OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
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CREEP PROPERTIES OF THE Nb–1% Zr ALLOY

H. E. McCoy

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ABSTRACT

Creep rupture properties of two heats of Nb-1% Zr have been determined. Although these materials have comparable chemical analyses and were reported to have the same fabrication history, their creep strengths are considerably different. It was found that, at a test temperature of 982°C and at high stresses, a fine precipitate formed which was oriented normal to the applied stress. At 1204°C, the precipitate was quite coarse and, again, was preferentially oriented normal to the applied stress. It was found that pretest annealing at temperatures up to 1700°C markedly improved the strength without decreasing the rupture ductility.

INTRODUCTION

In attempting to raise the temperature capabilities of nuclear power plants, attention has been focused upon the refractory metals and their alloys. Although the melting points of these materials indicate their potential usefulness at elevated temperatures, it is necessary that their properties be known more specifically before engineers can actually design such systems. One of the basic properties on which information is needed is the strength of the material at the service temperature. Because of the reactivity of the refractory metals with air, it is necessary that the test piece be protected during testing. This, in turn, requires leak-tight apparatus with associated systems for vacuum or for inert-gas purification. Hence, these tests are quite expensive and it is for this reason that reliable long-time creep data for refractory metals are almost nonexistent.

Another important aspect of refractory metal technology is that very few attempts have been made to optimize these materials. Optimization is being approached almost entirely from the standpoints of slight modifications in alloy additions and changes in fabrication schedules. These are indeed both important, but just as the properties of many iron- and nickel-base alloys have been found to be controlled by heat treatments, post-fabrication heat treatments can play an important role in refractory alloys.

The present study is concerned with making a thorough evaluation of the creep properties of the Nb–1% Zr alloy at elevated temperatures. This work includes a study of the influence on the creep properties of pretest annealing and supporting metallographic studies.

EXPERIMENTAL DETAILS

Chemical analyses for the two heats of Nb–1% Zr sheet included in this study are given in Table 1. Both heats of material were obtained from the Wah Chang Corporation in the form of 0.060–0.065-in. sheet. The following fabrication schedule was communicated by the vendor:

1. Stock material about 0.16 in. thick rolled at 427°C to 0.100 in.
2. Material conditioned and pickled.
3. Material cold rolled to 0.060–0.065 in.
4. Material sheared, inspected, and shipped.

Therefore, the material contained at least 40% cold working in the as-received condition.

The geometry of the test specimen used is shown in Fig. 1. The test apparatus is shown in Fig. 2. The test chamber is an alumina tube sealed by Viton gaskets to water-cooled metal fittings on both ends. The extension rods near the specimen are made of Nb–1% Zr and those extending outside the chamber are Inconel X. The pull rods are sealed at the top by an O-ring and at the bottom by a Viton U-cup seal. An alumina thermocouple well extends into the test chamber to allow temperatures to be read inside the chamber with a Pt_{100}-$Pt_{90}Rh_{10}$ thermocouple. The chamber is heated by a resistance furnace having a capability of 1200 or 1700°C. (Although furnace capabilities of 1700°C are available, the alumina tube presently poses a limitation of about 1200°C for long-term tests). The vacuum system utilizes a 750 liter/sec oil diffusion pump and an elbow

TABLE I

Analyses of Nb–1% Zr Test Materials[a]

Element	Content (wt %) Heat 912-1659	Content (wt %) Heat 1012-946	Element	Content (wt %) Heat 912-1659	Content (wt %) Heat 1012-946
Zr	1.05 (0.93±0.05)	0.95 (0.91±0.05)	Fe	< 0.010	< 0.010
O	0.0170 (0.015)	0.0205 (0.0160)	Hf	< 0.0080	Not Detected
N	0.0045 (0.0055)	0.0090 (0.0085)	Mn	< 0.0020	< 0.0020
H	0.0004 (0.0004)	0.0002 (0.0018)	Mo	0.0020	< 0.0020
C	0.0035 (0.011)	0.0065 (0.0045)	Ni	< 0.0020	< 0.0020
Al	< 0.0020		Pb	< 0.0020	< 0.0020
B	< 0.0001	< 0.0001	Si	< 0.010	< 0.0100
Cd	< 0.0005	< 0.0005	Ta	0.0650	0.0610
Co	< 0.0030	< 0.0020	Ti	< 0.0150	< 0.0150
Cr	< 0.0020		V	< 0.0020	< 0.0020
Cu	< 0.0040		W	0.0170	< 0.0300

[a]Numbers in parentheses are results of analyses at ORNL; other values are averages of the vendor's ingot analyses.

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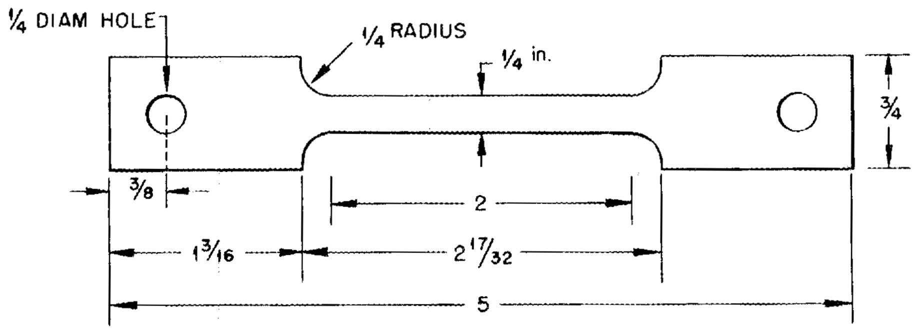

Fig. 1. Geometry of Test Specimen.

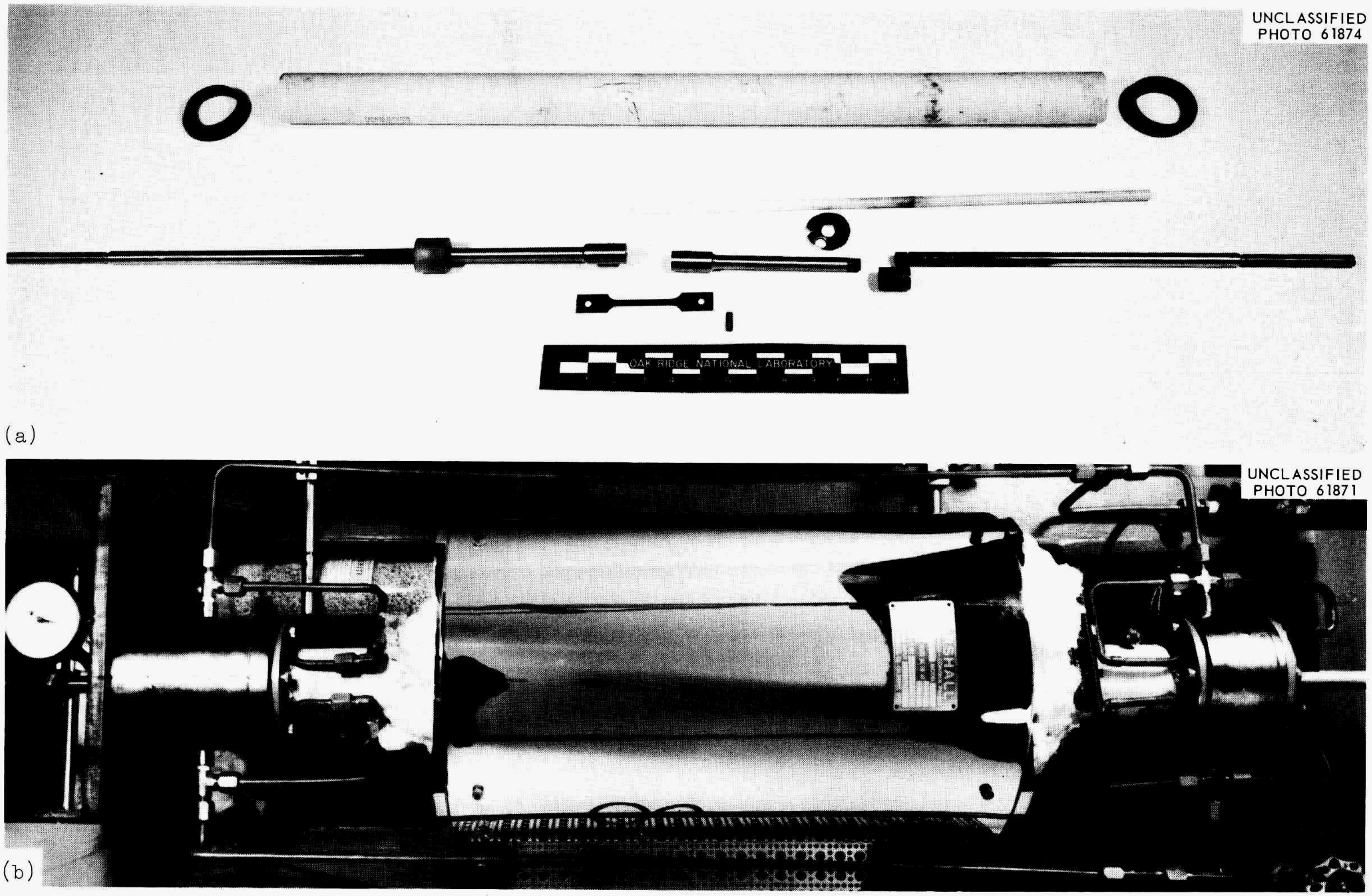

Fig. 2. High-Temperature Vacuum Test Chamber. (a) View of Disassembled Pull Rods and Test Specimens. (b) Assembled Test Chamber and Furnace.

trap cooled with dry ice. The vacuum is measured at the top of the test chamber with values of 1×10^{-7} torr being obtained routinely. A cylinder of thin Nb–1% Zr sheet is placed around the specimen to act as a getter. The machines are of the dead-load type, and extension of the specimen is measured by a dial gage which rests against the extension rod.

