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AN INVESTIGATION OF THE HIGH-TEMPERATURE PROPERTIES OF THE AISI TYPE 502 STEEL

W. R. Martin
H. E. McCoy, Jr.

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ABSTRACT

The mechanical properties of the AISI type 502 steel have been investigated at both room and elevated temperatures. Properties such as bend, tensile, hardness, and environmental creep have been evaluated as a function of various heat treatments. It was found that the room-temperature strength is increased as the solution annealing temperature is increased. The dependence of the mechanical properties at 1200°F on the heat treatment is not as significant as at room temperature although similar trends are observed. Creep tests in air, argon, and an Ar-5CO-5CO_2 mixture at 1200°F indicate an increase in the creep strength of this material in air and the Ar-5CO-5CO_2 environments as compared with that observed in argon. Room-temperature bend tests indicate that the rupture ductility is seriously reduced if the carbon content exceeds 0.1 wt %. Compatibility tests on unstressed specimens in an Ar-5CO-5CO_2 mixture show that the oxidation rate of the AISI type 502 steel is approximately linear with time at 1200°F. However, the oxide formed in the same mixture at 1500°F is more protective with a rate-time dependence of 0.73.

As a result of these studies, it was concluded that the AISI type 502 steel is unsatisfactory for service at 1200°F in an environment composed primarily of helium with small partial pressures of CO and CO_2.

INTRODUCTION

Although low-alloy steels are not usually thought of as being suitable for high-temperature nuclear applications, these metals fulfill a valuable role as structural materials in cooler parts of a nuclear reactor. The cost and availability of steels, coupled with their ease of fabricability, good weldability, and high strength make them desirable for use in any application where the service conditions are not too stringent. Both the loss of strength at elevated temperatures and the chemical incompatibility of low-alloy steels with their environment are factors which frequently yield these materials unsatisfactory. However, moderate alloy additions can be made to increase their range of usefulness. For example, chromium additions serve to increase the oxidation resistance of iron-base alloys. Hence, it becomes necessary to choose a steel having a composition which yields the material suitable for a particular set of service conditions.

The following report is concerned with the proposed use of a 5% Cr–0.5% Mo-alloy steel for the top grid structure in the Experimental Gas-Cooled Reactor (EGCR). This material, referred to as AISI type 502 steel, would be used at a maximum temperature of 1200°F and in an environment composed of helium containing small amounts of hydrocarbons, carbon monoxide, carbon dioxide, water, hydrogen, oxygen, and nitrogen. It was felt that the primary problem associated with the use of this material under these service conditions was its chemical reactivity with these impurities and the resultant effect of such reactions on its mechanical properties. Tests were run to determine the compatibility of the AISI type 502 steel with Ar-CO-CO_2 mixtures in the absence of stress. Stress-rupture tests were run in argon, air, and an Ar-5CO-5CO_2 mixture to determine the effects of these environments on the mechanical properties of the alloy. The effect of carbon on the rupture ductility of this alloy was determined since either carburization or decarburization may occur as the CO-CO_2 ratio varies during reactor operation. A series of heat-treating experiments was performed to determine the variation in properties of the alloy as the heat treatment is altered. Several heat treatments were chosen which yielded structures representative of those obtained by welding and other fabrication procedures. The following report is a summary of the findings of these experiments.

EXPERIMENTAL DETAILS

Test Material

All of the studies described in this report were carried out with one heat of the AISI type 502 steel. The composition of this alloy is given in Table 1. This material was received in the form of 0.0625 and 0.125-in.-thick sheets.

Table 1. Composition of Test Material in Weight Percent

C	Mn	P	Si	S	Cr	O	N	Mo	Fe
0.065	0.53	0.009	0.38	0.015	4.76	0.015	0.017	0.55	Balance

Stress-Rupture Tests

The specimens used in these tests were prepared from the 0.0625-in.-thick sheet. The specimens had a gage width of 0.500 in. and a 4-in.-gage length. Since many environments alter the mechanical properties of metals through surface reactions, sheet specimens were chosen because of their large surface area-to-volume ratio.

The environment surrounding the specimens during stress-rupture tests was controlled by means of a test chamber similar to that shown in Fig. 1. The test chamber is a type 310 stainless steel tube about which is placed a Nichrome wire heating element. The joint between the extension rod and test chamber is made with U-cup pressure-vacuum seals. These seals make it possible to maintain vacuum or pressure within the test chamber and not cause significant resistance to movement of the extension rods. Water jackets are provided at both the top and bottom of the chamber to cool the seals. Openings are made in the top of the chamber for bringing out four thermocouples for measuring the temperature of the test specimen. Other thermocouples are placed in wells which extend into the test chamber so that they are not influenced by the environment in the chamber.

The test components are assembled and the system leak checked to minimize the possibilities of the test environment being contaminated during the test. To reduce the effects which a particular environment may have upon the properties of a specimen before the stress is applied, all tests except those run in air were heated to the test temperature under a pressure of less than 1 μ. After the desired temperature was reached, the test environment was admitted and the load applied. Tests in the Ar-5CO-5CO_2 mixture were run in flowing environments since these gases react with the chamber walls and the specimens. Sufficient flow rates were used to ensure an ample supply of gas to the specimen. Tests in an argon environment were run under static conditions.

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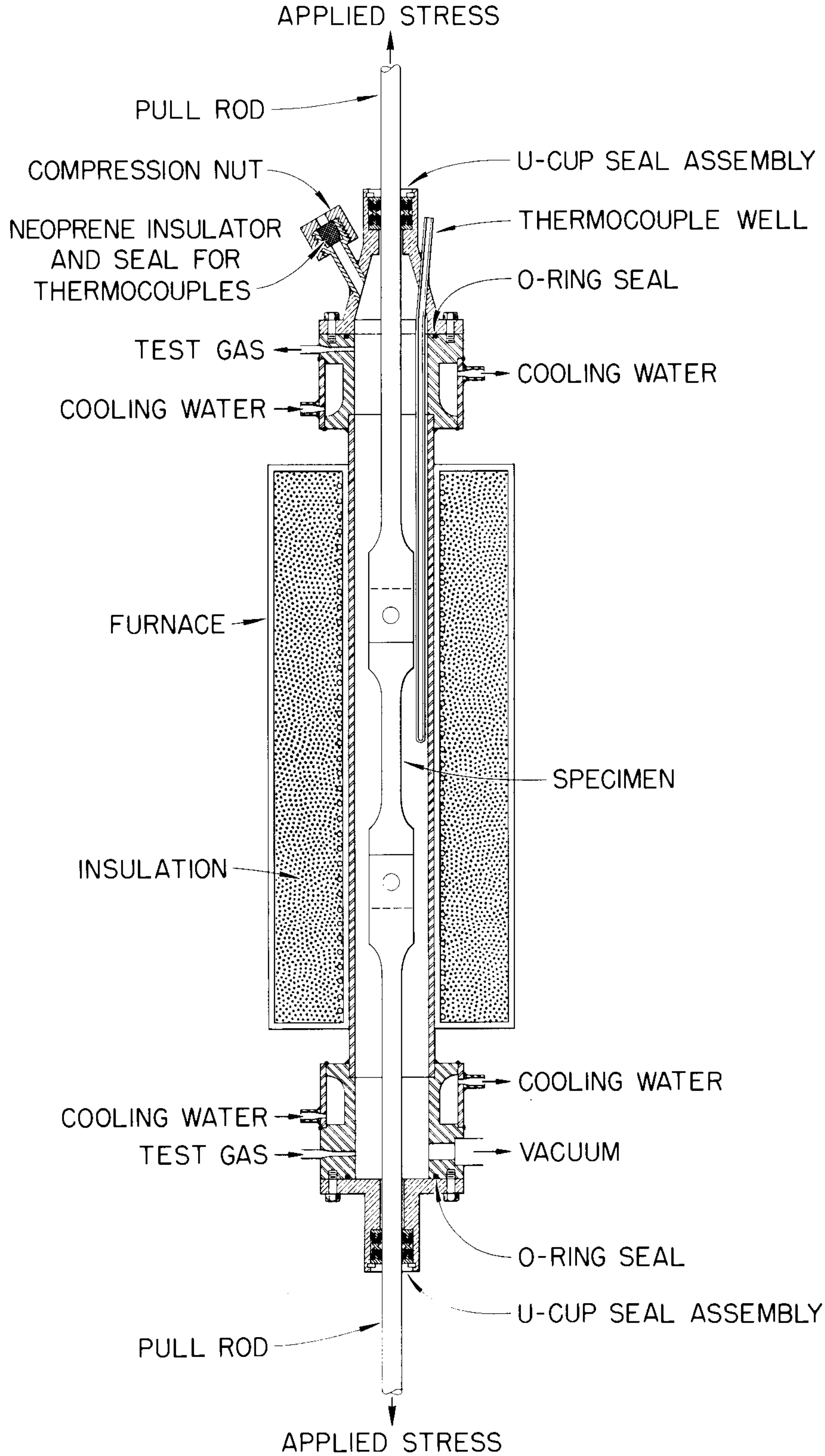

Fig. 1. Chamber for Environmental Stress-Rupture Tests at Elevated Temperatures.

Although high-purity argon was used, zirconium strips were suspended near the test specimen to act as a getter for further purification of the test environment.

Tensile Tests

The equipment used in running tensile tests at elevated temperatures is shown in Fig. 2. A test chamber similar to the one used for stress-rupture tests is suspended on the tensile machine. The experimental procedures for maintaining control of the test environment are similar to those used for environmental creep testing. The test specimens were identical to those used in stress-rupture tests. A hydraulic tensile machine was used for carrying out the tests. All tests were run at a deformation rate of approx 0.05 in./min.

Room-Temperature Bend Tests

The specimens used in these tests were nominally 1.00 x 1.25 x 0.0625 in. They were given various heat treatments and were tested in the apparatus shown in Fig. 3. The specimens were supported at two points, 0.75 in. apart and were centrally loaded using a tensile machine. A deflectometer was used to measure the deflection of the specimen and a load-deflection curve was recorded. Shear and tensile stresses are produced in the specimen during bending which follow the classical elastic-stress distribution pattern. Several parameters are conventionally used to describe the bend properties of a material depending upon whether it is brittle or reasonably ductile. Since the specimens in the present investigation range from very brittle to very ductile, the data will be represented by proportional limit values calculated from

$$S = \frac{Mc}{I}$$

where M is the maximum moment, c is the distance from the neutral axis to the outermost fiber, and I is the moment of inertia of the cross section; the deflection at failure; and the apparent ultimate strength values. The third method of representation is useful only on a comparative basis since equations which hold for elastic behavior are used in calculating the stresses when the beam is actually deforming plastically. All of the stress values presented for bend tests have a maximum range of uncertainty of approx ± 4% which is

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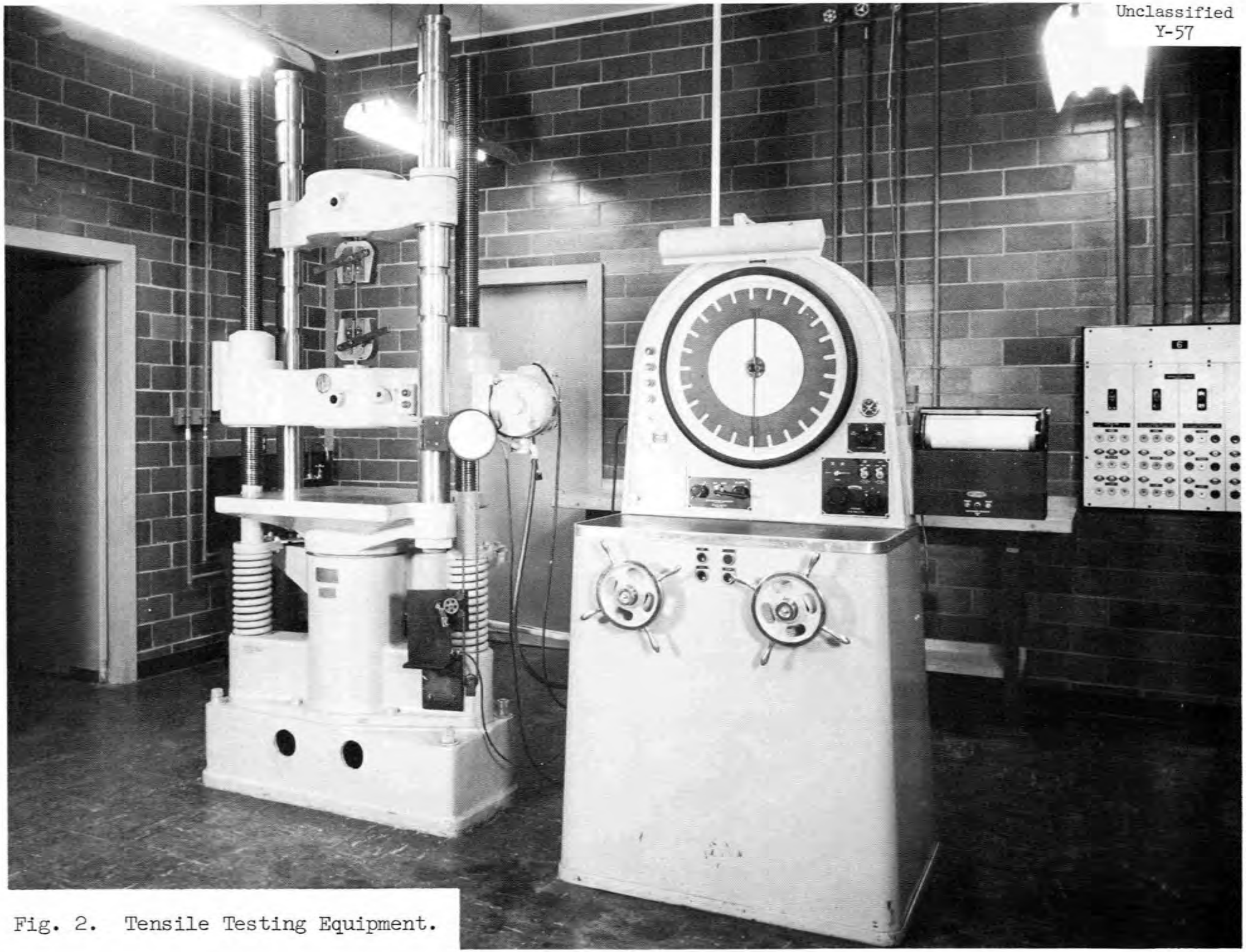

Fig. 2. Tensile Testing Equipment.

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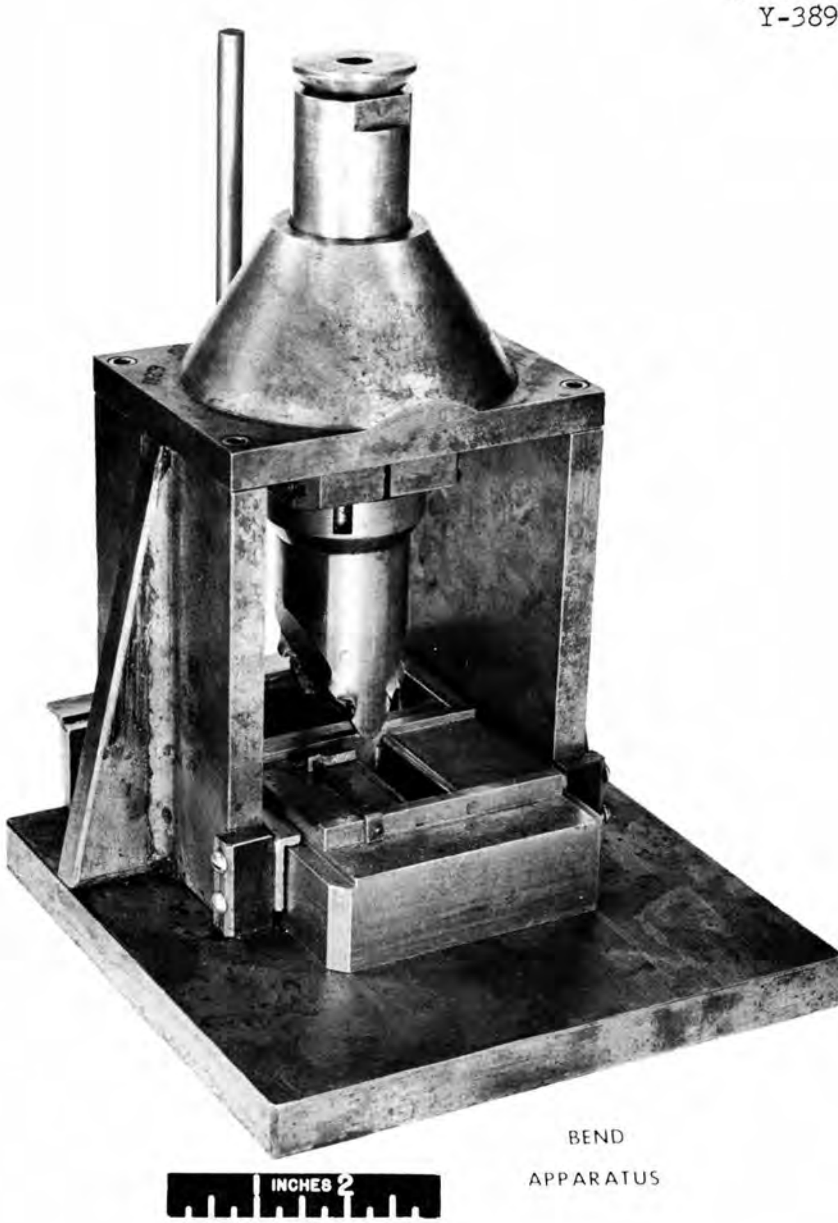

Fig. 3. Apparatus for Room-Temperature Bend Tests.

due principally to an uncertainty of ± 0.001 in. in the measurement of the thickness of the specimens.

