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HIGH TEMPERATURE PROPERTIES OF 2¼Cr-1Mo AND TYPE 316 STEELS IN AIR AND IN HELIUM BEFORE AND AFTER EXPOSURE TO LIQUID SODIUM

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INTRODUCTION

The proposed use of liquid sodium as a coolant and heat transfer medium in nuclear reactors has caused concern regarding the possible effects of the sodium environment on the mechanical properties of the materials with which it is in contact. The operating temperatures of such reactors will be sufficiently high so that the creep-rupture properties of the construction materials may be a limiting design criterion. Creep-rupture properties under some circumstances may be markedly affected by environment. As an example, it could be possible for liquid sodium at the operating temperatures of the reactor to contribute to mass transfer reactions which affect the elevated temperature properties of the materials with which it is in contact.

MSA Research Corporation has operated two sodium loops in which creep-rupture tests have been conducted for the Atomic Energy Commission. In one loop, 2¼Cr-1Mo steel specimens were tested in sodium at 1100°F. In the second, Type 316 (18-8+Mo) stainless steel specimens were tested at 1200°F. The sodium in the loops was reactor grade with helium used as the cover gas. Other samples of these two steels were exposed at the same temperatures, unstressed, for 4000 hours in different environments and then removed for various tests. The exposure environments were: sodium containing 20-30 ppm oxygen (hereafter referred to as low-oxygen sodium), sodium containing 200-300 ppm oxygen (high-oxygen sodium), sodium saturated with carbon, and helium. The helium exposures were included to evaluate the effect of the thermal history of the material on the properties, independent of the influence of sodium.

Under a sub-contract with MSA Research Corporation the University of Michigan conducted comparative tensile, creep and/or rupture tests on both unexposed and exposed samples of the 2¼Cr-1Mo and Type 316 steels.

This report presents a summary of the results of the testing program carried out at the University of Michigan. Some of the data presented herein were originally presented in an interim report (Ref. 1). These data, particularly from the original material, were described quite extensively in the interim report. Discussion of these data has been somewhat abbreviated in this report as well as discussion of the details of the experimental procedures.

EXPERIMENTAL MATERIALS

The 2¼ Cr-1Mo steel specimens were made from 0.080-0.085-inch thick sheets. The sheets had been reduced by cold rolling by the U.S. Steel Corporation Research Laboratory, Monroeville, Pennsylvania, from annealed ¼-inch x 4-inch blanks purchased from the McInnes Steel Company. After cold rolling the sheet was heated in an inert atmosphere at 400°F per hour to 1575°F, held one hour and cooled at 80°F per hour to 1330°F and then cooled at 350°F per hour to 600°F followed by air cooling to room temperature. The resulting Rockwell hardness was R_b 73-75. The chemical composition was as follows:

	C (%)	Mn (%)	P (%)	S (%)	Si (%)	Cr (%)	Mo (%)
Suppliers Analysis	0.097	0.56	0.007	0.022	0.33	2.17	1.01
Check Analysis by Allegheny Ludlum Steel Corporation	0.086	0.64	0.010	0.024	0.33	2.19	0.99

The Type 316 stainless steel had been purchased to ASTM Specification A240-54 from William M. Orr Company Inc., Pittsburgh, Pennsylvania, in the form of 16 gage sheet 48 inches wide by 96 inches long. It had been fully annealed and had a grain size of ASTM 5-7 and a Rockwell hardness of R_b82. The chemical composition was as follows:

	C (%)	Mn (%)	Si (%)	P (%)	S (%)	Cr (%)	Ni (%)	Mo (%)
Suppliers Analysis	0.045	1.94	0.45	0.019	0.012	18.00	12.84	2.33
Check Analysis	0.047	2.02	0.46	0.020	0.014	18.24	12.57	2.19

Specimen blanks were cut from the sheets in the direction of rolling. The blanks were milled to within 0.002-inch of the final dimensions. The final 0.002-inch was removed by wet grinding in small increments to a finish of 32 RMS or better. It is important to note that the flat surfaces were machined as well as the edges.

Both original specimens and specimens which had been exposed to sodium for 4000 hours were supplied by MSA Research Corporation.

Part of the specimens exposed to sodium for 4000 hours were washed to remove sodium and part were left unwashed by MSA Research Corporation before they were forwarded for testing. After exposure the "unwashed" specimens were sealed in plastic bags under a helium gas cover. The "washed" specimens were cleaned with alcohol, then with distilled water, and finally with alcohol again before being sealed in plastic bags under helium. These bags were placed in a desiccant jar which was then purged with helium and sealed.

EXPERIMENTAL PROCEDURES

The MSA Research Corporation supplied specimens in the various conditions of exposure with 1-inch gage length for rupture and creep-rupture tests and with 2-inch gage lengths for tensile and creep tests.

Tensile tests were conducted in a 60,000-lb. hydraulic testing machine. The load was applied in increments of 50 lbs. until the yield point was exceeded and then the test was completed at a constant head of speed of 0.01-inch per minute. Strain readings were taken for each increment of load and a stress-strain curve was plotted to establish the 0.2 percent offset yield strength. For the high temperature tests in air the specimen was mounted in an electric resistance furnace. The specimen was brought to temperature in about four hours after which the test was conducted. The tests in helium were conducted in the same manner except that the specimens were mounted in a chamber which was out-gassed and charged with helium. This procedure will be described later in the report.

Air Tests

The rupture tests in air were conducted in simple-beam creep-rupture units. The specimens were mounted in the automatically-controlled electric furnace and brought to temperature and equalized in about 4 hours. The load was then applied to the specimen. The time for rupture was automatically recorded. The creep tests were conducted in the same manner except that a modified Martens-type optical extensometer was attached to each of the specimens. After the initial loading of a specimen its creep extension was measured every 24 hours.

All tests were conducted in accordance with ASTM Recommended Practices E21-58T and E139-58T.

Helium Tests

The chamber used for the tests in helium was an inverted "T"-shaped retort constructed of Type 304 stainless steel tube. The actual construction of the retort is described in Ref. 1. Pull rods extended into the chamber through vacuum-tight seals, transmitting the load to the specimen. The extensometer was totally enclosed in the chamber and was read and adjusted from outside the chamber. The furnace and its controls were outside the retort.

The procedure used to minimize oxidation during creep and rupture testing can be summarized as follows:

1. The chamber was evacuated to a pressure of less than 5 microns of mercury and flushed 3 times with helium.
2. With the chamber at 5 microns pressure or less, the furnace temperature was raised to 650°F in about 6 hours and was maintained at that temperature for about 6 hours. Heating tape was used to warm those parts of the chamber not heated by the furnace. This prolonged heating at 650°F greatly facilitated outgassing of the chamber and reduced oxidation during testing. A closed system pressure rise of less than 3 microns per minute at a pressure of less than 5 microns was taken as

an indication than no leaks were present.

3. The temperature of the specimen was raised to the test temperature while the specimen was held in the vacuum overnight.
4. When the closed system leak rate was less than 3 microns per minute, the chamber was closed and helium was admitted to the specimen chamber and pumped out several times. The chamber was then filled with helium* at 3 psig, final temperature adjustments made and the test started.

The same type of retort and experimental procedures were used for the tensile tests conducted in helium.

RESULTS AND DISCUSSION

The data obtained for the Type 316 stainless steel and the 2¼Cr-1Mo steel are presented and discussed in separate sections.

Type 316 Stainless Steel

Tensile tests at room temperature and at 1200°F, creep tests and rupture tests at 1200°F were used to evaluate the properties of the original material in air and in helium. The results of these tests were discussed in detail in the Interim Report (Ref. 1). In the present report the data from the tests on the original material have been repeated to provide a base with which to compare the properties of the alloy after exposure to the various environments.

Samples of Type 316 steel were exposed at MSA Research Corporation for 4000 hours at 1200°F in low-oxygen sodium (20-30 ppm oxygen), high-oxygen sodium (200-300 ppm oxygen), carbon saturated sodium and helium.