All of the metallographic specimens in this study were polished by vibratory syntron polishing. The specimens were etched at 60°C in a solution of 250 ml H_2O, 60 g NaOH, 20 g tartaric acid, 50 ml lactic acid, 30 ml of 30% H_2O_2.

RESULTS

The experimental data obtained in this program are summarized in Tables 2 and 3. The data are presented graphically in different forms to illustrate several important points. Figures 3 and 4 are plots of stress vs time to certain amounts of strain and fracture for test temperatures of 982 and 1204°C, respectively. The plots represent the properties of heat 912-1659 in the as-received condition. It is significant that, at both test temperatures, large creep strains occur in a small fraction of the total rupture life (i.e., the minimum creep rate exists over a very short portion of the rupture life and acceleration into third stage creep occurs early in the test). The minimum creep rate is shown in Fig. 5 as a function of the applied stress. This property is adequately described by an expression of the form $\dot{\epsilon} = (\sigma/A)^n$, where ϵ is the strain rate, σ is the applied stress, and A and n are constants. With σ in pounds per square inch and $\dot{\epsilon}$ in percent per hour, the values of A were 15,400 and 5,200 at 982 and 1204°C, respectively; the values of n were 4.83 and 4.92

TABLE II

Creep Properties of 0.060-in.-thick Nb–1% Zr Sheet

Heat 912-1659

Test No.	Temperature (°C)	Stress[a] (psi)	Heat Treatment	Rupture Life (hr)	Rupture Strain (%)	Minimum Creep Rate (%/hr)	Chemical Analysis[b] H	O	N	C
2493	982	15,000	As-Received	14.8	29.69	0.820	4	130	59	70
2481	982	12,000	As-Received	64.8	38.28	0.200	1	170	56	120
2480	982	10,000	As-Received	148.1	50.78	0.129	3	360	60	130
2494	982	8,500	As-Received	260.8	65.63	0.101	7	280	58	140
2736	982	7,000	As-Received	1069.0	35.99	0.0182				
2536	982	6,500	As-Received	1732.8	61.72	0.0170	1	550	68	370
2986	982	4,500	As-Received	c		0.00152				
2377	1204	6,000	As-Received	9.8	64.84	2.35	6	260	65	
2447	1204	5,000	As-Received	23.4	88.28	1.15	4	220	62	
2600	1204	4,000	As-Received	96.9	87.66	0.290	2	150	58	
2613	1204	3,000	As-Received	541.5	103.9	0.0527	1	160	55	
2908	1204	2,700	As-Received	855.6	85.16	0.0318	1	130	57	50
2614	982	10,000	1 hr at 1200°C	110.9	56.72	0.0800	1	130	60	
2556	982	10,000	1 hr at 1400°C	790.2	38.28	0.00150	1	230	73	
2643	982	10,000	1 hr at 1500°C	856.6	42,97	0.00100	1	380	57	
2865	982	10,000	1 hr at 1700°C	1562.5	23.44	0.00024	1	350	55	80
2551	1204	4,000	1 hr at 1200°C	89.6	89.84	0.295	28	35	59	100
2594	1204	4,000	1 hr at 1400°C	152.1	65.62	0.0940	3	290	62	80
2555	1204	4,000	1 hr at 1500°C	233.4	80.16	0.0650	1	480	57	160
2864	1204	4,000	1 hr at 1700°C	536.0	53.13	0.00210	< 1	390	57	90
2489[d]	982	10,000	As-Received	158.7	56.25	0.184	7	390	70	90

[a]Stress applied parallel to rolling direction.
[b]See Table 1 for analysis of as-received material.
[c]Test discontinued prior to failure.
[d]Stress applied normal to rolling direction.

TABLE III

Creep Properties of 0.060-in.-thick Nb-1% Zr Sheet

Heat 1012-946

Test No.	Temperature (°C)	Stress[a] (psi)	Heat Treatment	Rupture Life (hr)	Rupture Strain (%)	Minimum Creep Rate (%/hr)
3156	982	17,500	As-Received	11.0	36.72	0.615
3123	982	15,000	As-Received	58.8	38.28	0.095
3092	982	12,500	As-Received	190.4	52.34	0.0124
3165	982	10,000	As-Received	468.9	52.34	0.0083
3126	1204	7,500	As-Received	5.15	78.13	5.40
3116	1204	6,000	As-Received	31.5	71.09	0.71
3078	1204	5,000	As-Received	82.7	85.94	0.293
3052	1204	4,000	As-Received	257.4	83.59	0.0675
3141	1204	3,000	As-Received	1074.2	106.3	0.0190
3159	982	12,500	1 hr at 1200°C	88.5	47.19	0.079
3148	1204	5,000	1 hr at 1200°C	78.2	76.09	0.28

[a]Stress applied parallel to rolling direction.

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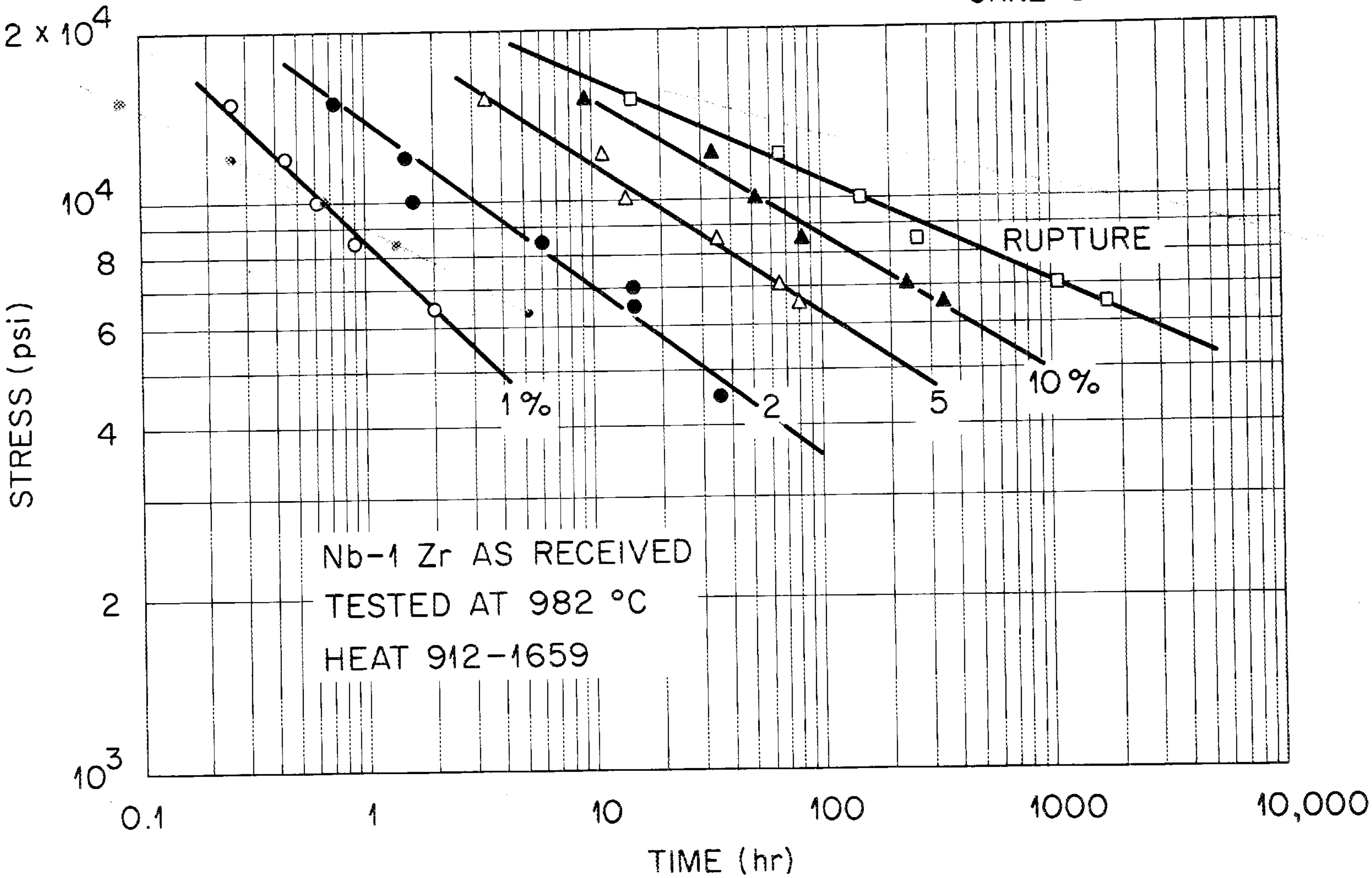

Fig. 3. Stress-Rupture Properties of Nb-1% Zr at 982°C.

