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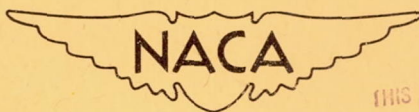
TECHNICAL NOTE 3108

RELATION OF MICROSTRUCTURE TO HIGH-TEMPERATURE
PROPERTIES OF A WROUGHT COBALT-BASE ALLOY

STELLITE 21 (AMS 5385)

By F. J. Clauss and J. W. Weeton

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Cleveland, Ohio



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RELATION OF MICROSTRUCTURE TO HIGH-TEMPERATURE PROPERTIES
OF A WROUGHT COBALT-BASE ALLOY, STELLITE 21 (AMS 5385)

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SUMMARY

An investigation was conducted to determine the effects of microstructure on the stress-rupture life of heat-treated wrought Stellite 21 under stress and temperature conditions comparable to those encountered during engine operation of turbine blades, and to correlate such properties as stress-rupture life, hardness, and ductility with microstructure. The microstructure of wrought Stellite 21, which may be considered a typical cobalt-base precipitation-hardening alloy, responds readily to solution treatment and to isothermal and aging heat treatments to form pearlitic and Widmanstätten structures as well as scattered precipitate. The results obtained in this investigation, although determined specifically for the alloy wrought Stellite 21, lead to conclusions that are believed to have general significance in the heat-treatment of other high-temperature alloys.

The optimum high-temperature properties are associated with a dispersion of fine precipitate scattered throughout the grains of the microstructure. Such a structure is obtained by a heat-treating cycle consisting of a solution treatment to produce a homogeneous solid solution, aging at a temperature low enough to produce scattered nucleation sites without permitting the growth of large particles, and aging a second time at a temperature slightly above that of the first aging in order to complete the precipitation process substantially by the growth of visible precipitates at the scattered nucleation sites.

Coarsening of the fine precipitates or the formation of a Widmanstätten structure by aging or isothermal transformation after solution treatment reduced the stress-rupture life but retained the low ductility. Formation of pearlite at still higher temperatures of aging or isothermal transformation favored lower stress-rupture life and higher ductility.

Hardness and elongation, which usually have a significance in predicting room-temperature properties, are less significant than

microstructure for predicting high-temperature properties. Neither hardness nor elongation provides a satisfactory criterion for correlating stress-rupture behavior, nor does overaging necessarily reduce the stress-rupture life.

INTRODUCTION

Previous investigations carried out at the NACA Lewis laboratory have reported the effect of heat treatment on the operating life of turbosupercharger blades of cast Stellite 21 alloy (refs. 1 to 3). This work was directed toward an immediate practical goal and was successful in showing that, by proper heat treatment, the service life of cast Stellite 21 blades could be doubled.

The present investigation is a continuation of work reported in reference 4, in which a survey was made of the microstructures produced by heat treatment in a number of high-temperature alloys. X-ray analyses made as part of that investigation showed that in several cases it was possible to develop sigma phase as a minor constituent in these alloys; but that, for the most part, the minor phases were carbides such as Cr_{23}C_6 , Cr_7C_3 , and M_6C , where M is any of the carbide-forming metals. At that time, particular attention was paid to the development of a pearlitic precipitate, since earlier work (ref. 3) had shown that best operating lives of turbine blades were associated with such a microstructure.

Later, the precipitation of the minor phases in wrought Stellite 21 by heat treatment was studied more extensively (ref. 5). In this study, the wrought alloy was first solution-treated 72 hours at 2250°F to dissolve all the carbides and other minor phases into the face-centered cubic matrix. The material was then aged or isothermally transformed at temperatures below the solution-treating range to cause reprecipitation of minor phases in a manner dependent upon the transformation temperature. It was demonstrated that a variety of microstructures could be produced, including pearlite, star-shaped Widmanstätten structures, and general precipitation. In water-quenched and aged specimens, precipitation occurred on slip planes and twin boundaries in the solid-solution matrix.

The present investigation was made to determine the effects of microstructure on the stress-rupture life of heat-treated wrought Stellite 21 under stress and temperature conditions comparable to those encountered during engine operation of turbine blades, and to correlate such properties as stress-rupture life, hardness, and ductility with microstructure.

This program was specifically limited to Stellite 21, a typical cobalt-chromium-base alloy, because of the large amount of information available from previous investigations. Wrought rather than cast material was used in order to avoid the scatter in properties and grain-size variables associated with castings. It is believed that the information obtained in this investigation pertains not only to the alloy studied but is of a fundamental character and will prove applicable to a large number of other high-temperature alloys.