Hot-Hardness Measurements

The hot-hardness measurements were performed using the instrument, shown in Fig. 4, which was developed by the Metallography Group. This instrument consists essentially of a 20-in. vertical capacity Rockwell tester with a direct reading dial gage to indicate Rockwell or superficial hardness readings and a furnace specifically designed for hot-hardness testing. The specimen and the indenter are protected during testing by an argon environment.

The specimens investigated were heated continuously at a heating rate of 7°F/min. Two to four hardness measurements were made at 90°F intervals, and the time of application of the load was 15 sec. Room-temperature hardness values obtained with the hot-hardness tester on all specimens investigated were essentially the same as those obtained with a conventional hardness tester.

Unstressed Oxidation Studies

The oxidation characteristics of the AISI type 502 steel were studied in the apparatus shown in Fig. 5. The weight change of the test specimen is determined by measuring the length of the spring as a function of time. The spring is made of 0.002-in.-diam piano wire and has a spring constant of 0.0769 g/in. A water jacket is provided to maintain the temperature of the spring constant. The test gas is passed over the specimen at a rate of approx 0.05 ft^3/hr. The test specimens are nominally 2 x 0.6 x 0.010 in. and weigh approx 1.5 g.

EFFECT OF HEAT TREATMENT ON THE MECHANICAL PROPERTIES OF AISI TYPE 502 STEEL

Hardness

The mechanical properties of this alloy can be altered greatly by heat treatment. To determine how these properties vary, a series of heat treatments was given to the AISI type 502 steel. These heat treatments with the corresponding hardness values are shown in Table 2. The effect of the solution

Fig. 4. Hot-Hardness Tester.

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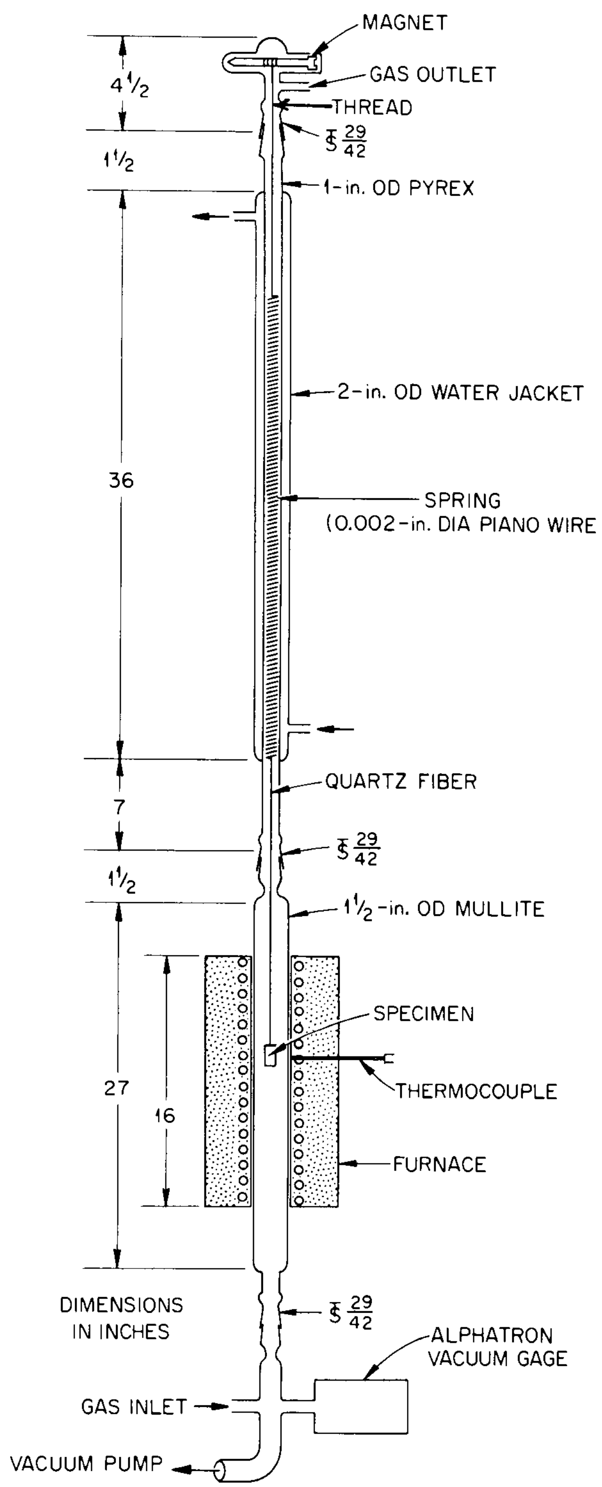

Fig. 5. Apparatus for Measuring Gas-Metal Reaction Rates.

Table 2. Effect of Heat Treatment on the Hardness of AISI Type 502 Steel

Heat Treatment		Hardness Values			
		Room Temperature			1200°F
No.	Type	Rockwell	DPH	15N	15N
1	As received (no further treatment).	87B	102	62	31
2	Solution heat treated for 1 hr at 1900°F in argon, cooled to room temperature, reheated to 1350°F in argon, and after 1 hr cooled to room temperature at a maximum rate of 500°F/min.	94B	121	--	--
3	Solution heat treated for 1 hr at 1700°F in argon, cooled to room temperature, reheated to 1350°F in argon, and after 1 hr cooled to room temperature at a maximum rate of 500°F/min.	91B	106	62.5	35
4	Solution heat treated for 1 hr at 1700°F in argon, cooled to room temperature at a maximum rate of 1000°F/min.	27C	127	--	--
5	Solution heat treated at 1700°F in argon, furnace cooled to 1350°F at a rate of 15°F/min, and held for two hours — then cooled to room temperature at a maximum rate of 500°F/min.	70B	83	51.5	23
6	Solution heat treated at 1900°F in argon for 1 hr, cooled to room temperature at a maximum rate of 1000°F/min.	35C	161	78	60
7	Solution heat treated for 1 hr at 1900°F in argon, furnace cooled to 1350°F, and held for 2 hr — then cooled to room temperature at a maximum rate of 500°F/min.	69B	89	53.5	25
8	Heat treated at 1500°F in argon for 1 hr, water quenched to room temperature.	17C	117	--	--
9	Solution heat treated at 1700°F in argon for 1 hr, water quenched to room temperature.	38C	172	--	--
10	Solution heat treated at 1900°F in argon for 1 hr, water quenched to room temperature.	39C	176	--	--

heat-treatment temperature is readily seen by comparing heat treatments Nos. 4 and 6 and heat treatments Nos. 8, 9, and 10. There is an appreciable increase in hardness as the solution heat-treatment temperature is raised from 1500 to 1900°F. The effect of cooling rate from the solution temperature is noted by comparing treatments Nos. 5, 4, and 9 and treatments Nos. 7, 6, and 10. The slower cooling rate results in a considerable decrease in the hardness of the base metal. Typical microstructures resulting from each heat treatment are shown in Figs. 6 through 10.

Hardness values were obtained for six heat treatments as a function of temperature. These values are shown in Fig. 11. Of the six heat treatments tested, all except the as-received material began losing appreciable strength at about 840°F. An appreciable reduction in the strength of the as-received material is noted at temperatures exceeding 660°F. On the basis of these hardness tests and metallographic examinations, heat treatments Nos. 1 through 5 were selected for further mechanical and physical property tests. The criterion for this selection was that these treatments represented the microstructures normally found in and about the weld area of a structural AISI type 502 steel welded member.

Tensile Properties

Tensile data were obtained at room temperature in an air environment and at 1200°F in a vacuum of less than 50 μ. These data are shown in Table 3. The differences in mechanical properties of the alloy at room temperature are due to different heat treatments. These differences are considerably smaller at the 1200°F testing temperature.

Correlation of Tensile Properties and Hardness Tests

Many investigators[1,2,3] have attempted to correlate the results of creep, short-time tensile, and short-time hardness tests. The effect of environment

[1]F. R. Larson and J. Miller, "A Time-Temperature Relationship for Rupture and Creep Stress," Trans. ASME 74, 765 (1952).

[2]J. H. Holloman and L. D. Jaffe, "Time-Temperature Relations in Temporary Steel," Trans. ASME 162, 223 (1945).

[3]E. E. Underwood, "Creep Properties from Short-Time Tests," Materials & Methods 45, 127 (April, 1957).

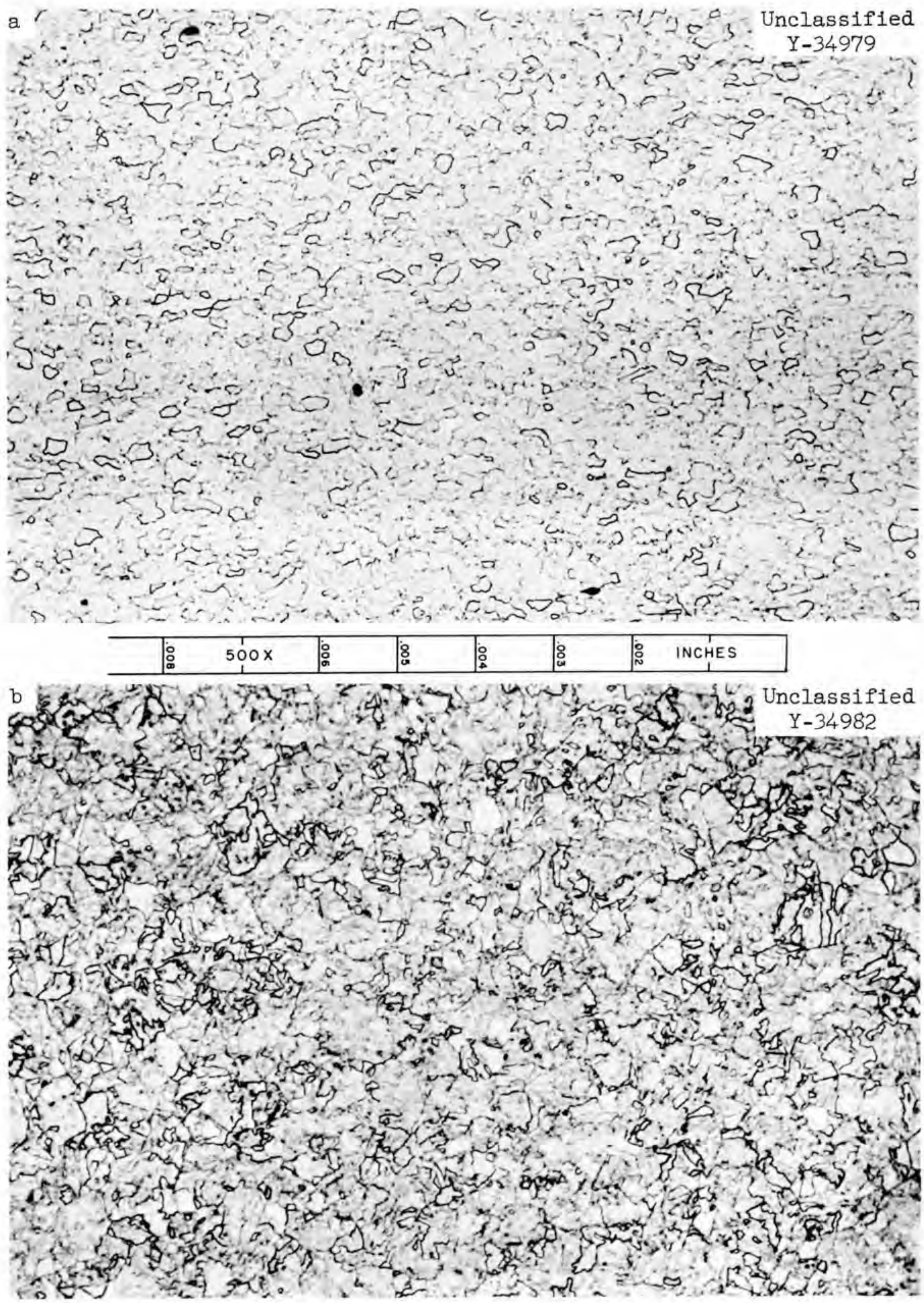

Fig. 6. Typical Microstructures of AISI Type 502 Steel. (a) Treatment No. 1 (b) Treatment No. 2.

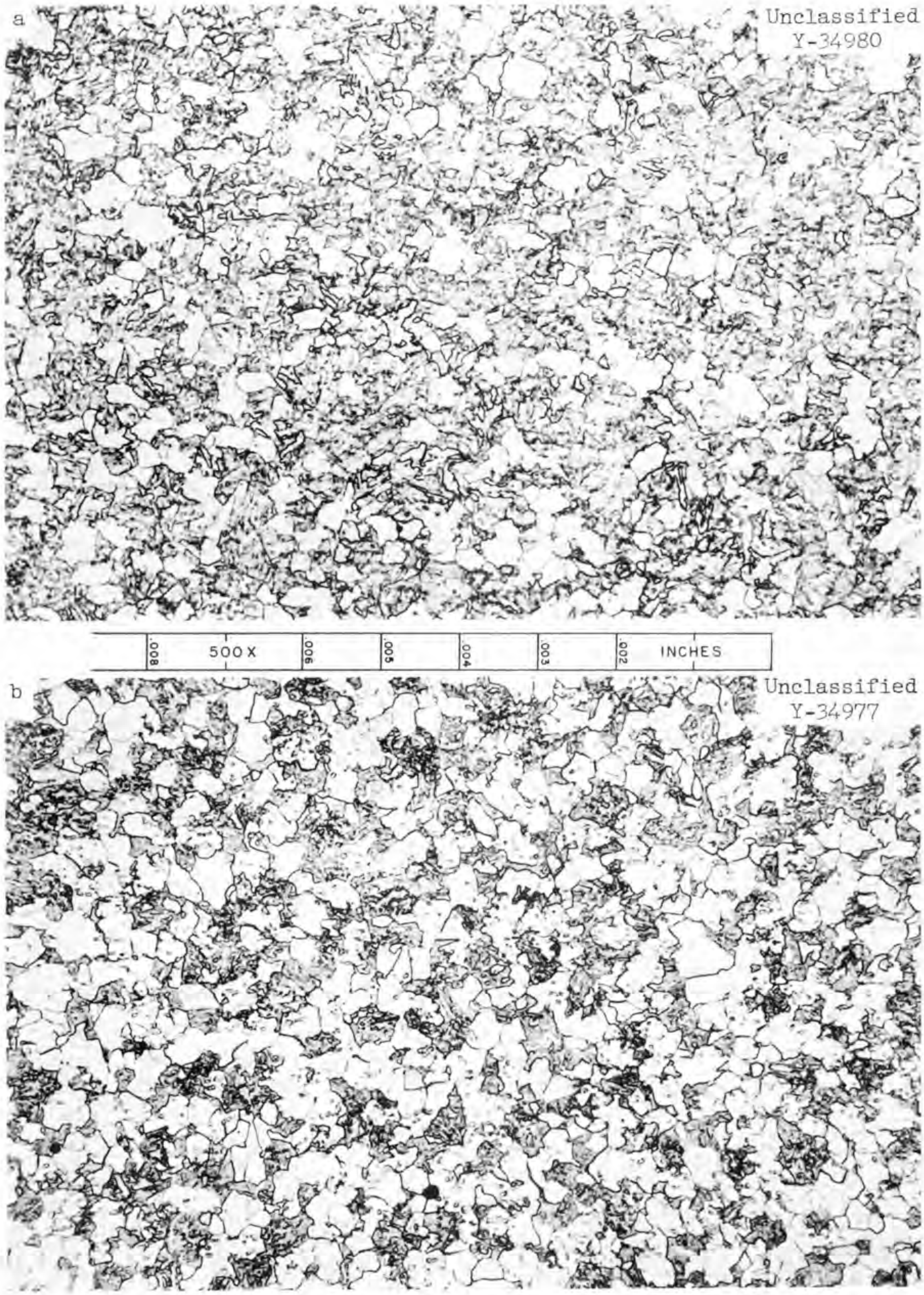

Fig. 7. Typical Microstructures of AISI Type 502 Steel. (a) Treatment No. 3 (b) Treatment No. 4.