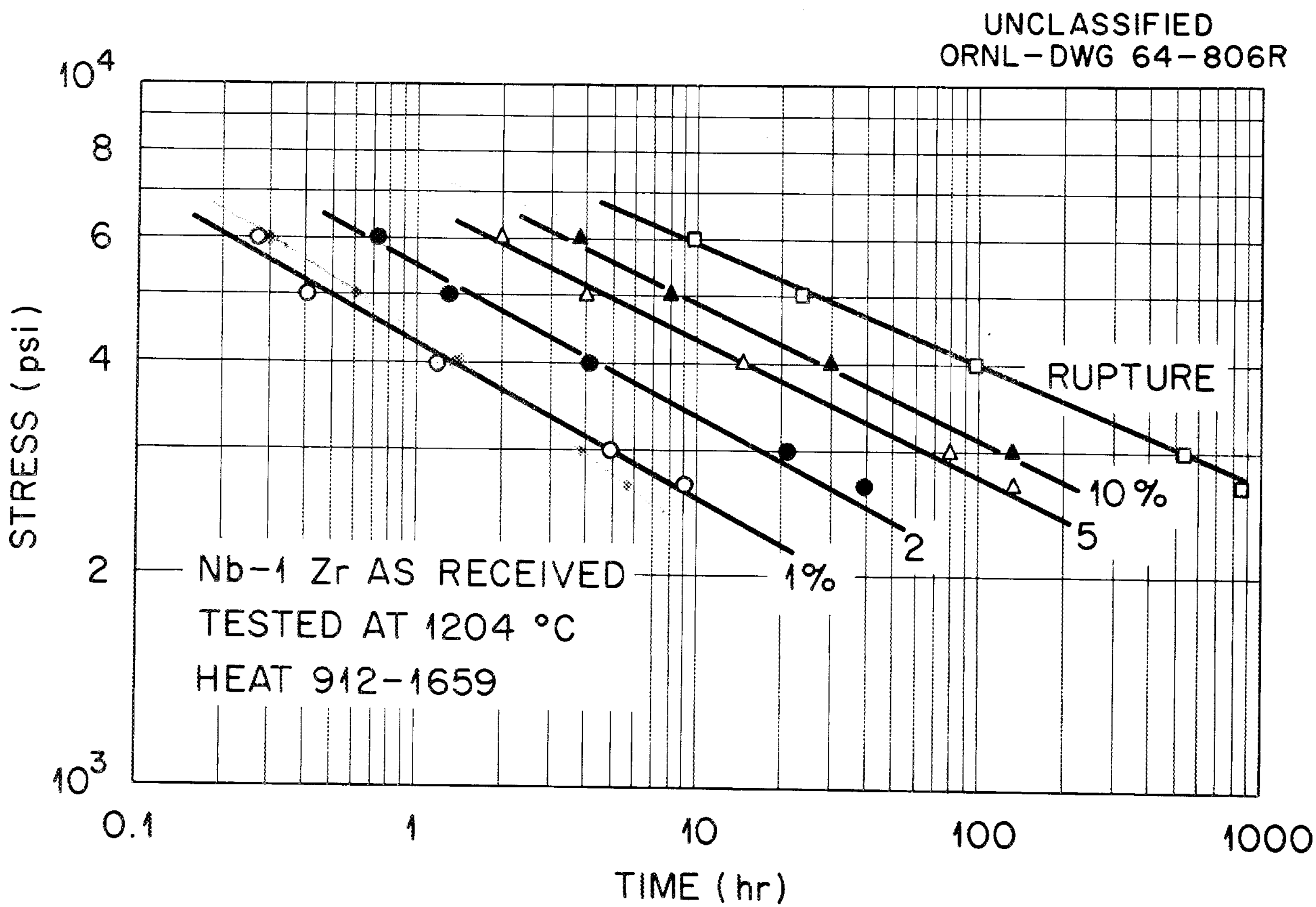

Fig. 4. Stress-Rupture Properties of Nb-1% Zr at 1204°C.

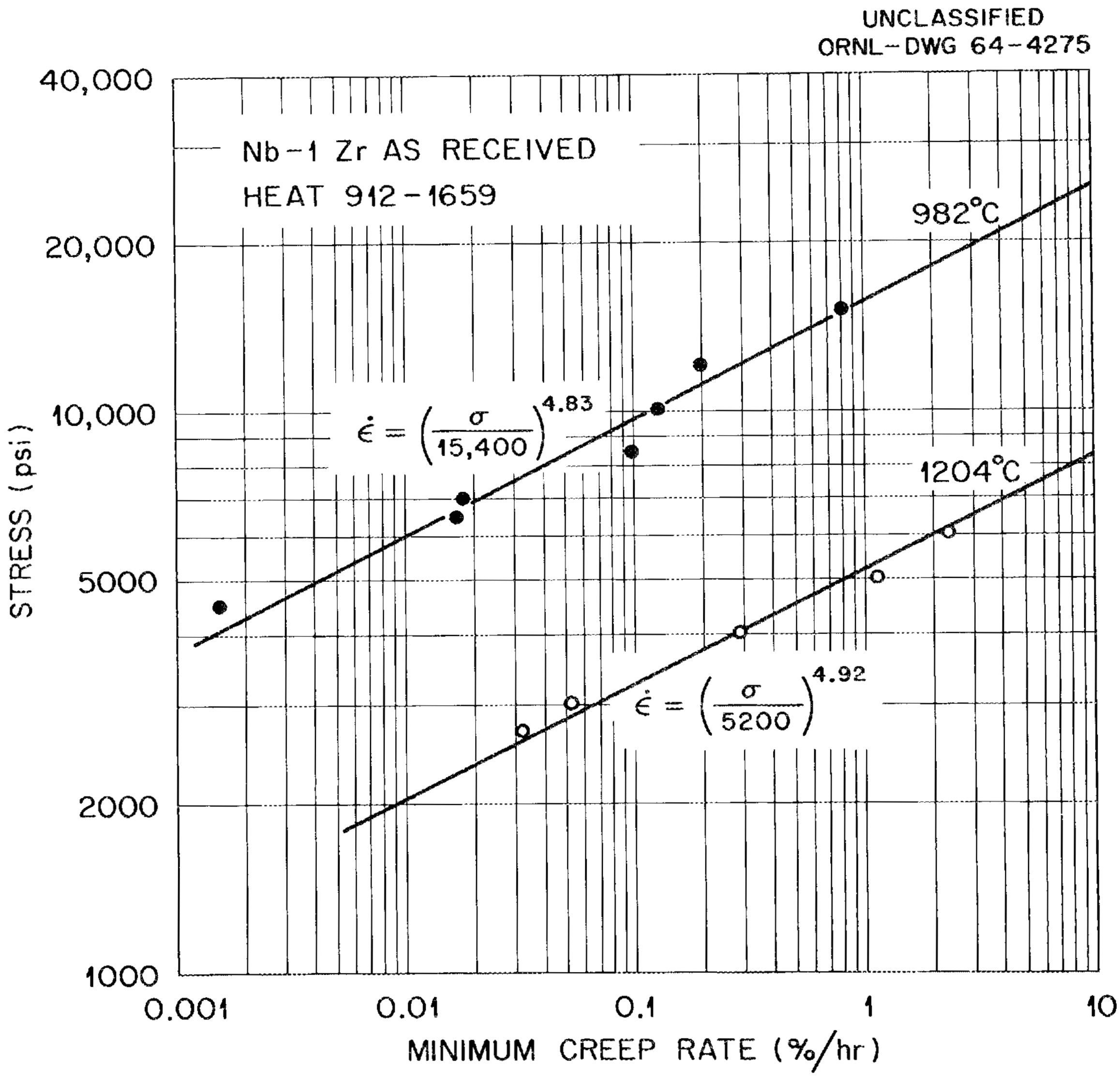

Fig. 5. Correlation of Stress and Minimum Creep Rate for Nb-1% Zr

at 982 and 1204°C, respectively. Another important relationship is shown in Fig. 6 where the logarithms of the minimum creep rate and rupture life are plotted. Machlin[1] predicted that these variables should correlate with a slope of unity for cases where the measured creep rate and the creep rate producing vacancies for voids are the same. The data at 1204°C obey this correlation quite well. At 982°C the short-time creep tests do not seem to fit, but longer tests do fit the prediction. This is probably because the starting material was cold worked and was not adequately annealed during the shorter creep tests at 982°C.

The stress-rupture properties of the two heats of material used in this study are compared in Fig. 7. Heat 1012-946 is considerably stronger than heat 912-1659 but the analytical data in Table 1 indicate that heat 1012-946 has the lower interstitial content. Thus, it does not seem possible to rationalize the improved strength on the basis of composition.

The influence on the creep strength of pretest annealing was investigated. The results of these studies are given in Table 2. Figure 8 illustrates the influence of pretest annealing on the minimum creep rate at test temperatures of 982 and 1204°C. At 982°C the minimum creep rate is reduced by pretest anneals of 1 hr at temperatures between 1200 and 1700°C. The creep rate of the alloy in the as-received condition is on the order of 10^3 greater than that of the alloy after annealing for 1 hr at 1700°C. The creep strength at 1204°C is improved by annealing above this temperature. At 1204°C the minimum creep rate of the material in the as-received condition is about 10^2 greater than that observed after annealing 1 hr at 1700°C. Not only was the minimum creep rate reduced by annealing