MATERIAL, APPARATUS, AND PROCEDURE

Material. - The wrought Stellite 21 used in this investigation had the following chemical analysis:

| Element | Percent |
|------------|--------------------|
| Chromium | 28.75 |
| Nickel | 3.01 |
| Cobalt | Balance (61.10) |
| Molybdenum | 5.52 |
| Iron | .33 |
| Silicon | .44 |
| Manganese | .56 |
| Carbon | .29 |

The alloy was supplied to the NACA by the manufacturer in the form of 3/4-inch-diameter round bar stock.

The "as-received" microstructure (fig. 1) consisted of equiaxed grains with large particles of the carbide $Cr_{23}C_6$ (ref. 4) scattered throughout. The grain size of the bar stock was very small (A.S.T.M. 8), and the hardness was Rockwell C-43.

Heat-treatment specimens. - Specimens for heat treatment were cut from the 3/4-inch-diameter bar stock in $2\frac{3}{4}$ - or $3\frac{1}{4}$ -inch lengths. The bars were cut into quarters so that four specimens could be obtained from each cross section. These specimens provided material for both stress-rupture testing and metallographic examination.

Heat-treatment apparatus. - The apparatus used for heat treatment included several tube furnaces heated by silicon carbide resistance elements. Specimens were inserted in a $2\frac{1}{4}$ -inch-inside-diameter

Inconel tube with radiation shields at each end of the heating zone. A stream of argon gas flowing through the Inconel tubes provided an inert atmosphere for heat treatment. The argon gas was 99.6 percent pure and was further purified before use by passing over titanium chips held at 1300° to 1400° F.

Specimen temperatures were measured with a platinum to platinum-plus-13-percent-rhodium thermocouple located within the tube directly over the specimens. Furnace temperatures were controlled to give a maximum variation of $\pm 10^\circ$ F from the nominal specimen temperature.

Heat treatments studied. - The material was studied in the "as-wrought" condition and in the conditions of heat treatment listed in table I.

Examination of heat-treated specimens. - Metallographic examinations were made of the microstructures developed by heat treatment. For this purpose, 5-percent aqua regia used electrolytically was generally employed as the etchant. Hardness measurements were taken on at least two specimens from each heat-treatment group.

Stress-rupture tests. - Figure 2 shows the type of specimen used for stress-rupture testing. A thermocouple was fastened to the gage section of the specimen, and the temperature was held at $1500^\circ \pm 10^\circ$ F throughout the test. The test temperature of 1500° F was selected because it is approximately the temperature in the failure zone of J33 turbine blades. The calculated centrifugal stress in the failure zone is about 21,000 pounds per square inch, so that stresses on this order were chosen for the stress-rupture tests of the present investigation.

The percentage elongation of the stress-rupture specimens was reported for a 1-inch-gage length, although punch marks were placed $\frac{1}{2}$ inches in opposite shoulders of the specimens.

Examination of fractured stress-rupture specimens. - Specimens representing short life (high stress, about 28,000 psi), medium life (medium stress, about 21,000 psi), and long life (low stress, about 16,000 psi) in stress-rupture were selected from each heat treatment and examined metallographically in the areas adjacent to the fractured edges in order to determine the final structures and paths of fracture. Rockwell C-hardness measurements were taken $\frac{1}{8}$ inch from the fractured edge on flat surfaces ground on the specimens.

Room-temperature tensile tests. - In addition to the stress-rupture tests, room-temperature tensile tests were carried out on specimens from heat-treatment groups 20 and 26. Yield strength, ultimate strength, and ductility were determined. Hardness before and after fracture was measured.

RESULTS

Stress-rupture life. - The stress-rupture lives of specimens of Stellite 21 tested at 1500° F in the as-wrought condition were as follows:

| | | | | | | | | |
|-------------|--------|---------------------|---------------------|---------------------|--------|--------|--------|---------------------|
| Stress, psi | 25,000 | ^a 21,000 | ^a 21,000 | ^a 20,400 | 20,000 | 19,000 | 18,000 | ^a 16,000 |
| Life, hr | 6.1 | >16.4 | >74.4 | >14.4 | 10.4 | 4.0 | 15.5 | >57.7 |

^aElongated beyond operating limit of test machines (equivalent to approximately 150-percent elongation) so that power was cut off at time indicated and specimens were removed unbroken.

Results for the stress-rupture specimens given the various heat treatments are plotted in figures 3, 12, and 13. Wherever possible, cross plots (such as those shown in fig. 11) were prepared to allow the greatest use to be made of the experimental data and to average out experimental errors. The procedure in preparing the stress-rupture plots was: (1) to plot the raw data on conventional plots of logarithm stress against logarithm life and construct an average line (figs. 3, 12, and 13); (2) to plot the average life from these plots for stresses of 15,000, 20,000, 25,000, and 30,000 pounds per square inch against the temperature of heat treatment (fig. 11); and (3) to balance discrepancies on both types of plot simultaneously in order to obtain a consistent trend of behavior.

