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## Postweld Heat Treatment Effects on Annealed 2 1/4 Cr-1 Mo Steel

R. L. Klueh

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R. L. Klueh

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# POSTWELD HEAT TREATMENT EFFECTS ON ANNEALED 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo STEEL\*

R. L. Klueh

## ABSTRACT

The tensile properties of four heats of 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel were determined over the range 25 to 566°C at strain rates of  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s after the heats were given a simulated postweld heat treatment (PWHT) for various times at 727°C. An annealed vacuum-arc-remelted (VAR) plate was tested after 0, 1, 5, 16, and 40 h at 727°C. An isothermally annealed electroslag-remelted (ESR) rod and an air-melted plate were tested after 0 and 40 h at 727°C. An isothermally annealed air-melted forging was tested after 0, 16, and 40 h at 727°C. The simulated PWHT decreased the yield strength (YS) and ultimate tensile strength (UTS) and increased the ductility of all heats. For the VAR plate the decrease in strength continued up to the 16-h exposure at 727°C; no further change occurred between 16 and 40 h. The VAR plate, the air-melted plate, and the ESR rod did not differ significantly in strength after 40 h at 727°C. For this same exposure, the air-melted forging was significantly stronger than the other three steels. After 40 h at 727°C, the room-temperature YS and UTS of all heats exceeded the 207- and 413-MPa minimum values required by ASME specifications. However, at elevated temperatures, all but the air-melted forging had a YS that fell below the expected minimum curve given in ASME Code Case N-47. The effect of PWHT on properties was explained in terms of precipitate strengthening.

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## INTRODUCTION

Since 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel is to be used as the structural material for liquid-metal fast breeder reactor (LMFBR) steam generators, substantial amounts of tensile<sup>1</sup> and creep<sup>2</sup> data have been collected on that steel. These data are being used for design studies and for ASME Code Case development. The steel investigated in those studies was in the annealed or isothermally annealed condition, since those were the heat treatment conditions established for the demonstration LMFBR—the Clinch River Breeder Reactor (CRBR). ASME Code Case N-47 allowable stresses used in the design with 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel were established on the basis of mechanical properties from steel given those heat treatments.

In the construction of a steam generator, there are a large number of 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel welds—especially tube-to-tubesheet welds. These welds will receive a postweld heat treatment (PWHT). For the CRBR, a PWHT temperature of 727°C was chosen.

During the PWHT, the strength of the weld metal and heat-affected zone (HAZ) will change. Since it is impossible to localize the PWHT to the weld metal and HAZ, the strength of the base metal heated during PWHT will also be affected. The 727°C PWHT temperature is quite high. Hence large mechanical property changes could occur, depending on the duration of the PWHT. It is conceivable that for annealed 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel the properties of the base metal could be reduced to values that approach the expected minimums given in Code Case N-47.

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\*Work performed under AF 15 10 15, Task OR-1.3, Mechanical Properties Design Data. Data reported herein represent information required in accordance with Clinch River Breeder Reactor DRS 53.40, Table IV.

To examine the effect of PWHT on base metal, we conducted tensile tests on  $2\frac{1}{4}$  Cr-1 Mo steel that was prototypic of material for the CRBR steam generators. These  $2\frac{1}{4}$  Cr-1 Mo steels are:

- Annealed vacuum-arc-remelted (VAR) steel—the melting process to be used for tubesheets of the CRBR steam generators.
- Isothermally annealed electroslog-remelted (ESR) steel—the melting process to be used for tubing in the CRBR steam generators.
- Annealed air-melted steel—the steel to be used for shell plate and other externals of the CRBR steam generators.

ASME Pressure Vessel and Piping Codes for Section III, Division 1, Class 1, require that a ferritic material be requalified after a PWHT. Although most welded components for the CRBR steam generators are expected to be given a PWHT of much less than 40 h, the 40-h time was chosen for requalification by the CRBR steam generator manufacturer (Rockwell International) to allow a safety margin to account for additional PWHT time over and above the shorter PWHT time to be used. Additional PWHT time can be required because of repair welds and multiple heat treatments of a region caused by overlap during the heat treatment of adjacent welds. Another possibility of a prolonged PWHT occurs because the time necessary to bring a region to the PWHT temperature may vary within the region being heat treated. Since the PWHT time is measured from the time the entire heat-treated region reaches temperature, there can often be a range of times at temperature for different parts of the component being heat treated.

Although a 40-h PWHT allows for all foreseen possibilities during fabrication, it would be useful to have information on the effect of PWHT time. For that reason, we examined the tensile properties of the annealed VAR plate after various shorter PWHT times. Some previously acquired data on an air-melted tubesheet forging are also presented.

## EXPERIMENTAL

The mechanical properties were determined on VAR plate (heat 56447), air-melted plate (heat 20017), and ESR rod (heat 91775). The VAR steel billet was obtained from Cameron Iron Works and subsequently was rolled to 25.4-mm-thick plate by Republic Steel Corporation. The 25.4-mm-thick air-melted plate was obtained from Babcock and Wilcox Corporation. The ESR 25.4-mm-diam rod was obtained from General Electric Company. Prior to heat treating it was swaged to a 16-mm diameter. The chemical compositions of these steels are given in Table 1.

The VAR and air-melted plates were given a full anneal as follows: austenitized by heating for 1 h at 927°C; cooled at a rate not exceeding 56°C/h to 316°C; air-cooled. The plates were given a simulated 40-h PWHT at 727°C. Parts of the VAR plate were also given a 1-, 5-, and 16-h PWHT.

The ESR rod was isothermally annealed as follows: austenitized by heating for 0.5 h at 927°C; cooled at a rate of approximately 280°C/h to 718°C; held for 1.5 h at 718°C; air-cooled. It was finally given a simulated 40-h PWHT at 727°C, cooled to 427°C at 56°C/h, then air-cooled to room temperature.

Previously, we did detailed studies on a prototypic air-melted tube-sheet forging<sup>3</sup> (Table 1). We also made tensile studies on that material after a 16- and 40-h PWHT. Since those data have not been reported and are pertinent to this study, they will be reported here. The forging was a 0.64-m-diam by 0.30-m-thick cylinder isothermally annealed by the National Forge Company as follows: austenitize at 727°C for 20 h; furnace-cooled to 750°C in about 2 h; held for 3 h at 750°C; air-cooled. Parts of this forging were tested after a simulated PWHT of 16 and 40 h.

**Table 1. Chemical composition of heats of 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel tested**

Analysis	Heat	Chemical composition (wt %)						
		C	Mn	S	P	Si	Cr	Mo
<b>VAR plate [25 mm (1 in.) thick]</b>								
Vendor	56447	0.10	0.49	0.007	0.010	0.23	2.15	1.03
ORNL		0.092	0.57			0.36	2.2	0.89
<b>Air-melted plate [25 mm (1 in.) thick]</b>								
Vendor	20017	0.11	0.55	0.011	0.014	0.29	2.13	0.90
ORNL		0.135	0.57	0.016	0.012	0.37	2.2	0.92
<b>ESR rod [22 mm (0.88 in.) in diameter]</b>								
Vendor	91775	0.098	0.47	0.002	0.009	0.28	2.33	1.02
ORNL		0.082	0.50	0.004	0.010	0.36	2.2	0.89
<b>Air-melted forging</b>								
Vendor	NF60-8746	0.09	0.39	0.009	0.009	0.35	2.40	1.01
ORNL		0.102	0.47	0.008	0.007	0.37	2.40	1.10

Tensile tests were performed in air on a 44kN Instron test machine according to the requirements of ASTM E 8-69 and E 151-64. The specimens were heated in a three-zone resistance furnace with the temperature controlled to  $\pm 1^\circ\text{C}$  and less than a  $2^\circ\text{C}$  temperature variation along the specimen gage length. The test machine was operated at constant crosshead velocity, and the reported strain rates were determined from the crosshead velocity and the initial gage length. Tests were made at nominal strain rates of  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$  /s (crosshead speeds of  $8.5 \times 10^{-5}$  and  $2.1 \times 10^{-2}$  mm/s). Tensile specimens had a 6.35-mm-diam by 31.75-mm-long reduced section. Total elongation was determined from gage marks on the specimen, while uniform elongation was determined from the load deflection charts.

## RESULTS

### Microstructures

Although below the reported  $A_{c1}$  temperature of  $777^\circ\text{C}$ ,<sup>4</sup> the  $727^\circ\text{C}$  PWHT temperature is quite high. At this temperature, diffusion of all species is rapid; the carbide precipitation reactions that occur in 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel can rapidly approach equilibrium.<sup>5</sup> Evidence of these changes can be seen by optical metallography.

The ferrite grain sizes of the various steels are given in Table 2. There are minor differences for the VAR heat and the two air-melted heats. The ESR heat, on the other hand, has a very fine grain structure.

Examples of the microstructural changes that occur during PWHT are shown in Figs. 1 and 2 for the VAR steel. After the anneal heat treatment, the steel is primarily proeutectoid ferrite with about 20% bainite (the dark-etching constituent in Figs. 1 and 2). There is also evidence of small carbide precipitates in the proeutectoid ferrite.

**Table 2. Estimated grain sizes for 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel with various PWHTs**

Heat	PWHT time (h)	ASTM grain size No.
VAR plate (56447)	0 <sup>a</sup>	5-6
	1	5-6
	5	5-6
	16	5-6
	40	4-5
Air-melted plate (20017)	0 <sup>a</sup>	5-6
	40	5-6
ESR rod (91775)	0 <sup>b</sup>	7-8
	40	6-7
Air-melted forging (NF60-8746)	0 <sup>b</sup>	4-5
	16	4-5
	40	4-5

<sup>a</sup>Annealed but no PWHT.

<sup>b</sup>Isothermally annealed but no PWHT.

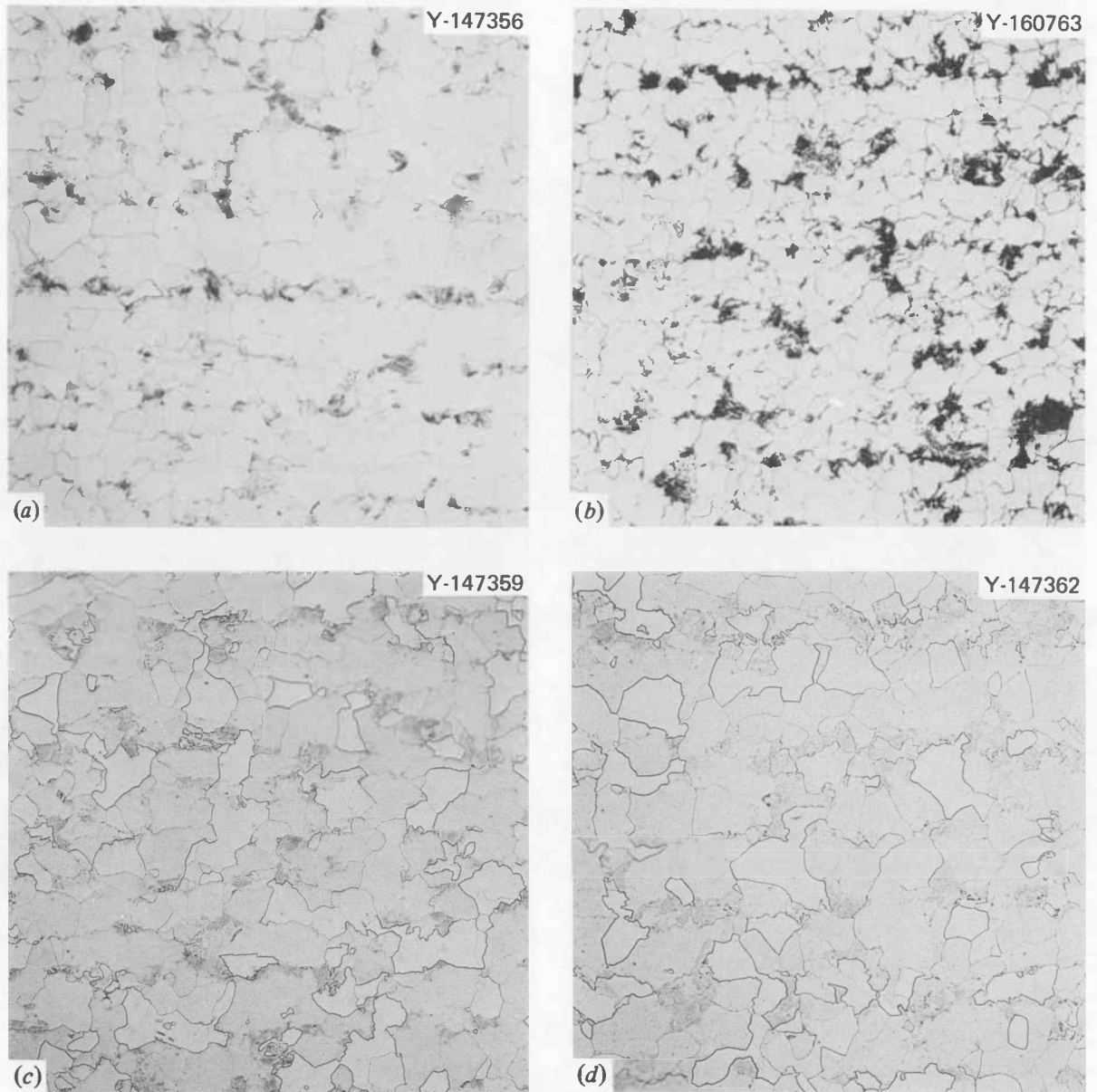
With time at 727°C, there is a general coarsening of the microstructure. The precipitates in the proeutectoid ferrite grow and are more easily resolved, while those that make up the former bainite regions become more diffuse. These changes are easily traced at a high magnification (Fig. 2).

Similar changes were detected for the air-melted plate and forging. The ESR steel showed a somewhat different microstructure, especially the grain size (Fig. 3). The grain size of the isothermally annealed steel was much finer than for the other steels [compare Figs. 1 and 3(a)]. Also, a much coarser precipitate morphology was present in the isothermally annealed ESR bar than was present after the anneal or isothermal anneal heat treatments for the plates and forging [compare Figs. 2(a) and 3(b)].