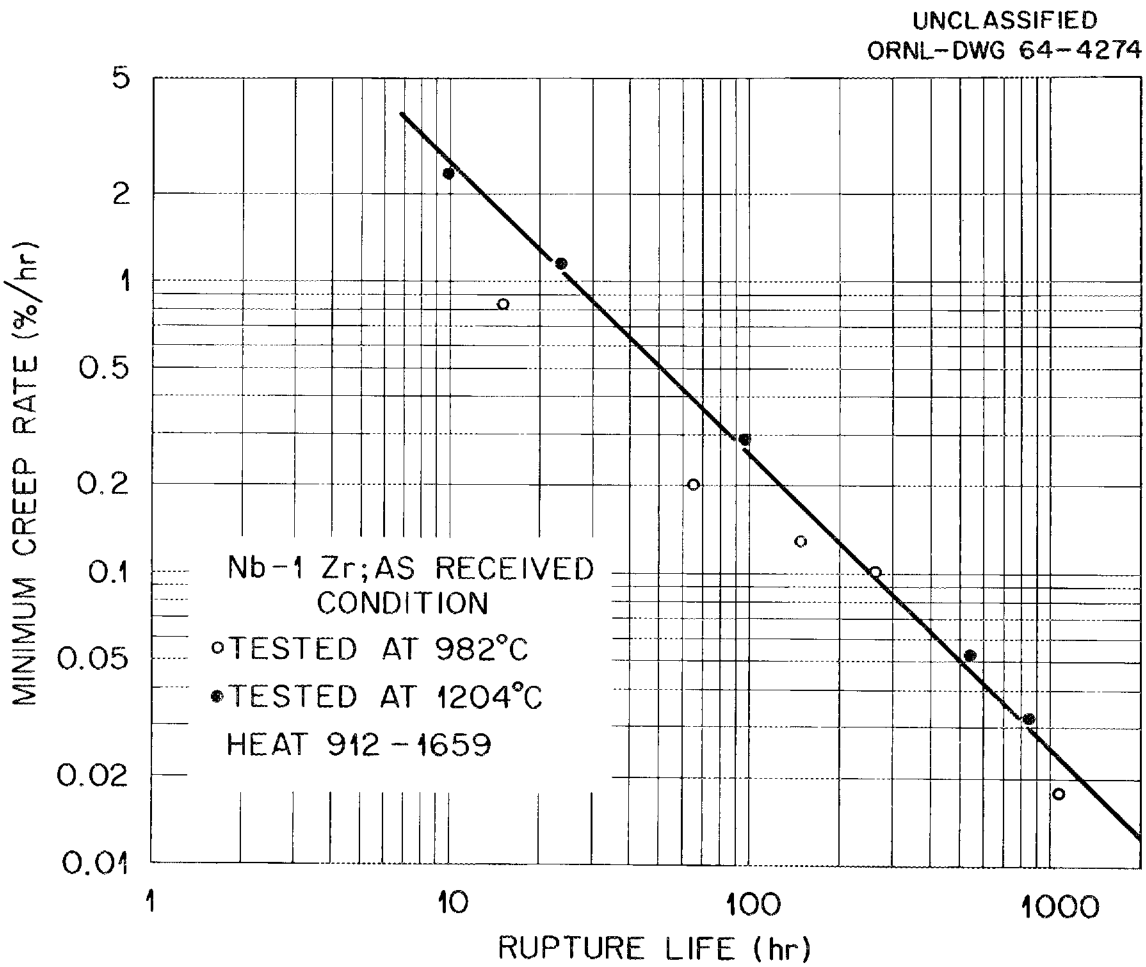

Fig. 6. Creep-Rupture Properties of Nb–1% Zr.

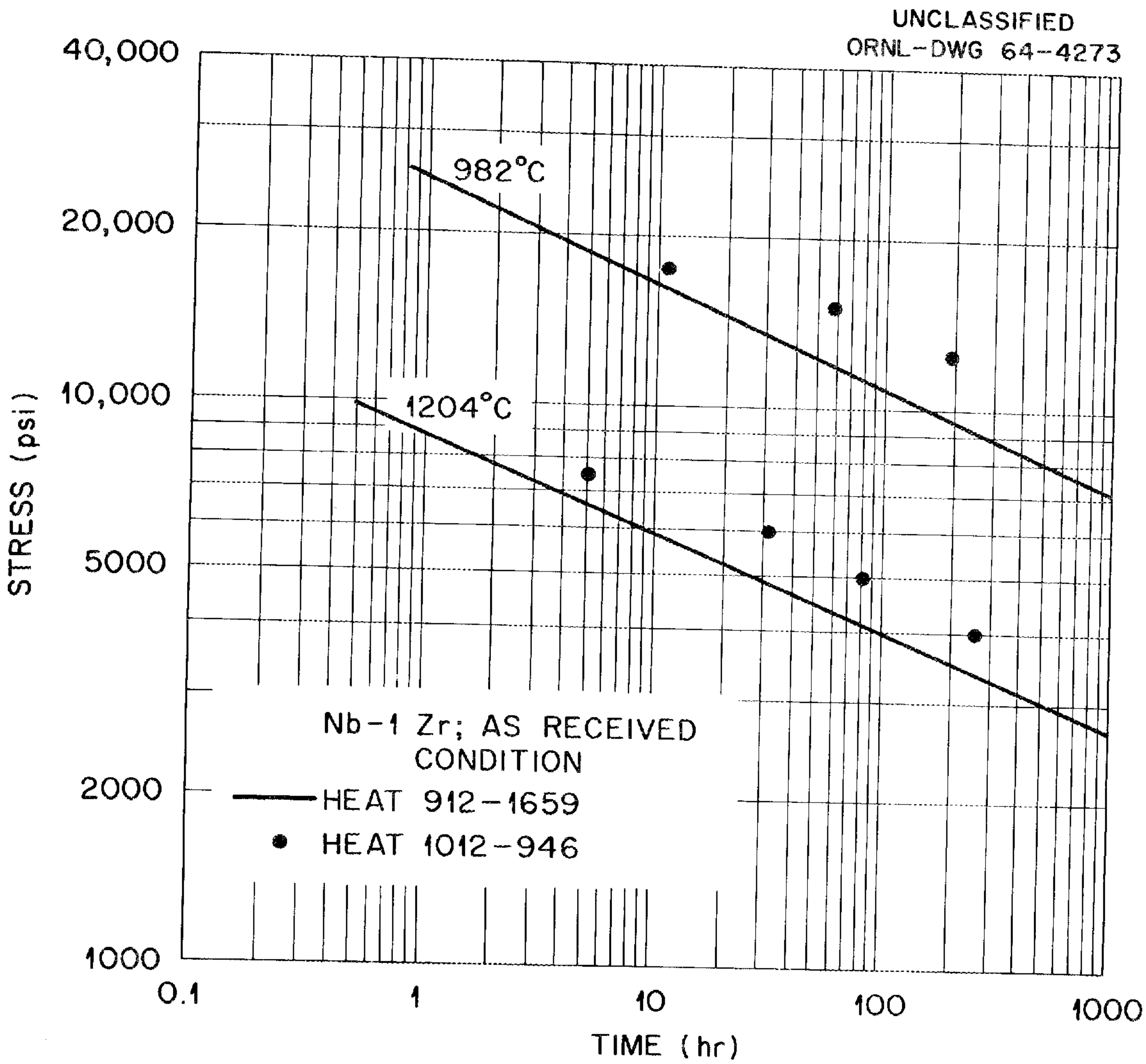

Fig. 7. Comparison of the Stress-Rupture Properties of Two Heats of Nb-1% Zr.

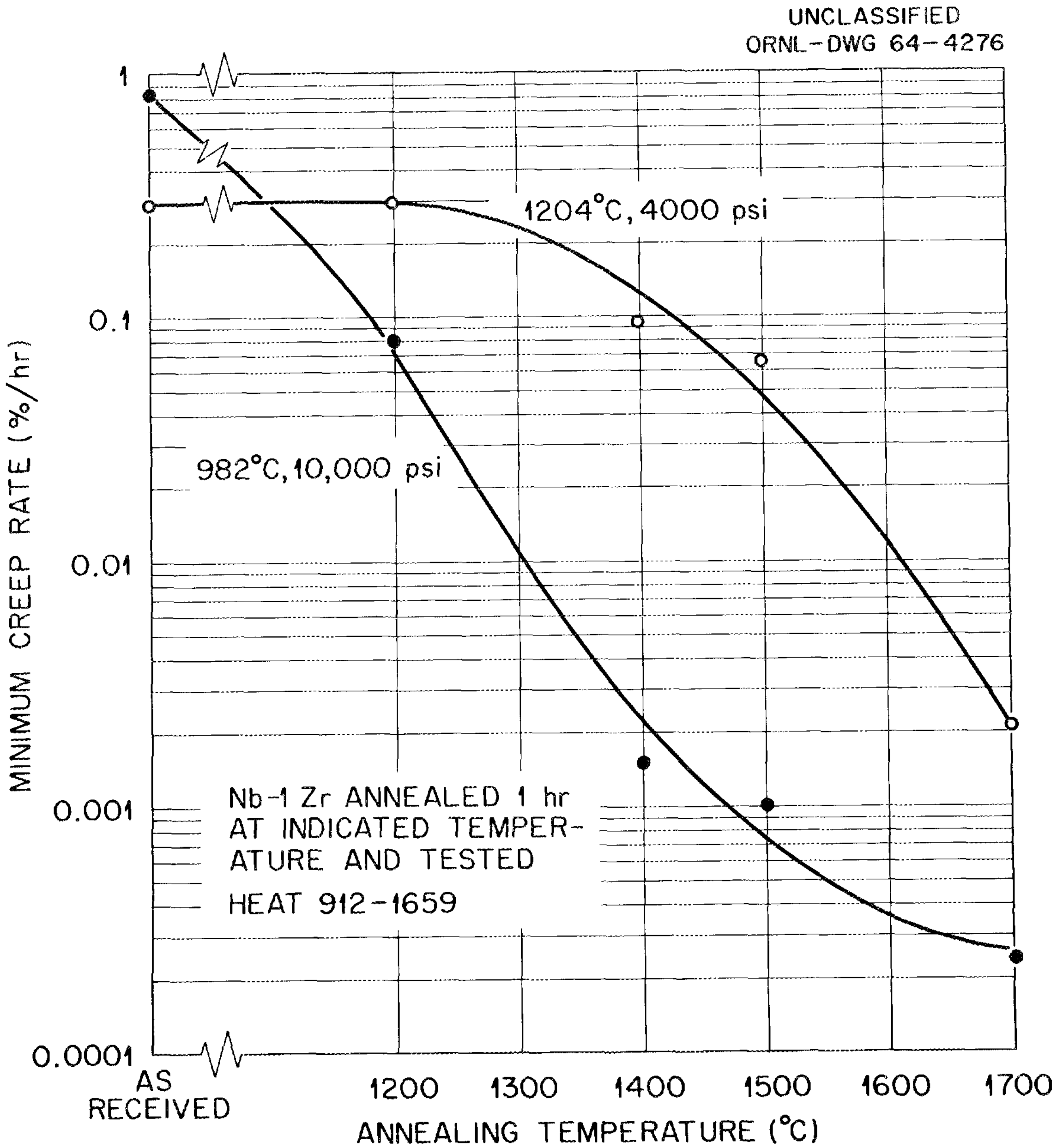

Fig. 8. Influence of Annealing on the Minimum Creep Rate of Nb-1% Zr.

but also the fraction of the test time for which the creep rate remained at the minimum value was increased. This is illustrated in Figs. 9 and 10. At 982°C (Fig. 9), with the material in the as-received condition, only about 10^{-3} of the rupture life is required to reach 1% strain. After a 1-hr anneal at 1700°C, the time required to reach 1% strain is about one-half the rupture life. At a test temperature of 1204°C (Fig. 10), the improvement is not as great but the same trends exist.