Elongation. - Total elongations of stress-rupture specimens are noted on the stress-rupture plots (figs. 3, 12, and 13). There were no consistent differences in the elongation values for specimens tested at high stress (short life) and those tested at low stress (long life). All specimens in any one heat-treatment group had approximately equal elongations at fracture, regardless of the stress or life. Average elongation values are presented in figure 4.

For isothermal transformation, increasing the time from 2 to 72 hours resulted in higher elongations for transformation temperatures above approximately 1600° F and lower elongations for transformation temperatures below 1600° F (fig. 4(a)). For the aging treatments, increasing the time from 2 to 72 hours resulted in higher elongations for aging temperatures above approximately 1400° F and approximately equal elongations for aging temperatures below 1400° F (fig. 4(b)). In all cases, specimens isothermally transformed gave equal or greater elongation than specimens aged for the same time and at the same temperature. Highest elongations were obtained by 72-hour isothermal transformation at 1750° F.

Hardness. - Hardness measurements made of specimens before and after fracture for the various heat treatments are noted in the stress-rupture plots (figs. 3, 12, and 13). As with elongation values, there were no consistent differences in the hardness values after fracture at 1500° F for specimens within any one heat-treatment group, regardless of the stress or life.

Average hardness measurements for the isothermal transformation treatments and the aging treatments are presented in figure 5 as a function of the temperature of heat treatment. From these figures, the following may be noted: First, the hardness behaviors of the isothermally transformed and the aged specimens are almost identical. Second, at temperatures above 1700° F, increasing the time from 2 to 72 hours resulted in lower hardness. Third, during stress-rupture testing at 1500° F, all specimens hardened to a narrow range of values above that for the condition as heat treated.

Metallographic examination. - The microstructures developed by the various heat treatments as well as the final microstructures and paths of fracture after stress-rupture testing at 1500° F are shown in figures 6 to 10.

The structures formed after isothermal transformation for 2 hours (groups 2 to 6) and 72 hours (groups 7 to 12) at various temperatures are shown in figures 7(a) and (b). For both transformation times and at all temperatures investigated, the structure is characterized by a pearlitic precipitation of the carbides. In general, the amount and the interlamellar spacing of the pearlite increase with increasing temperature, which is typical of most lamellar formations. At the shorter transformation time, the pearlite appears to be nucleated primarily at the grain boundaries; while, at the longer time, some precipitation within the grains is also evident, particularly at the higher temperatures. Also noted is a pronounced spheroidization of precipitates in 72 hours at 1950° F.

Microstructures after aging (fig. 7(c), groups 13 to 17, and fig. 7(d), groups 18 to 26) are generally similar to those after isothermal transformation, in that pearlite is formed at all temperatures. The amounts of pearlite, however, are less than for isothermal transformation; and, in addition, much more scattered precipitation within the grains is developed by aging. Increasing the time of aging from 2 to 72 hours results in an increase in the amount of this scattered precipitate within the grains rather than the growth of pearlite from the grain boundaries.

Photomicrographs of specimens fractured in stress-rupture at 1500° F are shown in figure 8. Comparison of the photomicrographs of the fractured specimens with those of the same specimens as heat treated

shows that precipitation occurred in all cases during stress-rupture testing by the formation of scattered particles throughout the grains. The amount of additional precipitation that occurs during testing varies inversely with the amount of precipitate present in the condition as heat treated. Fractures are observed to be both intercrystalline and transcrystalline.

Figure 9 shows the effect of the double-aging heat treatment (group 27) on the microstructure of wrought Stellite 21. After the first age of 72 hours at 1200° F, the only precipitate produced in the solution-treated structure consisted of small patches of densely packed pearlite along grain boundaries. Subsequent aging at 1500° F developed a fairly uniform scattering of fine precipitate throughout the grains. The second aging at 1500° F substantially completed the precipitation process, and little further precipitation after stress-rupture testing was visible. Fracture of this specimen was definitely intercrystalline.

Microstructures after aging the wrought Stellite 21 without a prior solution treatment are shown in figure 10. Aging for 72 hours at 1200° F produced no noticeable change in the microstructure from that of the as-wrought condition. After 72 hours at 1500° F, the microstructure consisted of both carbide "islands" and a scattered "salt-and-pepper" type of general precipitate. No noticeable grain growth had occurred at 1500° F. Solution of the grain boundaries and growth of carbide particles had taken place at 1950° F.