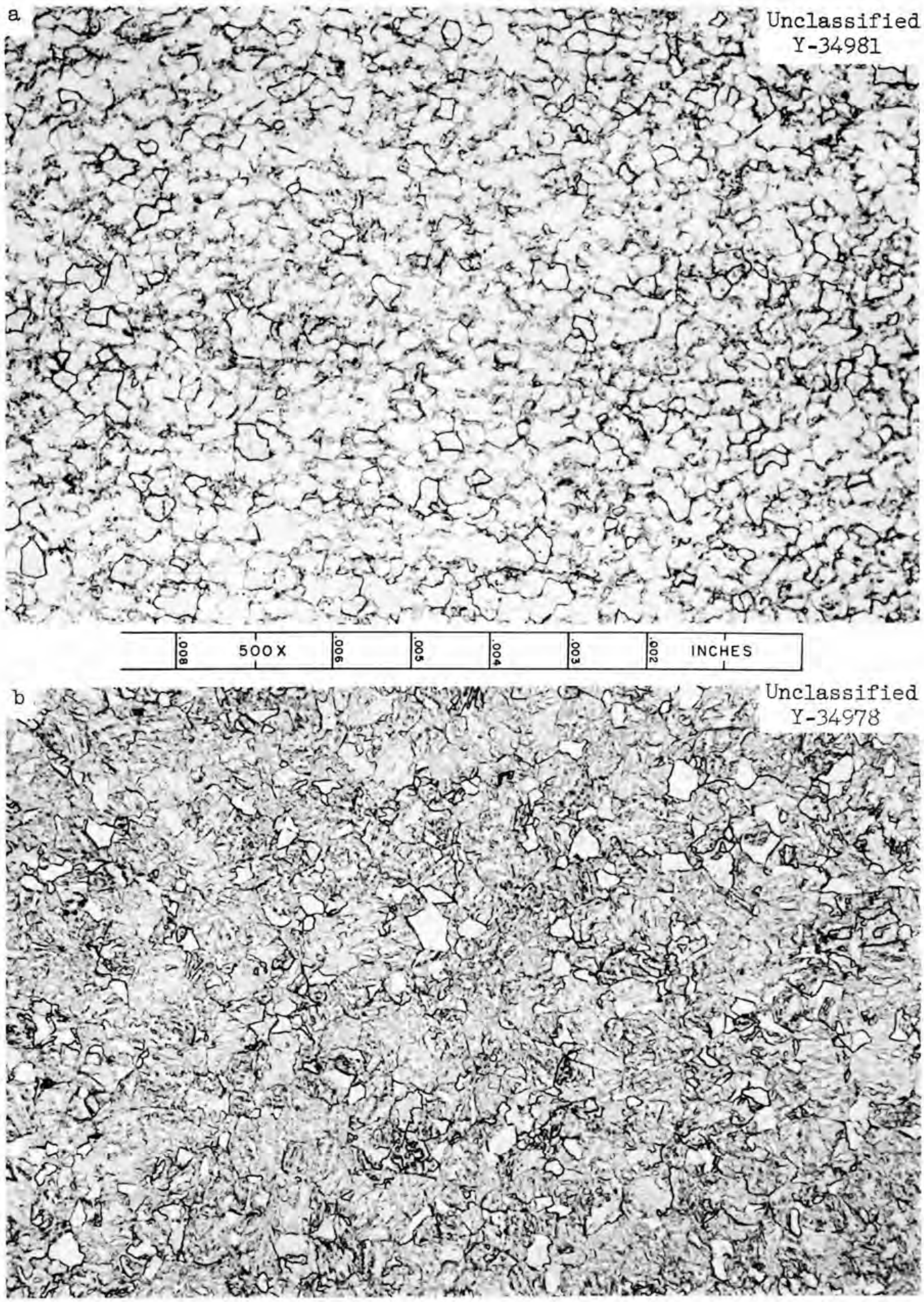

Fig. 8. Typical Microstructures of AISI Type 502 Steel. (a) Treatment No. 6 (b) Treatment No. 7.

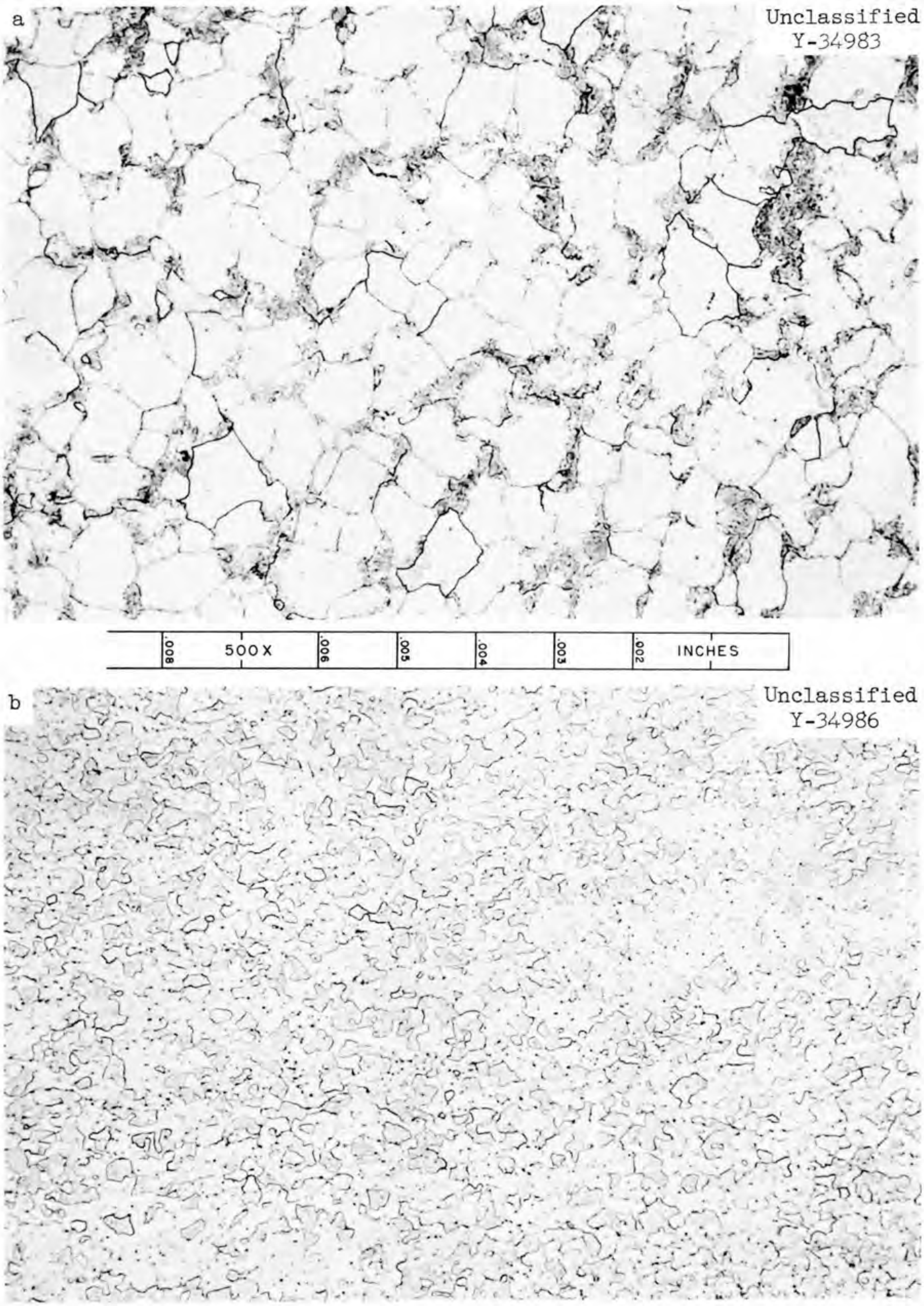

Fig. 9. Typical Microstructures of AISI Type 502 Steel. (a) Treatment No. 7 (b) Treatment No. 8.

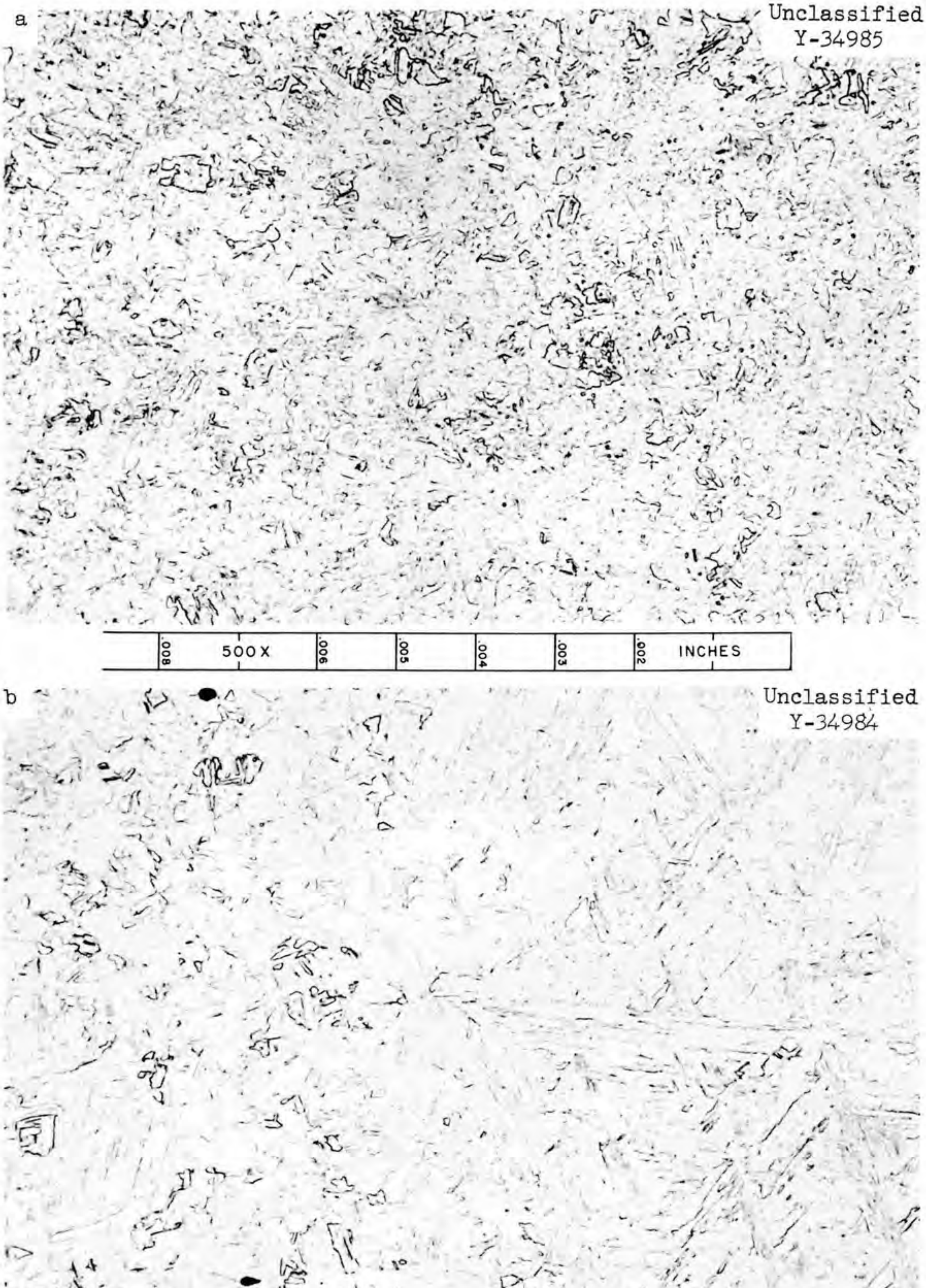

Fig. 10. Typical Microstructures of AISI Type 502 Steel. (a) Treatment No. 9 (b) Treatment No. 10.

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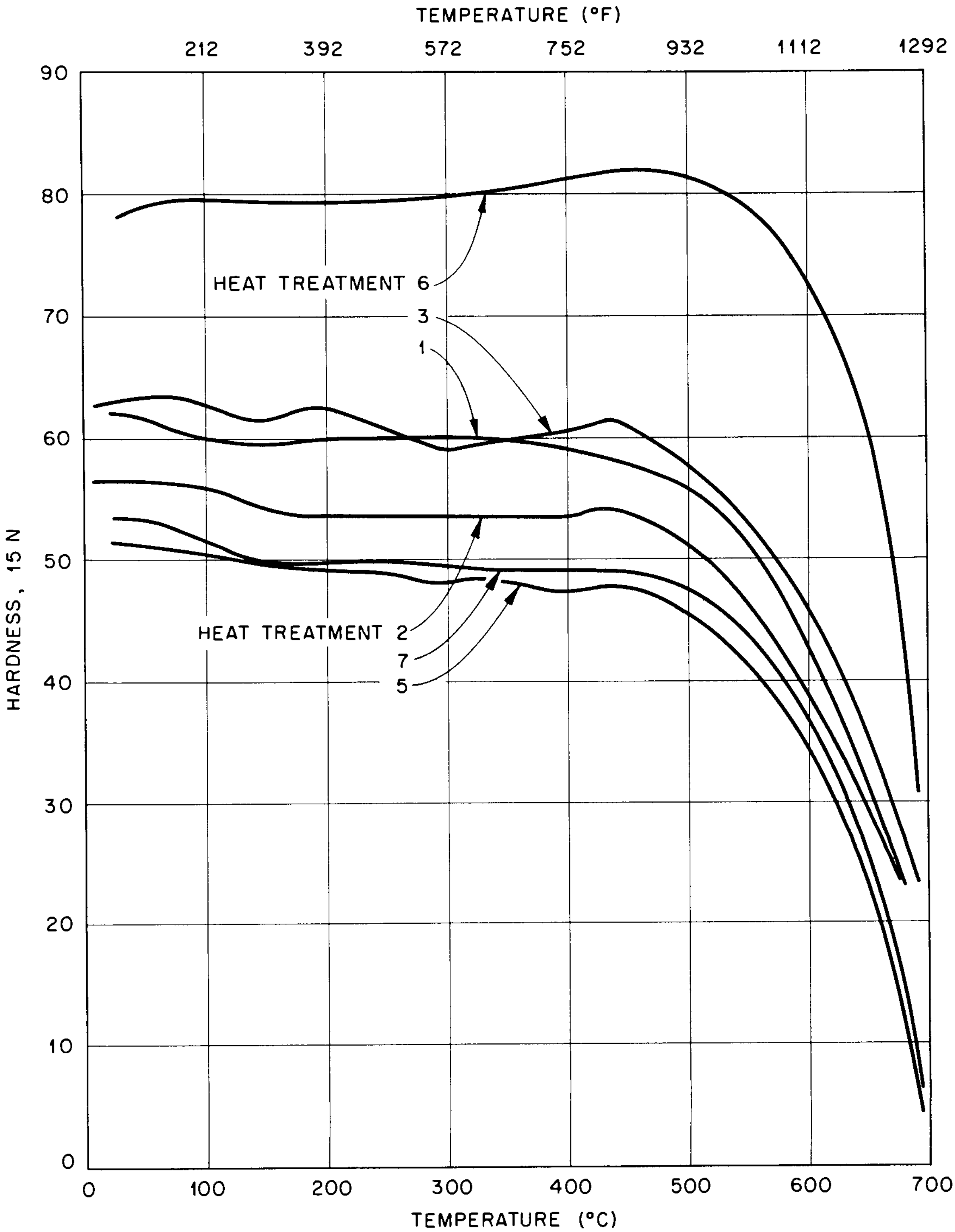

Fig. 11. Hardness at Temperature of AISI Type 502 Steel as a Function of Heat Treatment.

Table 3. Tensile Properties of AISI Type 502 Steel

Specimen Size — 0.5 x 0.060 x 4.0 in. Gage Length

Heat Treatment Type	Tensile Properties: Room Temperature: Yield Strength, 0.2% Offset (psi)	Room Temperature: Tensile Strength (psi)	Room Temperature: Ductility, % in 2 in.	1200°F: Yield Strength 0.2% Offset (psi)	1200°F: Tensile Strength (psi)	1200°F: Ductility, % in 2 in.
1	59,215	77,415	23	23,595	26,420	40
2	70,130	85,475	9	25,430	30,580	30
3	54,690	78,750	28	27,970	30,030	36
4	88,890	144,855	12	26,985	30,120	37
5	29,230	62,770	42	13,120	17,645	61

on the mechanical properties of a given alloy cannot normally be determined from these correlations. The effect of heat treatment on yield and tensile strengths of the AISI type 502 steel at room temperature and the effect on hardness are shown in Fig. 12. There is appreciable scatter about curves drawn to represent the best straight-line correlation. Similar plots of data obtained at 1200°F failed to show any correlation. An attempt was made to correlate the hardness, tensile, and creep data using the Larson-Miller parameters, and those plots are shown later in this report.

Properties in Bending

The proportional limit in bending for each of the ten heat treatments is tabulated in Table 4. Note the limited agreement between the change in proportional limit in bending measured at room temperature and the hardness of each specimen at room temperature.

Creep Properties

Creep tests were performed in environments of air, an Ar-5CO-5CO_2 mixture, and pure argon. Only the data obtained in argon and shown in Table 5 will be considered in this portion of the report. Two stresses and one temperature level were investigated as the experimental effort was directed toward determination of the effect of environment on materials with various heat treatments.