Large rupture ductilities were exhibited by all test specimens. At 982°C, the fracture strain of the as-received material ranged from 30 to 65%. Pretest annealing reduced the ductility slightly with the fracture strain of a specimen annealed at 1700°C being 23%. The ductility was even greater at 1204°C with fracture strains from 53 to 104% being obtained.

Several of the specimens were examined metallographically after testing. Figure 11 shows the microstructures of both heats of material in the as-received condition. Although the fabrication of the two materials was supposed to be identical, the microstructures indicate that heat 1012-946 was cold worked more heavily than heat 912-1659.

Figure 12 shows the microstructure of an Nb–1% Zr specimen tested at 15,000 psi and 982°C. Under these conditions, a precipitate is formed which is oriented normal to the stress direction. As the stress level is decreased, the orientation of the precipitate becomes more random until 10,000 psi where the precipitate appears completely random.

Figure 13 shows the structure of a specimen tested at 10,000 psi and 982°C after a pretest anneal of 1 hr at 1500°C. This anneal has increased significantly the grain size of the material. Some recrystallization occurred near the fracture. The microstructure of a specimen tested at 10,000 psi and 982°C after a pretest anneal at 1700°C is shown in Fig. 14.

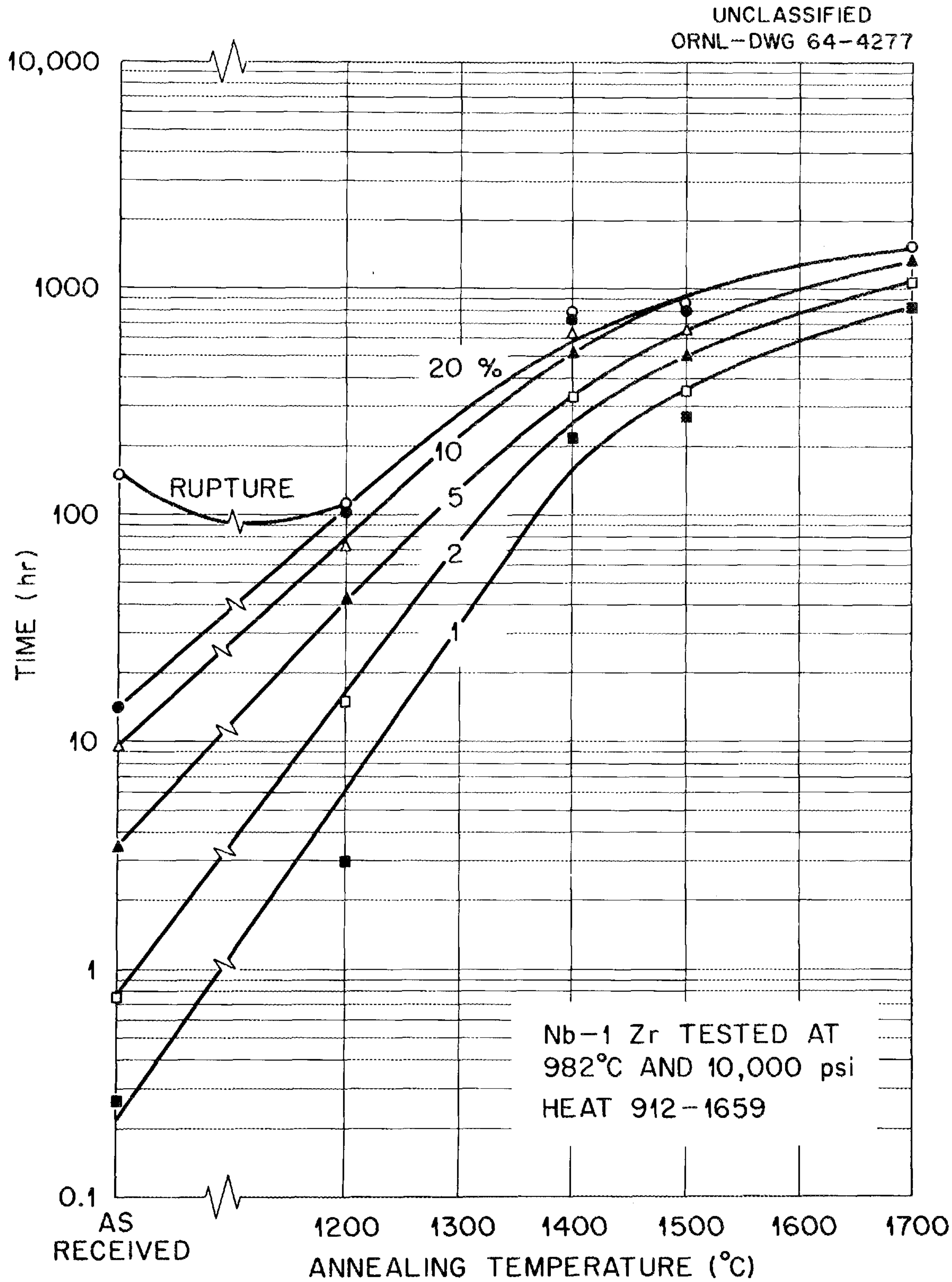

Fig. 9. Influence of Annealing on the Creep Properties of Nb–1% Zr at 982°C.

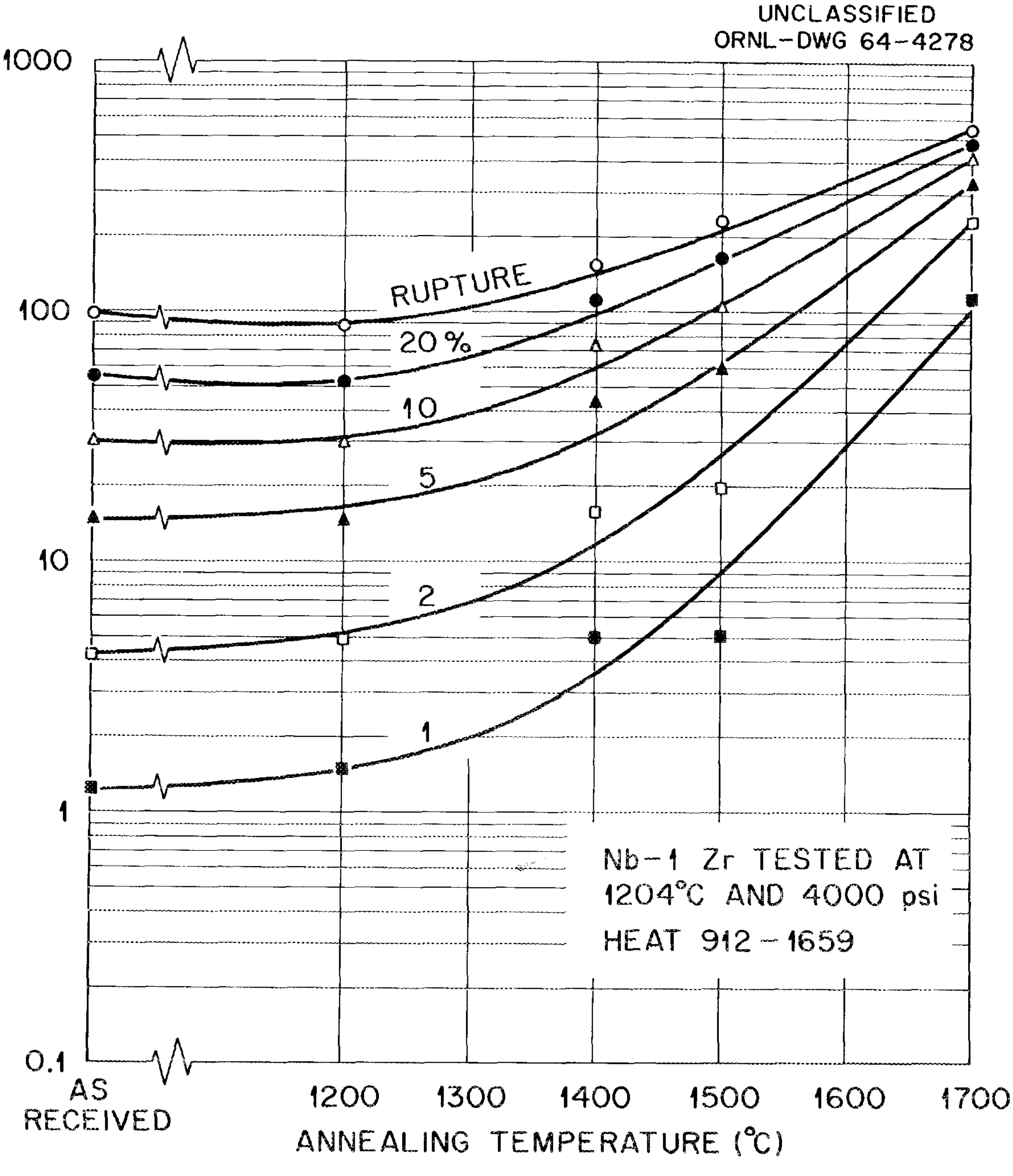

Fig. 10. Influence of Annealing on the Creep Properties of Nb-1% Zr at 1204°C.