Room-temperature tensile tests. - Results of the room-temperature tensile tests were as follows:

| Property | Group 20 (solution- treated, aged 72 hr at 1200° F) | Group 26 (solution- treated, aged 72 hr at 1950° F) |
|---|--|--|
| Yield point, psi | 70,000 | 88,000 |
| Ultimate strength, psi | 139,000 | 174,000 |
| Elongation, percent | 25.0 | 27.5 |
| Reduction in area, percent | 17.7 | 23.6 |
| Hardness, before testing, Rockwell C | 22 | 25 |
| Hardness, after fracture, Rockwell C | 43 | 44 |

DISCUSSION

Past work on Stellite 21 (ref. 5) showed that the minor constituents in this alloy can be taken into solid solution by treatment at elevated temperatures on the order of 2250° F. This work also showed that subsequent transformation at lower temperatures could be controlled to reprecipitate these minor phases to produce different microstructures, depending on the times and temperatures of transformation. The differences in the physical properties of the material after the various heat treatments studied in the present investigation are discussed here in terms of the microstructural differences.

In the as-wrought material, precipitation of the carbon as the carbide Cr_{23}C_6 (ref. 4) has been nearly completed during the rolling operations. Agglomeration and spheroidization of the carbide particles has occurred to the extent shown in figure 1. This figure also shows the arrangement of the carbide particles as "stringers." The poor strength of this structure is shown by the short rupture lives at 1500° F. The very erratic behavior did not permit drawing a representative stress-rupture curve for the as-wrought material.

The high-temperature stress-rupture behavior of the wrought material was considerably improved by the use of a solution treatment alone prior to testing. Figure 3(a) shows the stress-rupture curve at 1500° F for the solution-treated wrought material. It is impossible to say that the curve of this figure fairly represents the behavior of the alloy in a solution-treated condition throughout the test, since aging has occurred during the warm-up period prior to the application of the load and during the test itself. The development of precipitate from the solution-treated condition by this aging undoubtedly accounts for the increased stress-rupture lives and the lowered elongation as compared with the as-wrought material.

For convenience of comparison, the stress-rupture results of figures 3(b) to (e) are cross-plotted in figure 11, which is a plot of the stress-rupture life at 1500° F against the aging or isothermal transformation temperatures for lines of constant stress. There is a general similarity in the stress-rupture behavior, in that the curves for all four sets of heat treatments show a minimum at an aging or isothermal transformation temperature of about 1400° F. Improvement is obtained by aging or isothermally transforming the solution-treated material at temperatures either above or below 1400° F.

For the 2-hour isothermal transformation treatment, the curves are rather flat with a poorly defined minimum. The best stress-rupture life is obtained by transformation at 1950° F. This temperature is fairly close to the limit of improvement, as solution treatment alone

at 2250° F gave poorer stress-rupture life. Increasing the time of isothermal transformation from 2 to 72 hours gives a more pronounced minimum. At both 1200° and 1950° F, there was an increase in stress-rupture life over that obtained by a 2-hour isothermal transformation. This is particularly true at 1200° F (where the increase in time has approximately doubled the stress-rupture life), and the stress-rupture life for transformation at this temperature is now better than that for transformation at 1950° F. Between these two extremes of temperature, increasing the time of isothermal transformation from 2 to 72 hours actually decreased the stress-rupture lives.

In the case of the aging treatments, increasing the time of aging from 2 to 72 hours improves the stress-rupture lives for all temperatures of aging. By extending the range of 72-hour aging temperatures down to 1000° F, it was possible to identify a maximum in this set of curves at 1200° F, with a gradual decrease below 1200° F. The 72-hour aging treatment at 1200° F after solution treatment produced the highest stress-rupture life of all the heat treatments shown in figures 11 to 13.

The elongation of the specimens that had the best stress-rupture life (i.e., group 20, those aged 72 hr at 1200° F after solution treatment) was very small (about 2 percent). This follows the usual association of high stress-rupture life with low ductility. However, for other heat-treatment groups it was found that this concept generally did not hold true. For example, some specimens that had the poorest stress-rupture lives had equally small elongations (e.g., those aged 72 hr at 1350° F after solution treatment, which elongated 2 percent), while other specimens that had reasonably good stress-rupture lives had relatively high elongations (e.g., those aged 72 hr at 1950° F after solution treatment, which elongated 10 percent). Thus, it may be concluded from this investigation that elongation and stress-rupture life do not necessarily have a direct relation.