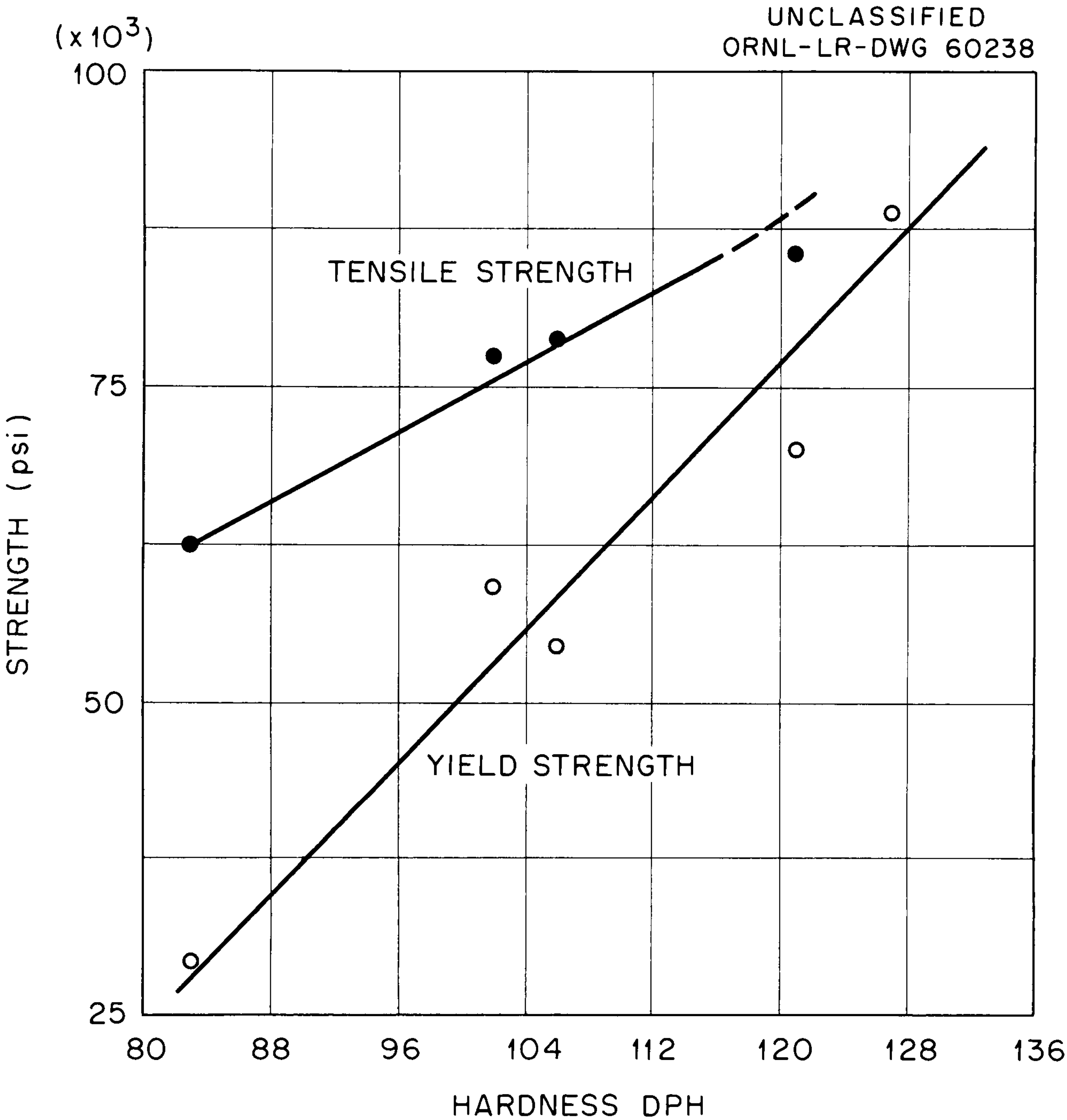

Fig. 12. Correlation of Room Temperature Hardness with Tensile Properties.

Table 4. Proportional Limit in Bending for AISI Type 502 Steel at Room Temperature

Heat Treatment*	1	2	3	4	5	6	7	8	9	10
Bend Proportional Limit, psi x 10^3	67	88	82	82	41	157	35	60	175	165
Hardness, DPH	102	121	106	127	83	161	89	117	172	176

*Numbers refer to heat treatments listed in Table 2.

Figure 13, a typical creep curve, is shown to define several terms. True elongation is defined as that elongation at the end of secondary creep. Total elongation is the elongation at fracture which is the sum of true elongation and the elongation that occurs during the ternary stage of creep. The true elongations for specimens which have been given heat treatments Nos. 1 through 5 are shown in Table 5. The true elongation of the as-received material far exceeds the other type heat treatments. This fact is further illustrated if the ratio of the true elongation to total elongation is plotted versus the total elongation as shown in Fig. 14. This points out that the differences in heat-treatment operations Nos. 2, 3, 4, and 5 do not affect the relationship between amount of strain during secondary creep for a given total elongation. However, the as-received material is somewhat different in that the true elongation is greater for a given total elongation.

The changes in rupture characteristics at 1200°F of the alloy brought about by heat treatment are significant as shown by the data in Table 5. Solution annealing at 1700 and 1900°F increases the creep strength over that observed for the as-received material. The relative effects of these two annealing temperatures, as well as cooling rates and tempering treatments, are not apparent from the limited number of tests which have been run.

The correlation of creep, tensile, and hardness data, using the Larson-Miller parameter, is shown in Fig. 15. More tests over various stress ranges for each heat treatment are needed before the scatter about the curve can be interpreted properly.

Table 5. Creep Properties of AISI Type 502 Alloy at 1200°F Tested in Argon

Heat Treatment No.	at 6000 psi					at 6500 psi				
	Rupture Time (hr)	Creep Rate (%/hr)	Total Elongation (%)	Elongation Attributable to Cracks (%)	True Elongation (%)	Rupture Time (hr)	Creep Rate (%/hr)	Total Elongation (%)	Elongation Attributable to Cracks (%)	True Elongation (%)
1	138	0.150	50.0	37.1	12.9	165	0.176	40.6	28.1	12.5
2	---	---	--	--	--	425	0.024	26.6	21.8	4.8
3	308	0.030	42.2	39.2	3.0	280	0.0314	39.8	36.5	3.3
4	359	0.030	36.3	34.7	1.6	290	0.056	40.6	35.1	5.5
5	412	0.036	39.8	37.2	2.6	559	0.022	43.0	39.5	3.5

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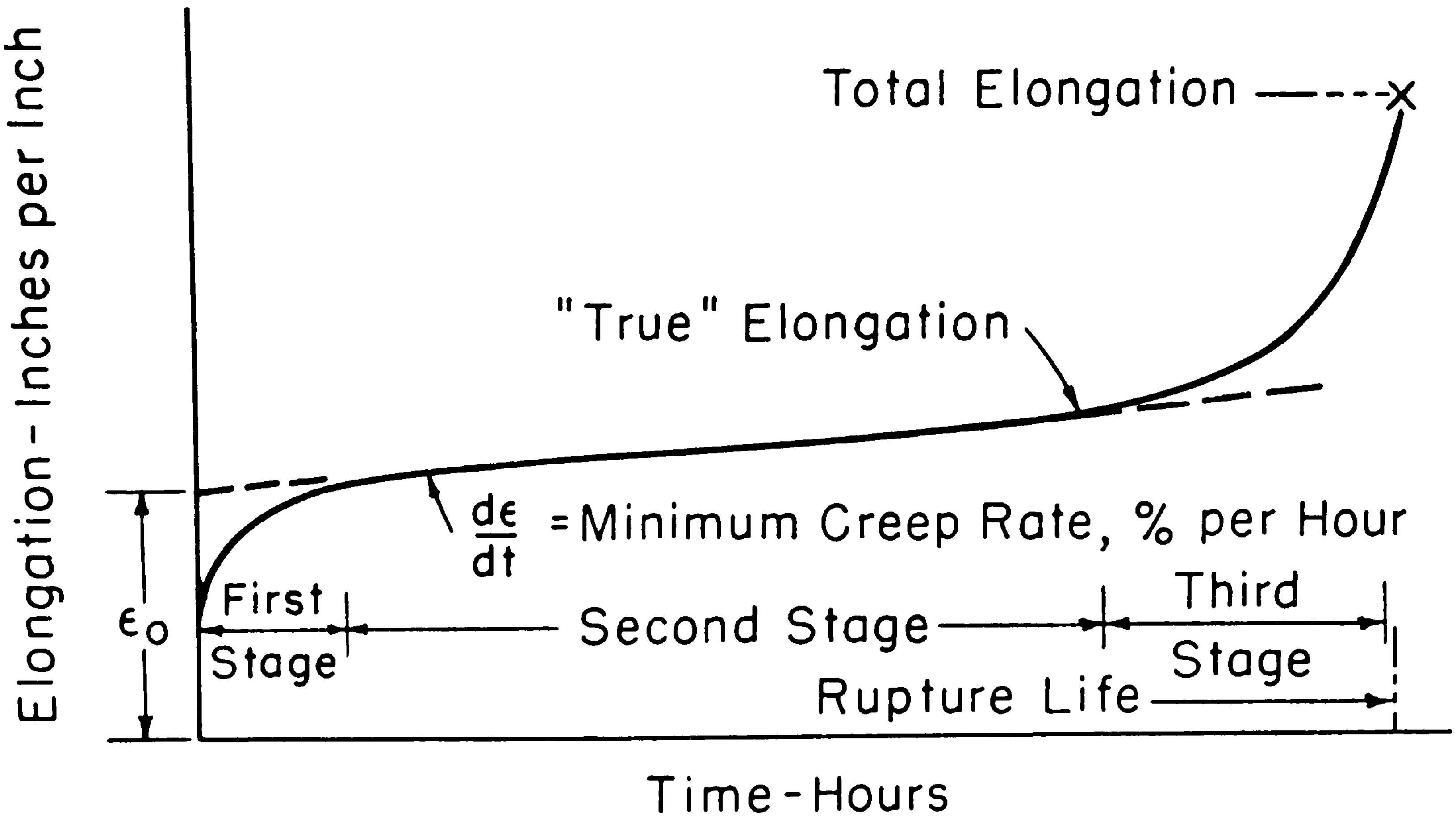

Fig. 13. Stress-Rupture Curve Showing Various Quantities.

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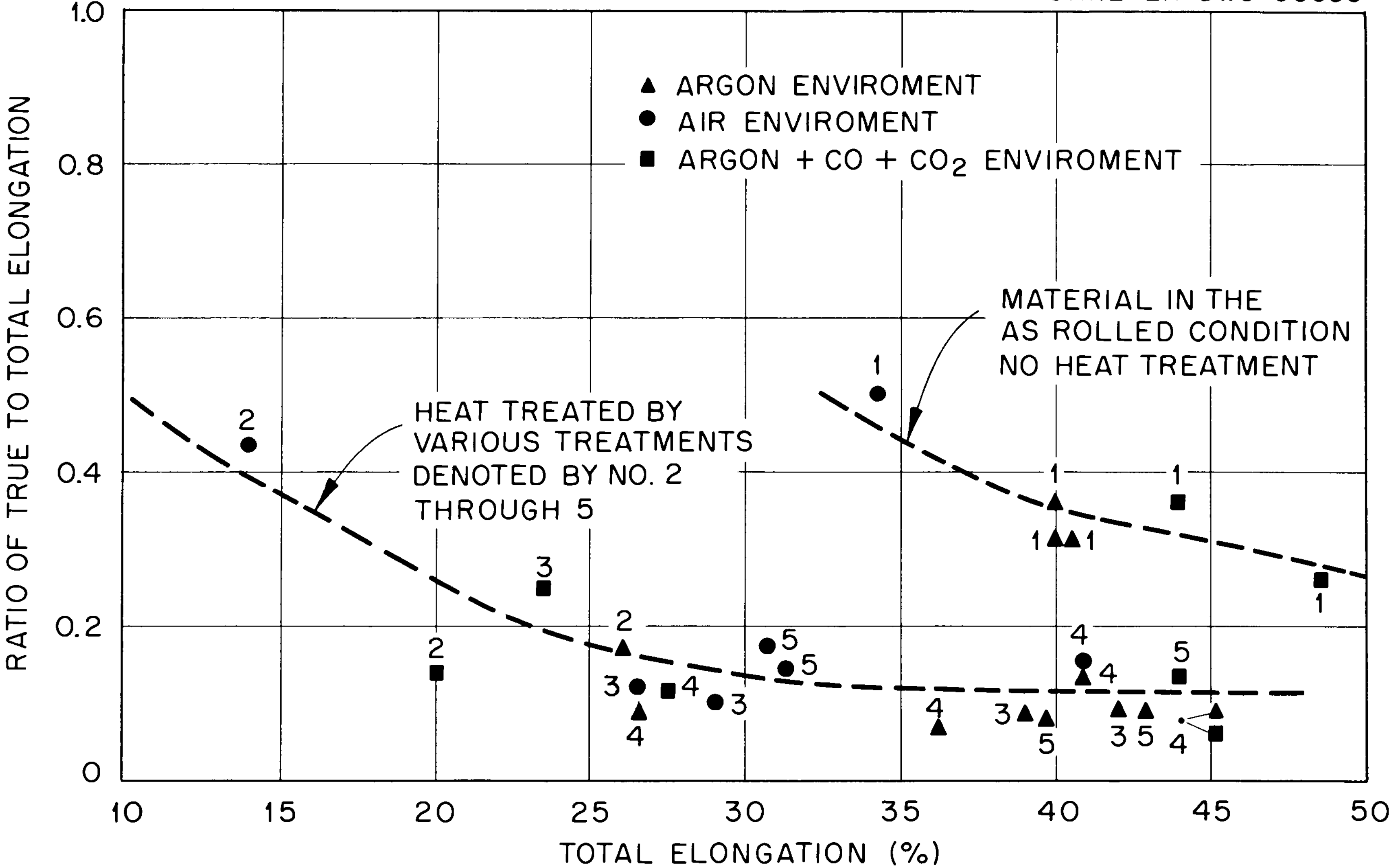

Fig. 14. Relationship Between Percent Elongation at the Beginning of the Final Stage of Creep and the Percent Elongation at Fracture.

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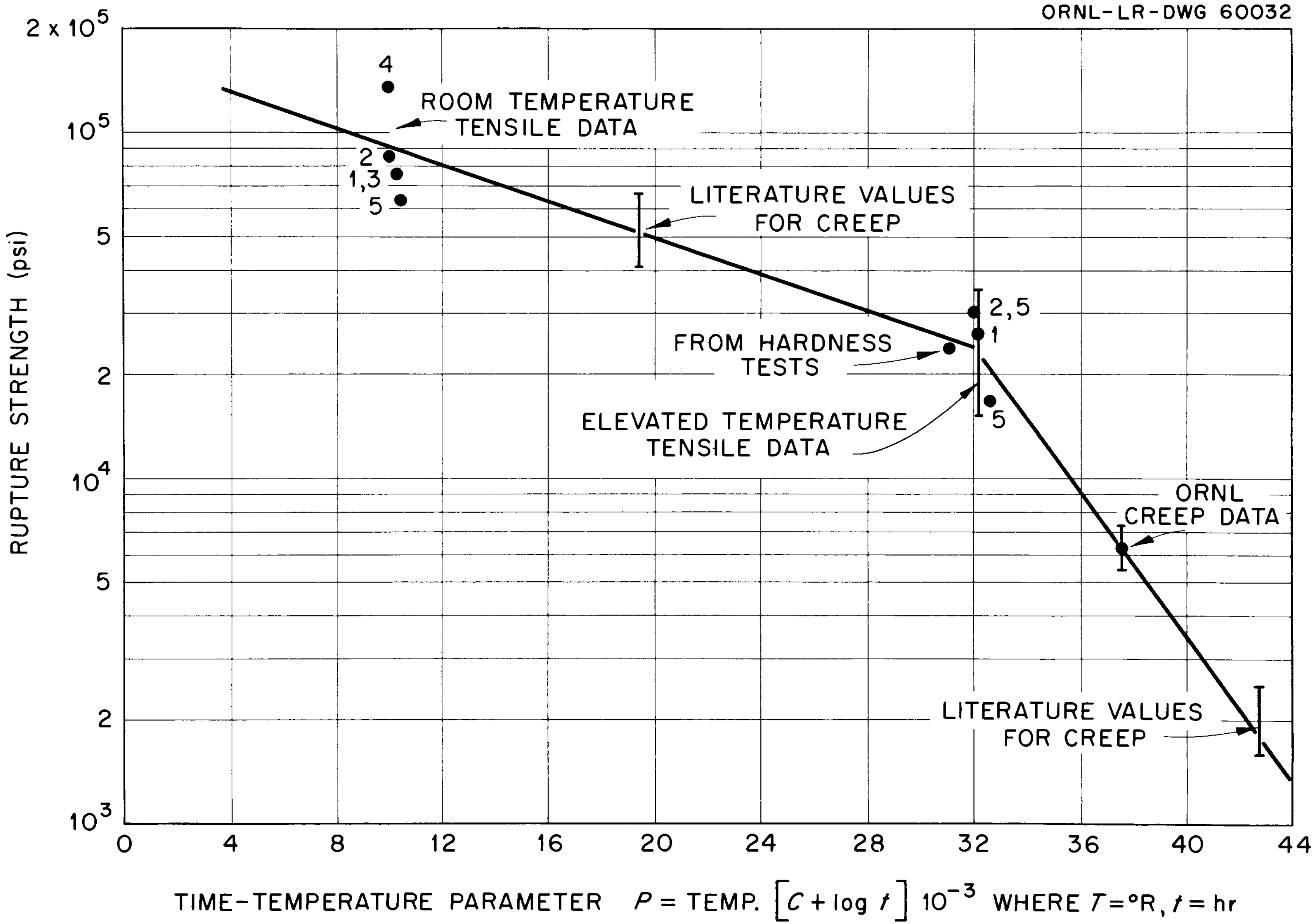

Fig. 15. Relationship Between Stress, Time, and Temperature for AISI Type 502 Steel.

COMPATIBILITY OF AISI TYPE 502 STEEL WITH AIR, Ar-CO-CO_2 ENVIRONMENTS – STRESSED

The role of the atmosphere at high temperature has been the object of numerous investigations.[4,5] For comparison with the argon-creep data, tests were run in both air and Ar-5CO-5CO_2 environments. These results are tabulated in Table 6. It is noted that air and Ar-5CO-5CO_2 environments are generally strengthening compared to the stress-rupture data in argon environments found in Table 5. The values of total elongation found in Tables 7 and 8 indicate that the total elongation is reduced in an air environment and that this reduction is found almost entirely in third-stage creep. The role of oxidation during the formation and propagation of cracks is one which appears to strengthen the metal. Oxygen dissolved into the newly exposed base metal strengthens the metal in the vicinity of fissures and either prevents or delays the propagation of the crack. The difference in true elongation between air and argon tests for a given heat treatment is not large. Its significance is difficult to determine since the location of true elongation is itself difficult to accurately determine. Therefore, any minute differences are questionable, possibly due to evaluation of the true elongation.