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* - The helium used was obtained from the Bureau of Mines. It was passed through a NaK purifier to a copper tube manifold which distributed the helium to the units. The NaK purifier was supplied by the MSA Research Corporation.

These samples were then subjected to tensile and creep-rupture tests. Tests were performed on both unwashed exposed specimens and exposed specimens which had been washed free of any adherent sodium.

Tensile Properties

The results of the tensile tests on the Type 316 austenitic steel are given in Table I; the averages of duplicate tests have been computed and are given in Table II. These data indicate that the tensile and yield strengths of the original material tested at room temperature in helium are slightly above the average for commercial material tested in air. It seems very unlikely, however, that the helium atmosphere could have influenced the room temperature test results.

The unexposed specimens tensile tested at 1200°F showed strength properties corresponding to the average expected values for this grade of steel. The yield strengths for the tests in air and in helium were identical, while the tensile strengths of the specimens tested in air were slightly higher than those of the specimens tested in helium at 1200°F.

With the exception of the material exposed in high-carbon sodium for 4000 hours at 1200°F, the prolonged exposures in varying environments had no significant influence on either the room temperature or the 1200°F tensile properties of the Type 316 steel. In the case of the material exposed in the high-carbon sodium, the room temperature tensile strength was reduced to an average of 69,980 psi (from 89,000 psi for unexposed material) and the yield strength increased to 57,830 psi (from 47,550 psi); the elongation decreased to an average of 1.6 percent and the reduction of area to 6.4 percent (from 65 and 59 percent respectively). At 1200°F the tensile strength was increased by the high-carbon sodium exposure to an average of 57,040 psi (from 47,790 psi) and the yield strength increased to 40,290 psi (from 25,930 psi); the elongation and reduction of area after the exposure averaged 2.1 percent and 3.1 percent respectively.

Creep and Rupture Properties

Original material. - The creep and rupture data for the original, unexposed Type 316 steel is summarized in Tables III and IV and in Figures 1 and 2. These data include specimens tested in air and in helium. As was observed in the previous report (Ref. 1), there was no detectable difference in rupture strength between the specimens tested in the two environments and there was relatively little scatter in the data. The creep tests at low stress levels exhibited slightly higher minimum creep rates for specimens tested in helium than in air. These differences, however, are not large enough to be considered significant.

Low-oxygen sodium exposure. - The data from specimens exposed 4000 hours in low-oxygen sodium and rupture tested in helium exhibited somewhat greater scatter than the specimens tested in air; see Figures 3 and 4a. Both the helium and the air rupture data for the material in this condition of exposure fell within the scatter band of the rupture data for the unexposed material.

High-oxygen sodium exposure. - The creep-rupture data for the Type 316 material exposed 4000 hours at 1200°F in high-oxygen sodium are given in part C of Table III and are also shown in Figures 3 and 4b. The rupture data for the material in this condition of exposure tested in helium also fell within the scatter band of the unexposed material. The slope, however, of the rupture curve (see Fig. 4b) was somewhat flatter than that of the unexposed material so that the rupture time at the lowest stress was on the high side of the unexposed rupture band. The results from the material tested in air appear to fall on a line parallel to but below the band of rupture strengths of unexposed material. The possible loss of carbon from the material during the exposure to high-oxygen sodium could possibly account for somewhat lower properties in the exposed specimens than in the unexposed. If carbon losses, however, were responsible for the reduced strength, a similar reduction of rupture strength should have been noted in the results of tests conducted in helium.

The relation between rupture properties in air and helium also existed for the creep rates. The creep rates of specimens tested in helium corresponded to those of unexposed material, but those of specimens tested in air were somewhat higher. The latter correspond to the creep rates of the material exposed 4000 hours in helium at 1200°F and creep-rupture tested in air at 1200°F.

High-carbon sodium exposure. - The material exposed to carbon saturated sodium exhibited similar rupture properties when subsequently tested in air and in helium (see Table III part D and Figures 3 and 4c). At low stress levels the rupture times were greater than those of the unexposed material. The minimum creep rates from these tests were an order of magnitude lower than those of the unexposed material; the helium tests exhibiting somewhat lower rates than the air tests. The rupture ductility and elongation (see Table III part D) for the specimens tested after this exposure were very low.

The behavior of the specimens after exposure to high-carbon sodium can apparently be accounted for on the basis of the greatly increased carbon content of the material resulting from exposure.

Helium exposure. - The rupture data for the Type 316 material exposed 4000 hours in helium at 1200°F and tested in air at 1200°F fell within the scatter band for unexposed material. The rupture data fell on a straight line, the slope of which was slightly less than that of the unexposed material (Figures 3 and 4d). The minimum creep rates of the alloy tested in air at 1200°F after exposure to helium were somewhat greater than those of the unexposed material. No subsequent creep-rupture tests were run in helium on the steel in this condition.

2¼Cr-1Mo Steel

Tensile tests at room temperature and at 1100°F, creep tests and rupture tests at 1100°F were used to evaluate the properties of the original material in air and helium. The results of these tests were discussed in detail in the Interim Report (Ref. 1). Some of these data and the discussion thereof have been repeated in this report to provide a base with which to compare the properties of the material exposed under various conditions.

Samples of the 2¼Cr-1Mo steel were exposed at MSA Research Corporation for 4000 hours at 1100°F in low-oxygen sodium (20-30 ppm oxygen), high-oxygen sodium (200-300 ppm oxygen) and in helium. Some of the exposed specimens, in both the washed and unwashed conditions, were tensile tested in helium at room temperature and at 1100°F. Other exposed specimens were creep-rupture tested in helium at 1100°F and still other specimens which had been exposed in low-oxygen sodium were rupture tested in air.

Tensile Properties

The results of the tensile tests on the 2¼Cr-1Mo steel are given in Table V; the averages of duplicate tests have been computed and are given in Table VI. These data indicate that the tensile and yield strengths of the original material tested in air at 1100°F were within but on the high side of the range for commercial 2¼Cr-1Mo steel; the ductility data for these tests were within but on the low side of the usual range of commercial material. The tensile strength of specimens of original material tested in helium at 1100°F were significantly lower than those tested in air. Their yield strengths, however, were only slightly lower.

Following the 4000 hour exposures at 1100°F in various environments, the strength of the 2¼Cr-1Mo steel at 1100°F was much lower than that of the original material when tested either in air or in helium (see Tables V and VI). The material exposed in low-oxygen sodium and that exposed in helium exhibited very similar properties; the former having a tensile strength in helium at 1100°F of 26,150 psi and a yield strength of 17,100 psi, the latter having values of 25,730 psi and 16,770 psi respectively. After the exposure to high-oxygen sodium, the tensile and yield strengths were 21,710 psi and 16,000 psi. The corresponding values of tensile and yield strength of the original material tested in helium were 38,550 and 23,700 psi respectively.

The similarity of the data for the material exposed in low-oxygen sodium and in helium suggests that the difference in tensile properties between the material in these conditions and the original unexposed material was probably due to thermally-induced structural changes and not to the environment. The lower level of the tensile data for the material exposed in high-oxygen sodium in comparison to the material subjected to the other exposures, can probably be attributed to decarburization during exposure.

Creep and Rupture Properties

Original material. - The creep and rupture data for the original unexposed 2¼Cr-1Mo steel are summarized in Tables VII and VIII. The stress versus rupture time and versus minimum creep rate curves are plotted in Figure 5. These figures include data from both air and helium tests. The 1100°F rupture tests in air on the original material exhibited relatively little scatter. While the extrapolated 100,000 hour rupture strength at 1100°F was low, it was within the expected range for this material. The data from rupture tests in helium were erratic but the average rupture curve for the helium tests was consistent with that of the original material tested in air at 1100°F. There does not appear to be any justification for drawing separate rupture curves for the two test conditions. The cause for the observed scatter in the results from the helium tests has not been determined. The following comments, which are essentially the same as those contained in Ref. 1, are offered concerning the scatter of the results:

1. No other series of tests of either steel in any condition of exposure exhibited this amount of data scatter. This fact indicates that the testing procedures and conditions alone were not responsible for the observed scatter.
2. Specimen 2DAX5, which ruptured very prematurely in 624 hours under 10,500 psi, fractured at a scribe mark used as a gage mark. This

suggests that the 2¼Cr-1Mo steel may be sensitive to stress concentrations when tested in helium. No obvious relationship between rupture time and surface condition was found in any of the other tests.