As with elongation, there is no simple relation between the stress-rupture life and the hardness, whether the hardness "as heat treated" or "after fracture" is used as the criterion. Low hardness may be associated with both good and bad stress-rupture lives. For example, the hardness as heat treated of the specimens that had the best stress-rupture lives (group 20, aged 72 hr at 1200° F after solution treatment) was only about Rockwell C-24; this is essentially the hardness of solution-treated specimens (group 1), which had among the poorest stress-rupture lives. Specimens with the highest hardness values had generally poor stress-rupture lives (e.g., group 21). Maximum hardness was developed at a temperature of 1750° F for 2-hour aging or isothermal transformation and at about 1400° F for 72-hour aging or isothermal transformation. The minimum stress-rupture lives, however, were for temperatures of about 1400° F, regardless of the time of heat treatment.

A further point to be noted from a comparison of the hardness results is that the overaging, or softening, which takes place at temperatures above 1700° F by increasing the time of aging from 2 to 72 hours, occurs with an increase in the stress-rupture life.

As noted, the visible precipitation during transformation of solution-treated wrought Stellite 21 at 1200° F is largely confined to formation of small patches of pearlite along grain boundaries. While the microstructure undergoes little apparent change by increasing time at 1200° F from 2 to 72 hours, the stress-rupture life at 1500° F is approximately doubled. Examination of specimens after fracture at 1500° F shows the development of considerable quantities of scattered precipitate throughout the grains. This precipitate is more uniformly scattered for the specimens transformed for 72 hours than for those transformed only 2 hours at 1200° F. The microstructure after heat treatment is not one that would ordinarily be expected to give high stress-rupture life, and the high values obtained can be attributed to the strengthening of the material by precipitation during testing. The size and the distribution of the precipitate developed during testing are apparently influenced both by small amounts of straining under load and by the existence of nucleation sites produced by the earlier transformation at 1200° F. Since the matrix is strengthened by this scattered precipitation, the development of the precipitate would tend to cause the fracture to occur along grain boundaries. This phenomenon is supported by the observation that fractures in these specimens were predominantly intergranular. Cracks within grains were also observed and may be due to localized straining before precipitation has had a chance to occur.

The fine, scattered precipitate associated with high stress-rupture life for specimens aged or isothermally transformed 72 hours at 1200° F may also be associated with low ductility. Specimens heat-treated at 1350° F had equally low ductilities but very poor stress-rupture lives. The precipitate developed at 1350° F consisted of small amounts of pearlite together with a Widmanstätten structure. Specimens isothermally transformed 72 hours at 1750° F (group 10) had large amounts of pearlite in their microstructures and showed very large elongations and relatively poor stress-rupture lives. Relatively little scattered precipitate was developed during stress-rupture testing of these specimens. By aging 72 hours at 1750° F (group 24) in place of the isothermal transformation, scattered precipitates were developed along with the pearlite during heat treatment, the elongation was reduced, and the stress-rupture life improved. Aging or isothermal transformation at 1950° F developed considerable quantities of pearlite in the microstructures, but precipitation was not completed at this temperature (1950° F is high enough, as already noted, to cause partial solution treatment of the as-wrought material). As a result,

additional precipitate was developed during stress-rupture testing at 1500° F to strengthen the material. Specimens heat-treated at 1950° F combined relatively high ductilities with good stress-rupture lives.

The general relation between structure and properties that may be deduced from these and similar observations is that a fine, uniformly scattered precipitate favors high stress-rupture life and low ductility; whereas, a predominantly pearlitic structure favors lower stress-rupture life and higher ductility. The fine precipitate can be developed during heat treatment prior to testing or during the stress-rupture test itself. As the scattered precipitate is coarsened or changed to a Widmanstätten type, the low ductility remains and the stress-rupture life is drastically reduced. The performance of specimens that contain both fine, scattered precipitate and pearlite is a compromise based on the relative quantities of the different structures.

In order to confirm these conclusions and to apply them for further improving the properties of the material, several additional tests were conducted. These tests were the room-temperature tensile tests and the double-aging heat treatments described in previous sections.

Room-temperature tensile tests were made on specimens that had been solution-treated and aged 72 hours at 1200° F (group 20) in order to test the proposition that this material after heat treatment was not inherently strong but owed its high stress-rupture life to the formation of scattered precipitate during testing at 1500° F. While it was recognized that strain-hardening could occur during the test and strengthen the material, the test was conducted at room temperature in order to avoid thermal aging or precipitation and growth of carbides. As a basis for comparison, another room-temperature tensile test was made on material having the next best stress-rupture life (group 26, solution-treated and aged 72 hr at 1950° F) and containing a large quantity of visible precipitate. Results showed that the material that had been aged 72 hours at 1200° F after solution treatment had a lower yield point and lower ultimate strength than the material that had been aged 72 hours at 1950° F after solution treatment. Both materials underwent a considerable amount of strain-hardening during the test and had surprisingly high room-temperature ductility (25.0 and 27.5 percent elongations). While not conclusive in themselves, these results support the belief that the high stress-rupture life of specimens aged 72 hours at 1200° F after solution treatment is not due to the structure of the material after heat treatment, but rather to changes in the microstructure during testing.