Oxygen and nitrogen analyses of several of the test specimens are found in Table 9. The average amount of oxygen found in the fracture area of the specimens which contained several fissures has no apparent relationship with creep rate, but seems to be a function of time in test as shown in Fig. 16. These analyses show no increase in nitrogen content. The carbon analyses of the 6500-psi stress series are shown in Table 10. It is noted that there is no apparent relationship between percent carbon and time in test; however, the higher carbon content results in lower creep rate.

The after-test appearances of the test specimens are compared in Fig. 17. The specimens tested in argon were bright or only slightly discolored. Relatively thick oxide films were formed on the specimen tested in the Ar-5CO-5CO_2 mixtures. Very thick nonadherent films were formed on the specimens tested in air and the cross section of the test specimen was reduced significantly.

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[4] R. F. Hehemann and G. Mervin Ault (eds.) High Temperature Materials, p. 429–97, John Wiley and Sons, New York, 1959.

[5] H. E. McCoy, Jr. and D. A. Douglas, Jr., "Effect of Environment on the Creep Properties of Type 304 Stainless Steel at Elevated Temperatures," p. 748 in Proceedings of the US/UK Meeting on the Compatibility Problems of Gas-Cooled Reactors, Held at Oak Ridge National Laboratory, Feb. 24–26, 1960, TID-7597, Book 2 (March 3, 1961).

Table 6. Creep Properties of AISI Type 502 Steel at 1200°F in Air and Ar-5CO-5CO_2 Environments

Test No.	Environment	Heat Treatment	Stress (psi)	Rupture Time (hr)	Creep Rate (%/hr)
408	Air	1	6500	141.2	0.125
428	Ar-5CO-5CO_2	1	6500	155.7	0.164
426	Air	2	6500	686.2	0.016
561	Ar-5CO-5CO_2	2	6500	839.7	0.016
425 or 678	Air	3	6500	259.7	0.036
515	Ar-5CO-5CO_2	3	6500	429.1	0.030
441	Air	4	6500	309.0	0.056
484	Ar-5CO-5CO_2	4	6500	336.8	0.033
463	Air	5	6500	318.0	0.056
442	Ar-5CO-5CO_2	5	6500	227.3	0.058
496	Air	1	6000	232.0	0.129
739	Air	1	6000	170	0.100
526	Air	3	6000	586.6	0.011
755	Ar-5CO-5CO_2	3	6000	338.8	0.036
587	Air	4	6000	462.9	0.014
665	Ar-5CO-5CO_2	4	6000	925.9	0.010
559	Air	5	6000	306.5	0.054
817	Ar-5CO-5CO_2	5	6000	332.4	0.024

Table 7. Contribution of Cracking to Total Elongation for AISI Type 502 Steel at 6000 psi, 1200°F

Test No.	Type of Heat Treat-ment	Environ-ment	Total Elongation at Fracture (ϵ_t, %)	Elongation During Tertiary Creep (ϵ_c, %)	True Elongation (ϵ_t-ϵ_c,%)
496	1	Air	48.43	35.73	12.50
598	1	Argon	50.00	37.10	12.90
526	3	Air	26.55	23.35	3.20
755	3	Ar-5CO-5CO_2	35.93	29.73	6.20
624	3	Argon	42.18	39.18	3.00
583	4	Argon	36.33	34.68	1.65
587	4	Air	26.60	24.10	2.50
665	4	Ar-5CO-5CO_2	37.50	33.30	4.20
628	5	Argon	39.84	37.24	2.60
559	5	Air	31.25	26.45	4.80
817	5	Ar-5CO-5CO_2	42.18	39.78	2.40

EFFECT OF CARBON ON THE DUCTILITY OF THE AISI TYPE 502 STEEL

The gain or loss of carbon by a ferrous alloy can have large effects on its mechanical and physical properties. For example, both the maximum hardness and the hardenability can be increased by the addition of carbon and similarly decreased by the removal of carbon. Because of the proposed use of this material at elevated temperatures in environments composed of an inert carrier gas and small amounts of carbon monoxide and carbon dioxide, the effect of carbon on the room ductility was investigated.

Several specimens of the AISI type 502 steel were annealed in carbon monoxide at 1700°F for various lengths of time to increase their carbon contents. They were subsequently annealed for one hour in argon for homogenization and were pulled into the cold zone of the furnace. Room-temperature bend tests were used to evaluate the ductility of each specimen. The results of these

Table 8. Contribution of Cracking to Total Elongation for AISI Type 502 Steel at 6500 psi, 1200°F

Test No.	Type of Heat Treatment	Environment	Total Elongation at Fracture (ϵ_t, %)	Elongation During Tertiary Creep (ϵ_c, %)	True Elongation (ϵ_t-ϵ_c, %)
427	1	Argon	40.62	28.12	12.50
408	1	Air	34.37	16.87	17.50
428	1	Ar-5CO-5CO_2	43.75	27.35	16.40
506	2	Argon	26.56	21.76	4.80
426	2	Air	14.06	8.06	6.00
561	2	Ar-5CO-5CO_2	20.31	17.21	3.10
448	3	Argon	39.84	36.54	3.30
425	3	Air	28.90	26.10	2.80
678	3	Air	26.55	23.95	2.60
515	3	Ar-5CO-5CO_2	23.50	17.50	6.00
440	4	Argon	40.62	35.12	5.50
441	4	Air	40.60	34.30	6.30
484	4	Ar-5CO-5CO_2	43.70	39.80	3.90
514	5	Argon	43.00	39.50	3.50
463	5	Air	30.31	24.41	5.90
442	5	Ar-5CO-5CO_2	43.75	37.85	5.90

tests are summarized in Table 11. Photomicrographs of the bend area of each specimen are shown in Figs. 18 through 23. These results illustrate the decrease in ductility of the AISI type 502 steel in the hardened condition as the carbon concentration is increased. This property may be problematic in the instance where repair welds have to be made which cannot be tempered. Although the present tests leave many variables uninvestigated, it is felt that the presence of a problem is illustrated which should be explored further before this material is used in environments containing carbon monoxide and carbon dioxide.

Table 9. Analysis for AISI Type 502 Steel Creep Specimens After Test at 6500 psi, 1200°F in Air

Heat Treatment No.	Wt % O_2	Wt % N_2	Creep Rate (%/hr)	Time of Test (hr)
1	0.091	0.014	0.0176	141
2	1.18	0.017	0.0155	686
3	0.15	0.015	0.0359	260
4	0.40	0.012	0.0579	309
5	0.80	0.008	0.0583	318
Before Test	0.015	0.017	--	--

Table 10. Carbon Analysis of AISI Type 502 Steel Tested in Ar-5CO-5CO_2 at 6500 psi, 1200°F

Heat Treatment	Wt % C	Creep Rate (%/hr)	Time (hr)
1	0.18	0.0164	155.7
2	0.17	0.0156	---
3	0.11	0.0296	429
4	0.15	0.0291	336
5	0.11	0.0675	237
Before Test	0.065	--	--

COMPATIBILITY OF AISI TYPE 502 STEEL WITH Ar-CO-CO_2 ENVIRONMENTS — UNSTRESSED

Because of the thick oxide films which were observed on creep specimens after testing in Ar-CO-CO_2 mixtures, several gas-metal compatibility tests were run in the absence of externally applied stress. A summary of the tests is given in Table 12, and the observed weight gain versus time curves are presented in Figs. 24 and 25. It was found that the reaction rates of all the tests could be represented by an equation of the form $\Delta W = Kt^n$, where ΔW is the weight increase in mg/cm^2, and t is the time in hours. The quantities K

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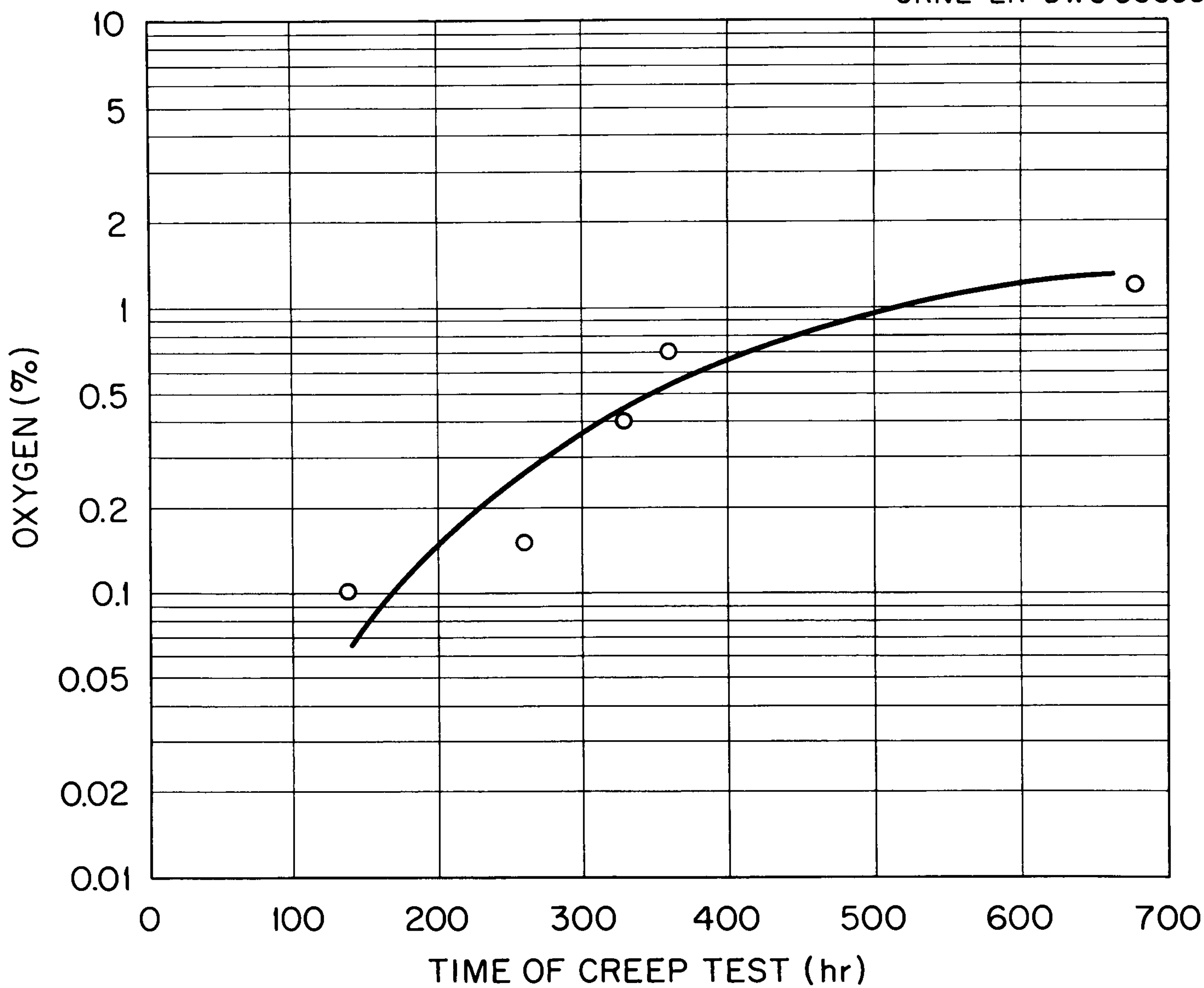

Fig. 16. Composition Change of AISI Type 502 Steel During Air Creep at 1200°F.

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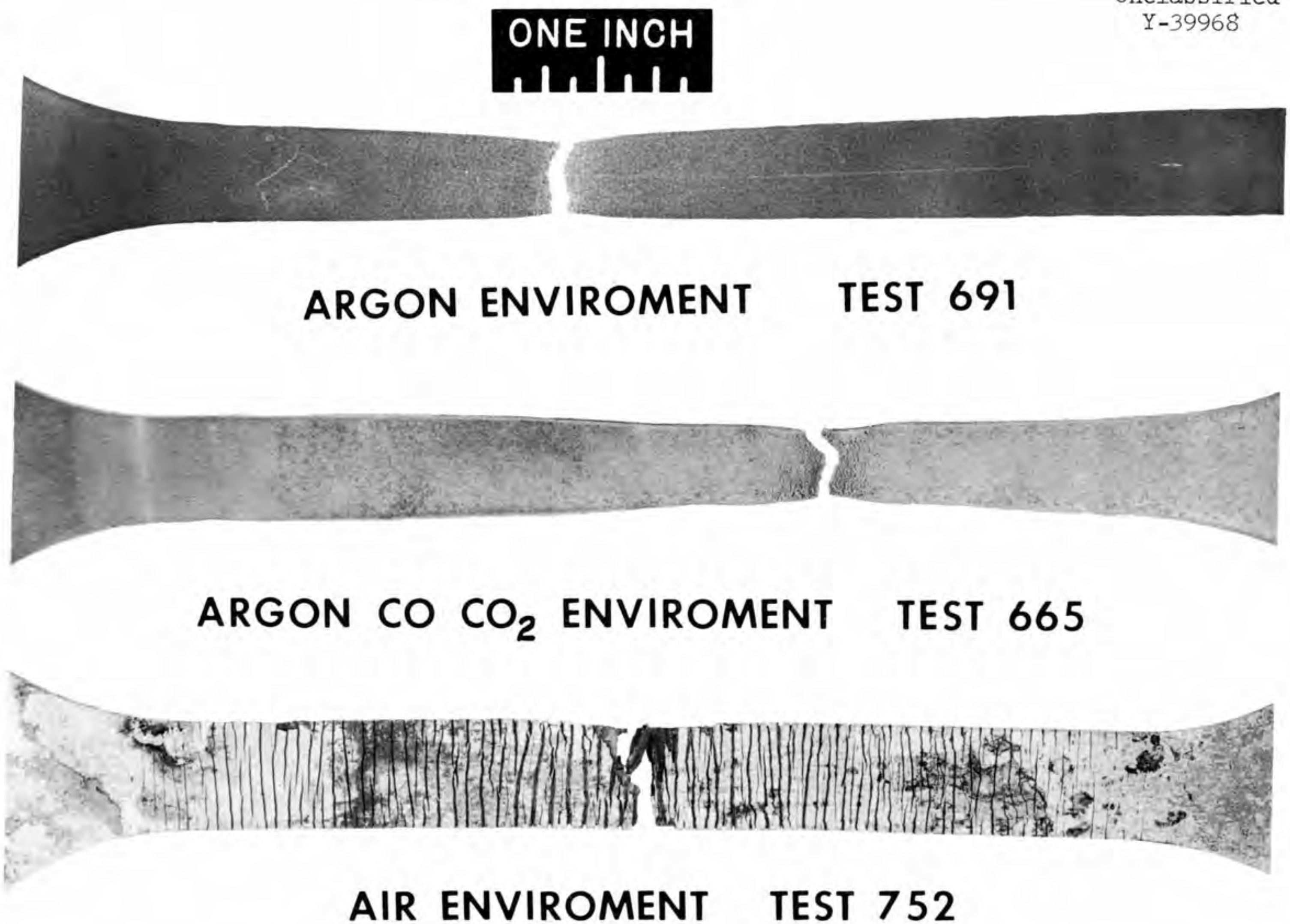

Fig. 17. Typical Appearance of Surface of AISI Type 502 Steel after Creep Test.

Table 11. Effect of Carbon on Room-Temperature Bend Properties of AISI Type 502 Steel

Heat Treatment	Carbon Content (wt %)	Hardness R_c	Maximum Deflection (in.)	Stress at Proportional Limit (psi)
Annealed 1 hr at 1900°F in argon	0.065	35	0.25	157,000
1 hr at 1700°F in CO Annealed 1 hr at 1900°F in argon	0.083	41	--[a]	164,400
2 hr at 1700°F in CO Annealed 1 hr at 1900°F in argon	0.13	46	--[a]	172,200
3.4 hr at 1700°F in CO Annealed 1 hr at 1900°F in argon	0.29	53	0.060	393,400
19 hr at 1700°F in CO Annealed 1 hr at 1900°F in argon	0.77	67	0.014	164,400[b]
26 hr at 1700°F in CO Annealed 1 hr at 1900°F in argon	0.96	66	0.013	155,900[b]

[a]Exact deflection at which cracks formed not detectable, but cracks were present after 0.25-in. deflection.

[b]Brittle failure.