3. The use of the helium atmosphere did not prevent oxidation. The extent of oxidation encountered in the creep and rupture tests in helium at 1100°F varied among the specimens from a slight discoloration to appreciable oxide (never as great, however, as encountered in the tests in air). There appears to be no relation between the degree of oxidation and the scatter in the data of the tests run in helium.

It may also be noted that the latter comment and the similarity between the average rupture properties in air and in helium indicate that the oxidation which occurred during the tests in air was not very influential toward the rupture characteristics of the material.

At the time that the above tests were run, creep measurements were not being made during the rupture testing, so these data are not available for evaluation. Low stress creep tests of about 4000 hours duration were run on the original material at 1100°F in air and in helium. The stress dependence of the minimum creep rates of these tests is shown in the lower part of Figure 5. The stress dependence of the minimum creep rate (i.e. the slope of the stress versus minimum creep rate curve in this figure) of the tests in air was as expected for this material. The slope of the curve for the tests in helium, however, was flatter than that of the tests in air and at the two lowest stresses the minimum creep rates in helium were less than those measured in the air tests. This is somewhat unusual in that it has been generally considered that an inert atmosphere lowers creep strength.

It was noted in Ref. 1 in connection with these data that the tests in helium at 6000 and 7000 psi exhibited an early onset of third-stage creep which was not found in the similar tests in air. Considering the limited amount of creep data and the uncertainty of the properties reflected by the erratic rupture data, the early onset of increasing creep rate is the strongest suggestion of an effect which may be attributable to the helium atmosphere.

Helium exposure. - The data from the specimens exposed 4000 hours in helium at 1100°F and then creep-rupture tested in helium at 1100°F are given in Table VIII and plotted in Figure 6. Note that in contrast to the original material these data exhibit very little scatter. The significant feature of these data is the reduction in short-time strength as a result of the 4000 hour exposure. This behavior has been found to be typical of 2¼Cr-1Mo steel.

It is thought that the prolonged heating of this alloy at 1100°F caused structural changes to occur. These microstructural changes caused the short-time rupture properties of the exposed material to be much lower than characteristic of unexposed virgin material. As would have been expected, however, the long-time properties of the exposed specimens were similar to the long-time properties of the unexposed specimens. Since most of the microstructural changes occur in a few thousand hours at 1100°F and are essentially the same in either low stress or unstressed exposure, their influence on rupture properties should be greatly reduced after these relatively short-time periods.

The result of this type of behavior is that while the short-time rupture strength can be drastically reduced, the long-time rupture strength may not be significantly changed by the prior exposure. This is precisely the behavior exhibited in Figure 6 for the material exposed in helium for 4000 hours prior to creep-rupture testing. Note that at about 2500 hours the extrapolated rupture curves for the original material and the material prior-exposed in helium, appear to intersect. After about 2500 hours the prior-exposed material is assumed to be as strong as the original material.

The creep data for the helium exposed material is such that the minimum creep rates are somewhat lower than either those of the unexposed material or of the material exposed in high-oxygen sodium. The stress dependence of the minimum creep rate of the material subjected to the helium exposure and tested in helium appears to be the same as that of the unexposed material tested in helium and the material exposed in high-oxygen sodium tested in helium.

Low- and high-oxygen sodium exposure. - The creep and/or rupture data from the specimens exposed 4000 hours in low and high-oxygen sodium at 1100°F are given in Table VIII and plotted in Figure 6. The rupture data at 1100°F from the tests in air and in helium on the material subject to the low-oxygen sodium exposure are almost identical and exhibited virtually no scatter. The rupture curve fitted to this data fell below that of the original material and had a flatter slope. There were no rupture tests run in air on the material exposed in high-oxygen sodium; the rupture data from tests in helium on this material laid somewhat below the rupture data from the material exposed in low-oxygen sodium and the slope of the curve was even less steep than that of the material after the low-oxygen exposure. There was also no scatter in the data from the material exposed in high-oxygen sodium.

As a consequence of the difference in the slopes of the rupture curves of the original and the exposed materials, extrapolation of the curves indicate that the curves of the exposed material would intersect that of the original material; the material exposed in low-oxygen sodium at about 35,000 hours and the material exposed in high-oxygen sodium at about 15,000 hours. Assuming that such extrapolations are valid, as well as the extrapolation of the rupture curve of the original material, the exposed material would be at least as strong as the original at times greater than those indicated by the intersection of the two curves.

This general type of behavior, as mentioned previously, is not unusual in 2¼Cr-1Mo steel. It results from thermally-induced structural changes accompanying the prolonged heating of the steel at 1100°F. These structural changes usually reduce the short-time strength but not the long-time strength relative to unexposed material. Consequently, to evaluate the influence of the sodium environment to which the material was exposed, the test results must be compared not to the original material but to the material exposed 4000 hours at 1100°F in helium. The latter, it is assumed, has undergone the same thermally-induced structural changes as the material exposed in the sodium environments. The following features are evident in the stress-rupture curves of Figure 6:

1. At short times the rupture behaviors of the material exposed in helium and in low-oxygen sodium appear to be very similar; this is also supported by the 1100°F tensile tests on these materials.
2. At short times there was a significant difference in rupture strength between the material exposed in low-oxygen sodium and that exposed in high-oxygen sodium. This same trend was noted in the results of tensile tests on the alloy in these conditions of exposure.
3. At long times only slight differences were noted in the rupture strengths of the steel subjected to the two sodium exposures.
4. There was a divergence of the rupture curves of the material exposed to low-oxygen sodium and that exposed in helium. At long time periods the rupture strength of the material exposed to helium was significantly higher than that of the alloy exposed to low-oxygen sodium for 4000 hours.

The differences between the materials subject to the two sodium exposures may be due to decarburization of the steel during the high-oxygen sodium exposure. Carbon analysis performed by MSA Research Corporation on exposed specimens indicated that the material had been decarburized during both exposures, the high-oxygen sodium resulting in somewhat greater decarburization than the low-oxygen sodium environment.

The differences between the test results shown in Figure 6 for the materials subjected to the three types of exposure suggest that the sodium environment with which the material was in contact during the exposure did contribute to a change in properties larger than that associated with thermally-induced structural changes alone.

GENERAL DISCUSSION

Microstructural Instability

Most commercially-produced materials used in elevated temperature applications are microstructurally unstable. The degree of the structural instability is reflected in the slope of the rupture curve, since the processes through which creep acts on an alloy to cause rupture are inter-related with the processes through which the microstructure acts to attain its stable state. In most cases the higher the degree of instability in the microstructure, the greater the slope of the rupture curve.

Unstressed exposure for 4000 hours at 1100°F in the case of 2¼Cr-1Mo steel and 1200°F in the case of Type 316 stainless steel should have caused each alloy to approach a stable state. Type 316 stainless steel, however, in the fully annealed condition, is a rather stable alloy. For this reason only minor structural changes would be anticipated during 4000 hours at 1200°F. Examination of Figure 4 shows that with one exception the rupture properties of the steel in either air or helium after 4000 hours of exposure in high-oxygen sodium, low-oxygen sodium or helium were within the range expected of unexposed material; the slopes of the rupture curves of the steel after the different exposures were slightly flatter than that of the unexposed material, indicating that the microstructure did change somewhat during the prolonged exposure.

The effect of the approach of microstructural stability during exposure was much more apparent in the results from the 2¼Cr-1Mo steel than in the Type 316 steel data. Figure 6 shows a comparison of the average properties of the unexposed alloy with rupture properties after 4000 hours exposure at 1100°F in different environments. In every case exposure drastically reduced short-time rupture properties and caused the alloy to exhibit a much flatter rupture curve than typical of new material. Such behavior is characteristic of the influence of long-time low-stress service on the rupture properties of this type of alloy (Ref. 2).