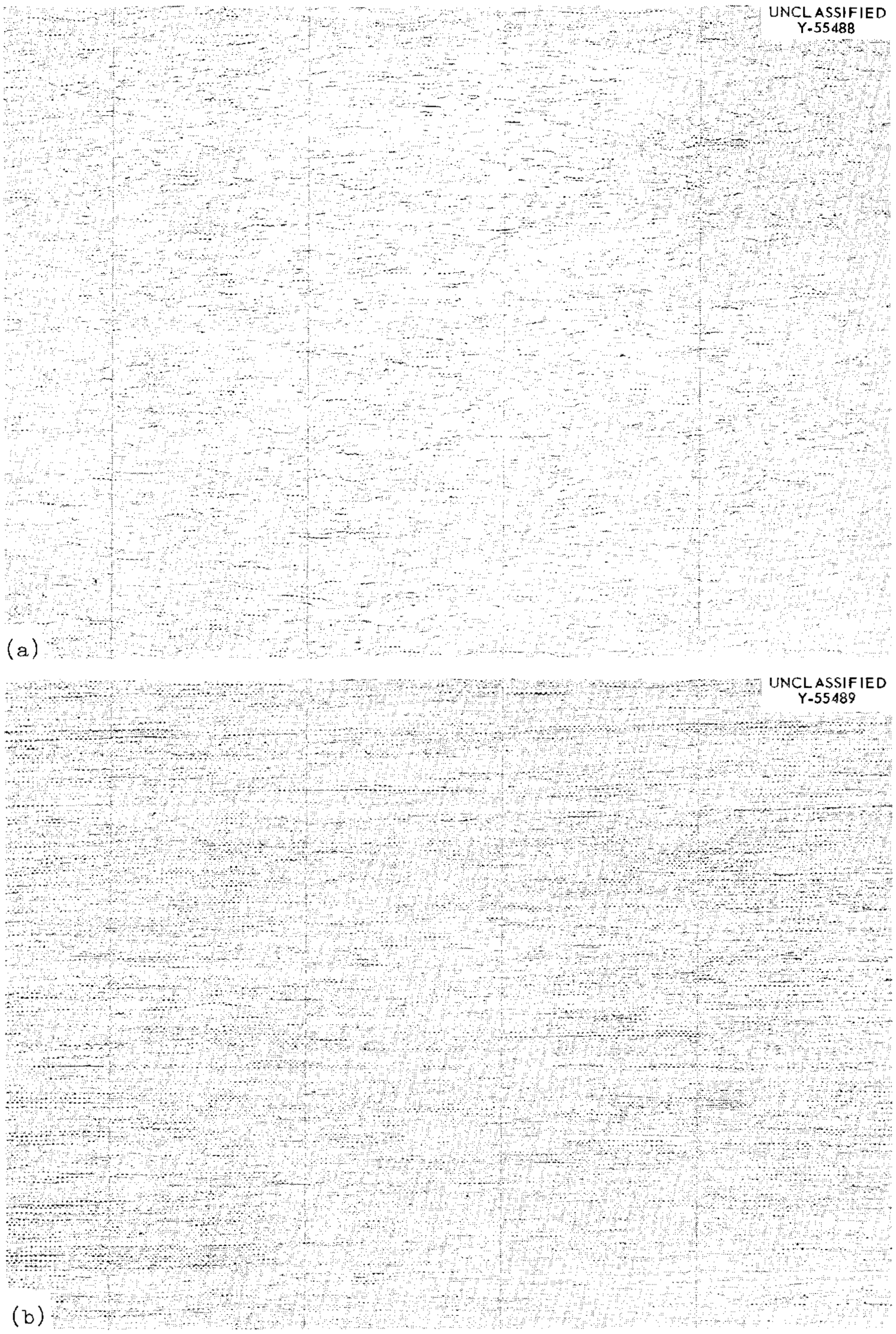

Fig. 11. Microstructures of Nb–1% Zr in the As-Received Condition. (a) Heat 912-1659. (b) Heat 1012-946. 100×. Reduced 17%.

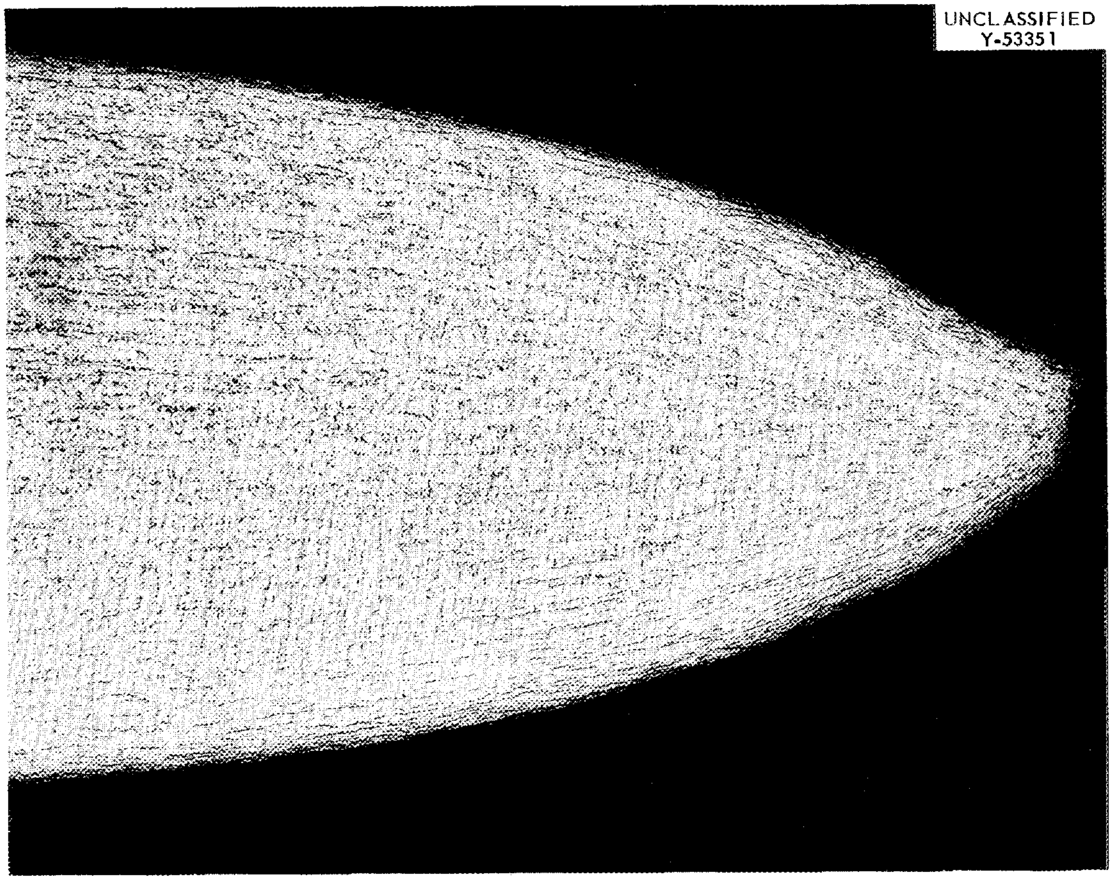

Fig. 12. Photomicrograph of the Fracture of Nb–1% Zr Specimen Tested in the As-Received Condition at 982°C and 15,000 psi. Rupture life: 14.8 hr. Heat 912-1659. 100×

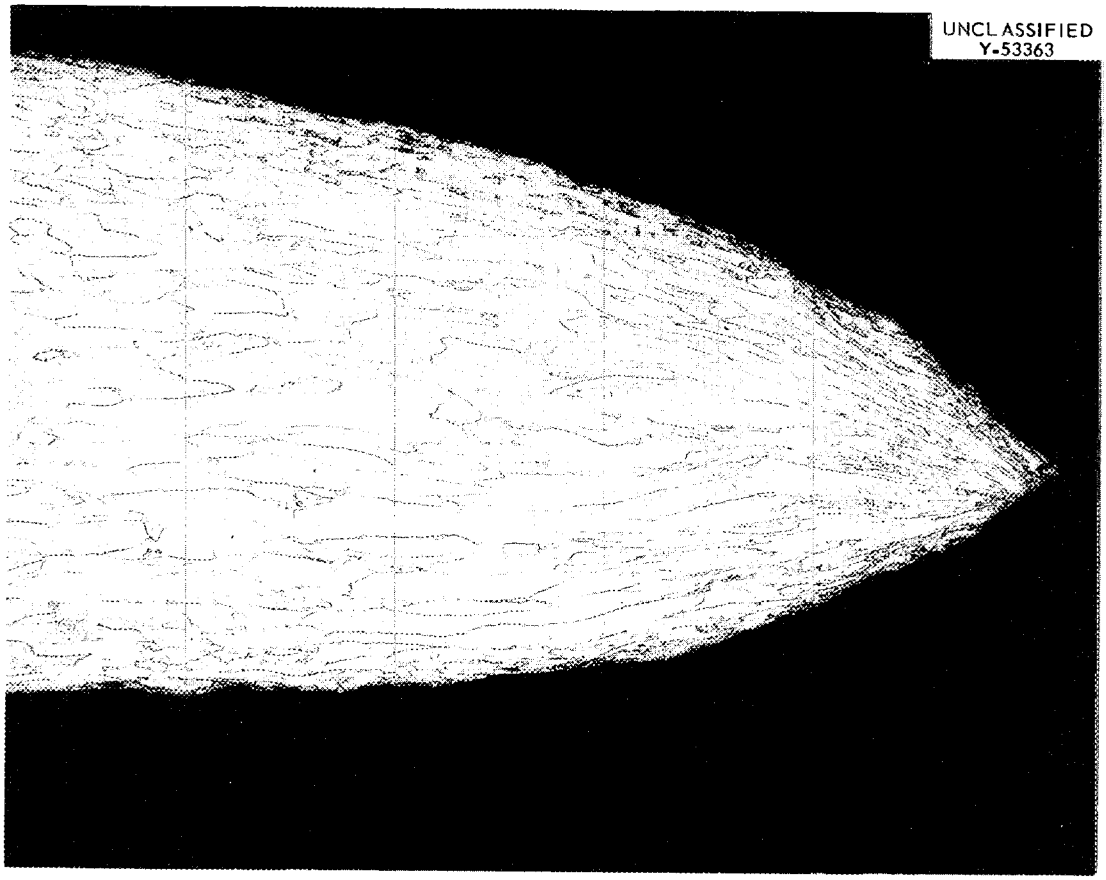

Fig. 13. Photomicrograph of the Fracture of a Nb–1% Zr Specimen Tested at 982°C and 10,000 psi, Annealed 1 hr at 1500°C Prior to Testing. Rupture life: 856.6 hr. Heat 912-1659. 100×