A more direct test of this belief, in which an attempt was made to take advantage of such changes in microstructure, consisted in the use of a double-aging heat treatment (group 27, solution-treated, aged

72 hr at 1200° F plus 24 hr at 1500° F). The first aging at 1200° F was expected to produce a large number of nucleation sites at which subsequent precipitation and growth at the temperature of stress-rupture testing would occur, and the second aging at 1500° F was expected to strengthen the matrix by precipitation at these nucleation sites before the specimens were deformed under load. This, it was further felt, might result in an additional increase in the stress-rupture life.

The results of this test confirmed these beliefs: The microstructure did consist of fine, uniformly scattered precipitate (fig. 9); and, on the basis of the limited number of points, the stress-rupture life of the material was the highest obtained for all heat treatments studied.

The three curves in figure 12 illustrate the improvement in stress-rupture life obtained by aging wrought Stellite 21 from the solution-treated condition. The lower curve represents the stress-rupture life after solution treatment but without any aging before testing at 1500° F (group 1); the middle curve shows the best results obtained for a single age (group 20, solution-treated, aged 72 hr at 1200° F); and the upper curve shows the additional improvement obtained by adding a second age at 1500° F. The stress-rupture life of the fine-grained wrought Stellite 21 given the last heat treatment is slightly longer than the best stress-rupture lives for the coarse-grained cast Stellite 21 reported in reference 6.

Many of the carbide precipitates present in cast cobalt-base alloys similar to Stellite 21 are not in completely stable forms and may be transformed to more stable structures by simple aging at elevated temperatures. This type of aging is described in reference 7. In addition to such carbide reactions, precipitation of carbon from the supersaturated matrix of the cast structure can also occur during aging. Both of these changes may be involved in the development of the scattered precipitates observed about the original carbide islands in many cast cobalt-base alloys after aging; and, together or singly, these changes may account for the improvement in the stress-rupture life of these materials obtained by simple aging. On the other hand, the carbides in wrought Stellite 21 have already been reduced to a relatively stable form (Cr_{23}C_6 , ref. 4); and precipitation of carbon from the matrix solid solution has been substantially completed during the rolling operations. Thus, substantial improvement in the stress-rupture life by aging wrought Stellite 21 might not be expected unless preceded by some degree of solution treatment. This is shown by the results for specimens of wrought Stellite 21 aged for 72 hours at temperatures of 1200°, 1500°, and 1950° F without prior solution treatment (groups 28, 29, and 30, respectively). These results are plotted in figure 13, where they are compared with the results obtained when the same aging treatments were preceded by solution treatment.

Aging the as-wrought alloy for 72 hours at 1200° F (fig. 10) produced no perceptible change in the microstructure of the material. The stress-rupture behavior remained the same as without heat treatment - that is, poor lives (e.g., 0.7 hr at 23,000 psi and 1500° F), erratic behavior, and very high elongation at fracture (e.g., 127 percent for the specimen tested at 21,000 psi). In all cases, the specimens failed in shorter times than when aging at 1200° F was preceded by solution treatment.

Aging the wrought alloy for 72 hours at 1500° and 1950° F gave some improvement in the stress-rupture life over that of the as-wrought condition, but again the lives were poorer than when the aging was preceded by solution treatment. It may be added that some solution treatment was effected by "aging" at 1950° F. Precipitates in the grain boundaries were removed and carbides within the grains were spheroidized by aging at this temperature. Whether temperatures as high as 1950° F are to be considered aging or solution-treating temperatures is a matter of definition and the condition of the alloy. With a solution-treated material, "aging" is the more properly descriptive term for holding at 1950° F; while, if the alloy is in the as-wrought condition, the term "solution treatment" is probably more applicable.

SUMMARY OF RESULTS

Studies were made of heat-treated wrought Stellite 21 in order to correlate stress-rupture life, hardness, and ductility with microstructure. The results of these studies may be summarized as follows:

1. Best stress-rupture life for wrought Stellite 21 was obtained by solution-treating (16 hr at 2250° F) followed by double-aging (72 hr at 1200° F plus 24 hr at 1500° F).
2. Specimens of as-wrought Stellite 21 tested without any prior heat treatment exhibited erratic behavior in stress-rupture testing. Lives were generally low and elongations high.
3. The stress-rupture life of wrought Stellite 21 was improved by solution treatment alone prior to testing. Precipitation from the solution-treated structure occurred during testing and strengthened the matrix.
4. The stress-rupture life could be further increased by aging or isothermally transforming the solution-treated material before testing. Transformation at temperatures between 1300° and 1700° F gave poorer stress-rupture lives (minimum at 1400° F) than solution treatment alone, while transformation at temperatures from 1700° to 1950° F and from 1000° to 1300° F gave better lives. The best single transformation temperature was 1200° F. By aging 72 hours at 1200° F

following solution treatment, the stress-rupture life was more than doubled over that for solution treatment alone (90 hr compared with 37.5 hr at 21,000 psi and 1500° F). Increasing the time of aging from 2 to 72 hours improved the stress-rupture life for all aging temperatures. For isothermal transformation, the increase in time was significantly beneficial only for transformation at 1200° F.