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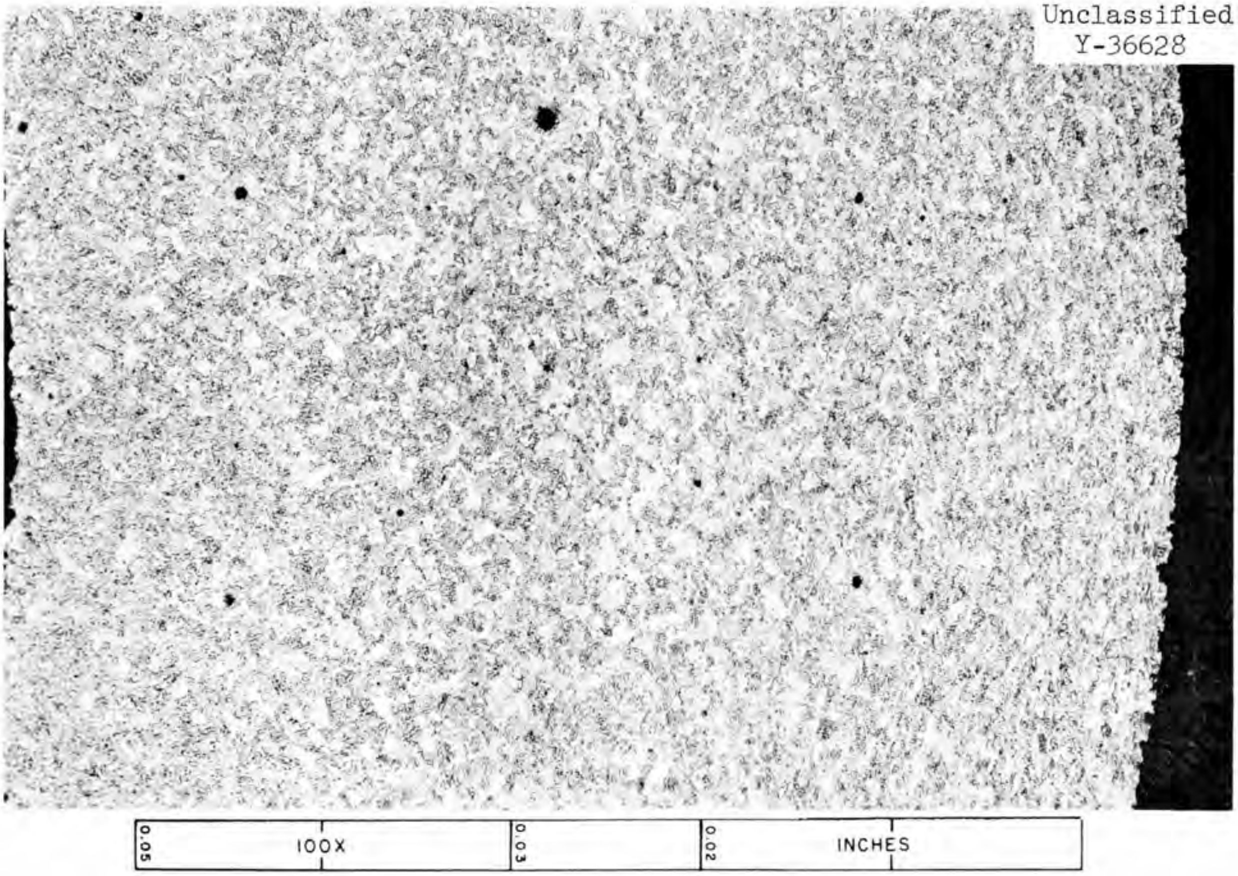

Fig. 18. Photomicrograph of the Bend Section of an AISI Type 502 Steel Specimen Containing 0.065 wt % C. Etchant: Picric-HCl.

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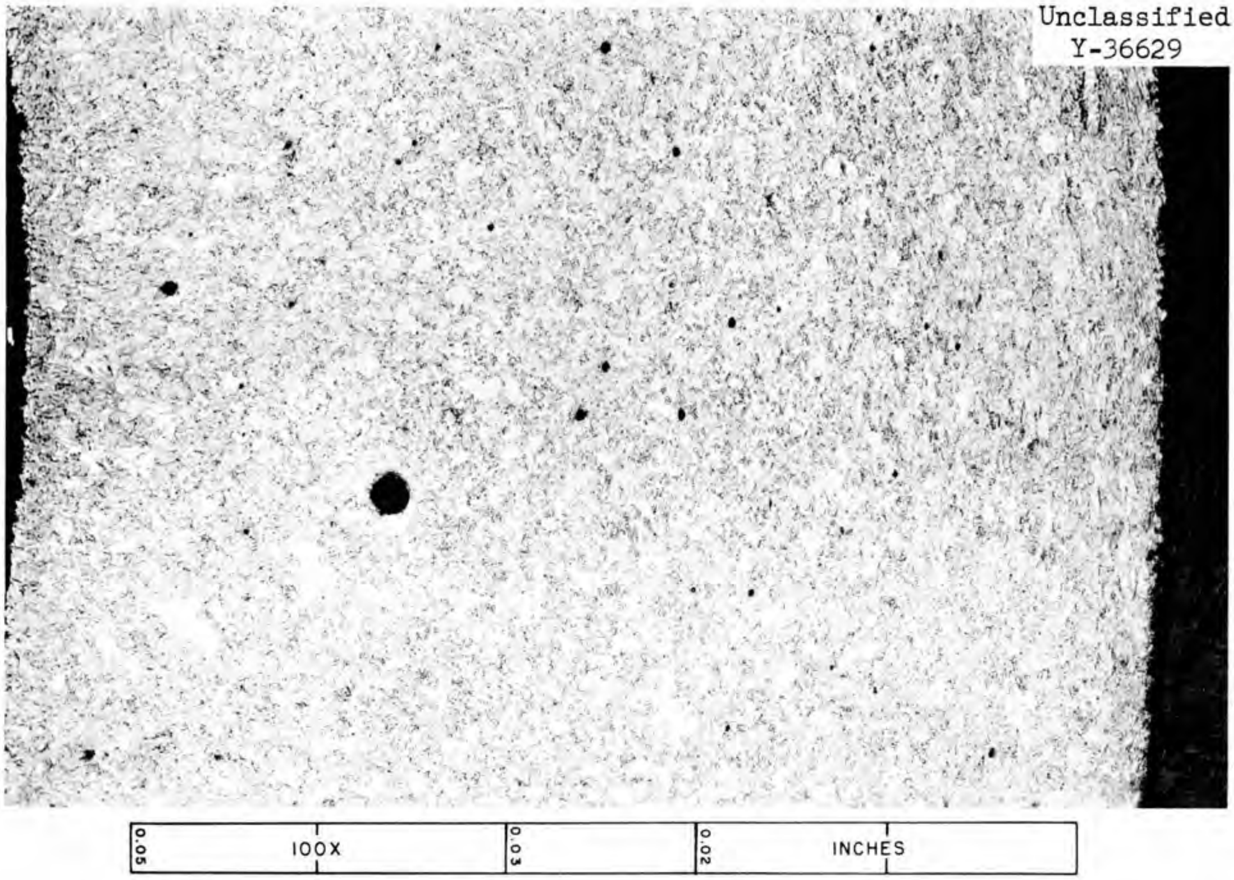

Fig. 19. Photomicrograph of the Bend Section of an AISI Type 502 Steel Specimen Containing 0.083 wt % C. Etchant: Picric-HCl.

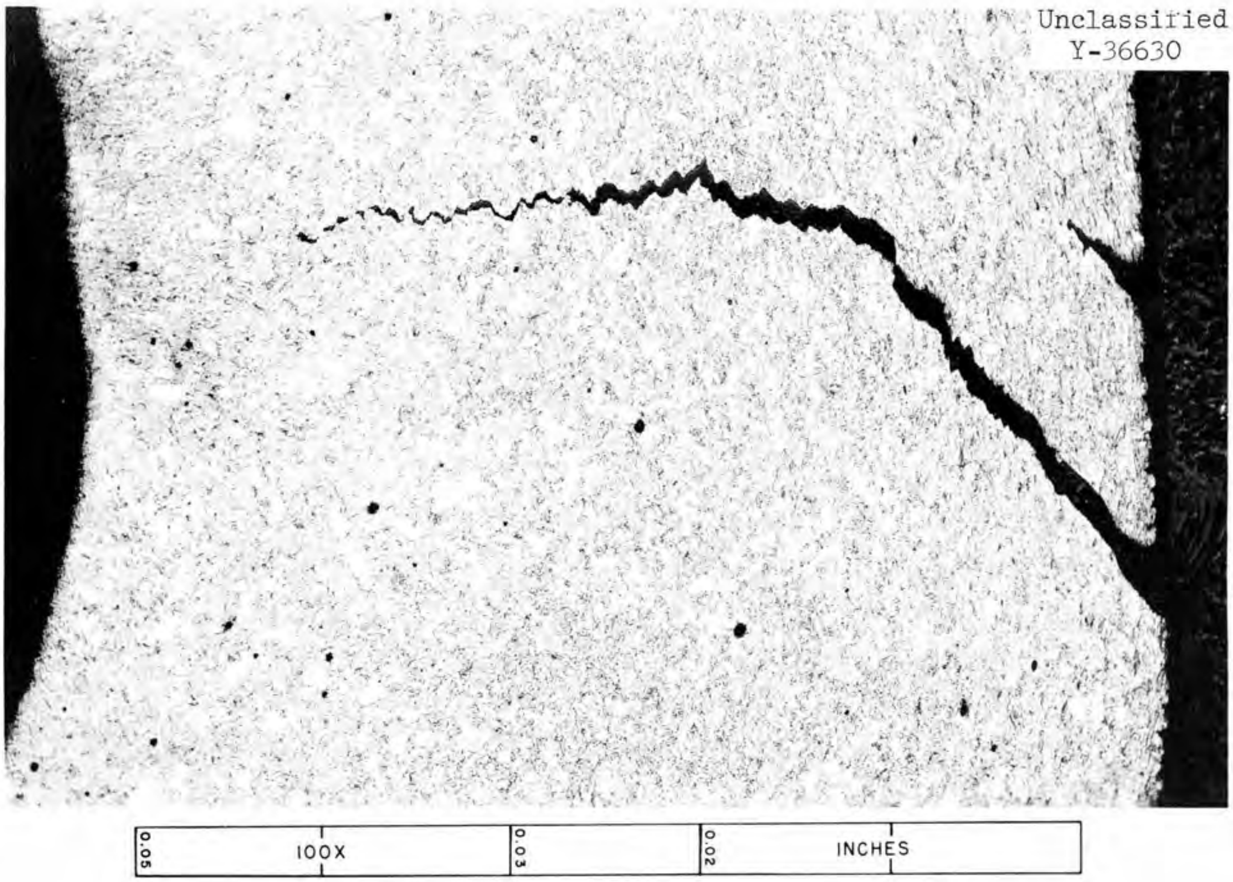

Fig. 20. Photomicrograph of the Bend Section of an AISI Type 502 Steel Specimen Containing 0.13 wt % C. Etchant: Picric-HCl.

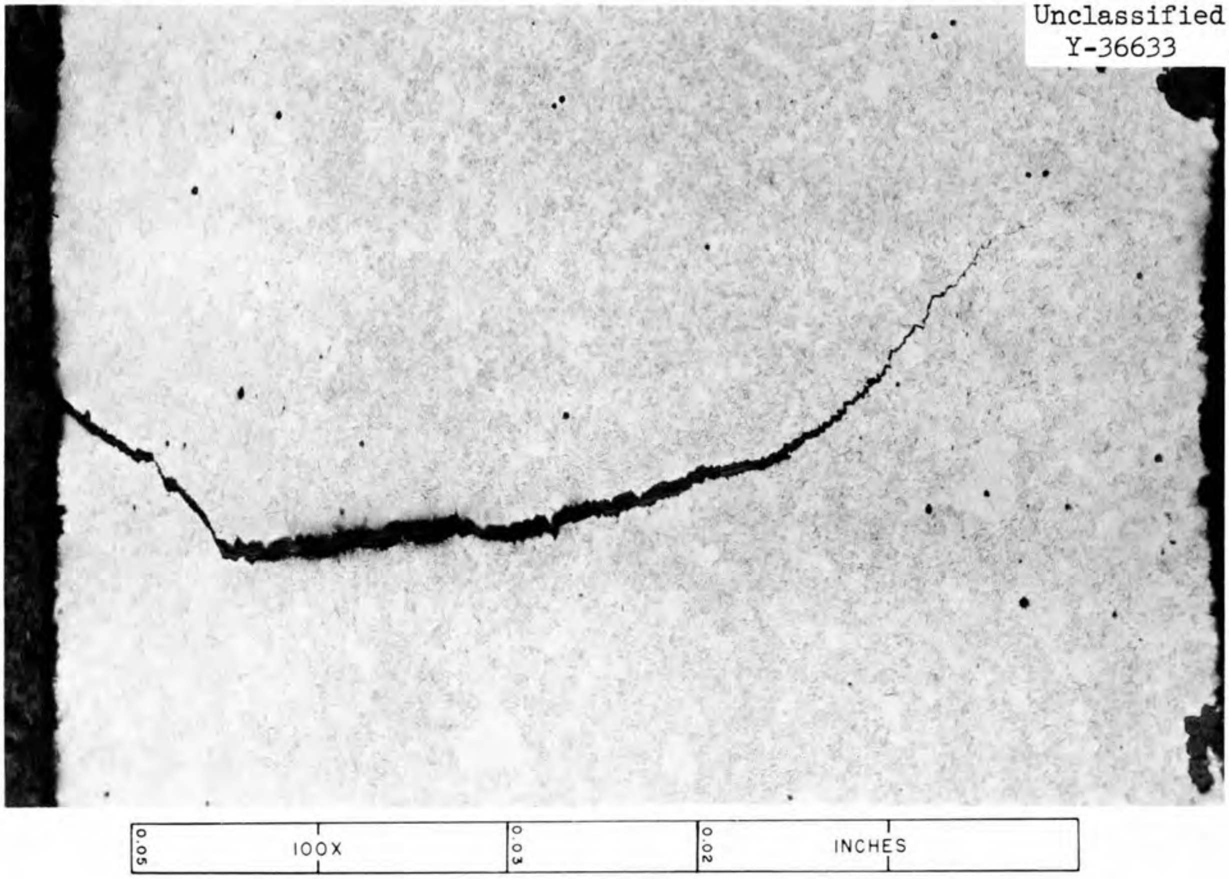

Fig. 21. Photomicrograph of the Bend Section of an AISI Type 502 Steel Specimen Containing 0.29 wt % C. Etchant: Picric-HCl.

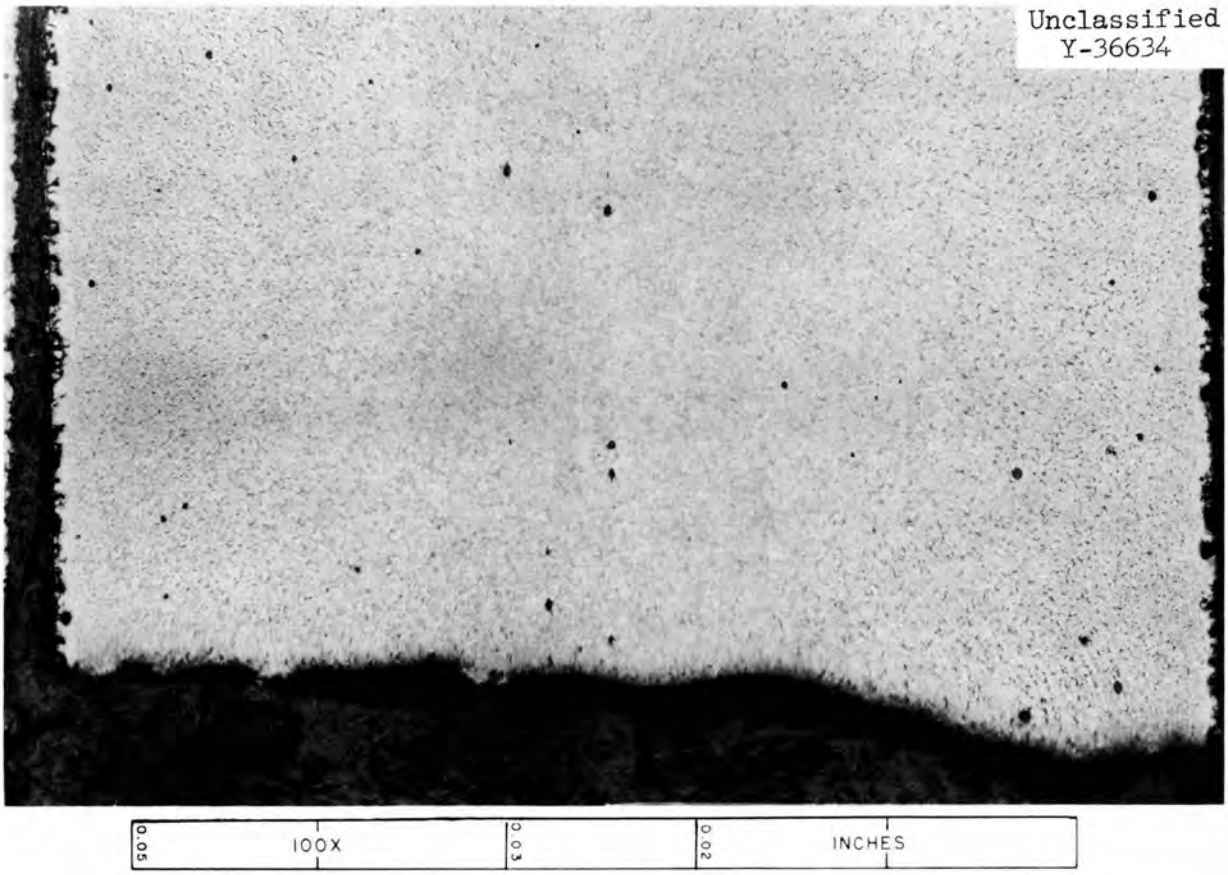

Fig. 22. Photomicrograph of the Bend Section of an AISI Type 502 Steel Specimen Containing 0.77 wt % C. Etchant: Picric-HCl.

