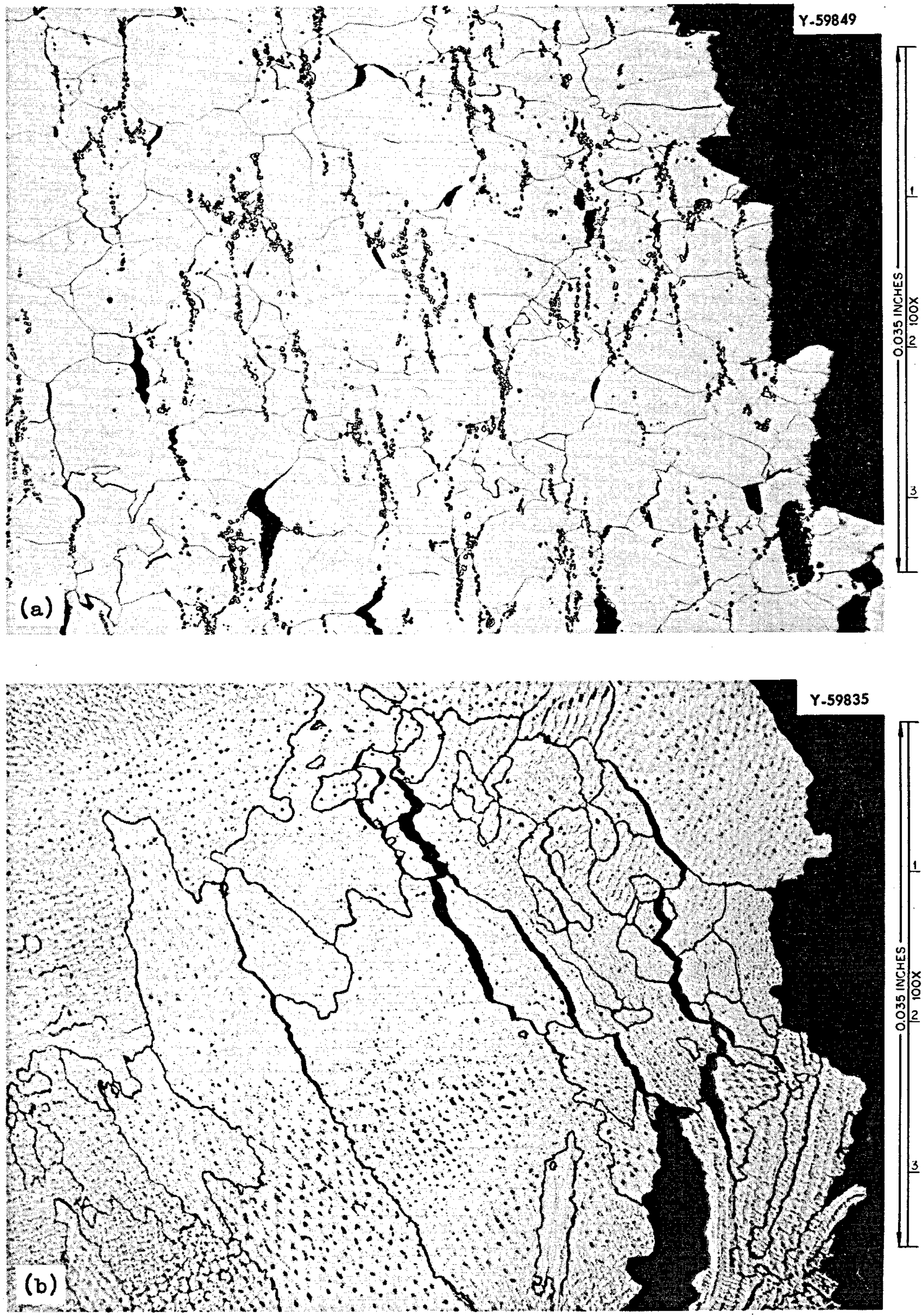

Fig. 16 — Creep-rupture failures of MSRE INOR-8 transverse weld specimens. A. Base metal failure in heat 5075, tested at 1100°F; applied stress 54,000 psi, rupture time 53.0 hr, total strain 1.4%. B. Weld metal failure in heat 5072, tested at 1300°F; applied stress 45,000 psi, rupture time 7.2 hr, total strain 7.5%. Etchant: CrO_3, HCl, H_2O. 100x.