Exposure Environment

In order to evaluate the influence of sodium environment during exposure on the subsequent properties of the two steels, the properties have to be compared with the alloy properties after helium exposure. This comparison is necessary in order to eliminate any influence of thermally-induced microstructural changes on properties. Such changes should only be a function of time and temperature and should be independent of environment.

Exposure of Type 316 stainless steel in low- or high-oxygen sodium had very little influence on subsequent tensile or creep-rupture properties of the alloy. Exposure to high-carbon sodium, however, did markedly affect the properties of the steel. While rupture strength was relatively unaffected by this exposure, creep resistance was improved significantly. Rupture and tensile ductility was reduced to a very low level. Tensile properties were also influenced greatly by the exposure. At room temperature the strength was reduced but at 1200°F it was raised. All these effects are most probably related to carbon pick-up by the steel during the exposure to carbon-saturated sodium.

The tensile properties of 2¼Cr-1Mo steel exposed to low-oxygen sodium were similar to the properties exhibited by the helium-exposed material. Exposure to high-oxygen sodium presumably decarburized the alloy and thereby lowered its tensile strength properties. Some decarburization may also have occurred during exposure to the low-oxygen sodium, since the rupture properties of the steel after this exposure were somewhat lower than those of the steel after exposure to helium at 1100°F. The rupture properties of the specimens exposed to high-oxygen sodium were even lower, due to the still greater degree of decarburization. In all cases the tensile and rupture ductility were quite high, indicating little influence of exposure conditions on this property.

CONCLUSIONS

The conclusions drawn from this research on the influence of exposure to different sodium environments can be summarized as follows:

1. Thermally-induced microstructural changes occurring during 4000 hours of exposure at 1200°F for Type 316 stainless steel and at 1100°F for 2¼Cr-1Mo steel caused a reduction in the subsequent short-time strength of each alloy and a flattening of the stress-rupture curve.
2. The degree of reduction of short-time strength properties was much greater in 2¼Cr-1Mo steel than in Type 316, due to its greater microstructural instability.
3. Exposure to low- or high-oxygen sodium did not have any greater influence on the properties of Type 316 steel than did exposure to helium.
4. Exposure to high-carbon sodium improved the creep resistance but lowered the rupture ductility of Type 316 steel.
5. Low-oxygen sodium exposure had relatively little influence on the properties of 2¼Cr-1Mo steel.
6. Exposure to high-oxygen sodium reduced the strength of 2¼Cr-1Mo steel due to decarburization of the alloy.

REFERENCES

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2. Cullen, T. M., Rohrig, I. A., and Freeman, J. W.: Creep-Rupture Properties of 1.25Cr-0.5Mo Steel After Service at 1000°F; ASME Paper 65-WA/Met-3, Presented at ASME Winter Annual Meeting, Chicago, Illinois, November 1965.

TABLE I

Results of Tensile Tests on Type 316 Stainless Steel Sheet Specimens

Specimen No./Code	Temp. (°F)	Test Atmosphere	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
A) Original Material						
271/3BHX 10	Room	Helium	89,500	49,700	65	59
272/3BHX 11	Room	Helium	88,500	49,400	66	59
3BHX 1	1200	Helium	47,200	26,600	46	58
3BHX 2	1200	Helium	47,500	25,900	52	54
3BHX 3	1200	Helium	48,600	25,300	44	53
3BAX 1	1200	Air	51,200	26,300	47	44
3BAX 3	1200	Air	50,250	25,550	41	48
3BAX 4	1200	Air	49,400	26,100	43	47
B) Material exposed 4000 hours in low oxygen sodium at 1200°F						
86/3BHX 4	Room	Helium	93,200	44,600	47	46
87/3BHX 5	Room	Helium	92,000	43,750	48	50
88/3BHX 6	Room	Helium	92,500	42,000	46	40
89/3BHX 7	Room	Helium	91,400	43,700	49	52
90/3BHX 8	Room	Helium	92,500	43,900	49	52
91/3BHX 9	Room	Helium	95,500	45,600	46	40
52/3BHL1C	1200	Helium	48,850	24,500	39	46
53/3BHL2C	1200	Helium	46,900	24,800	52	52
54/3BHL3C	1200	Helium	50,000	26,200	41	46
55/3BHL1U[b]	1200	Helium	49,100	26,750	41	56
56/3BHL2U[b]	1200	Helium	46,400	25,200	46	55
57/3BHL3U[b]	1200	Helium	49,900	25,650	46	51
C) Material exposed 4000 hours in high oxygen sodium						
470	Room	Air	85,200	40,600	34	32
471	Room	Air	85,900	41,250	48	34
474	Room	Air	84,900	39,750	39	34
473	Room	Air	85,000	39,750	38	30
474	Room	Air	85,600	41,250	42	32
475	Room	Air	82,900	41,600	40	32

continued ....

Specimen No./Code	Temp. (°F)	Test Atmosphere	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
C) Material exposed 4000 hours in high oxygen sodium (continued)						
476	1200	Helium	46,700	24,400	27	46
477	1200	Helium	47,800	24,000	31	42
509	1200	Helium	49,100	24,200	25	46
473[a]	1200	Helium	49,500	23,300	25	38
479[a]	1200	Helium	49,250	25,000	24	26
480[a]	1200	Helium	48,500	24,600	21	36
D) Material exposed 4000 hours in high carbon sodium						
360	Room	Air	67,806	53,750	<1	<1
361	Room	Air	66,500	57,500	1.0	5.5
364	Room	Air	78,100	57,250	2.4	7.4
369	Room	Air	68,800	57,750	2.0	8.5
378	Room	Air	68,400	62,250	1.2	9.2
379	Room	Air	69,000	58,500	2.2	7.0
366	1200	Helium	57,300	39,700	2.1	2.1
367	1200	Helium	54,300	40,500	1.7	4.3
368	1200	Helium	57,000	40,300	2.7	4.5
362[a]	1200	Helium	59,000	38,000	2.3	1.5
363[a]	1200	Helium	56,375	41,250	1.7	3.6
365[a]	1200	Helium	58,250	42,000	2.0	2.6
E) Material exposed 4000 hours in helium at 1200°F						
353	Room	Air	81,100	40,250	53	57
354	Room	Air	86,400	38,600	59	56
355	Room	Air	87,500	41,250	49	39
511	Room	Air	87,000	38,500	55	54
512	Room	Air	87,700	29,300	53	56
512	Room	Air	87,200	40,500	53	58
356	1200	Helium	48,100	23,600	14	62
357	1200	Helium	46,400	23,400	31	67
358	1200	Helium	46,100	23,500	25	62
514	1200	Helium	48,000	23,500	23	48
515	1200	Helium	54,250	27,600	24	47
516	1200	Helium	65,000	27,750	26	50

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a - Unwashed Specimens

TABLE II

Averages of Tensile Data for Type 316 Steel

Prior Exposure	Temp. (°F)	Test Atmosphere	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
None	Room	Helium	89,000	49,550	66	59
None	1200	Helium	47,790	25,930	47	55
None	1200	Air	53,620	25,980	44	46
Lo-O_2-Na	Room	Helium	92,850	43,925	48	47
Lo-O_2-Na	1200	Helium	48,580	25,170	44	48
Lo-O_2-Na[a]	1200	Helium	48,470	25,870	44	54
Hi-O_2-Na	Room	Air	84,920	40,700	40	32
Hi-O_2-Na	1200	Helium	47,870	24,200	28	45
Hi-O_2-Na[a]	1200	Helium	49,080	24,300	23	33
Hi-C-Na	Room	Air	69,980	57,830	1.6	6.4
Hi-C-Na	1200	Helium	56,200	40,170	2.2	3.6
Hi-C-Na[a]	1200	Helium	57,880	40,420	2.0	2.6
Helium	Room	Air	86,150	38,070	55	57
Helium	1200	Helium	51,310	24,890	24	56
Average Properties of Commercially Produced Type 316 Steel:						
None	Room	Air	81,000	39,000	45	55
None	1200	Air	50,000	22,000	39	52