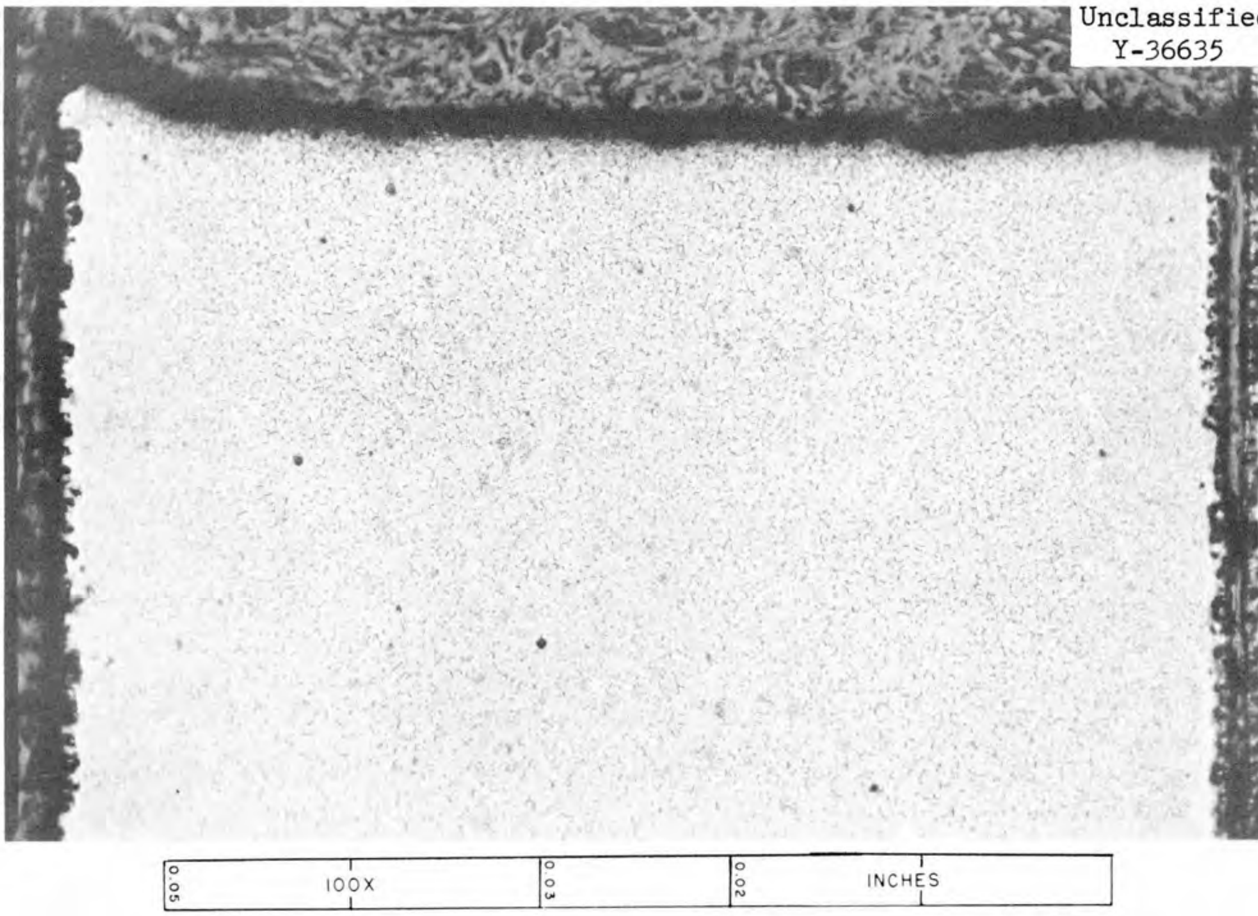

Fig. 23. Photomicrograph of the Bend Section of an AISI Type 502 Steel Specimen Containing 0.96 wt % C. Etchant: Picric-HCl.

Table 12. Summary of AISI Type 502 Steel Compatibility Tests

Test No.	Specimen History	Test Environment	Test Temperature (°F)	Duration of Test (hr)	K, mg/cm^2-hr	n
454	Annealed 1 hr at 1700°F in Ar, Tempered 1 hr at 1350°F in Ar	Ar-1CO-1CO_2	1500	450.1	0.146	0.268
530	Annealed 1 hr at 1700°F in Ar, Tempered 1 hr at 1350°F in Ar	Ar-5CO-5CO_2	1500	329.6	0.0480	0.718
589	Annealed 1 hr at 1700°F in Ar, Tempered 1 hr at 1350°F in Ar	Ar-5CO-5CO_2	1200	746.7	0.00319	1.210
669	Annealed 1 hr in air at 1500°F Tempered 1 hr at 1350°F in Ar	Ar-5CO-5CO_2	1200	247.5	0.00200	1.243
711	Annealed 1 hr at 1500°F in N_2 Tempered 1 hr at 1350°F in Ar	Ar-5CO-5CO_2	1200	377.2	0.00381	0.701

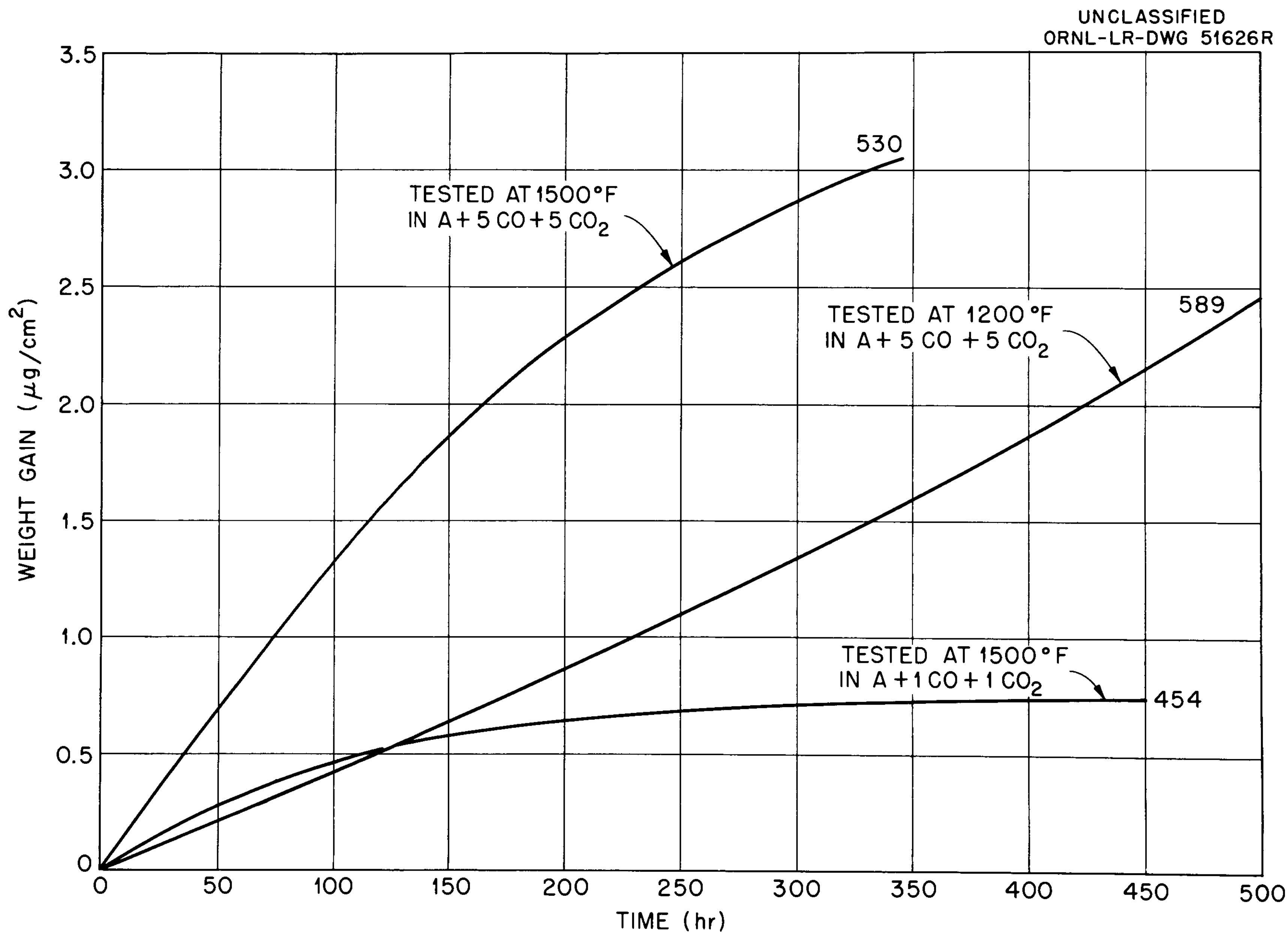

Fig. 24. Oxidation of the AISI Type 502 Steel in Ar-CO-CO_2 Mixtures.

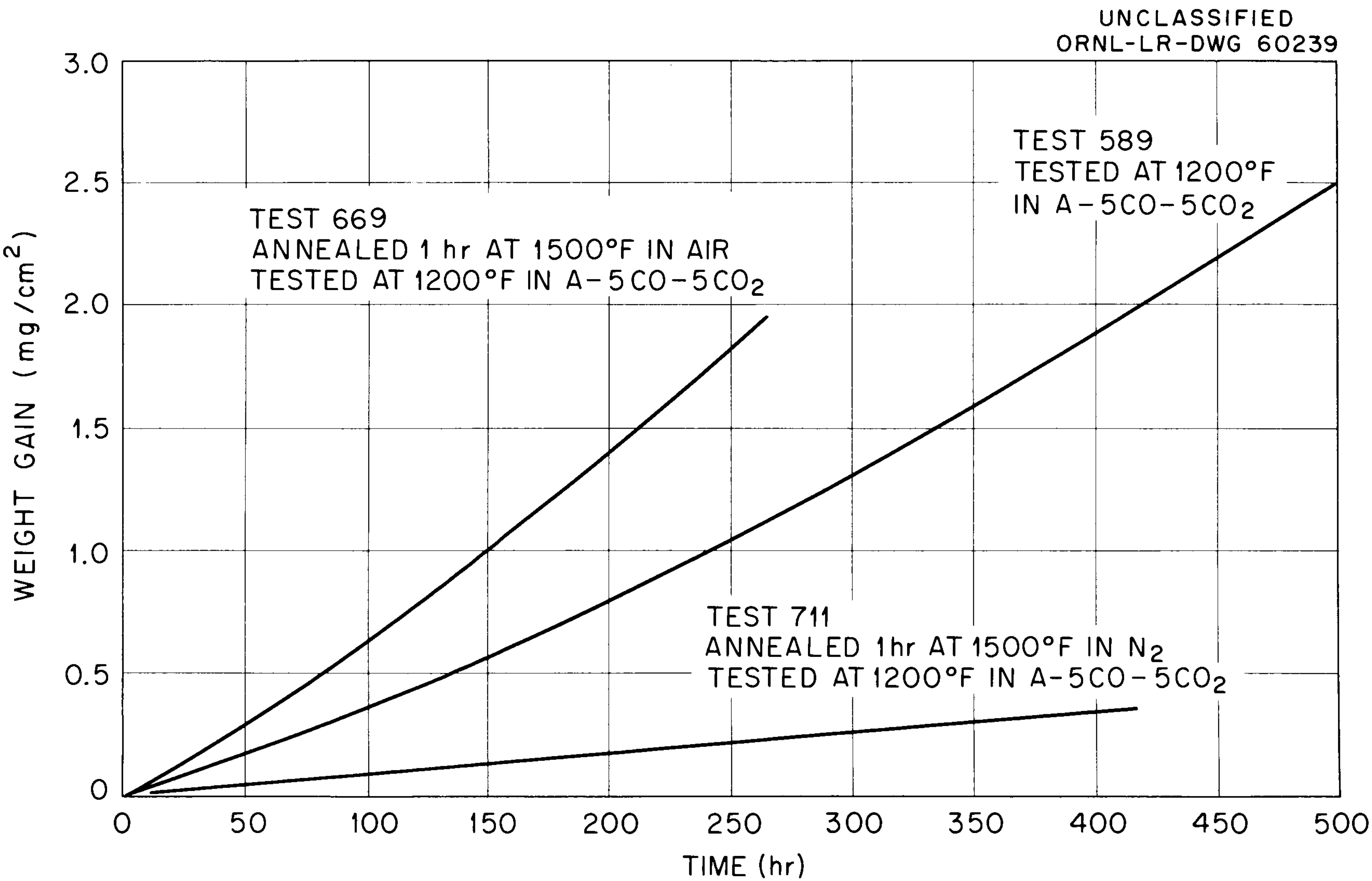

Fig. 25. Oxidation of AISI Type 502 Steel in A-CO-CO_2 Mixtures.

and n are constants, and specific values are tabulated in Table 12 for the various test conditions used. The chemical composition of several of the specimens was determined after testing and relevant analyses are given in Table 13. The test specimens were examined metallographically and representative photomicrographs are shown in Figs. 26 through 32.

Table 13. Summary of Analytical Data
Obtained From AISI Type 502 Steel Compatibility Tests

Test No.	Before Test			After Test		
	O_2, wt %	N_2, wt %	C, wt %	O_2, wt %	N_2, wt %	C, wt %
454	0.018	0.018	0.065	---	---	0.018
530	0.018	0.018	0.065	---	---	0.036
589	0.018	0.018	0.065	---	---	0.29
669	0.63	0.018	0.009	0.30	0.019	0.146
711	0.078	0.017	0.058	0.24	0.015	0.067

It was observed that the oxide film formed on this material in an Ar-CO-CO_2 mixture at 1200°F is nonprotective and spalling of the oxide is observed after several hours of exposure. This is illustrated by the weight change versus time plot for test No. 589 shown in Fig. 24, and the corresponding value of the rate constant given in Table 12. The value of 1.21 indicates that the reaction rate increases slightly with time. This is probably due to an increase in reactive surface area. Figures 26 and 27 illustrate the nature of the surface films formed at these test conditions. Note also from the data in Table 13 for test No. 589 that the AISI type 502 steel was carburized during its exposure to an Ar-5CO-5CO_2 environment at 1200°F.

At 1500°F the oxide formed in an Ar-5CO-5CO_2 environment was observed to be somewhat more protective with a rate constant of 0.718 being measured in test No. 530. No spalling was noted during or after the test. The nature of the oxide formed is illustrated in Fig. 28. A specimen was also tested at 1500°F in an Ar-1CO-1CO_2 mixture to determine the effect of concentration on the oxidation characteristics of this alloy (test No. 454). The oxide formed under these test conditions was also protective and adherent with a rate constant

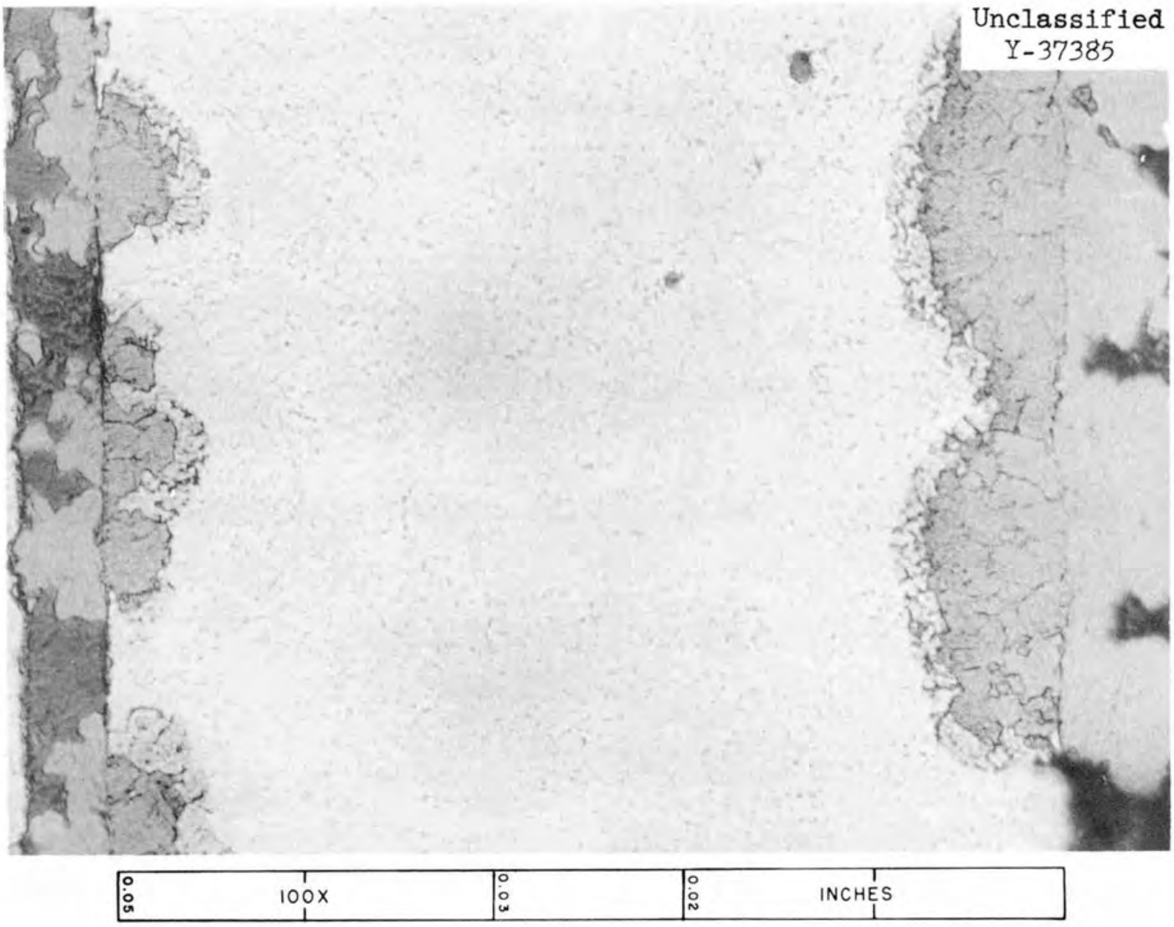

Fig. 26. Representative Photomicrograph of Specimen from Test No. 589. Unetched.