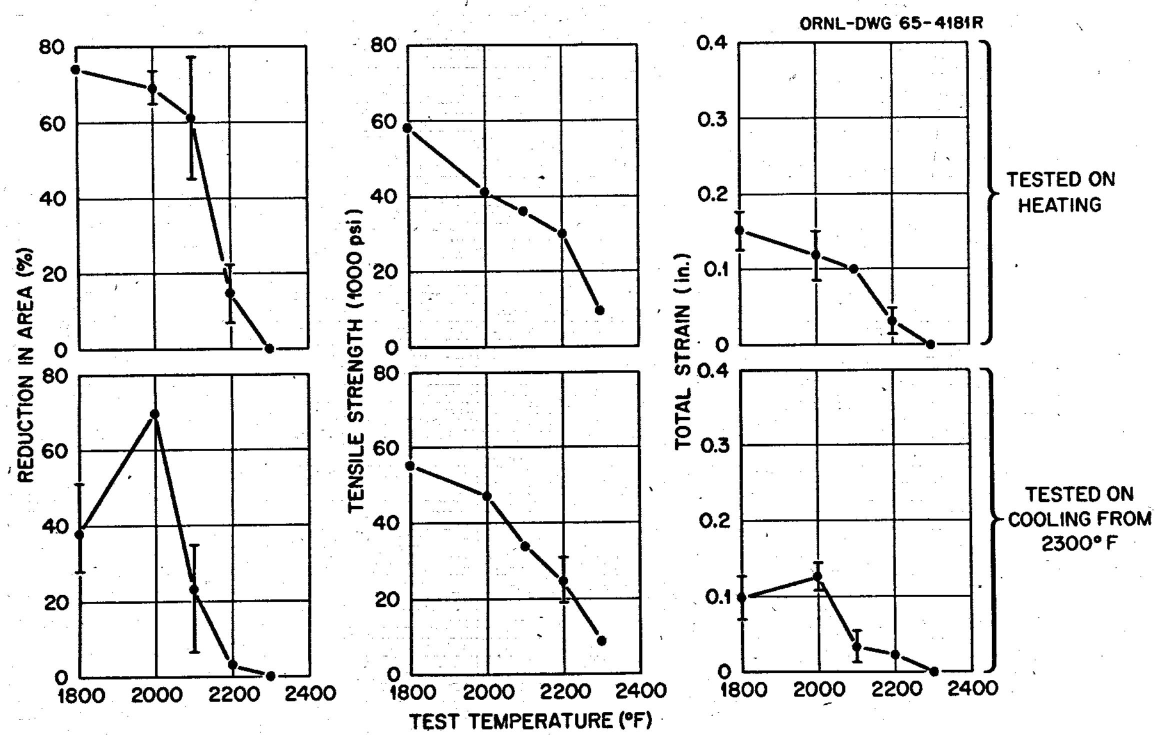

Fig. 17 — Results of hot-ductility tests on MSRE reactor-grade INOR-8. The nil-ductility temperature for the material was found to be 2300°F.

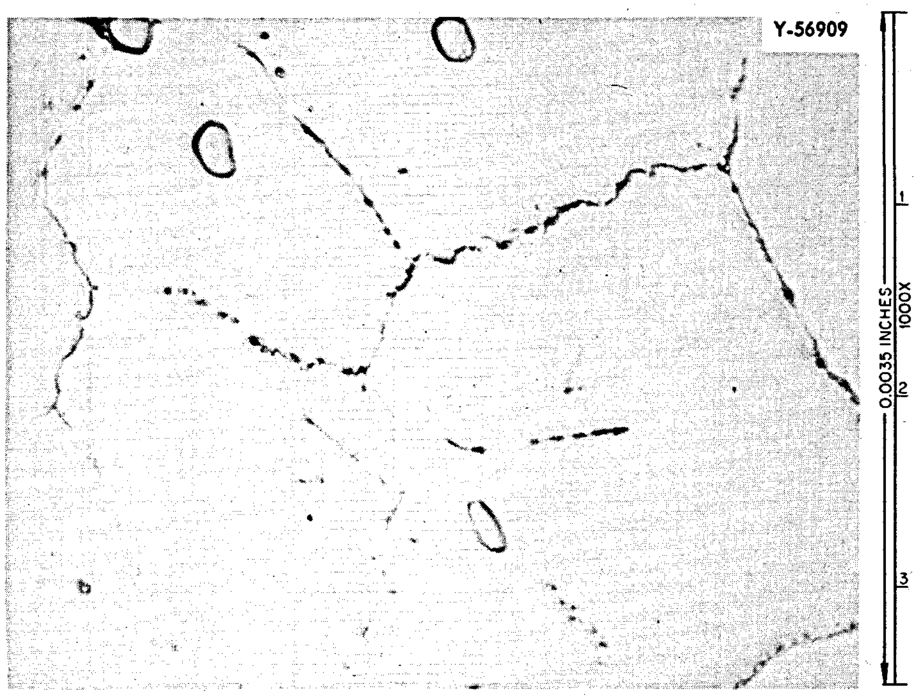

Fig. 18 — Micrograph of the structure of MSRE INOR-8 which has experienced a welding thermal cycle with a peak temperature of 2300°F (NDT). Etchant: H_3PO_4, H_2O. 1000X.

the motion of the grain boundaries and cause them to be quite irregular. These boundaries etch rapidly and appear quite broad, indicating that, possibly, an impurity layer is present. Earlier studies have shown that by very rapid cooling from very high temperatures (2500°F), the precipitate or stringer particles appear to be converted to an intergranular lamellar product. Gross formation of this product throughout the heat-affected zone of INOR-8 welds could cause permanent damage, resulting in almost certain failure under high restraint. The hot ductility tests conducted here do not indicate that this condition is a serious problem for the material investigated.

CONCLUSIONS

It appears from this study that the room- and elevated-temperature mechanical properties of welds in INOR-8 compare favorably with the base metal properties. Tensile testing of these transverse specimens showed that this material possessed a good combination of strength and ductility. Stress relieving produced the general effect of lowering the 0.2% offset yield strength. The creep studies of as-welded samples indicated that they possessed stress-rupture properties equivalent to those of base metal at all test temperatures. Stress relieving, using a hydrogen atmosphere, caused a significant improvement in the creep properties of samples tested at 1300°F. Heat-affected zone hot ductility experiments established the nil-ductility temperature for this material to be 2300°F, with reasonable mechanical property recovery after experiencing this temperature.

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