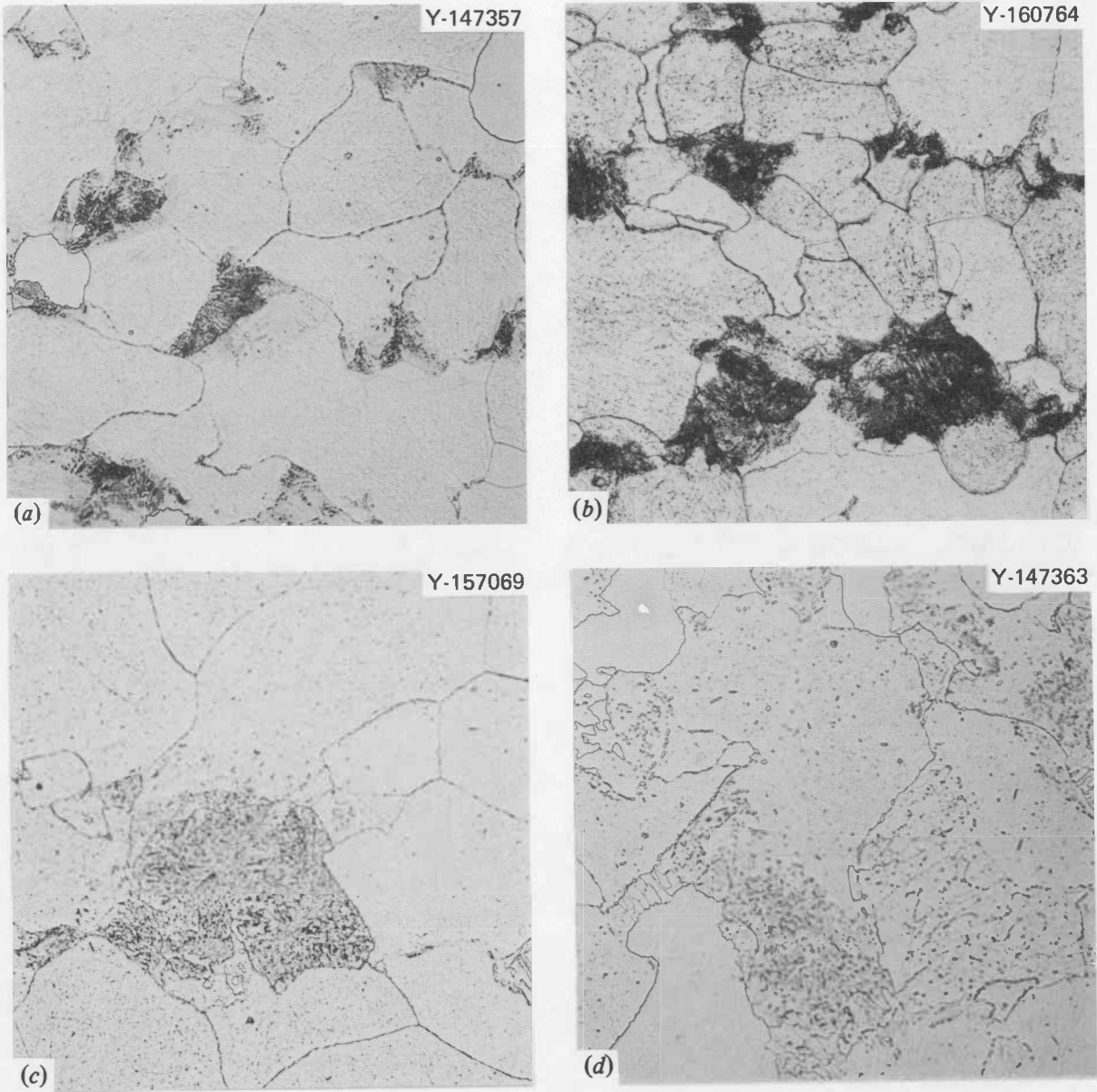
A still more unusual microstructure appeared after the 40-h PWHT [Fig. 3(c)]. There appeared to be small regions of bainite that formed during the 40-h PWHT. These regions were in bands along the direction of the rod, probably in the bands of bainite present in the isothermally annealed steel [Fig. 3(a)]. When these regions were examined at a higher magnification [Fig. 3(d)], they were found not to be bainite formed during the PWHT. That is, the elevated-temperature PWHT had coarsened the carbides in the bainite formed during the isothermal anneal.

### Tensile Behavior

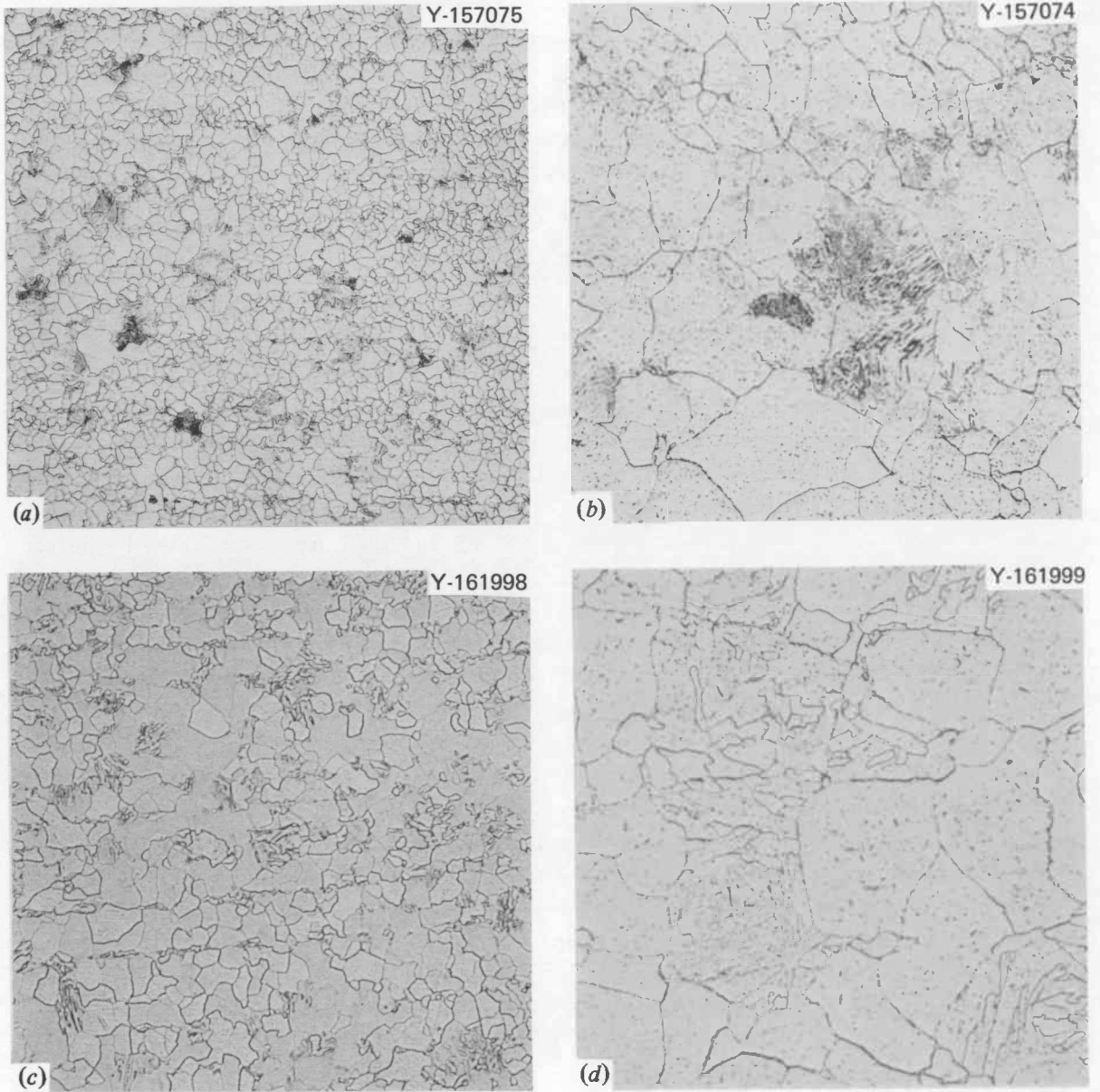
Tensile tests were made at strain rates of  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s for each of the heats of steel after the 40-h PWHT at 727°C. In addition, tests were made on the VAR plate (heat 56447) after a 0-, 1-, 5-, and 16-h PWHT at 727°C. The air-melted plate (heat 20017) was previously tested in the annealed condition<sup>1</sup> (no PWHT). We also previously did extensive testing of the air-melted forging (heat NF60-8746) in an isothermally annealed condition.<sup>3</sup> Parts of this forging were also given a 16- and 40-h PWHT, after which the tensile properties were again determined. The results for each of these heats will be examined separately, after which the properties for all heats will be compared.



**Fig. 1. Microstructures of VAR steel (a) annealed (A), (b) A + 1-h PWHT, (c) A + 16-h PWHT, (d) A + 40-h PWHT. 100 $\times$ .**



**Fig. 2.** Microstructures of VAR steel (a) annealed (A), (b) A + 1-h PWHT, (c) A + 16-h PWHT, (d) A + 40-h PWHT. 500 $\times$ .



**Fig. 3. Microstructures of ESR steel (a) and (b) isothermally annealed (IA), (c) and (d) IA + 40-h PWHT. 100 $\times$ .**

### VAR Plate (Heat 56447)

The most extensive testing was done on the VAR plate (Figs. 4-6). Regardless of the PWHT, the ultimate tensile strength (UTS)-temperature relationship displayed a dynamic strain-aging peak [Figs. 4(a), 5(a), and 6(a)]. For the tests at  $6.67 \times 10^{-4}$ /s [Fig. 4(a)] the effect of the PWHT was to reduce the height of this peak and move it to lower temperatures. After 1 h, the height of the peak was unchanged, but the peak had shifted from about 450 to 325°C. After the 5-h PWHT, the peak height in the UTS relationship was again slightly reduced. A substantial reduction in UTS peak height occurred after the 16-h PWHT. However, after 40 h, there was little further change. Thus, somewhere between 5 and 16 h there is a point where further PWHT has little effect on the UTS. Note also that the temperature at which the dynamic strain-aging peak occurs does not change significantly after the 1-h PWHT for the tests at  $6.7 \times 10^{-4}$ /s [Fig. 4(a)].

From 25 to 316°C for tests at  $6.67 \times 10^{-4}$ /s, the 1-h PWHT raised the 0.2% yield strength (YS) above that for the annealed steel with no PWHT [Fig. 4(a)]. Above 350°C the YS after the 1-h PWHT falls slightly below that for the annealed steel. Between 25 and 250°C the 5-h PWHT raised the YS above that of the annealed steel; at higher temperatures the YS falls below those for the annealed steel and the steel that received the 1-h PWHT. The 16- and 40-h PWHT data are essentially the same from 25 to 593°C. In all cases the YS for the steels with the 16- and the 40-h PWHT falls below those of the annealed steel. The differences increase as temperature increases. The YS of the steel with the 16- and the 40-h PWHT also falls below those with the 1- and the 5-h PWHT. Thus it appears that there is a time between 5 and 16 h at 727°C after which the YS and UTS are little affected by further PWHT.

Strain rate effects on annealed  $2\frac{1}{4}$  Cr-1 Mo steel have been studied in detail.<sup>2</sup> As the strain rate decreases, the dynamic strain-aging peak height in the UTS-temperature relationship increases and moves to lower temperatures.<sup>2</sup> This is easily seen for the annealed steel of this study by comparing the UTS data of Figs. 4(a) and 5(a). For the  $2.67 \times 10^{-6}$ /s tests, the peak height is about 525 MPa at 260°C and about 480 MPa at 455°C for a strain rate of  $6.67 \times 10^{-4}$ /s. A similar strain rate effect is noted for tests on a steel with any given PWHT. Figure 6 shows the behavior at  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s for the steel given the 40-h PWHT. Note, however, that although there was a slight increase in peak height and a decrease in the temperature of the peak for the steel with the 1- and the 5-h PWHT, the peaks occurred at higher temperatures for these two steels than for the annealed steel [Fig. 5(a)].

In addition to changes in the dynamic strain-aging behavior in the UTS-temperature relationship, strain rate also leads to significant changes at temperatures above the dynamic strain-aging peak. Here there is a decrease in strength with increasing temperature and decreasing strain rate. This loss of strength is extremely large for the steels given the 16- and the 40-h PWHT and tested at the low strain rate [Fig. 6(a)]. At 566°C for the steel with the 40-h PWHT, the UTS drops from 262 MPa to 156 MPa when the strain rate is decreased from  $6.67 \times 10^{-4}$ /s to  $2.67 \times 10^{-6}$ /s.

Strain rate affects the YS much less than the UTS. At room temperature and above about 400°C, there is a slight decrease in YS with decreasing strain rate.

Total elongation and reduction of area [Figs. 4(b), 5(b), and 6(b)] are quite large under all test conditions. The decrease in UTS with decreasing strain rate and increasing temperature is accompanied by large increases in total elongation and reduction of area. For the low-strain-rate tests, there is a rather large decrease in uniform elongation with increasing temperature.