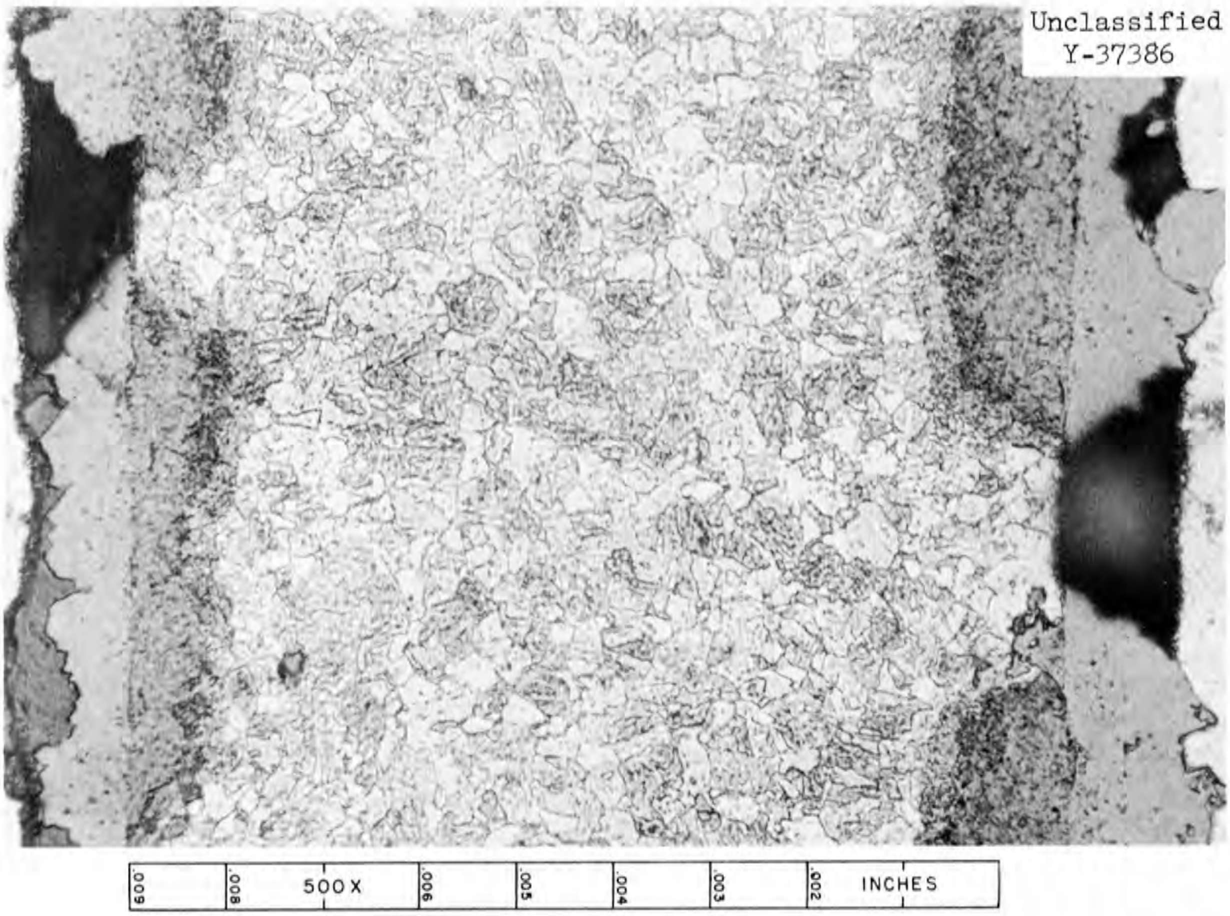

Fig. 27. Representative Photomicrograph of Specimen from Test No. 589. Etchant: 2% Nital.

Fig. 28. Photomicrograph of the Edge of the AISI Type 502 Steel Specimen from Test No. 530. As Polished.

Fig. 29. Photomicrograph of the Edge of the AISI Type 502 Steel Specimen from Test No. 454. As Polished.

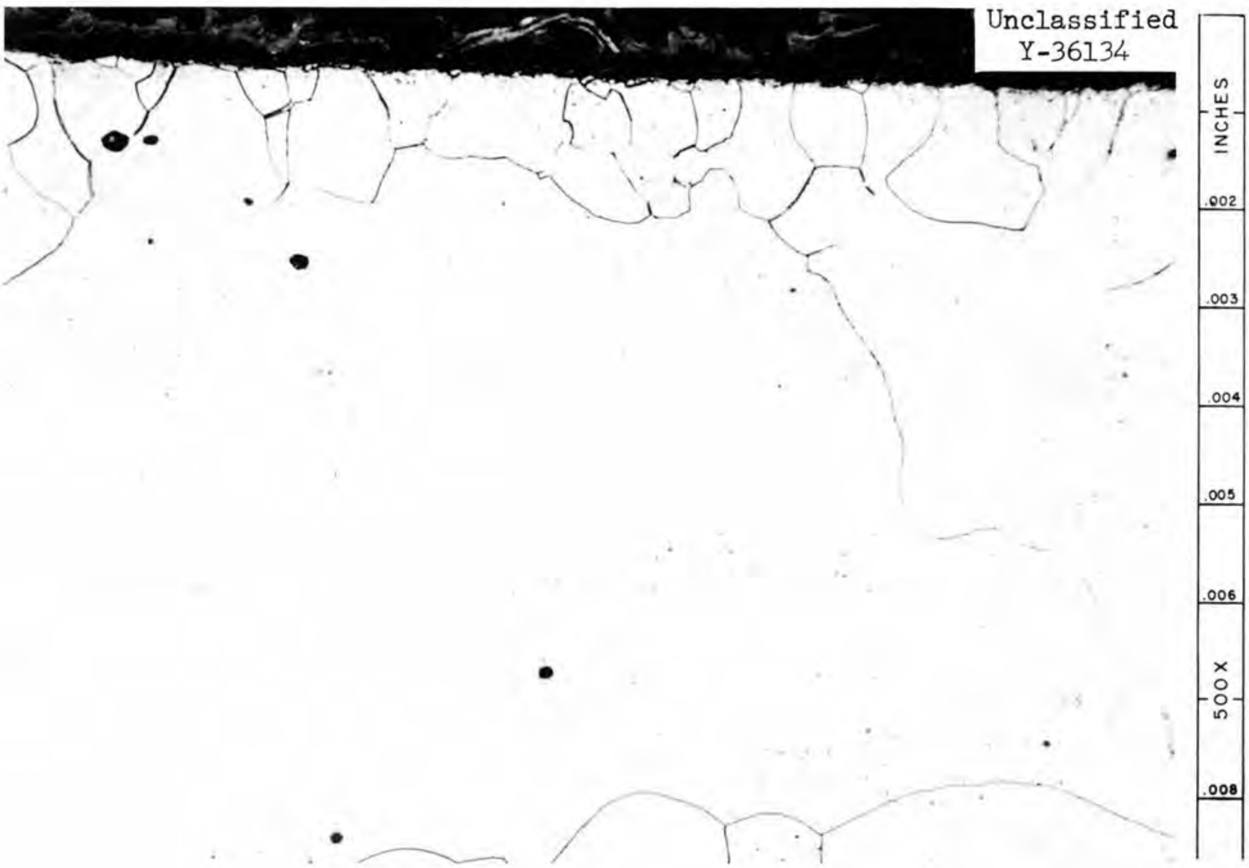

Fig. 30. Edge Photomicrograph of the Specimen from Test No. 454. Etchant: 2% Nital.

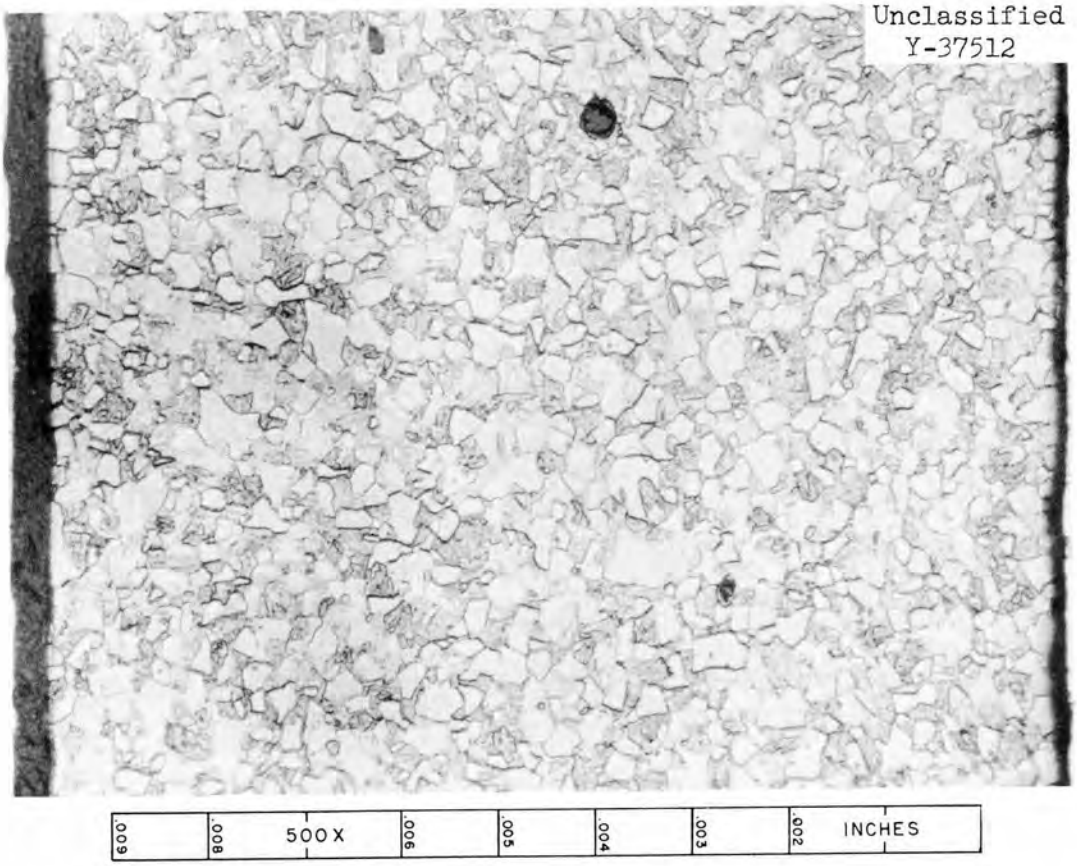

Fig. 31. Representative Photomicrograph of the AISI Type 502 Steel after Annealing 1 hr at 1500°F in Nitrogen. Etchant: Picral-HCl.

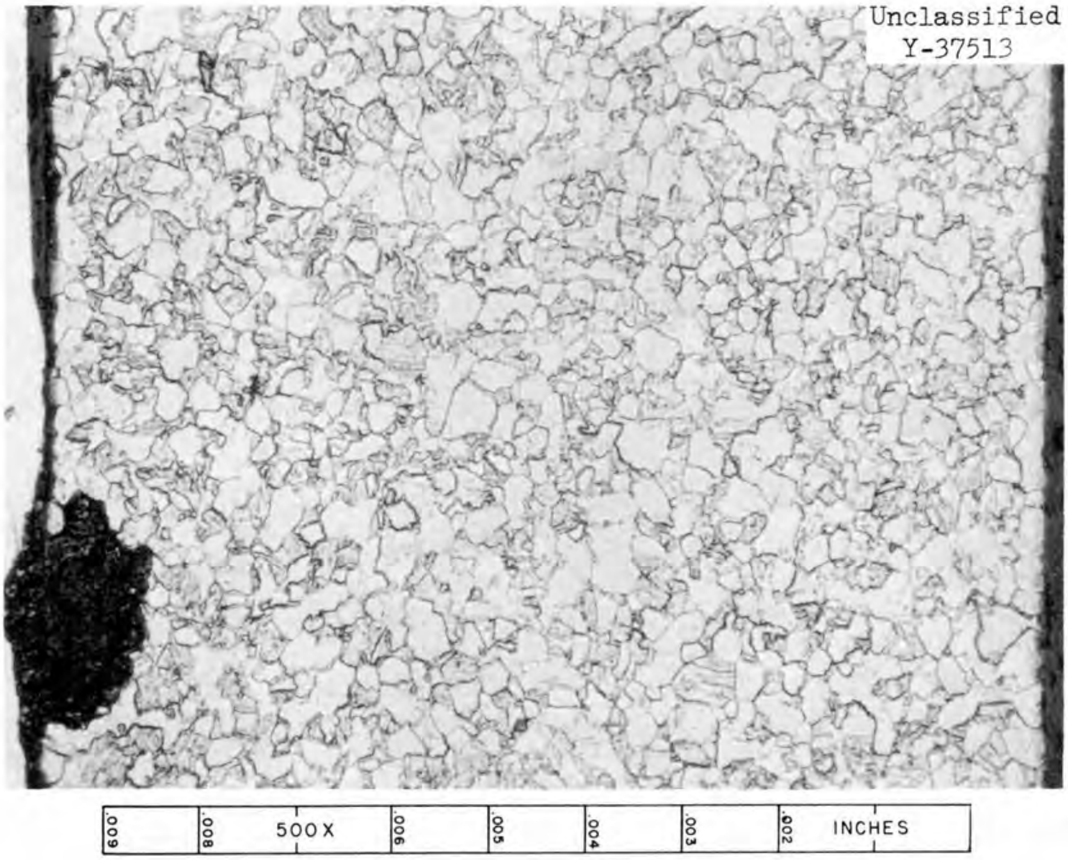

Fig. 32. Representative Photomicrograph of the Specimen from Test No. 711. Etchant: Picral-HCl.

of 0.268 being measured. Figures 29 and 30 are representative photomicrographs taken of the specimen from test No. 454. Note in Fig. 29 that, although the average oxide thickness is very thin, the maximum oxide penetration is quite deep. Figure 30 shows evidence of severe decarburization and excessive grain growth. That decarburization occurred in both the Ar-5CO-5CO_2 and Ar-1CO-1CO_2 mixtures is shown by the analytical data in Table 13 of tests Nos. 530 and 454. However, the test specimens run at 1200°F in the Ar-5CO-5CO_2 mixtures were carburized.

Because of the very poor oxidation resistance of this material at 1200°F, the effects of preoxidation and nitriding treatments were investigated. The oxidation characteristics at 1200°F in an Ar-5CO-5CO_2 mixture of a specimen annealed in argon are compared in Fig. 25 with those of specimens Nos. 669 and 711, which were annealed one hour in air and nitrogen, respectively. The nitriding treatment improves the oxidation resistance; whereas, the preoxidation treatment slightly enhances the oxidation rate. Figures 31 and 32 are representative photomicrographs of specimen No. 711 after it was annealed in nitrogen and after it was exposed to the Ar-5CO-5CO_2 mixture at 1200°F. Observe that, although the surface oxide is in general very thin, some areas are heavily oxidized.

SUMMARY

The various heat treatments which were used in this investigation illustrate the dependence on this variable of room-temperature hardness and tensile properties of the AISI type 502 steel. The strength is increased as the solution annealing temperature is increased. This material is air hardenable and no significant differences in strength occur as a result of more rapid cooling rates such as water quenching. Tempering reduces the strength although the toughness of the material is increased. Reasonably good correlations were obtained between hardness and tensile properties.

The dependence of the mechanical properties of this material at 1200°F on heat treatment is not as significant as that observed at room temperature. It was found that the only heat treatments which significantly changed the tensile properties were those involving solution annealing treatment at temperatures of less than 1700°F. The creep properties were observed not to be significantly altered by heat treatment although the limited number of tests run makes it difficult to make a conclusion of this importance.

Creep tests in air, argon, and an Ar-5CO-5CO_2 mixture at 1200°F indicate an increase in the creep strength of this material in environments of air and the Ar-5CO-5CO_2 mixtures as compared with that observed in argon. Again, the number of tests run makes a conclusion difficult.

A series of bend tests was run to evaluate the effect of carbon content on the room-temperature ductility of this alloy. It was found that the ductility was reduced considerably if the carbon content exceeded 0.1 wt %. The suggestion is made that welding procedures take into consideration that the specification for this material allows a maximum of 0.10 wt % C.

Compatibility tests on unstressed specimens show that the oxidation rate of the AISI type 502 steel in an Ar-5CO-5CO_2 mixture is approximately linear with time at 1200°F. However, the oxide formed in the same mixture at 1500°F is more protective with a rate-time dependence of 0.73. Preoxidation in air was found to have little, if any, effect on the oxidation rate of this material in the Ar-5CO-5CO_2 mixture. A preliminary anneal in nitrogen was found to significantly reduce the oxidation rate.

CONCLUSIONS

As a result of these and other studies, it was concluded that the AISI type 502 steel is unsatisfactory for service at 1200°F in an environment composed primarily of helium with small partial pressures of carbon monoxide and carbon dioxide. This conclusion is based primarily upon the poor oxidation resistance of this material. Although mechanical-property tests do not exhibit a loss of strength due to exposure to this environment, it is felt that tests for longer times would show adverse effects. The loss of material due to scaling is within itself a significant factor. The effects of other impurities such as water vapor have not been investigated. However, it is felt that most other trace impurities present in the system would only serve to increase the oxidation rate.

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