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a - Unwashed specimen

TABLE III

Results of Stress-Rupture Tests at 1200°F on Type 316 Stainless Steel

Specimen No./Code	Test Atmosphere	Stress (psi)	Rupture Time (hours)	Elongation (%)	Reduction of Area (%)	Min. Creep Rate (%/hr.)
A) Original Material						
97/3CAX 10	Air	27,500	173.6	61	45	
3CAX 2	Air	27,500	152.8	58	49	
98/3CAX 9	Air	27,500	146.5	52	46	
3CAX 3	Air	27,500	143.0	63	50-60	0.12
3DAX 4	Air	24,000	476.2	40	38	0.034
94/3CAX 6	Air	24,000	426.3	60	43	
95/3CAX 7	Air	24,000	449.7	44	39	
3CAX 1	Air	22,000	863.5	33	31	
96/3CAX 8	Air	21,500	1000.6	33	28	
93/3CAX 5	Air	21,500	971.0	35	38	
3DAX 3	Air	20,500	1387.8	35	29	
3DAX 1	Air	18,500	2363.0	34	29	
3DAX 2	Air	17,750	1969.5	39	20	
3AAX 4	Air	17,750	3695.1	18	18	
3CAX 4	Helium	27,500	166.3	63	46	
3CHX 2	Helium	27,500	152.8	53	49	
3CHX 3	Helium	24,000	697.7	48	49	
3DHX 3	Helium	24,000	483.1[c]	65	54	
3CHX 1	Helium	21,500	894.3	56	47	
3DHX 1	Helium	20,000	1509.5	34	37	
3DHX 2	Helium	18,000	2619.9[b]	44	32	
104/3DHX 4	Helium	17,750	3068.1	40	67	
B) Material exposed 4000 hours in low-oxygen sodium at 1200°F						
3EAL 1	Air	27,500	144	62	46	
3EAL 2	Air	24,000	574	54	44	
3EAL 3	Air	21,500	985.2	59	42	
3EHL 1	Helium	27,500	57.6	57	52	
3EHL 2	Helium	24 000	675.0	48	44	
3EHL 3	Helium	21,500	822.2	49	42	

continued ....

Specimen No./Code	Test Atmosphere	Stress (psi)	Rupture Time (hours)	Elongation (%)	Reduction of Area (%)	Min. Creep Rate (%/hr.)
C) Material exposed 4000 hours in high-oxygen sodium at 1200°F						
491	Air	27,500	87.6	36	64	0.151
490	Air	21,500	575.3[a]	29	29	0.029
489	Air	18,500	1269.4	23	27	0.0088
492	Helium	24,000	369.8[a]	32	37	0.032
494	Helium	22,500	736[a]	42	48	0.019
493	Helium	20,000	1915	42	45	0.0055
D) Material exposed 4000 hours in high-carbon sodium at 1200°F						
380	Air	27,500	163.5	14.6	13.0	0.017
381	Air	22,500	940.6	6.0	9.1	0.0037
382	Air	21,000	1601.5[a]	5.9	5.4	0.0015
481	Helium	27,500	167.7	7.5	13.0	0.0094
482	Helium	23,000	924[a]	4.5	15.5	0.0012
483	Helium	20,500	3396	11.0	17.5	0.00067
E) Material exposed 4000 hours in helium at 1200°F						
496	Air	27,500	102.1	66	79	0.0239
469	Air	25,000	300.5[a]	55	56	0.074
506	Air	23,000	542[a]	70	92	0.034
370	Air	21,500	1003.8	51	56	0.0248
507	Air	20,500	1076	71	69	0.019
468	Air	20,000	1543.7	34	52	0.013

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a - Specimen broke in gage mark or fillet

b - Test interrrupted at 2097 hours

c - Test brought up to temperature twice before test could be run

TABLE IV

Summary of Creep Test Results at 1200°F on Original Type 316 Steel

Specimen Code	Atmosphere	Stress (psi)	Minimum Creep Rate %/1000 hours	Discontinued after: (hrs.)
3AAX3	Air	11,500	0.040	3987
3BAX2	Air	12,500	0.143	4243
3AAX2	Air	13,000	0.215	3693
3AHX3	Helium	10,500	0.08	
3AHX2	Helium	11,000	0.18	
3AHX1	Helium	12,000	0.23	4008

TABLE V

Results of Tensile Tests on 2¼Cr-1Mo Steel

Specimen No./Code	Temp. (°F)	Test Atmosphere	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
A) Original Material						
2BAX1	1100	Air	56,800	28,800	32	57
2BAX4	1100	Air	49,170	25,800	28	64
2AAX4	1100	Air	59,400	29,370	32	62
2BHX3	1100	Helium	39,700	25,000	26	63
2BHX4	1100	Helium	38,120	22,600	31	44
2BHX5	1100	Helium	37,820	23,500	41	74
B) Material exposed 4000 hours in low-oxygen sodium at 1100°F						
208	1100	Helium	26,620	17,200	45	54
209	1100	Helium	25,230	-	46	55
210	1100	Helium	26,580	16,800	47	41
206	1100	Helium	26,160	17,300	41	50
211[a]	1100	Helium	25,800	16,150	40	65
242[a]	1100	Helium	26,410	17,950	42	56
243[a]	1100	Helium	29,200	19,400	38	57
C) Material exposed 4000 hours in high-oxygen sodium at 1100°F						
331	Room	Helium	61,600	28,600	23	56
332	Room	Helium	69,280	25,800	33	66
333	Room	Helium	57,910	24,400	28	63
334	Room	Helium	59,550	28,300	21	61
335	Room	Helium	59,700	27,300	28	60
337	Room	Helium	60,520	27,300	32	64
324	1100	Helium	23,800	18,300	46	66
325	1100	Helium	20,600	14,700	38	61
326	1100	Helium	20,740	15,000	39	54
328[a]	1100	Helium	23,400	14,500	37	64
329[a]	1100	Helium	22,300	-	36	64
330[a]	1100	Helium	22,000	13,200	37	64

continued ....

Specimen No./Code	Temp. (°F)	Test Atmosphere	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
D) Material exposed 4000 hours in helium at 1100°F						
436	Room	Helium	67,300	29,700	28	57
437	Room	Helium	64,500	26,900	23	65
438	Room	Helium	66,200	29,700	28	62
433	1100	Helium	20,800	13,500	33	81
434	1100	Helium	28,200	18,000	30	63
435	1100	Helium	28,200	18,800	47	76

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a - Unwashed specimen

TABLE VI

Averages of Tensile Data for 2¼Cr-1Mo Steel

Prior Exposure	Temp. (°F)	Test Atmosphere	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
None	1100	Air	55,120	27,990	31	61
None	1100	Helium	38,550	23,700	32	60
Lo-O_2-Na	1100	Helium	26,150	17,100	45	50
Lo-O_2-Na[a]	1100	Helium	27,140	17,833	40	59
Hi-O_2-Na	Room	Helium	61,430	26,950	27	62
Hi-O_2-Na	1100	Helium	21,710	16,000	41	61
Hi-O_2-Na[a]	1100	Helium	22,570	13,850	37	64
Helium	Room	Helium	66,000	28,770	27	61
Helium	1100	Helium	25,730	16,770	37	73
Average Properties of Commercially Produced 2¼Cr-1Mo Steel:						
None	Room	Air	73,000	42,000	35	50
None	1100	Air	42,000	27,000	44	88

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a - Unwashed specimens

TABLE VII

Results of Creep Tests at 1100°F on 2¼Cr-1Mo Steel

Specimen Code	Atmosphere	Stress (psi)	Minimum Creep Rate %/1000 hours	Discontinued after: (hrs.)
2AAX1	Air	5,500	0.076	4003
2AAX2	Air	6,000	0.12	4010
2BAX2	Air	8,000	0.36	3957
2AHX2	Helium	5,500	0.023	
2AHX1	Helium	6,000	0.075	4009
2AHX3	Helium	7,000	0.28	