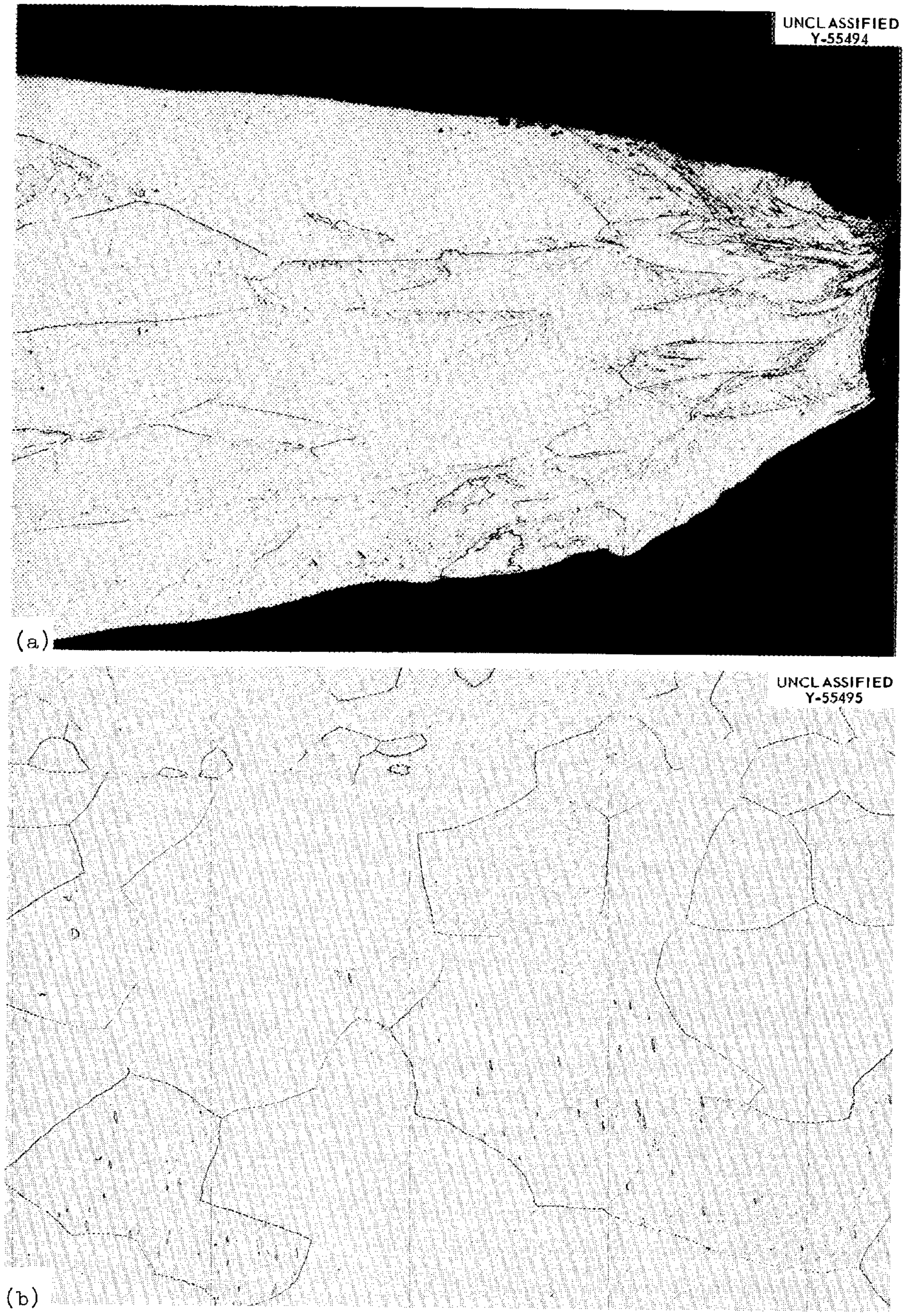

Fig. 14. Photomicrographs of a Nb–1% Zr Specimen Tested at 982°C and 10,000 psi, Annealed 1 hr at 1700°C Prior to Testing. Rupture life: 1562.2 hr. (a) Rupture; (b) 1 in. from rupture. Heat 912-1659. 100X. Reduced 19%.

In addition to the increased grain size resulting from annealing, the tested specimen appears to have developed an extensive substructure. A relatively coarse precipitate oriented normal to the applied stress is formed under these test conditions. Since this precipitate was not present in the material after annealing at 1700°C, it must have nucleated during testing.

Figure 15 shows the microstructure of a specimen tested in the as-received condition at 4,000 psi and 1204°C. The material recrystallized during testing and considerable grain growth occurred near the fracture. There also appears to be a coarse precipitate in this specimen which is oriented normal to the stress direction. Figure 16 illustrates the microstructure of a specimen after a pretest anneal at 1700°C and testing at 1204°C and 4,000 psi. Significant features are the large grain size, the coarse precipitate normal to the applied stress, and the substructure.

Although all of the metallographic results presented here have been for heat 912-1659, it was found that heat 1012-946 exhibited similar structures.

DISCUSSION OF RESULTS

Creep studies have been conducted to evaluate the creep properties of the Nb–1% Zr alloy at 982 and 1204°C. It was found that the as-received material exhibits creep behavior characterized by a very short period at the minimum creep rate followed by a continuously increasing creep rate. Annealing the material at various temperatures before testing decreases the minimum creep rate, increases the rupture life, and increases the fraction of the rupture life spent at the minimum creep rate. These changes

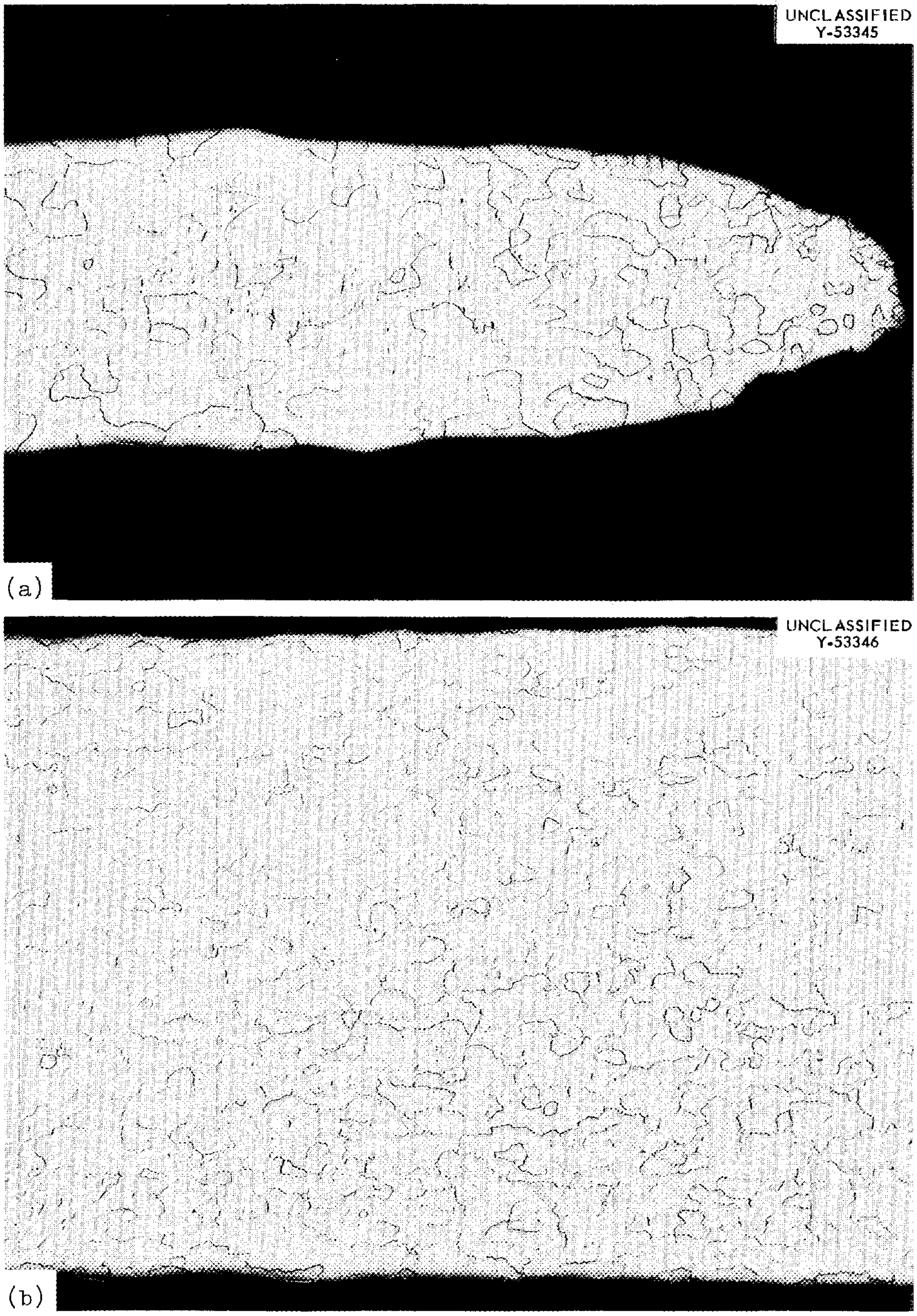

Fig. 15. Photomicrographs of a Nb–1% Zr Specimen Tested in the As-Received Condition at 1204°C and 4000 psi. Rupture life: 96.9 hr. (a) Fracture; (b) 1 in. from fracture. Heat 912-1659. 100×. Reduced 12%.