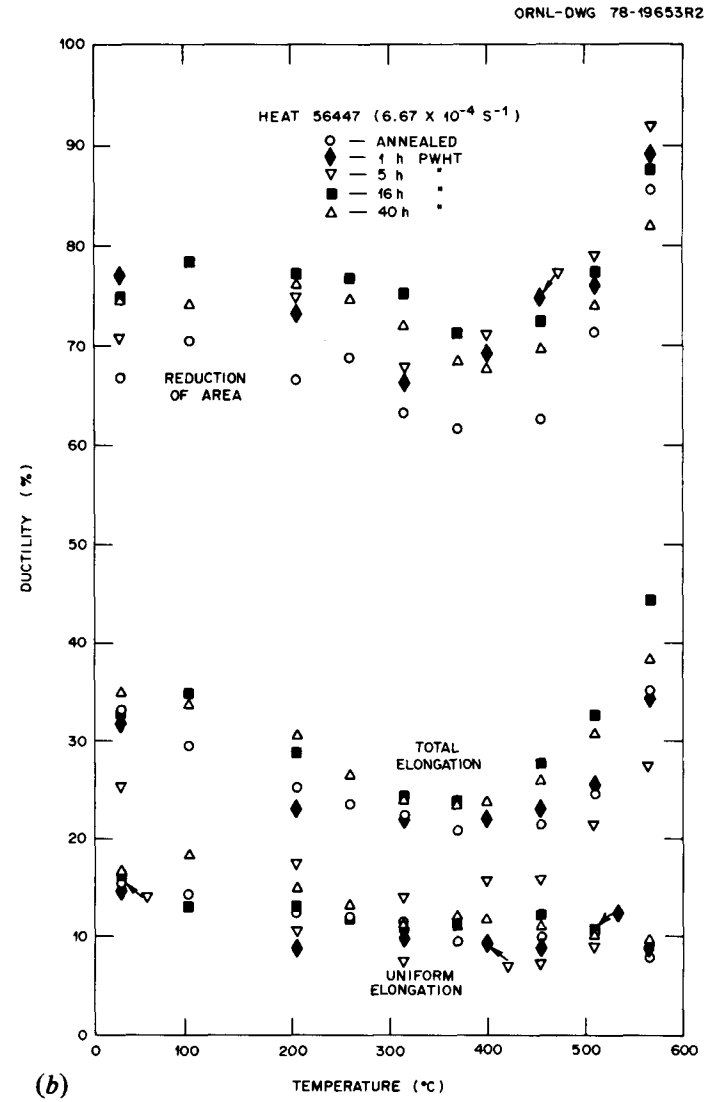
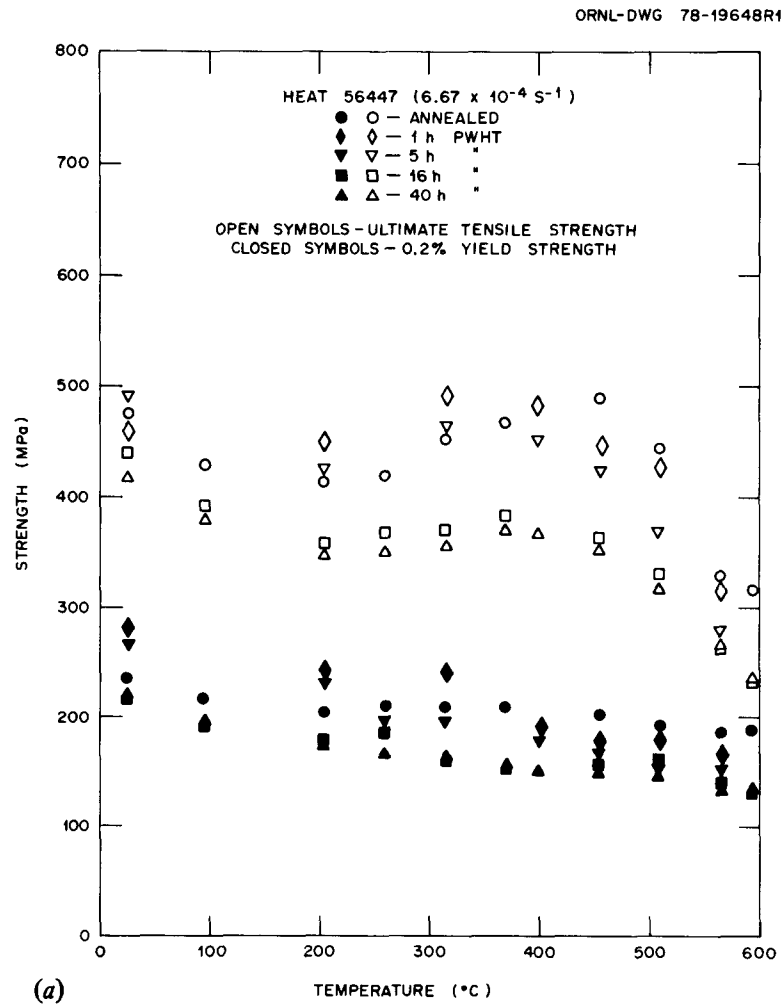


Fig. 4. Tensile properties at  $6.67 \times 10^{-4}$  /s as a function of temperature for VAR steel (heat 56447) with various PWHTs: (a) yield strength and ultimate tensile strength, (b) ductility.

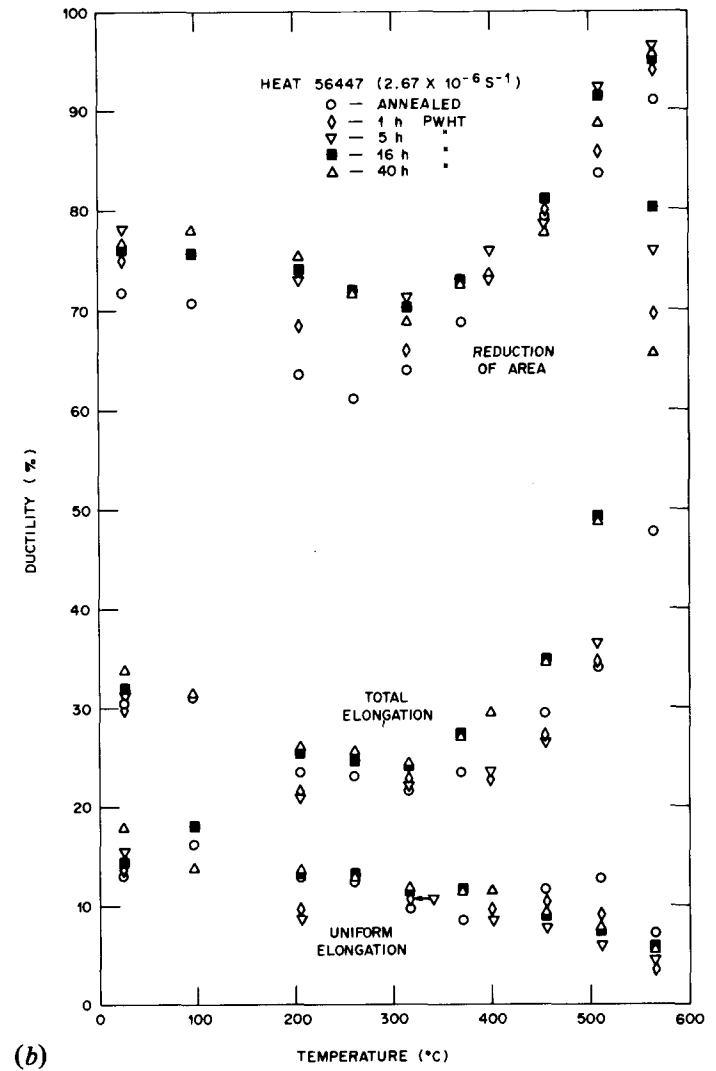
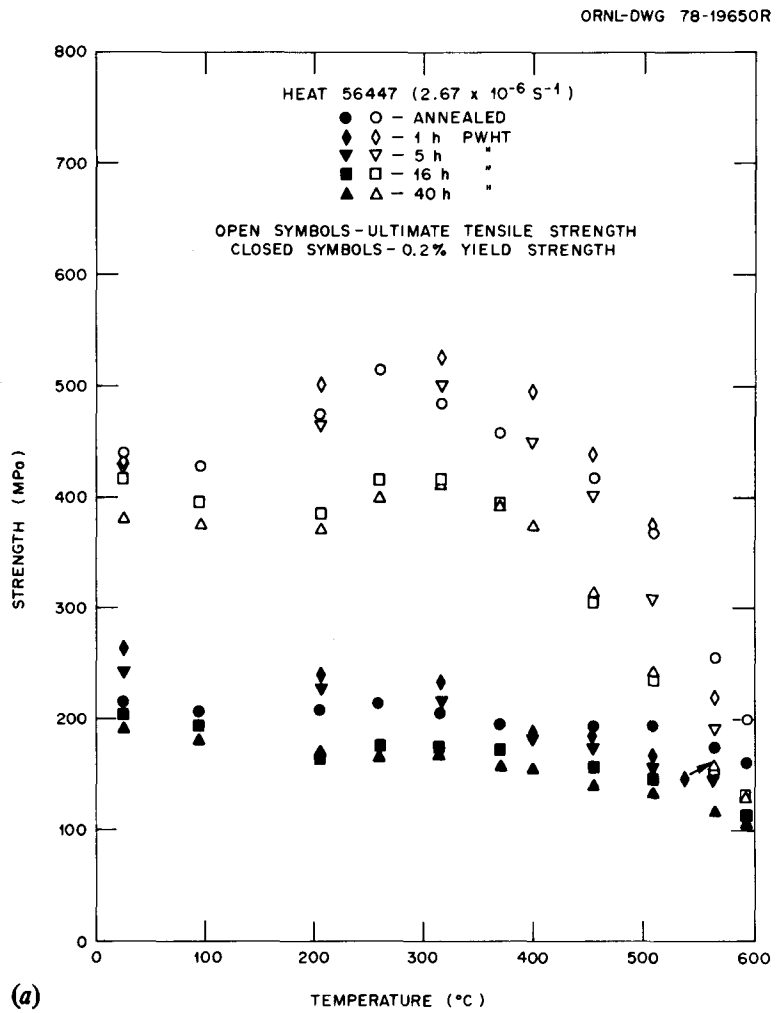
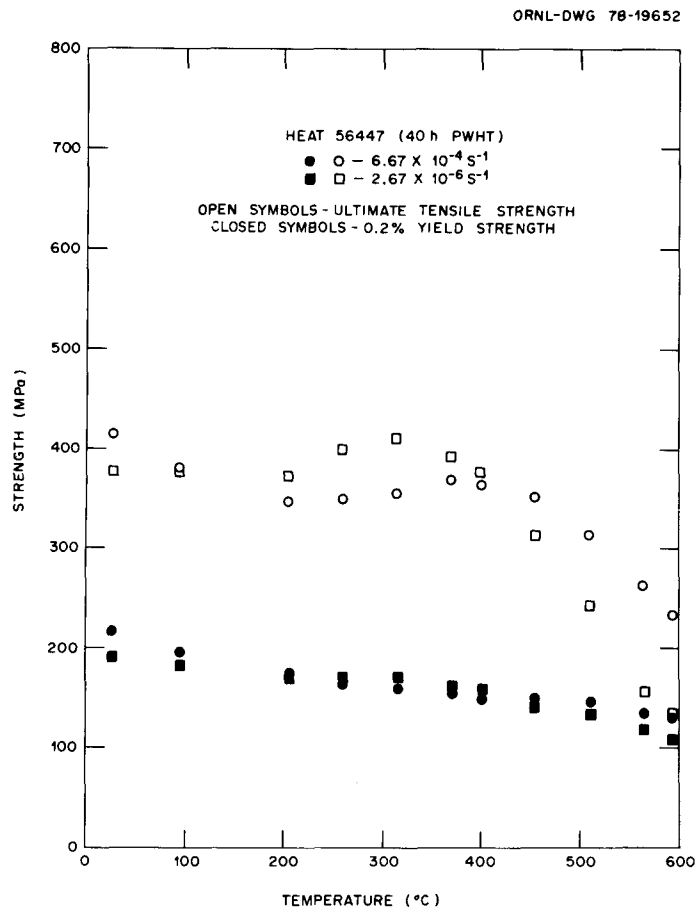
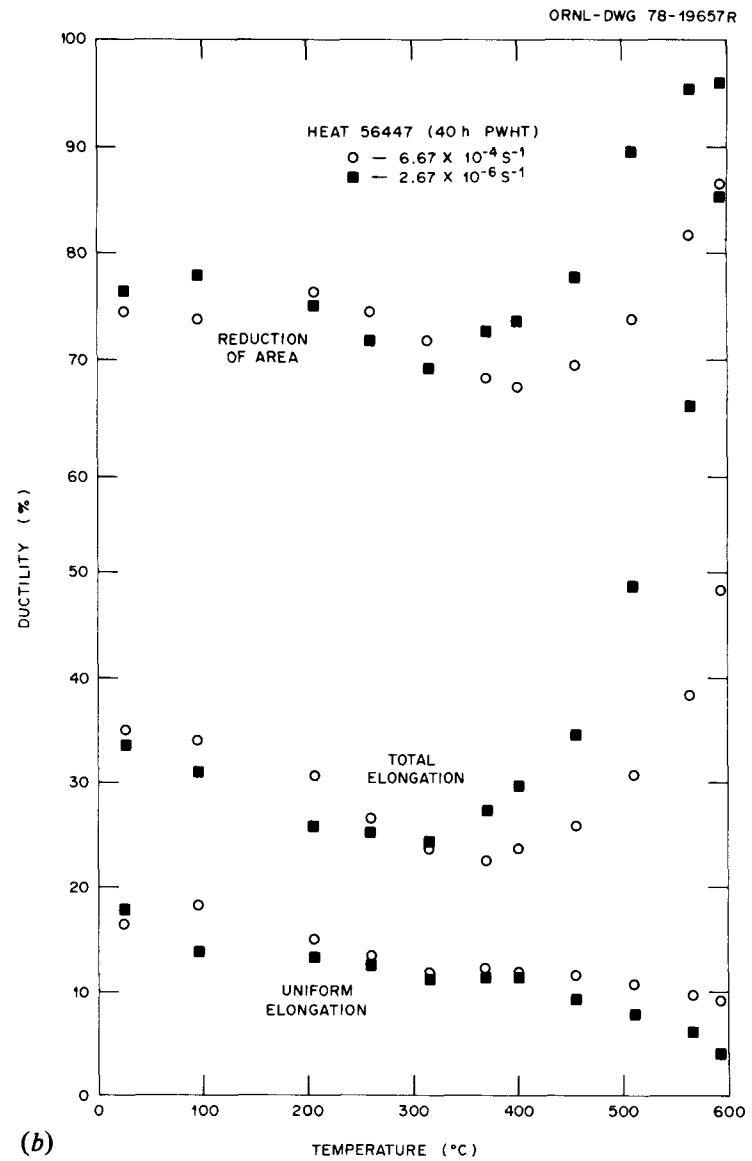


Fig. 5. Tensile properties at  $2.67 \times 10^{-6}$  /s as a function of temperature for VAR steel (heat 56447) with various PWHTs: (a) yield strength and ultimate tensile strength, (b) ductility.