TABLE VIII

Results of Stress-Rupture Tests at 1100°F on 2¼Cr-1Mo Steel

Specimen No./Code	Test Atmosphere	Stress (psi)	Rupture Time (hours)	Elongation (%)	Reduction of Area (%)	Min. Creep Rate (%/hr.)
A) Original Material						
2CAX 4	Air	20,000	90.8	64	58	
2CAX 2	Air	17,500	139.2	42	36	
2CAX 3	Air	15,000	302.3	41	32	
2CAX 1	Air	12,000	1390.5	23	15	
2CHX 1	Helium	20,000	108.0	55	55	
2CHX 3	Helium	20,000	86.5	56	40	
2DHX 1	Helium	16,500	246.6	47	41	
2DHX 3	Helium	16,500	98.4	47	54	
2DAX 1	Helium	14,500	303.8	36	41	
2DAX 2	Helium	14,000	479.2[c]	28	-	
2DAX 4	Helium	12,500	1545.0	29	27	
219/2DAX 5	Helium	10,500	624.0[a]	22	27	
220/2CHX 4	Helium	10,500	1398.0	27	28	
B) Material exposed 4000 hours in low-oxygen sodium at 1100°F						
215	Air	14,000	74.3	26	-	
214	Air	12,500	195.2[a]	44	-	
213	Air	10,000	1205.0[b]	16[d]	11	
2EHL 1	Helium	14,000	78.8	36	31	
2EHL 2	Helium	12,500	180.4	31	21	
2EHL 3	Helium	10,000	1078.4	27	27	
C) Material exposed 4000 hours in high-oxygen sodium at 1100°F						
222	Helium	13,720	18.0	41	45	0.612
188	Helium	12,000	68.3	32	29	0.0487
223	Helium	10,000	780.8	13	18	0.00816
D) Material exposed 4000 hours in Helium at 1100°F						
257	Helium	13,000	312.1[a]	31	48	0.031
344	Helium	11,500	961.5	37	39	0.0086
258	Helium	10,800	2374[c]	33	32	0.0031

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a - Specimen broke in gage mark or fillet

b - Heavily oxidized

c - Oxidized

d - Estimated from shoulder to shoulder length of specimen

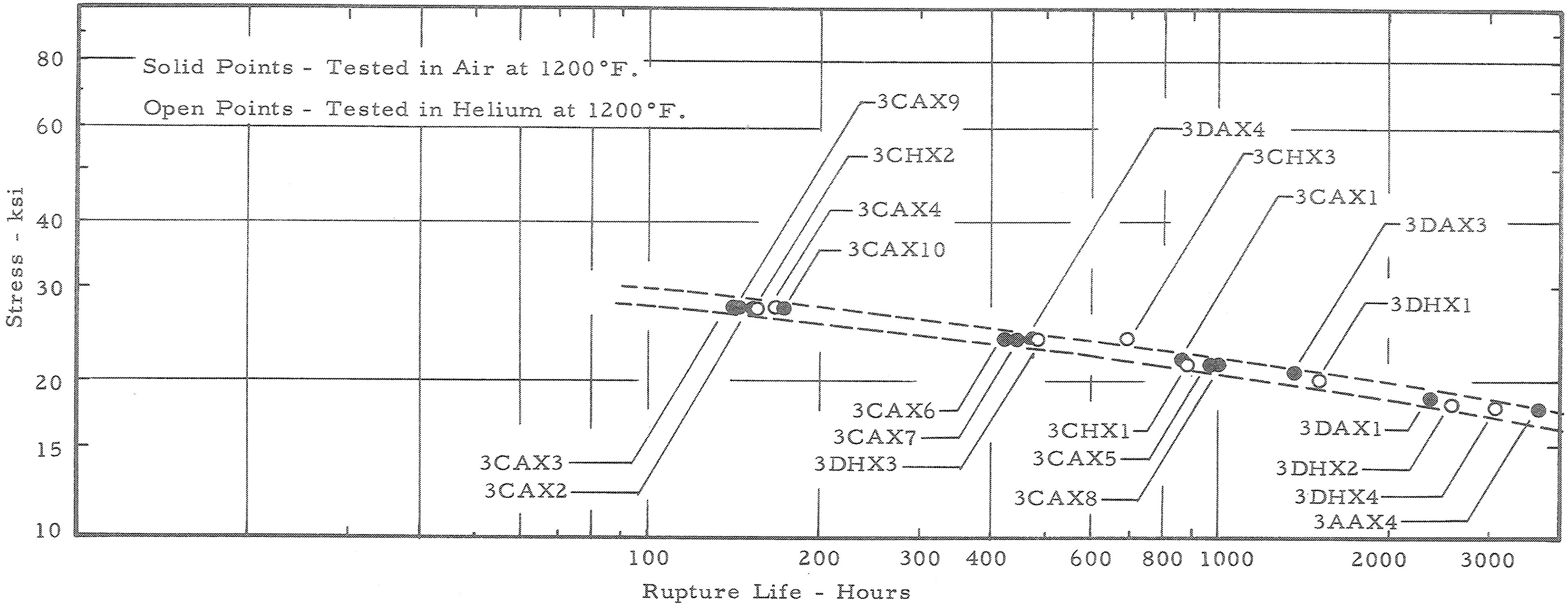

Figure 1. Stress-rupture time relationship for Type 316 austenitic steel tested at 1200°F in air and helium with no prior exposure.

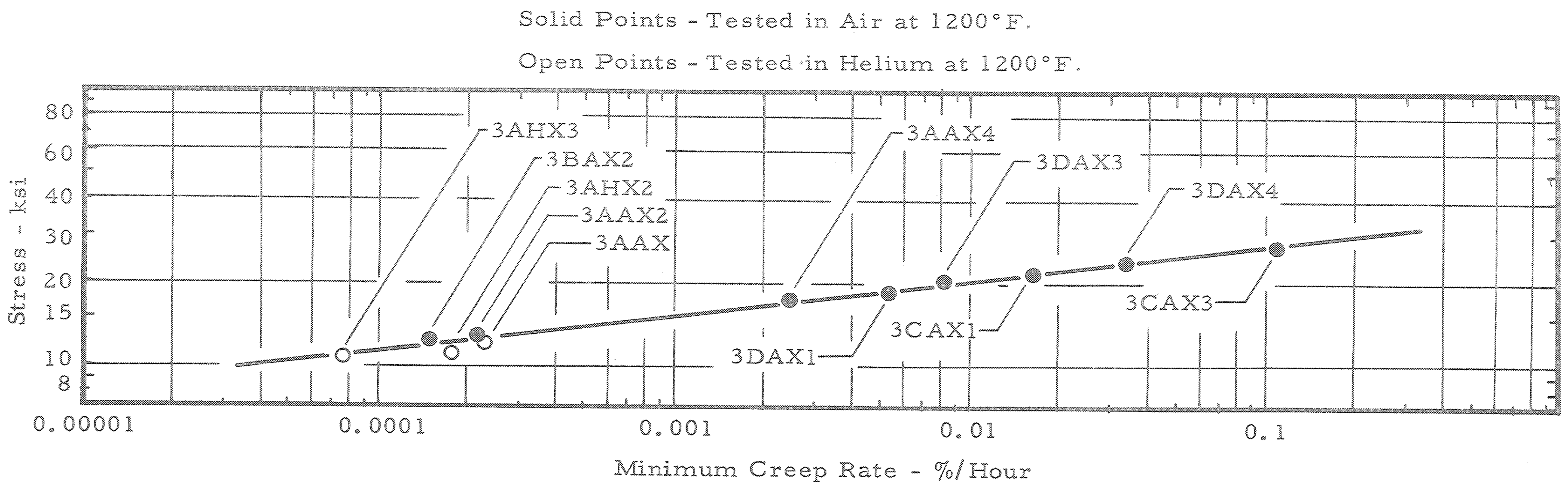

Figure 2. Stress versus minimum creep rate relationship for Type 316 austenitic steel tested at 1200°F in air and helium with no prior exposure.