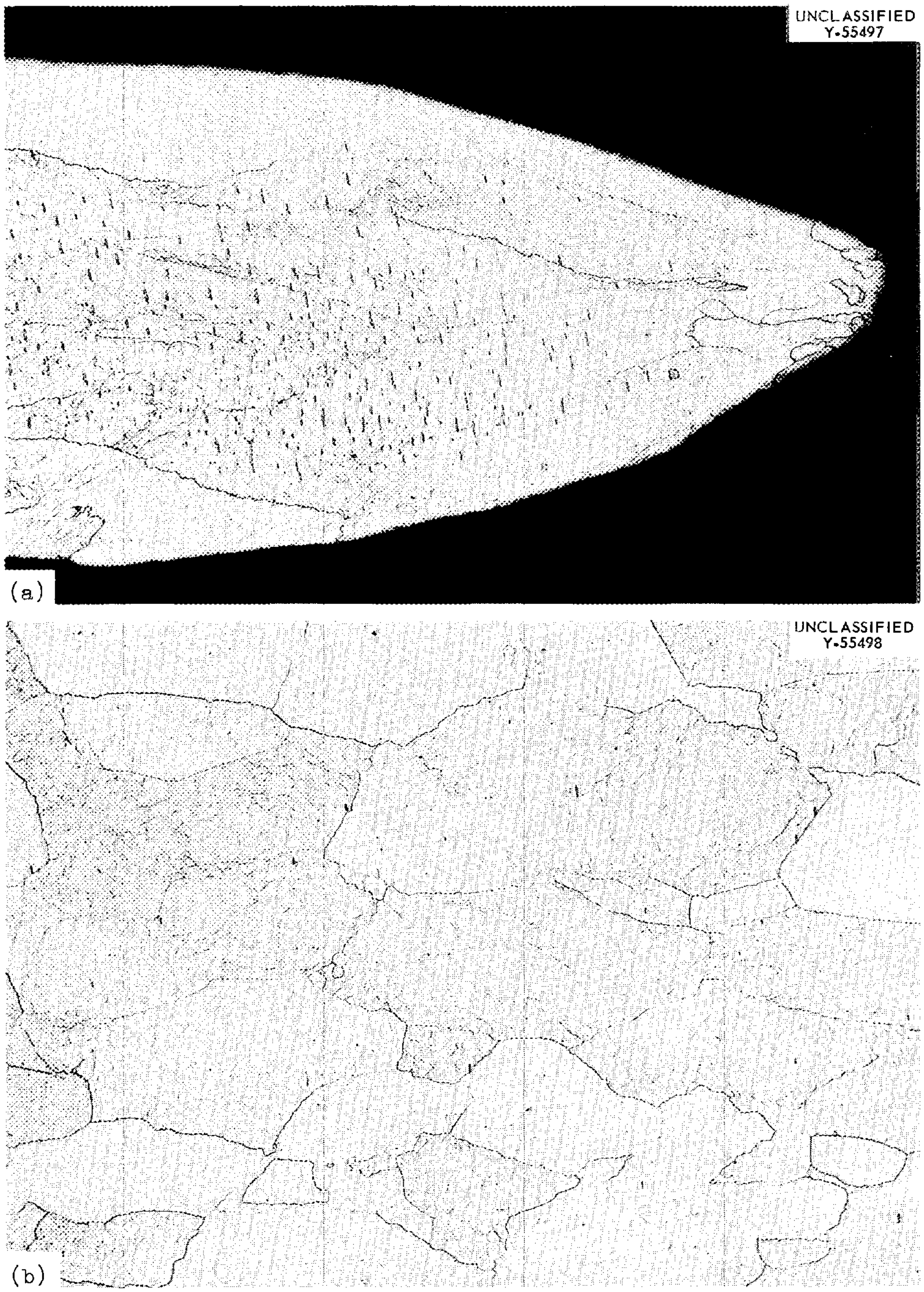

Fig. 16. Photomicrographs of a Nb–1% Zr Specimen Tested at 4000 psi and 1204°C, Annealed 1 hr at 1700°C Prior to Testing. Rupture life: 536 hr. (a) Fracture; (b) 1 in. from fracture. Heat 912-1659. 100X. Reduced 17%.

are desirable from an engineering viewpoint since components are normally designed to small strains rather than to fracture. For example, a part constructed of Nb–1% Zr in the as-received condition and put in service at 982°C and 4,000 psi would deform 2% after only 0.75 hr. If the material had been annealed 1 hr at 1200°C, the time to 2% strain would be increased to 15 hr. Annealing at 1700°C would increase the time to 2% strain up to 1150 hr. Heat treatment can obviously be used to an advantage in the Nb–1% Zr alloy.

Although a clear explanation of why annealing improves the creep strength of this alloy cannot be given at this time, it is possible to postulate the responsible mechanism on the basis of what is known about other materials. In annealing a solid solution alloy, at least three processes can occur: grain growth, solution of impurities, and reorientation of grains. The first process is generally accepted to cause improvement in high-temperature creep resistance, but Parker[2] has shown that for copper this improvement is actually brought about by the last process and that coarse-grained material is inherently weaker than fine-grained material at all temperatures. When a material is annealed, its internal energy is reduced by reduction of the angles between adjacent grains. This reduction in angle also makes the boundaries less effective as sources and sinks of vacancies. Since the rate-controlling process in high-temperature creep is the motion of dislocations around obstacles and this process is, in turn, dependent upon the presence of vacancies, a reduction in the effectiveness of the vacancy sources reduces the creep rate. Although this argument has been developed for copper, the same reasoning may apply to the Nb–1% Zr alloy. The solution of interstitial precipitates may also be an important process. These precipitates may be taken into solution during annealing and may reprecipitate at the test temperature in a distribution which is effective in impeding dislocation motion. The photomicrographs presented in this study show considerable evidence of reprecipitation during testing.

The specimens tested at 982°C and high stresses showed a selective orientation of the precipitate along planes normal to the applied stress. The impurities present in this precipitate were probably initially in solution, and this does not represent a case of a precipitate reorienting itself under a stress. The precipitates formed during testing at 1204°C were quite coarse and were oriented normal to the applied stress. Similar selectively oriented precipitates have been observed in other materials.[3]

Many of the test specimens were observed to contain a substructure. Weissmann, Lement, and Cohen[4] have summarized for refractory metals the available information on the experimental detection of substructures, types, and the relation of mechanical properties and substructures. The formation of substructures in the refractory metals is well documented as well as the fact that these substructures improve the strength. It is known[5] also that substructures are not eliminated by anneals which would result in complete conventional recrystallization. Thus the substructures noted in the present study are a function of (1) the fabrication procedure of the alloys, (2) the pretest anneal, (3) the test temperature, and (4) the strain rate.

Another significant metallographic feature of the test specimens is that no intergranular voids were formed. Intergranular voids have been observed in many other materials after being deformed at homologous temperatures comparable with those of the Nb–1% Zr alloy in the present study.[6,7] Several investigators[1,8] have been concerned with the nucleation of these voids, and the general opinion seems to be that grain boundary irregularities, such as slip steps, are plausible nuclei. These nuclei are then pictured as growing by the stress-motivated diffusion of vacancies to these sites[9] or by the condensation of excess vacancies produced by the deformation.[1] These nuclei grow and join to form cracks. Conditions which produce fracture by this mode normally result in low-fracture strains. The absence of voids in the Nb–1% Zr alloy may result from the mobility of the grain boundaries. This mobility, in turn, may result from grain boundary migration per se or by motion through recrystallization. Thus, the boundaries are not stationary long enough for void nuclei to grow. This may account for the extremely good ductility of this alloy. It is also interesting that the model proposed by Machlin[1] for creep rupture by vacancy condensation seems to fit the experimental results even though no visible voids are present.

Another point of significance is that the properties of the two heats of material are so different. The interstitial analyses are quite similar for the two heats with the carbon content of the stronger heat being slightly lower. The photomicrographs of the as-received materials (Fig. 11) indicate that the stronger heat was cold worked more than the weaker heat. It is felt that this apparent difference in fabrication technique is the major cause for the difference in properties. This variable of fabrication procedure has been shown[10] to be very important in controlling the properties of the material. However, suppliers of materials and experimenters who evaluate these materials appear to be so far removed that control of properties by fabrication schedule is not progressing very rapidly.

The properties of refractory metals can depend on contamination during testing. As is obvious, several specimens from the present tests were found to contain about 300-ppm oxygen after testing. It was found that points representing these tests did not consistently fall ahead of the average stress-rupture curve. However, some specimens were heavily contaminated with oxygen and carbon, and these specimens had rupture lives much longer than would be expected. Although this conclusion is not supported by detailed observations, it would appear that small (about 300 ppm) oxygen contamination does not modify the creep strength appreciably but that a comparable amount of carbon increases the strength.

SUMMARY AND CONCLUSIONS

Tests have been run which evaluate the creep properties of the Nb–1% Zr alloy at 932 and 1204°C. Solution anneals of 1 hr at 1200 to 1700°C were found to significantly improve the creep properties. It is felt that the improvement in strength is a result of changes in grain boundary structures and not in grain size alone. Rupture ductilities were quite high under all test conditions. A fine precipitate was formed normal to the applied stress at 932°C when the stress was quite high. At low stresses, the precipitate appeared random. At 1204°C the precipitate was quite coarse but was oriented normal to the stress direction. Deformation was accompanied by recrystallization and grain growth in some areas as well as extensive substructure formation. A second heat of material which was supposedly similar with respect to fabrication history and interstitial content was found to be considerably stronger. This difference is ascribed to the fabrication procedure.

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