(a)



(b)

Fig. 6. Tensile properties at  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$  /s as a function of temperature for VAR steel (heat 56447) annealed and given a 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

### Air-Melted Plate (Heat 20017)

We previously tested heat 20017 in an annealed condition.<sup>1</sup> In Figs. 7 (tests at  $6.67 \times 10^{-4}$ /s) and 8 (tests at  $2.67 \times 10^{-6}$ /s) these data are compared with those from the steel after a 40-h PWHT. In Fig. 8 the effect of strain rate on the steel with the 40-h PWHT is presented. As was true for the VAR heat, there was a considerable decrease in strength due to the 40-h PWHT. Likewise, the effect of strain rate was similar to that described for the VAR heat, as was the effect on ductility [Figs. 7(b), 8(b), and 9(b)].

### ESR Rod (Heat 91775)

The ESR steel was tested after the isothermal anneal and after the 40-h PWHT (Figs. 10–12). The changes with strain rate were similar to those observed for the VAR heat. One curious fact was that there was little difference in properties for the isothermally annealed steel and the one given the 40-h PWHT.

### Air-Melted Forging (NF60-8746)

We previously reported on the tensile properties of a typical air-melted  $2\frac{1}{4}$  Cr-1 Mo steel steam generator tubesheet forging.<sup>3</sup> Tests were made on the steel as received and after small pieces of it had been given an isothermal anneal in the laboratory. As received, the forging was in an isothermally annealed condition: austenitized 20 h at 927°C; furnace-cooled to 750°C, where it was held for 3 h; then air-cooled. Blanks from this forging were given the 16- and the 40-h PWHT and then tested.

We found that the microstructure of the as-received forging was nonuniform. The bainite content increased from approximately 10% at the external cylindrical surface to about 30% three-quarters of the way to the forging center. About 25% was present at the forging center. The variations in microstructure and strength with distance into the forging were concluded to be the result of nonuniform cooling of the forging. We concluded that a more uniform microstructure would result for such a large forging if it were normalized (air-cooled). The tensile properties reflected the microstructural changes and showed a maximum in the strength for steel taken at progressive intervals into the interior of the forging.

We gave the 16- and the 40-h PWHT to specimens taken different distances along the cylinder diameter and compared the tensile properties at 25 and 566°C with those for the as-received specimens taken from similar positions<sup>3</sup> (Fig. 13). For tests at 25°C, there was little effect of PWHT on YS, while the UTS showed a decrease and a small effect of PWHT time. After the 40-h PWHT, there was essentially no longer an effect of position on the room-temperature UTS.

Large changes in YS and UTS occurred for the PWHT steel tested at 566°C. In this case there was essentially no difference in the UTS for the steel given the 16- and the 40-h PWHT. However, as the center of the cylinder was approached, the YS after the 16-h PWHT was larger than that for the steel given the 40-h PWHT.

A series of specimens taken from near the center of the forging were tested in the range 25 to 566°C at  $6.67 \times 10^{-4}$ /s (Fig. 14). As was true for the other three heats, the YS and UTS after the 16- and the 40-h PWHT showed little difference [Fig. 14(a)]. Reduction-of-area values increased after the PWHT, but there was little change in total and uniform elongation; the uniform elongation decreased slightly at the elevated temperatures [Fig. 14(b)].

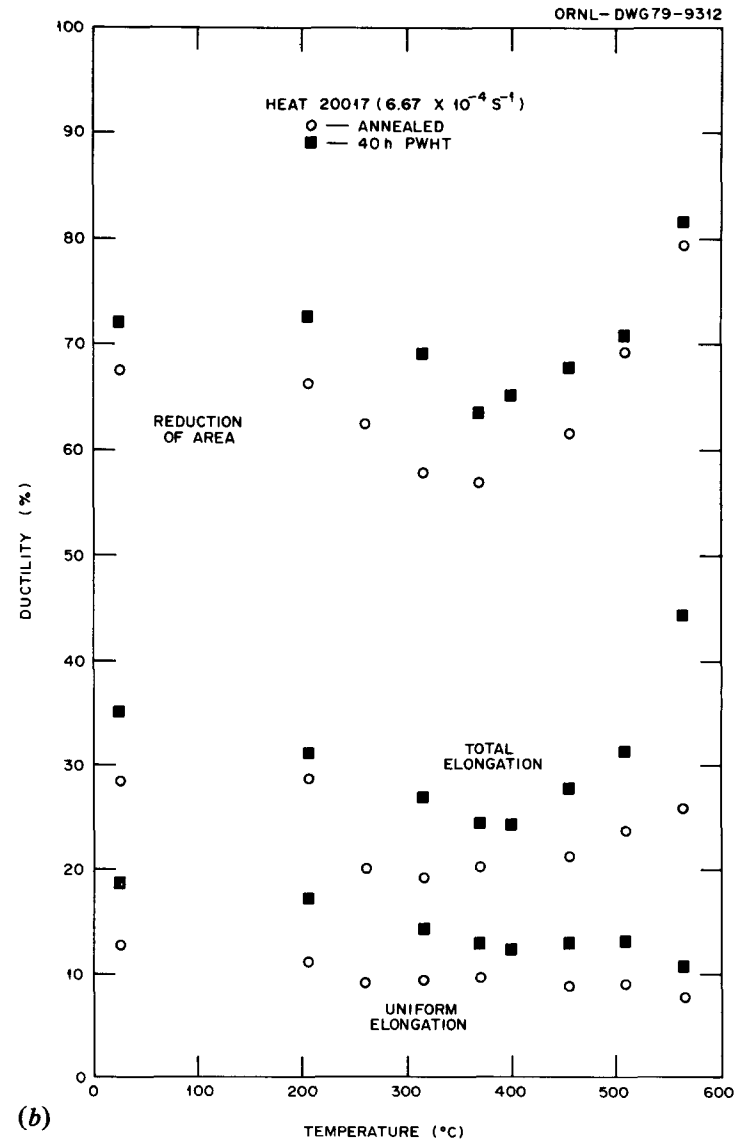
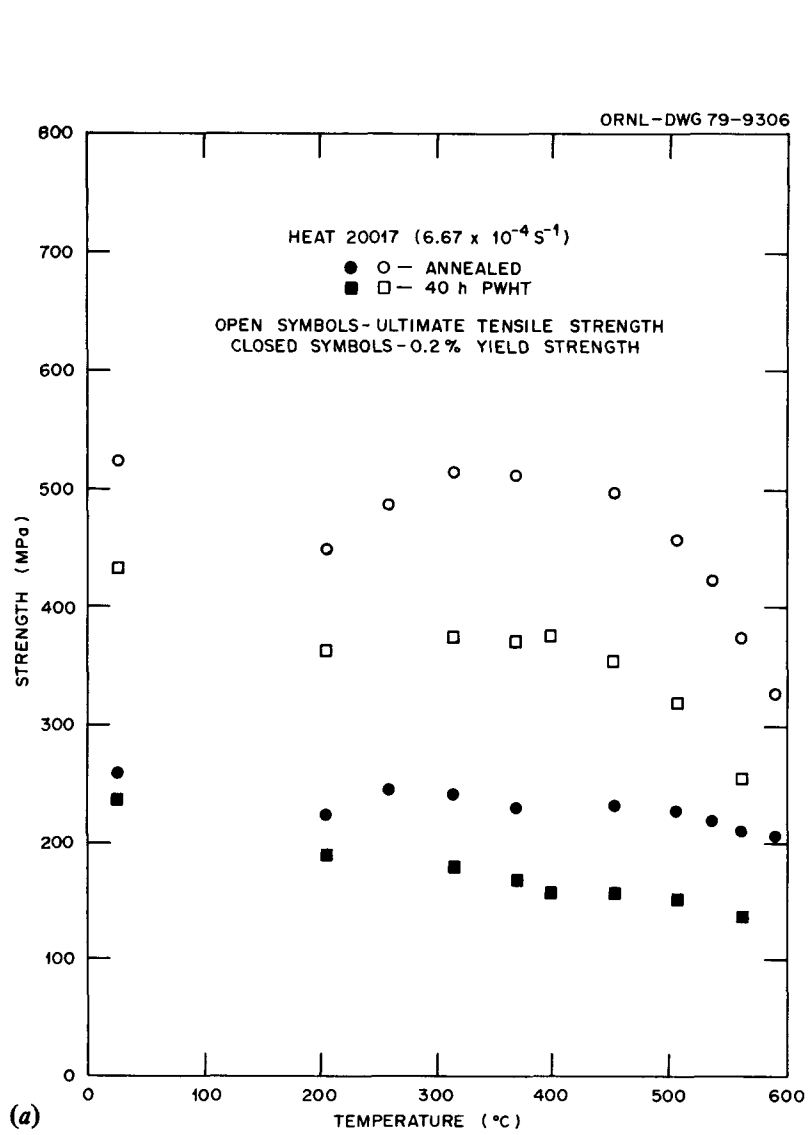


Fig. 7. Tensile properties at  $6.67 \times 10^{-4} \text{ s}^{-1}$  as a function of temperature for air-melted steel (heat 20017) as annealed and annealed plus 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