5. All specimens given the same heat treatment had approximately equal elongations after fracture at 1500° F, regardless of the stress or life. Specimens solution-treated and isothermally transformed gave higher elongations than those solution-treated and aged under the same conditions of time and temperature. Increasing the time from 2 to 72 hours resulted in higher elongations for isothermal transformation at temperatures above 1600° F and for aging above 1400° F. Highest elongations were obtained by 72-hour isothermal transformation at 1750° F.

6. Both the highest and lowest stress-rupture lives were associated with low ductility (0 to 2 percent elongation). Many specimens with relatively high ductility (15 percent elongation) had good stress-rupture lives. In general, the elongation and the stress-rupture life of heat-treated wrought Stellite 21 showed no direct relation to each other.

7. The hardness behaviors of specimens aged or isothermally transformed after solution treatment were similar. A maximum hardness of Rockwell C-42 was developed by aging 72 hours at 1350° F after solution treatment. Overaging or softening took place at temperatures above 1700° F by increasing the time from 2 to 72 hours. All specimens hardened during stress-rupture testing at 1500° F to a narrow range of values above that for the heat-treated condition, regardless of the heat treatment.

8. In general, the hardness and the stress-rupture life of heat-treated wrought Stellite 21 showed no direct relation to each other. For example, overaging or softening during heat treatment did not necessarily shorten the stress-rupture life.

9. Isothermal transformation for 2 hours after solution treatment occurs principally with the formation of pearlite along grain boundaries. Increasing the time of isothermal transformation from 2 to 72 hours at temperatures in the range 1350° to 1950° F causes an increase in the amount of pearlite, divorcing of the pearlite, and/or the formation of scattered precipitates within the grains, depending upon the temperature.

10. Less pearlite and more general precipitation are formed by aging than by isothermal transformation.

11. Heat-treated specimens underwent additional precipitation during stress-rupture testing by the formation of fine precipitates scattered randomly throughout the grains, along slip lines or twin boundaries.

12. In general, scattered fine precipitates favored high stress-rupture life and low ductility. Coarsening of the fine precipitate or the formation of a Widmanstatten structure decreased the stress-rupture life but still favored low ductility. Pearlite favored lower stress-rupture life and higher ductility.

13. Scattered precipitate in the final microstructure was best formed in wrought Stellite 21 by an initial solution treatment at high temperature (e.g., 2250° F), followed by formation of nucleation sites at a low temperature (e.g., 1200° F), and finally the development of the visible precipitates at an intermediate temperature (e.g., 1500° F). The development of the visible precipitates may take place as the final step in the heat-treating cycle or, if omitted there, may occur during the stress-rupture test. The highest stress-rupture life was obtained when the development of the visible precipitates was essentially completed before stress-rupture testing by the use of a double-aging treatment (16 hr at 2250° F, air cool, age 72 hr at 1200° F plus 24 hr at 1500° F).

14. Paths of fracture in heat-treated specimens were predominantly intercrystalline, though some evidences of transcrystalline failure were also found.

CONCLUSIONS

The results obtained in this investigation, although determined specifically for the alloy wrought Stellite 21, lead to conclusions that are believed to have general significance in the heat treatment of other high-temperature alloys. Hardness and elongation, which usually have significance in predicting room-temperature properties, are less significant than microstructure for predicting high-temperature properties. Neither hardness nor elongation provides a satisfactory criterion for correlating stress-rupture behavior, nor does overaging necessarily reduce the stress-rupture life.