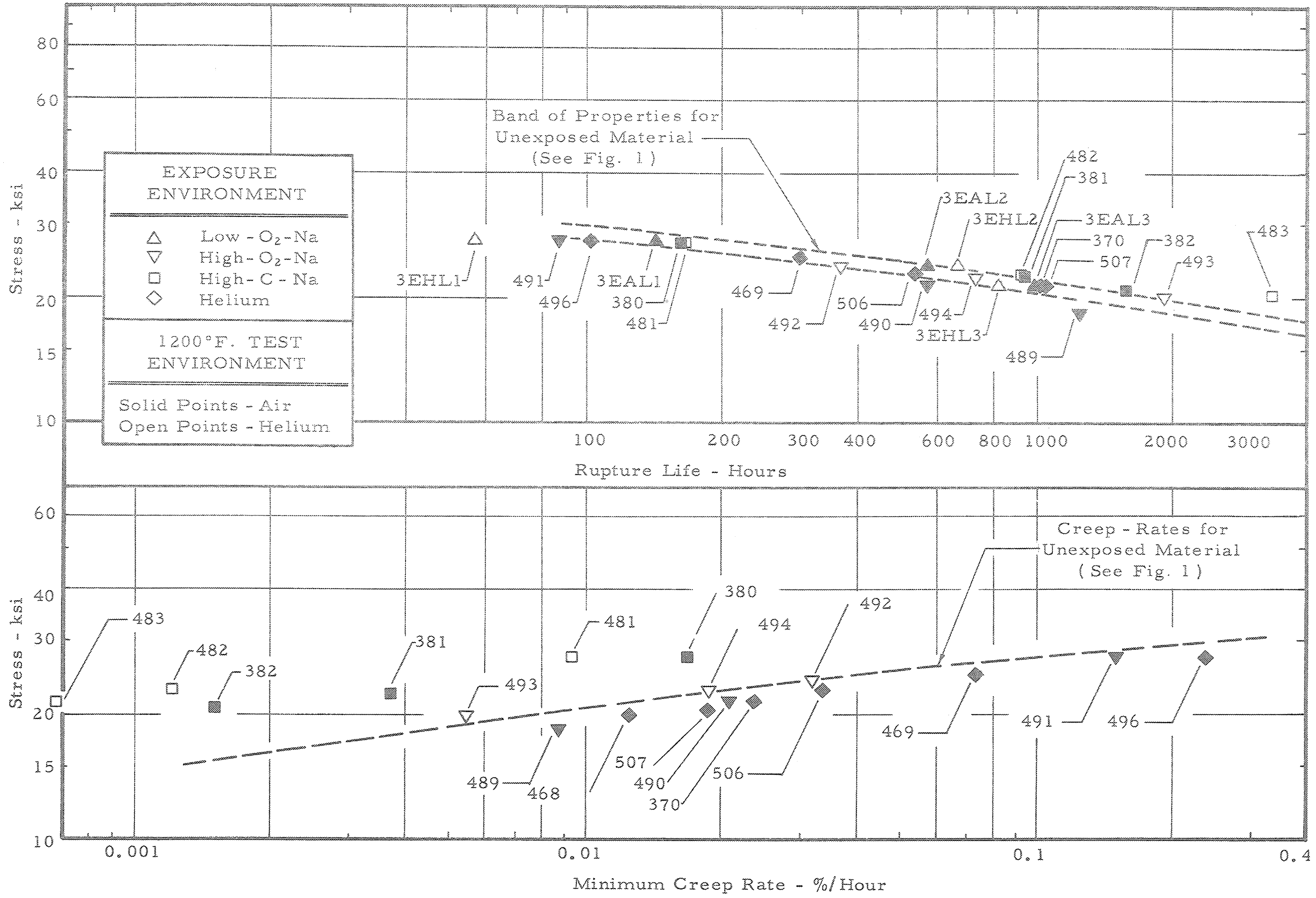

Figure 3. Stress-rupture time and minimum creep rate relationships for Type 316 austenitic steel tested at 1200°F in air and helium after exposure at 1200°F for 4000 hours in various environments.

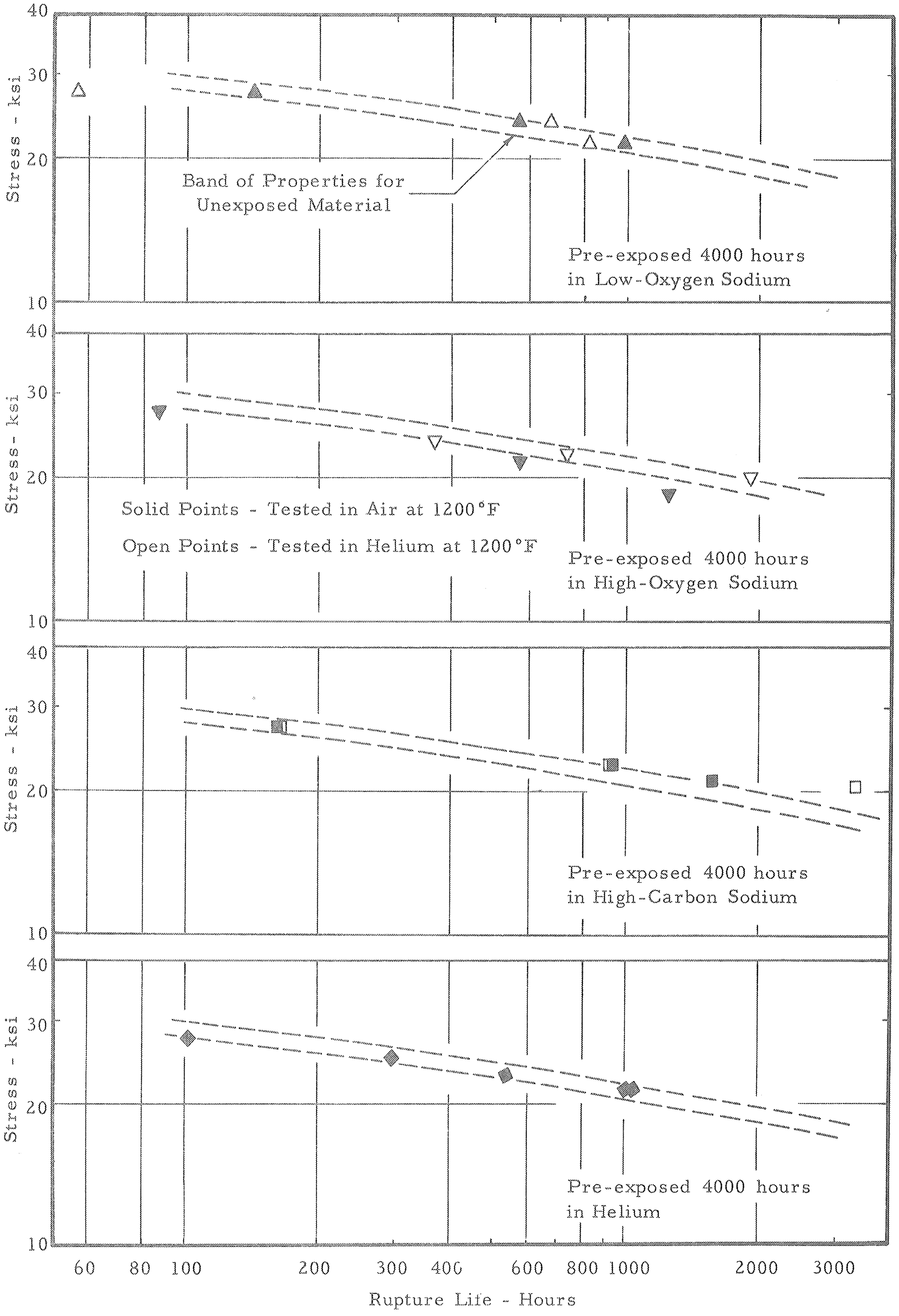

Figure 4. Stress-rupture time relationships for Type 316 austenitic steel tested at 1200°F in air and helium after various exposures.