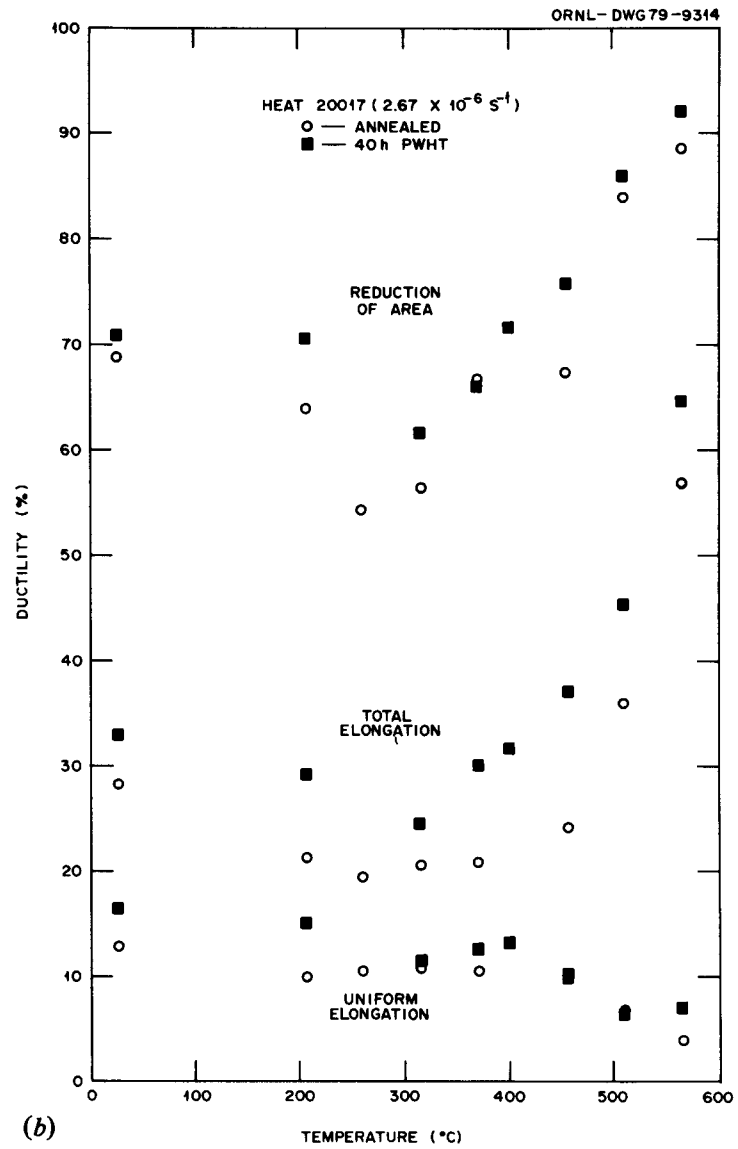
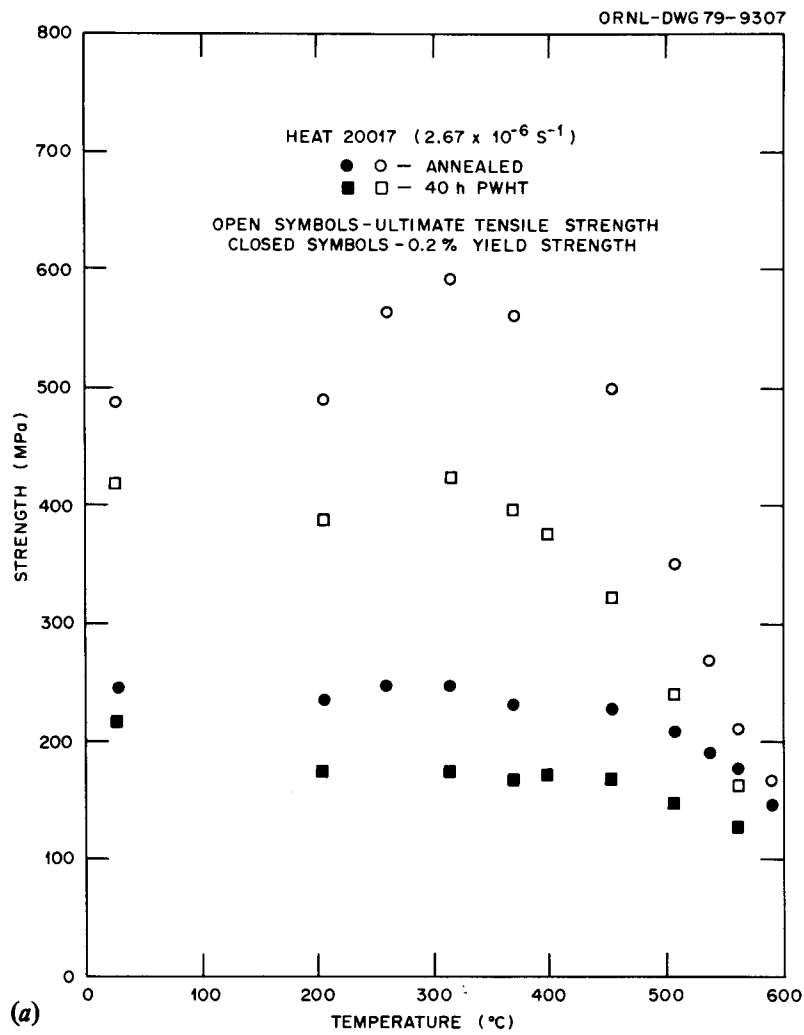
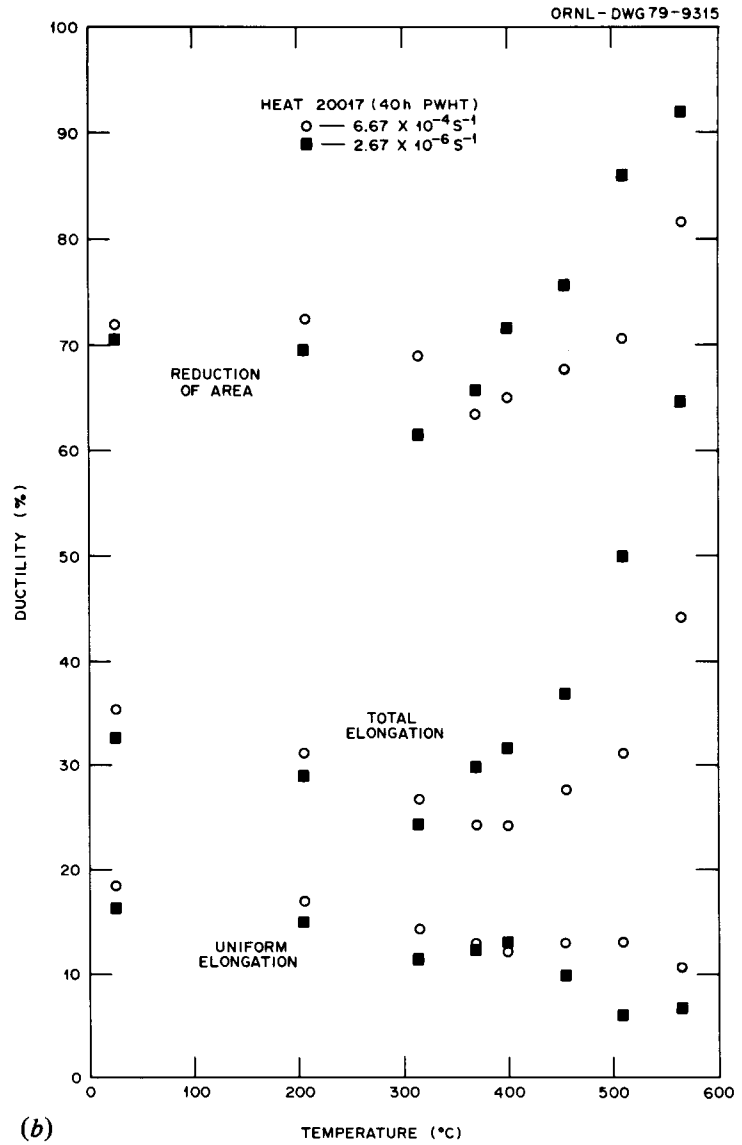
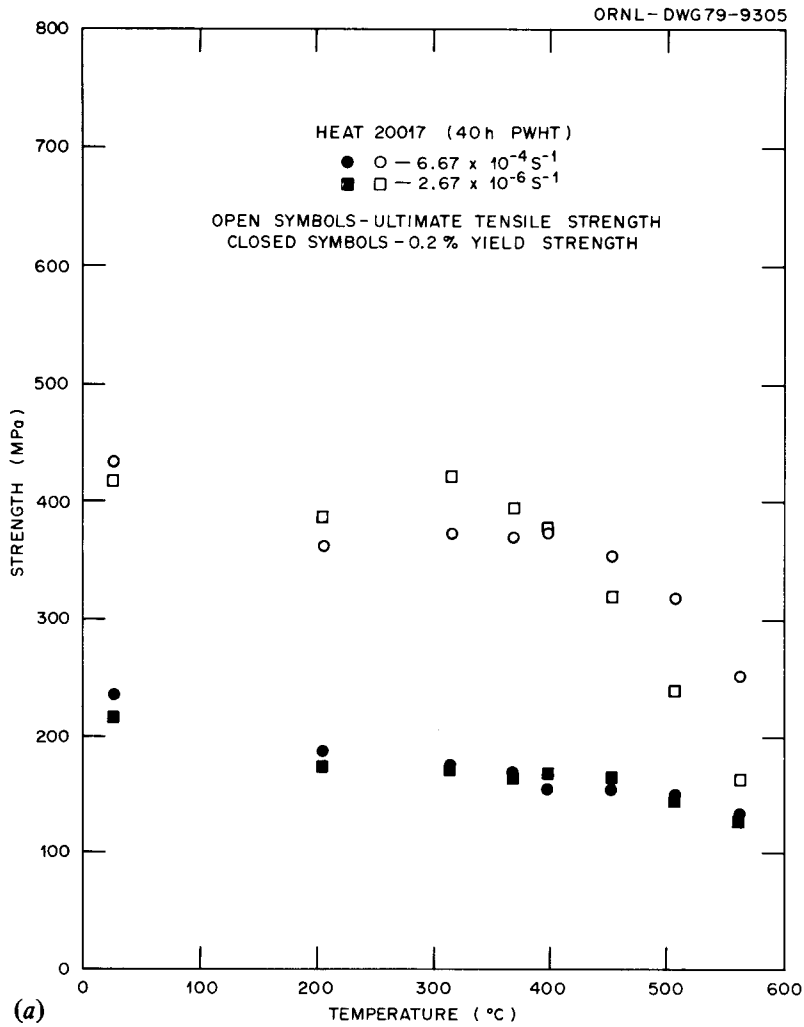


Fig. 8. Tensile properties at  $2.67 \times 10^{-6} \text{ s}^{-1}$  as a function of temperature for air-melted steel (heat 20018) as annealed and annealed plus 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.



**Fig. 9. Tensile properties at  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s as a function of temperature for air-melted steel (heat 20017) annealed plus 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.**

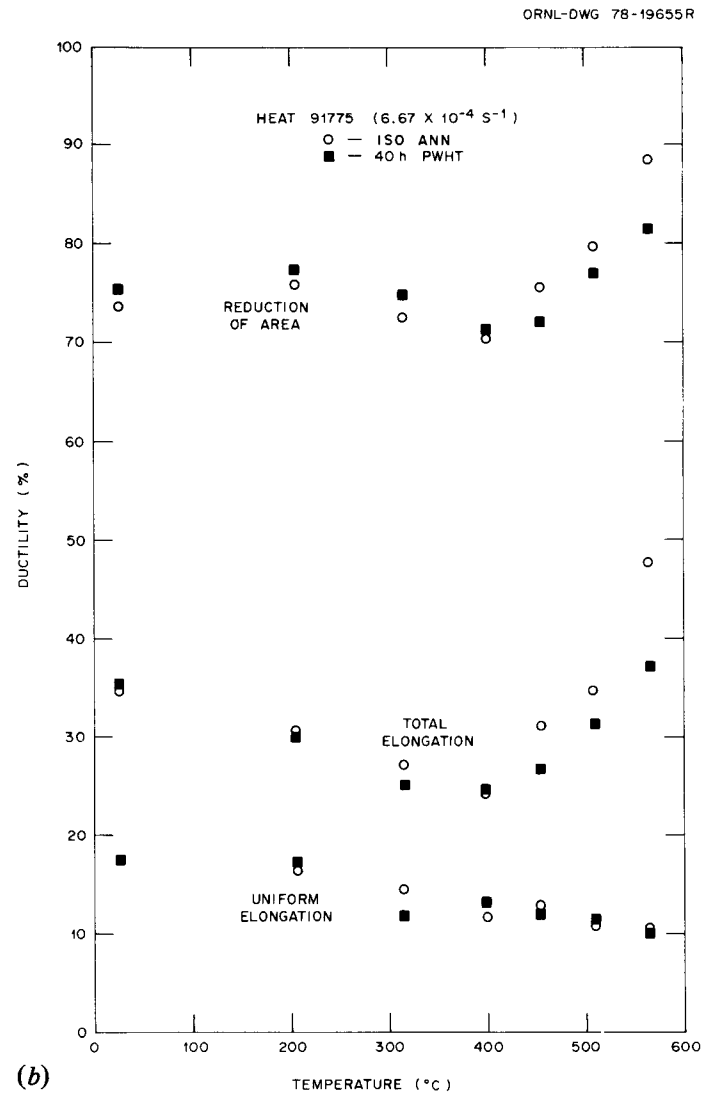
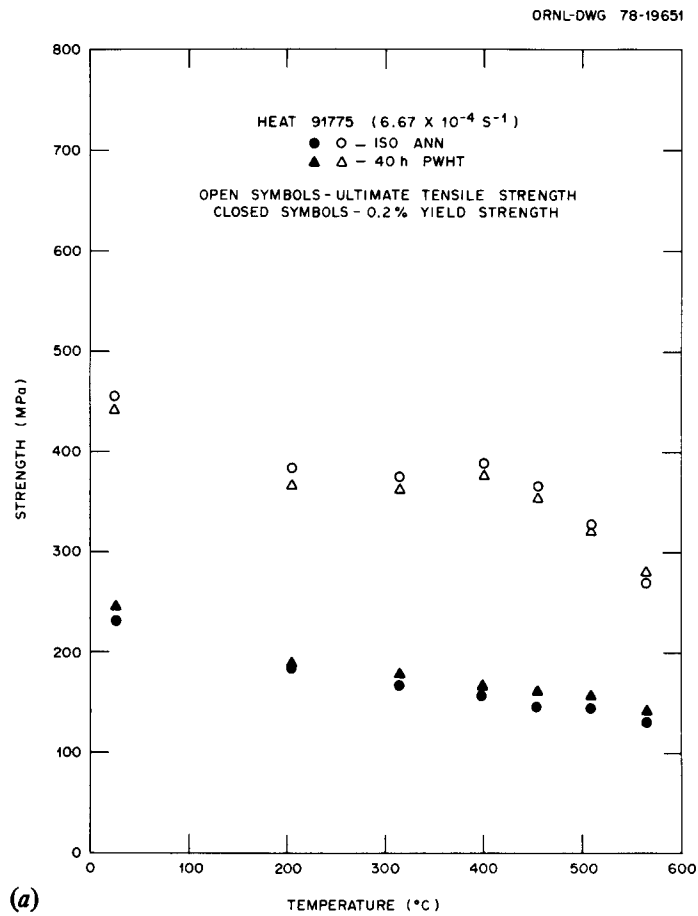


Fig. 10. Tensile properties at  $6.67 \times 10^{-4} \text{ s}^{-1}$  as a function of temperature for ESR steel (heat 91775) as isothermally annealed and isothermally annealed plus 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

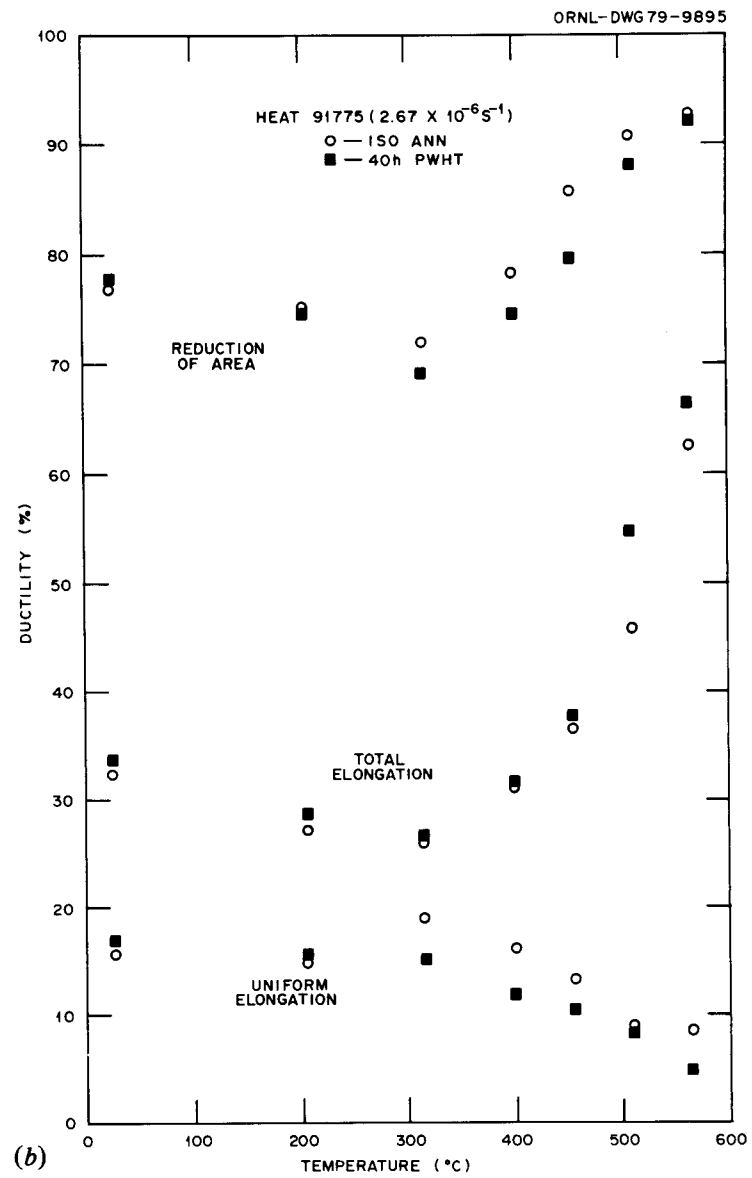
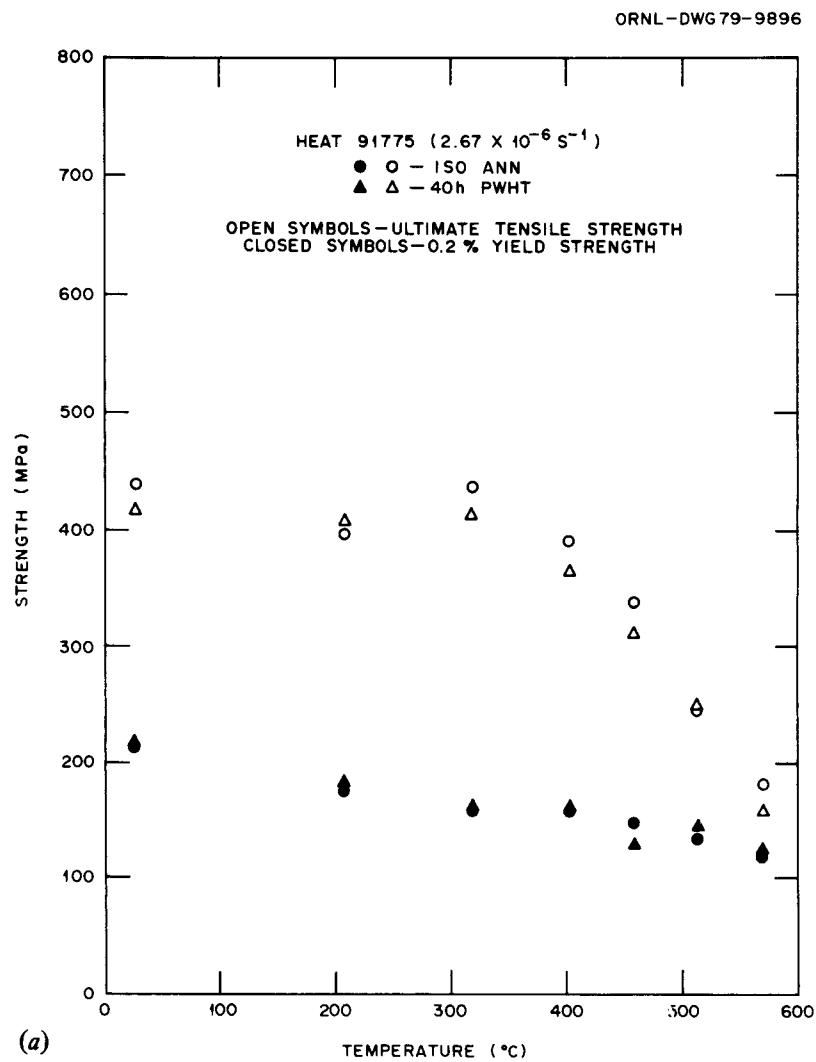


Fig. 11. Tensile properties at  $2.67 \times 10^{-6} \text{ s}$  as a function of temperature for ESR steel (heat 91775) as isothermally annealed and isothermally annealed plus 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

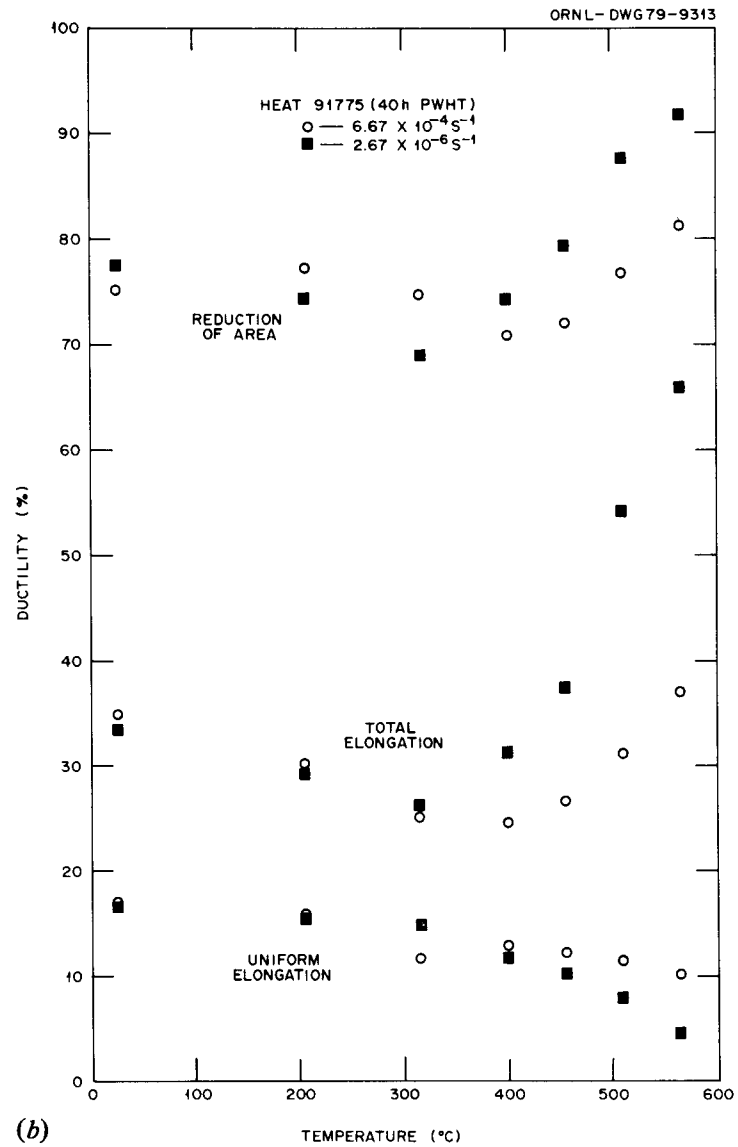
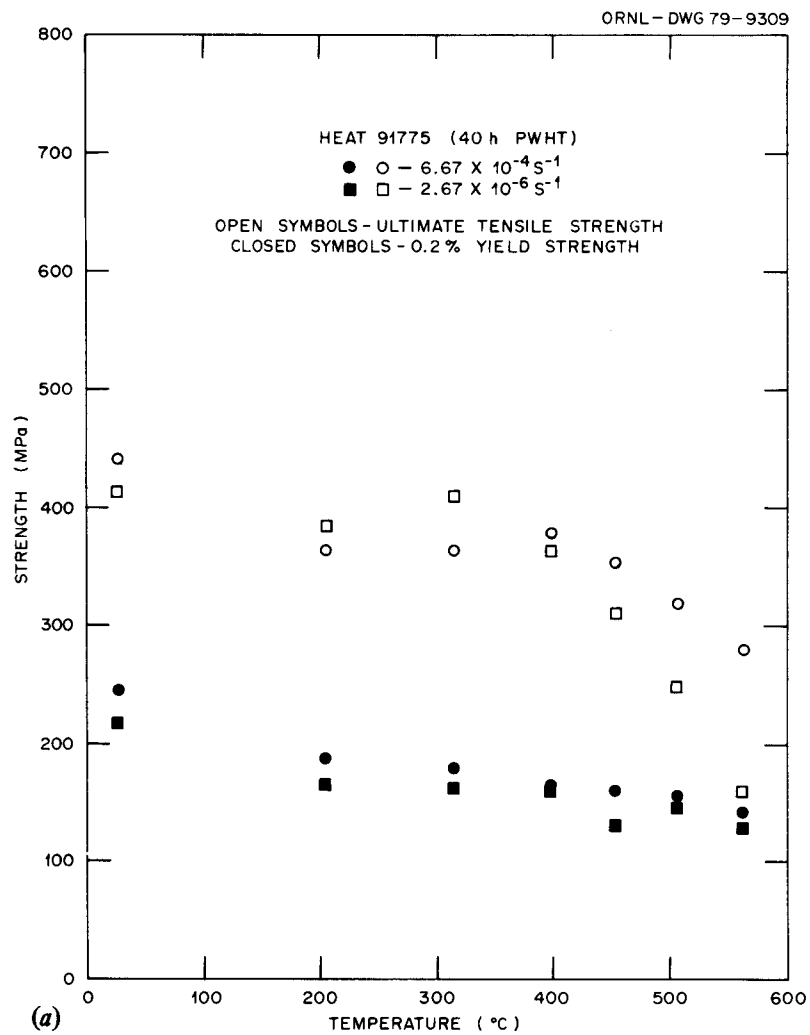


Fig. 12. Tensile properties at  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s as a function of temperature for ESR steel (heat 91775) as isothermally annealed plus 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

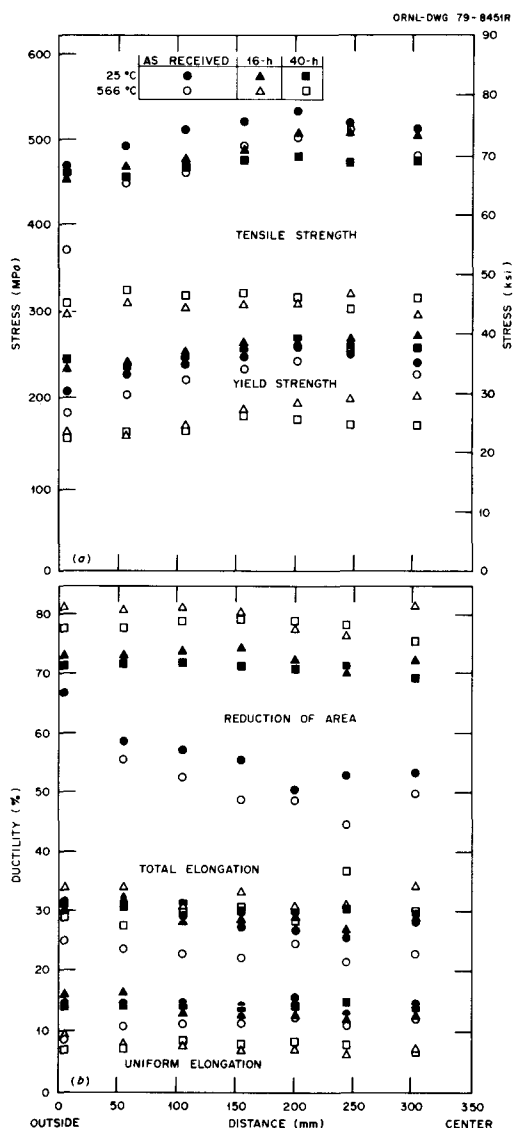


Fig. 13. Tensile property variation of air-melted forging (heat NF 60-8746) along radius as isothermally annealed and after isothermally annealed plus 16- and 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

### Comparative Tensile Properties

For all four heats, we obtained data on steel given a 40-h PWHT. In Fig. 15(a) the YS and UTS are compared for tests at  $6.67 \times 10^{-4}$ /s. There is essentially no difference in the VAR (heat 56447) and air-melted (heat 20017) plates and the ESR rod (heat 91775). However, the air-melted forging is considerably stronger after the 40-h PWHT. This strength advantage persists over the entire temperature range. Ductility for the four heats shows little difference over the range 25 to 566° C [Fig. 15(b)].

### DISCUSSION

Since one of the objectives of this work was to determine whether the  $2\frac{1}{4}$  Cr-1 Mo steel with a 40-h PWHT still qualified according to Code Case N-47, we have compared the properties with the