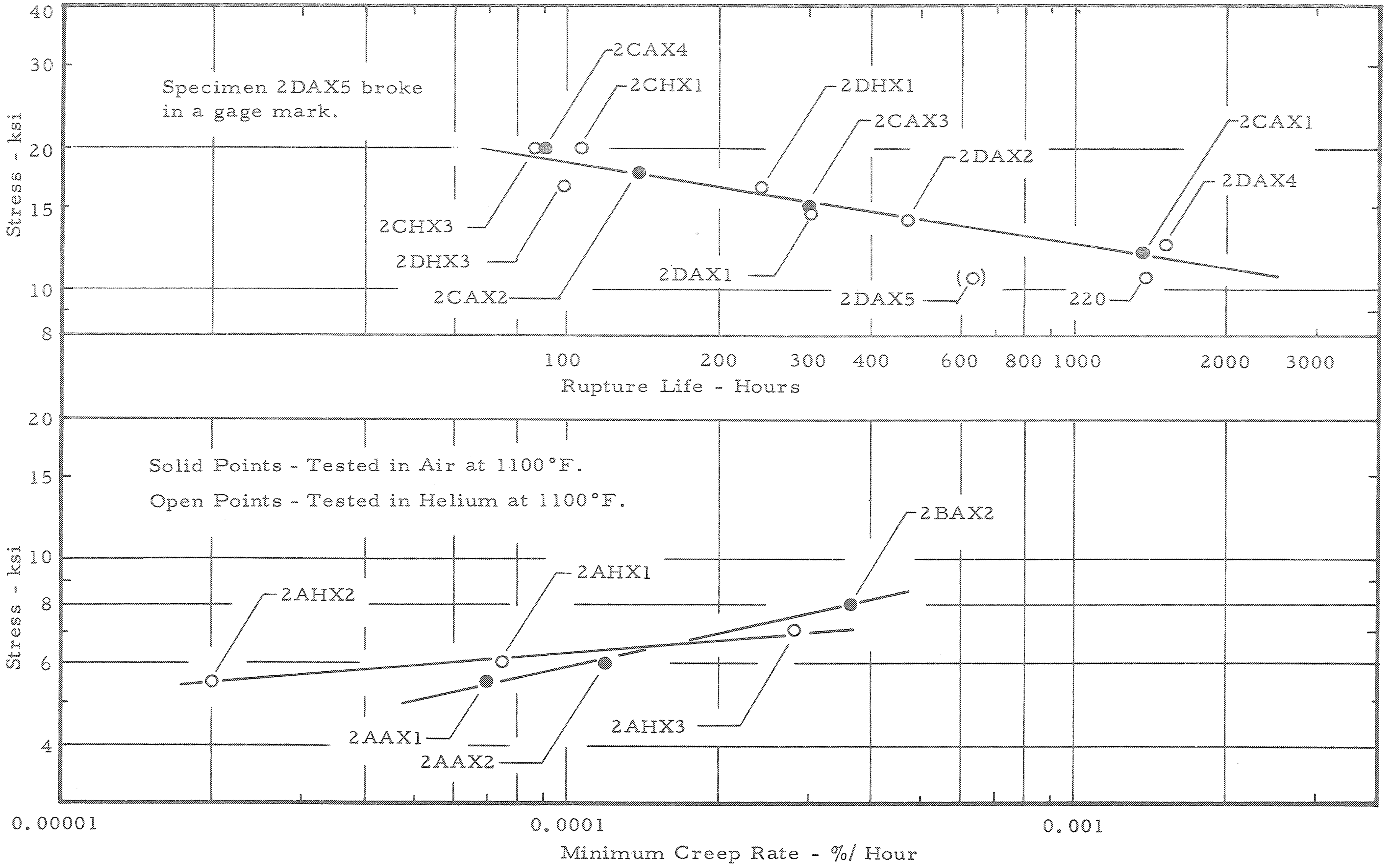

Figure 5. Stress-rupture time and minimum creep rate relationships for 2¼Cr-1Mo steel tested at 1100°F in air and helium with no prior exposure.

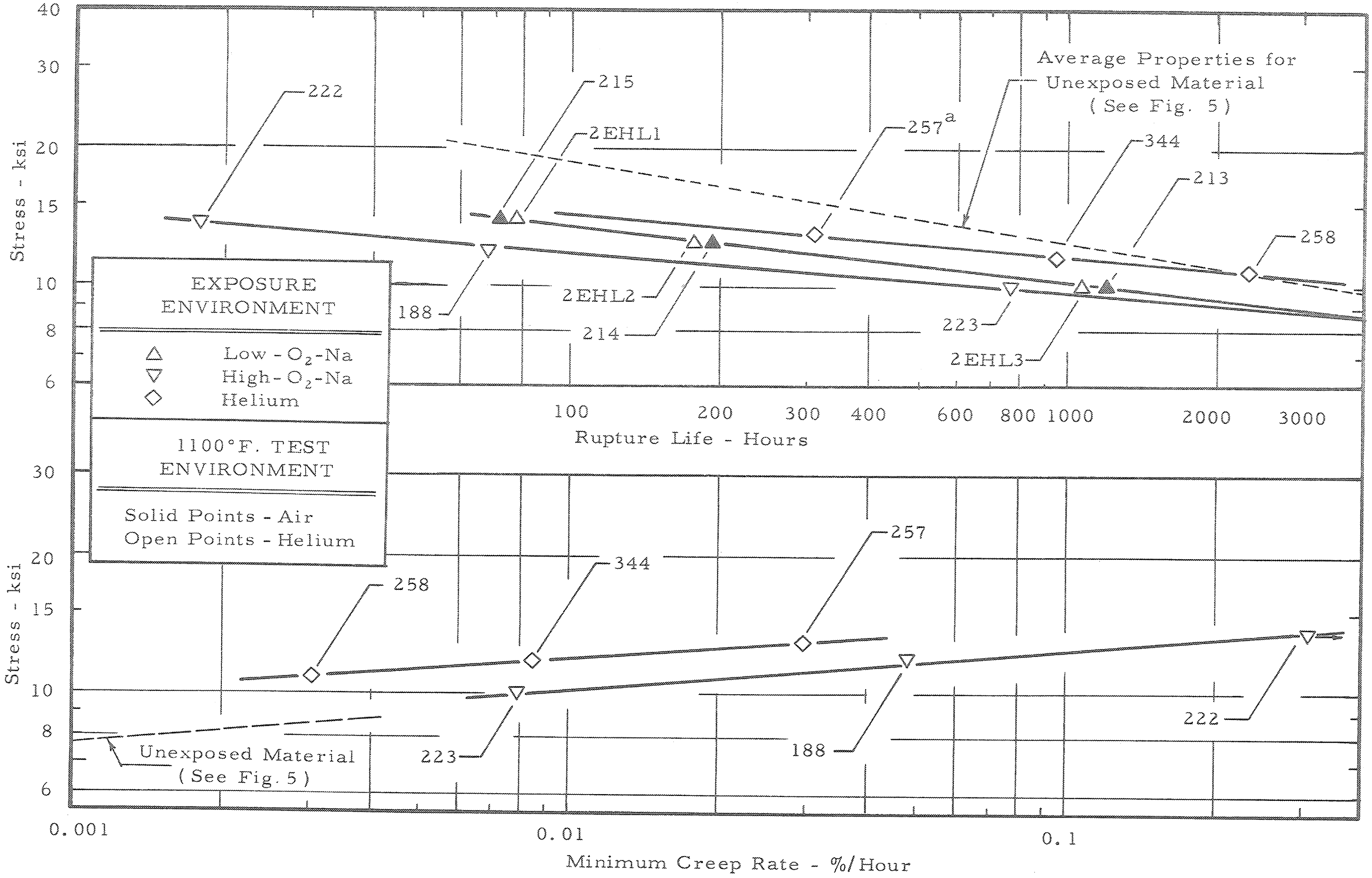

Figure 6. Stress-rupture time and minimum creep rate relationships for 2¼Cr-1Mo steel tested at 1100°F in air and helium after exposure at 1100°F for 4000 hours in various environments.