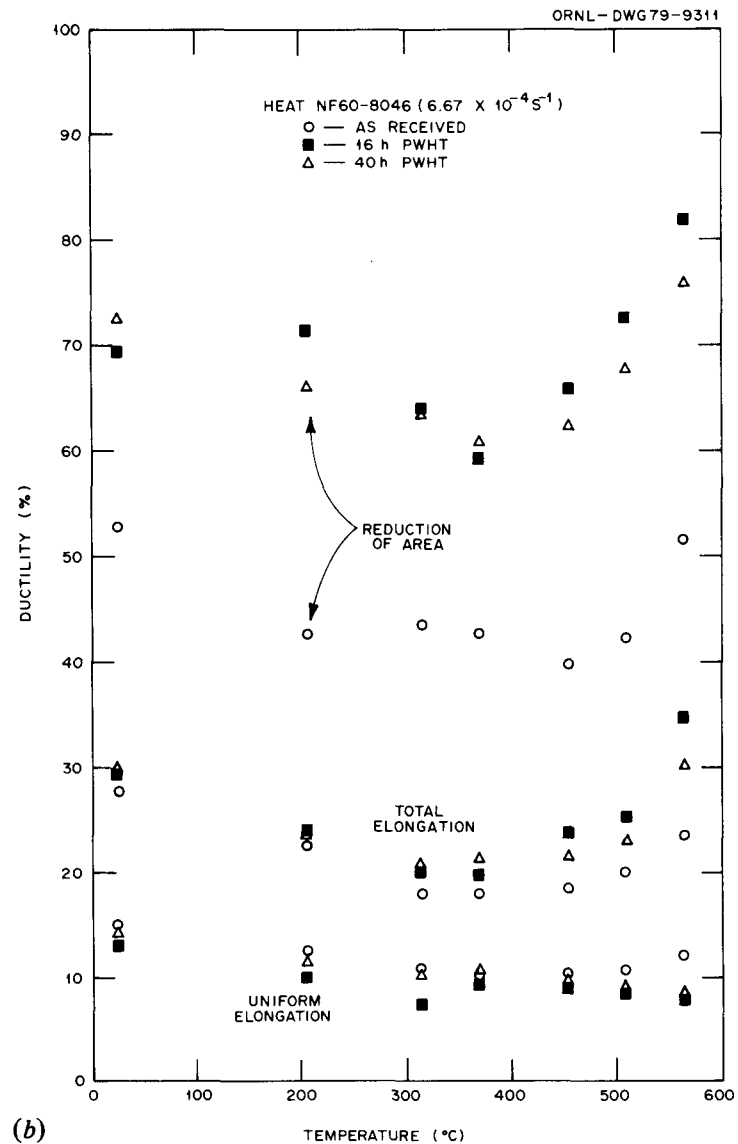
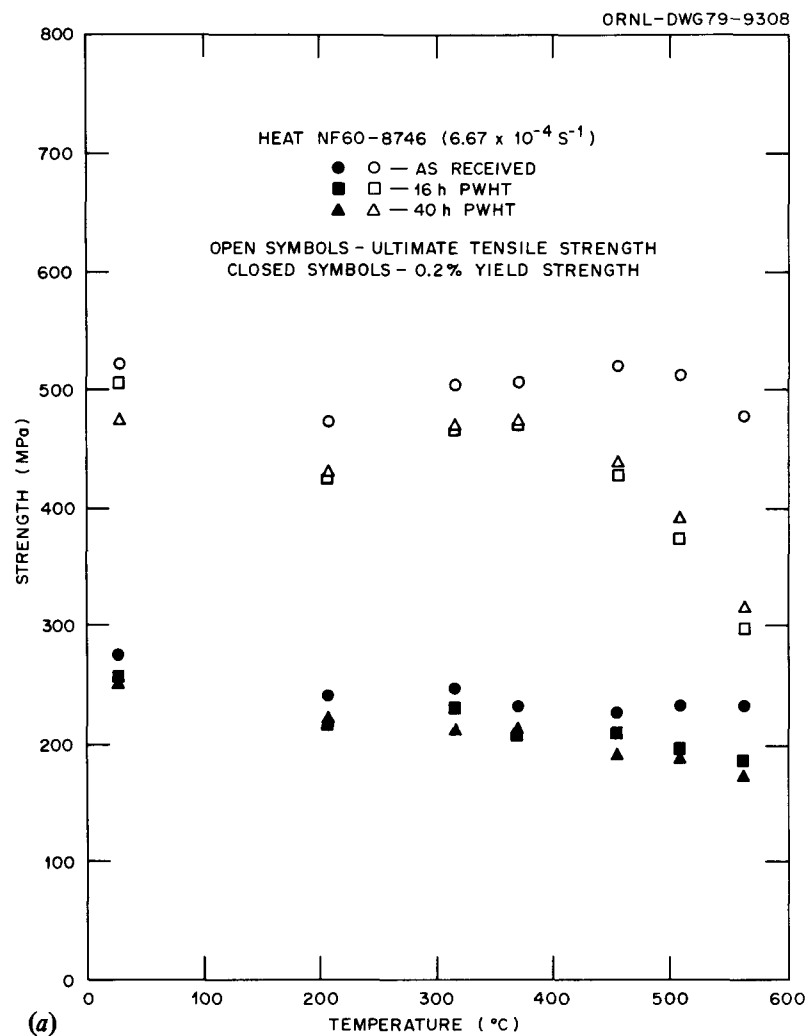


Fig. 14. Tensile properties at  $6.67 \times 10^{-4} \text{ s}^{-1}$  as a function of temperature for air-melted forging as isothermally annealed and isothermally annealed plus 16- and 40-h PWHT: (a) yield strength and ultimate tensile strength, (b) ductility.

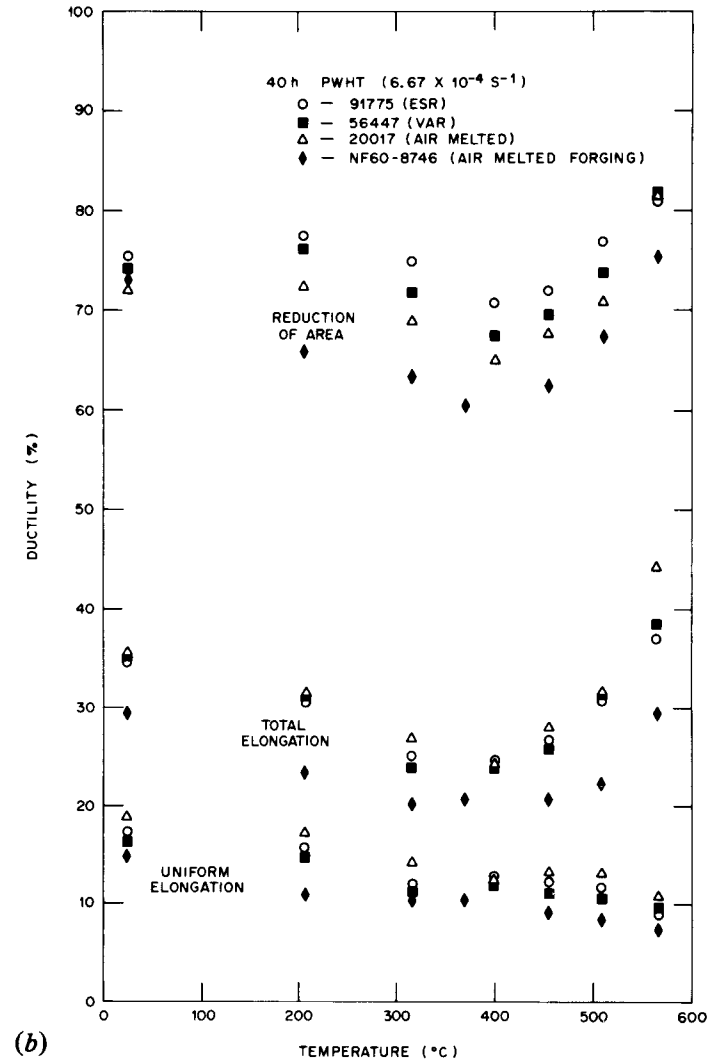
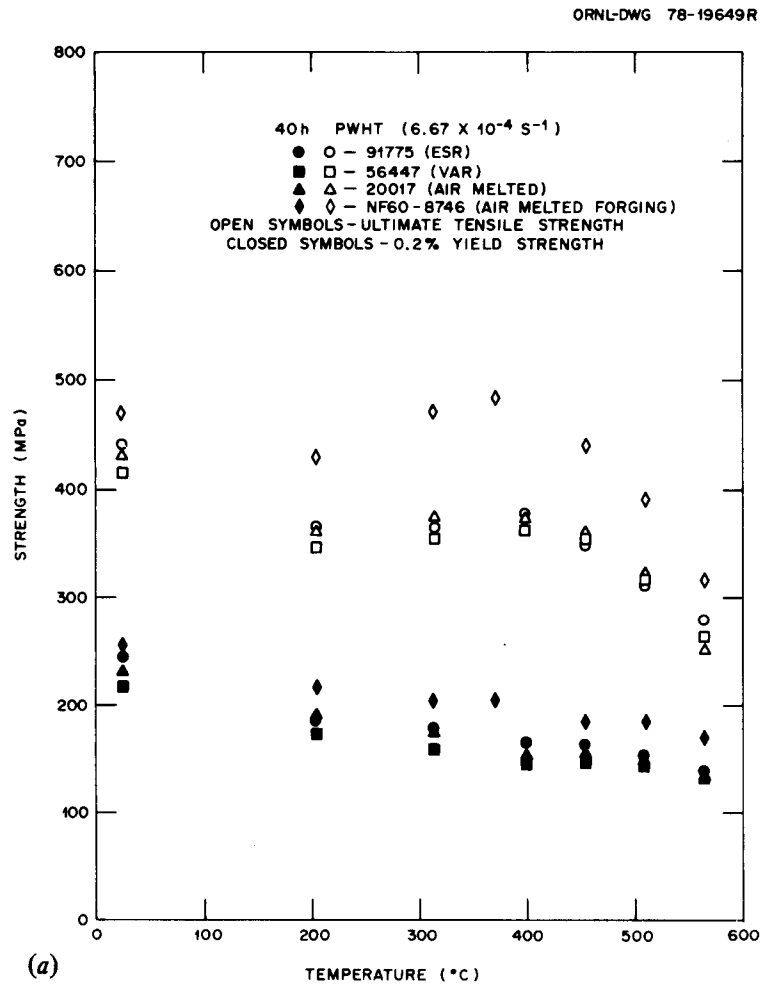


Fig. 15. Yield strength and ultimate tensile strength as a function of temperature at  $6.67 \times 10^{-4} \text{ s}^{-1}$  for heats 91775 (ESR), 56447 (VAR), 20017 (air-melted), and NF60-8746 (air-melted forging): (a) yield strength and ultimate tensile strength, (b) ductility.

expected minimum YS given in Code Case N-47 (Fig. 16). This comparison is for the VAR heat, but since there is little difference between the VAR plate and the ESR rod and air-melted plate after the 40-h PWHT, the observations also apply for the latter two heats.

After the 40-h PWHT, the room-temperature YS and UTS of all heats exceeded 207 and 413 MPa respectively. These are minimum values required by the appropriate ASME specifications that must be met for the steel to qualify for Code Case N-47.

Above about 100°C the YS for the VAR steel with a 40-h PWHT falls slightly below the expected minimum curve given in Code Case N-47. Lowering this strain rate increases this difference slightly at the lowest and highest test temperatures. Although not shown in Fig. 16, we found that the annealed steel and that given only a 1-h PWHT always fall well above the expected minimum. After a 5-h PWHT at 727°C, the YS was above the expected minimum below 400°C, after which it fell slightly below. The 16-h PWHT material paralleled that given the 40-h PWHT.

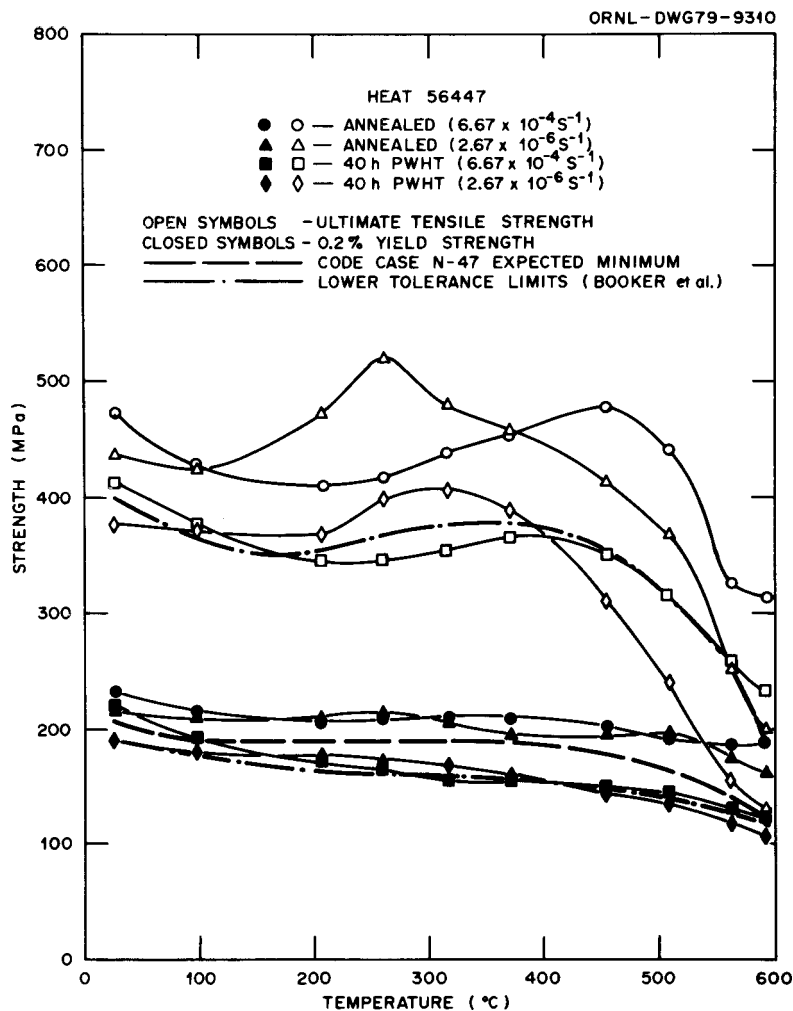


Fig. 16. Yield strength and ultimate tensile strength as a function of temperature for VAR plate (heat 56447) as annealed and after the 40-h PWHT. Data are compared with expected minimum yield strength curve from ASME Code Case N-47 and lower tolerance limits from analysis of available annealed data.

Code Case N-47 gives no expected minimum for the UTS. However, Booker et. al.<sup>6</sup> statistically analyzed the available 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel data in the annealed condition and established lower tolerance limits. These are shown in Fig. 16 for both UTS and YS (only at the highest temperatures do the YS values for the 40-h PWHT data fall below the lower tolerance limits). These curves differ very little from the minimum-trend curves that can be extracted from data compiled by Smith.<sup>7</sup> It was from the Smith data that Code Case N-47 values were determined.

When our UTS data are compared with the tolerance limits, we find that at the high strain rate ( $6.67 \times 10^{-4}$ /s, the 40-h PWHT data fall slightly below the lower tolerance limit between 200 and 400°C. Elsewhere, there is little difference between the two curves. At the lower strain rate ( $2.67 \times 10^{-6}$ /s), the 40-h PWHT data fall slightly below the lower tolerance limit at 25°C and quite substantially below it above 400°C. This large discrepancy at the high temperatures is probably the result of the data used to generate the lower tolerance limits. These data were biased against low-strain-rate data, since most data used for these correlations were obtained at strain rates greater than  $2.67 \times 10^{-6}$ /s. Note that at high temperatures and the low strain rate, the annealed VAR steel UTS falls on the lower tolerance limit.

From this discussion it appears that a PWHT of greater than 16 h at 727°C will give elevated-temperature YS values below those of the expected minimum from Code Case N-47. However, code-allowable stresses are calculated from the expected minimum with a conservative safety factor. Thus the small differences after a 16- or 40-h PWHT may not be that important.

The strengthening mechanisms for 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel have been discussed in detail.<sup>1,5,8,9</sup> Enhanced strength results from both solid-solution effects<sup>9,10</sup> and precipitation-strengthening effects.<sup>5,8</sup> It is the effect of PWHT on these processes that gives rise to the observed degradation of tensile properties.

After most anneal heat treatments, proeutectoid ferrite makes up the bulk of the microstructure and determines the strength of the steel.<sup>1</sup> Under most conditions the proeutectoid ferrite is super saturated with respect to carbon and molybdenum.<sup>1</sup> The dynamic strain-aging peak in 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel is the result of the interaction of dislocations with carbon-molybdenum atom pairs or clusters.<sup>1,9,10</sup> These clusters inhibit dislocation motion, thus requiring a larger stress to move them through the lattice. This dynamic strain-aging process has been termed interaction solid-solution hardening by Baird and Jamieson<sup>10</sup> and is analogous to the dynamic strain aging that occurs in a carbon or nitrogen steel. In the latter case, there is an interaction between the dislocations and the interstitial carbon or nitrogen atoms. The main difference in the processes is that interaction solid-solution hardening causes dynamic strain aging to occur at higher temperatures. This follows because dynamic strain aging is a diffusion-controlled process. In one case a single carbon or nitrogen interstitial atom has to diffuse, which can occur at relatively low temperatures. Much higher temperatures are required for interaction solid-solution hardening, since the diffusion of a cluster of atoms is required; thus the higher temperature of the peak in the latter case.

Since the carbon and molybdenum are in supersaturated solution in the proeutectoid ferrite, they eventually precipitate. The Mo<sub>2</sub>C is the first carbide to form.<sup>5</sup> However, this carbide is unstable and is eventually replaced by eta-carbide<sup>5</sup>—a molybdenum-rich carbide stoichiometrically near M<sub>4</sub>C.<sup>11</sup> All indications are that only the Mo<sub>2</sub>C provides substantial dispersion strengthening, since it forms as a relatively small high-density precipitate<sup>2,5,8</sup> (at least when it is formed below 566°C). The eta-carbide forms as large globular particles that contribute little to the strength.

The equilibrium microstructure in 2<sup>1</sup>/<sub>4</sub> Cr-1 Mo steel is a ferrite matrix that contains large globular M<sub>23</sub>C<sub>6</sub> and eta-carbide precipitates. The M<sub>23</sub>C<sub>6</sub>, along with more eta-carbides, forms in the bainitic regions<sup>5</sup> and in the few pearlitic regions that are present. In the bainitic regions the first precipitates to form are M<sub>3</sub>C and Mo<sub>2</sub>C; they are eventually replaced by the M<sub>23</sub>C<sub>6</sub> and eta-carbide.<sup>5</sup>

The difference between the annealed steel and the one given a PWHT is thus the state of supersaturation of molybdenum and carbon and the state of the precipitate. Since diffusion is rapid at a temperature as high as 727°C, the Mo<sub>2</sub>C precipitation reaction occurs rapidly<sup>5</sup> and is accompanied by the loss of molybdenum and carbon from solution. Thus, although the solubility of carbon and molybdenum is greater at 727°C than at the test temperature, the amount of supersaturation that remains after a long-time PWHT at 727°C is much less than was present prior to the PWHT (that supersaturation was established in a rather rapid furnace cool during the anneal). For a prolonged PWHT, the Mo<sub>2</sub>C will be replaced by the large globular particles of M<sub>23</sub>C<sub>6</sub> and eta-carbide.

The decrease in the height of the dynamic strain-aging peak after a PWHT is the result of the reduction of supersaturation and the agglomeration of precipitate particles. The temperature of the peak remains unchanged after the 1 hr PWHT (Fig. 4). This must mean that equilibrium for carbon and molybdenum in solution is achieved by 1 hr at 727°C (this appears reasonable, considering diffusion is rapid at 727°C). This equilibrium establishes the supersaturation at the test temperature (the saturated concentration at 727°C determines the supersaturation at the test temperature) and thus establishes the temperature of the peak for all steels given a longer PWHT. The continued decrease in peak height with time at 727°C reflects the formation and growth of the large globular carbides.

The large decrease in UTS with temperature above the dynamic strain-aging peak is largely due to recovery processes. This gives rise to the rapid loss of strength with decreasing strain rate. The still larger decrease in UTS for the material with the 16- and the 40-h PWHT is expected because of the precipitate morphology.

The observation on the ESR steel that there was essentially no strength difference between the isothermally annealed steel and the isothermally annealed steel that was given the 40-h PWHT was quite unexpected. A possible explanation involves the grain size, which was much finer for the ESR steel than for the other steels (Table 2). This smaller grain size is probably the result of the way this steel was fabricated (i.e., swaging). Hardenability of a steel decreases with decreasing grain size.<sup>12</sup> Hence, for the small grain size, the proeutectoid ferrite forms more quickly during the isothermal anneal, and the precipitation reactions proceed during the isothermal anneal. Further heat treatment at 727°C then has little effect. This possibility is supported by comparing the precipitate distribution in Figs. 2(a) and 3(b). The precipitate density of the ESR steel is much lower than that of the VAR steel. Although there are microstructural changes in the ESR steel during the 40-h PWHT [compare Figs. 3(b) and 3(d)], they are not as large as those for the VAR steel treated similarly [Figs. 2(a) and 2(b)].

The significantly greater strength of the air-melted forging after all steels had the 40-h PWHT is not understood. We previously showed that in the as-received condition, the dynamic strain-aging peak for this steel occurred at a higher temperature than for the annealed air-melted plate<sup>3</sup> (heat 20017). Evidently, this excess supersaturation persists even after the prolonged PWHT at 727°C. There is a difference in microstructures of these two steels after the 40-h PWHT (Fig. 17). The air-melted forging has a much higher density of small precipitates and would thus be expected to be stronger. It is difficult to understand, however, why the 40-h PWHT did not have more of an effect on the air-melted forging and cause the properties to approach those for the other steel. Something in this steel reduces the rate of the precipitate agglomeration.

Although the PWHT temperature for the CRBR steam generators has been established, it would appear profitable to examine whether a temperature as high as 727°C is required for future nuclear applications. Since the metallurgical processes that determine the strength degradation are thermally activated, even rather small reductions in the PWHT temperature could have significant effects on the

subsequent strength of the steel (ASME specifications require that any tempering treatment exceed 677°C). Studies should be made to determine whether sufficient stress relief can be achieved at temperatures of 680 to 700°C. If a PWHT at such temperatures was possible, a substantial improvement in strength may be achieved.

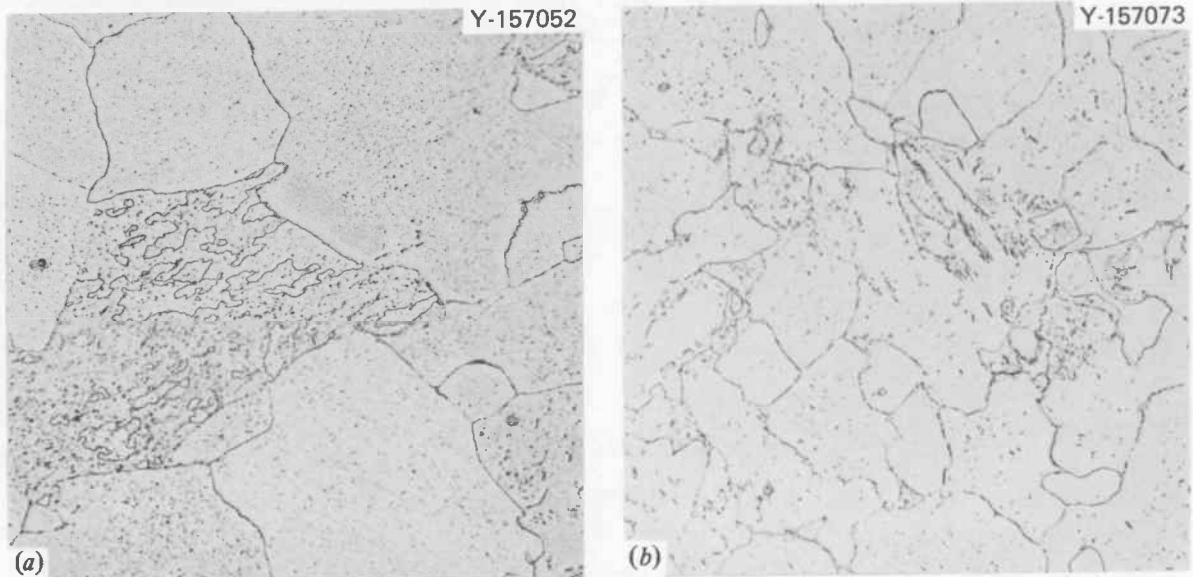


Fig. 17. Comparison of microstructures of (a) air-melted plate and (b) air-melted forging after the 40-h PWHT. 500 $\times$ .

### SUMMARY AND CONCLUSIONS

The effect of postweld heat treatment on the tensile properties of annealed and isothermally annealed 2 $\frac{1}{4}$  Cr-1 Mo steel was determined; PWHT was at 727°C. Tests were made at  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s over the range 25 to 566°C. The heats tested were a vacuum-arc-remelted plate in the annealed condition and after a PWHT of 1, 5, 16, and 40 h; an air-melted plate in the annealed condition and after a 40-h PWHT; an electroslag-remelted rod after an isothermal anneal and after a 40-h PWHT; and an air-melted forging as received (isothermally annealed) and after a 16- and 40-h PWHT. Following are our observations and conclusions.

1. The PWHT at 727°C significantly decreased the yield strength and ultimate tensile strength and increased the ductility. After 40 h at 727°C, there was no difference in the YS and UTS for the VAR plate, the ESR rod, and the air-melted plate. Even after the 40-h PWHT, all steels displayed a dynamic strain-aging peak in the UTS-temperature relationship.

2. For the VAR steel given a PWHT of 1, 5, 16, and 40 h, the YS and UTS changed after 1, 5, and 16 h. There was essentially no change between 16 and 40 h. The temperatures at which the dynamic strain-aging peak occurred in the UTS-temperature relationship decreased during the 1-h PWHT and remained relatively unchanged for a longer PWHT. The peak height decreased during the 5- and the 16-h PWHT; the height after the 40-h PWHT was the same as that after the 16-h PWHT.

3. Even after the 40-h PWHT, the YS and UTS of the air-melted forging remained significantly greater than for the other three steels. The microstructure of the air-melted forging after the 40-h PWHT contained a finer precipitate of higher density than did the other three steels.

4. After the 40-h PWHT, the room-temperature YS and UTS of all heats exceeded the 207- and 413-MPa minimum values required by ASME specifications.

5. Above 100°C the YS for the VAR plate, the ESR rod, and the air-melted plate with a 40-h PWHT fell slightly below the expected minimum curve given in ASME Code Case N-47.

6. It is recommended that the need for the 727°C PWHT temperature be reconsidered for future applications. It is expected that a lower-temperature temper (680 to 700°C) could relieve stresses and not reduce the strength as drastically as a 727°C PWHT.

### ACKNOWLEDGMENTS

Thanks are due the following people who aided in this work: J. L. Griffith and L. T. Ratcliff for carrying out the experimental work; C. W. Houck for metallography; and R. W. Swindeman, W. J. Stelzman, C. R. Brinkman, and G. M. Slaughter for reviewing the manuscript.

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**APPENDIX**

The work in this report was completed in accordance with the Development Requirement Specification (DRS) 53.40 issued by the CRBRP Project Office. The tests were specified in DRS 53.40, Table IV, The Monotonic Stress-Strain Test Matrix.

Part 5.0 of DRS 53.40 lists the data requirements. Below, those data requirements are listed along with an explanation of how they were met.

Specimen size and geometry: Fig. A-1.

Test material composition and processing history: Table 1 and Experimental section, pp.

Test material strength and ductility: Results section, pp. 2-3.

Test temperature: 25, 204, 316, 371, 399, 454, 510, 538, and 566°C.

Method of measuring strain: Standard Procedure (ASTM E 8-69 and E 151-64).

Stress-strain curves: Stored at ORNL.

Strain rates:  $2.67 \times 10^{-6}$  and  $6.67 \times 10^{-4}$ /s.

Test environment: Air.

Mathematical model of monotonic stress-strain curves—Voce equation: Data being analyzed.

Yield strength: Results section, pp. 4-19.

Ultimate tensile strength: Results section, pp. 4-19.

Percentage elongation: Results section, pp. 4-19.

Percentage reduction in area: Results section, pp. 4-19.

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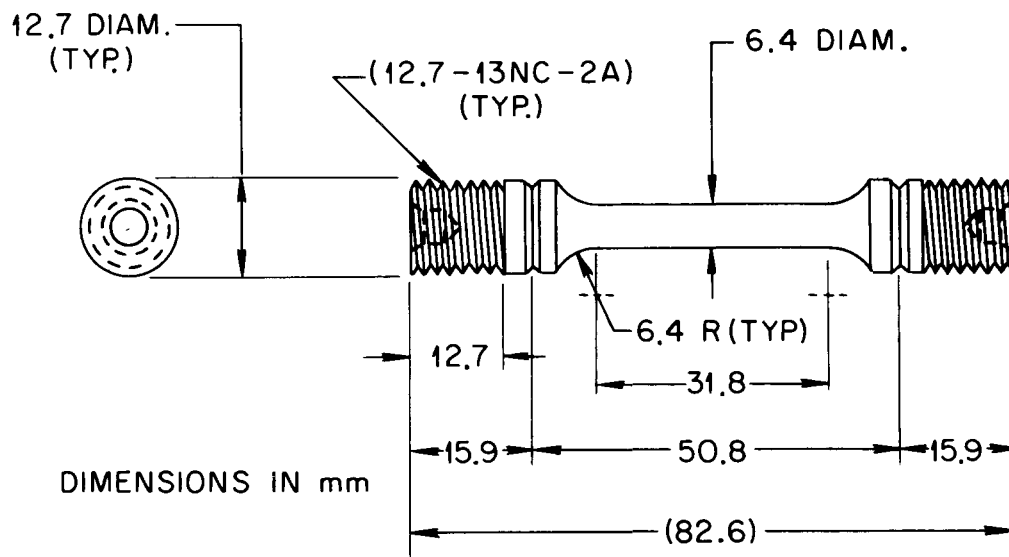


Fig. A-1. Drawing of tensile specimen used in study.

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