The optimum high-temperature properties are associated with a dispersion of fine precipitate scattered throughout the grains of the microstructure. Such a structure is obtained by a heat-treating cycle consisting of the following steps: (1) solution treatment to produce a homogeneous solid solution, (2) aging at a temperature low enough to produce scattered nucleation sites without permitting the

growth of large particles, and (3) a second aging at a temperature slightly above that of the first aging in order to complete the precipitation process substantially by the growth of visible precipitates at the scattered nucleation sites.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 15, 1953

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2. Yaker, C., and Hoffman, C. A.: Effects of Some Solution Treatments Followed by an Aging Treatment on the Life of Small Cast Gas-Turbine Blades of a Cobalt-Chromium Base Alloy. I - Effects of Solution-Treating Temperature. NACA TN 2320, 1951.
3. Hoffman, C. A., and Robards, C. F.: Effects of Some Solution Treatments Followed by an Aging Treatment on the Life of Small Cast Gas-Turbine Blades of a Cobalt-Chromium-Base Alloy. II - Effect of Selected Combinations of Soaking Time, Temperature, and Cooling Rate. NACA TN 2513, 1951.
4. Weeton, J. W., and Signorelli, R.: An Investigation of Lamellar Structures and Minor Phases in Eleven Cobalt-Base Alloys Before and After Heat Treatment. NACA TN 3109, 1954.
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6. Anon.: Haynes Alloys for High-Temperature Service. Haynes Stellite Co. (Kokomo, Ind.), 1948.
7. Lane, J. R., and Grant, N. J.: Carbide Reactions in High Temperature Alloys. Trans. Am. Soc. Metals, vol. 44, 1952, pp. 113-134; discussion, pp. 134-137.

TABLE I. - SCHEDULE OF HEAT TREATMENTS STUDIED

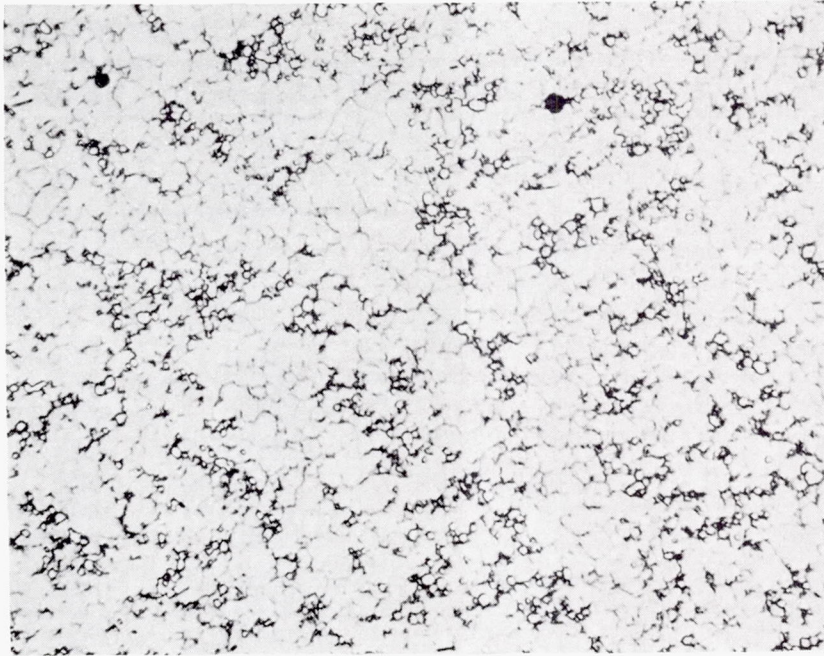
| Group | Solution treatment | | Heat treatment (aging or isothermal transformation) | | |
|-------|--------------------------|-----------------|--|--------------------|--------------------|
| | Temperature, °F | Time, hr | Isothermal or aging | Temperature, °F | Time, hr (a) |
| 0 | As-wrought | | No heat treatment | | |
| 1 | 2215 | ^a 16 | No heat treatment | | |
| 2 | 2250 | ^b 16 | Isothermal | 1200 | 2 |
| 3 | | | | 1350 | |
| 4 | | | | 1600 | |
| 5 | | | | 1750 | |
| 6 | | | | 1950 | |
| 7 | | | | 1200 | |
| 8 | | | 1350 | | |
| 9 | | | 1550 | | |
| 10 | | | 1750 | | |
| 11 | | | 1830 | | |
| 12 | | | 1950 | | |
| 13 | | | 2250 | ^a 16 | Aging |
| 14 | 1600 | | | | |
| 15 | 1750 | | | | |
| 16 | 1830 | | | | |
| 17 | 1950 | | | | |
| 18 | 1000 | 72 | | | |
| 19 | 1100 | | | | |
| 20 | 1200 | | | | |
| 21 | 1350 | | | | |
| 22 | 1550 | | | | |
| 23 | 1600 | | | | |
| 24 | 1750 | | | | |
| 25 | 1830 | | | | |
| 26 | 1950 | | | | |
| 27 | 2250 | ^a 16 | Aging | 1200 | 72 |
| | | | | 1500 | 24 |
| 28 | No solution treatment | | Aging | 1200 | 72 |
| 29 | | | | 1500 | |
| 30 | | | | 1950 | |

^aAir-cooled.

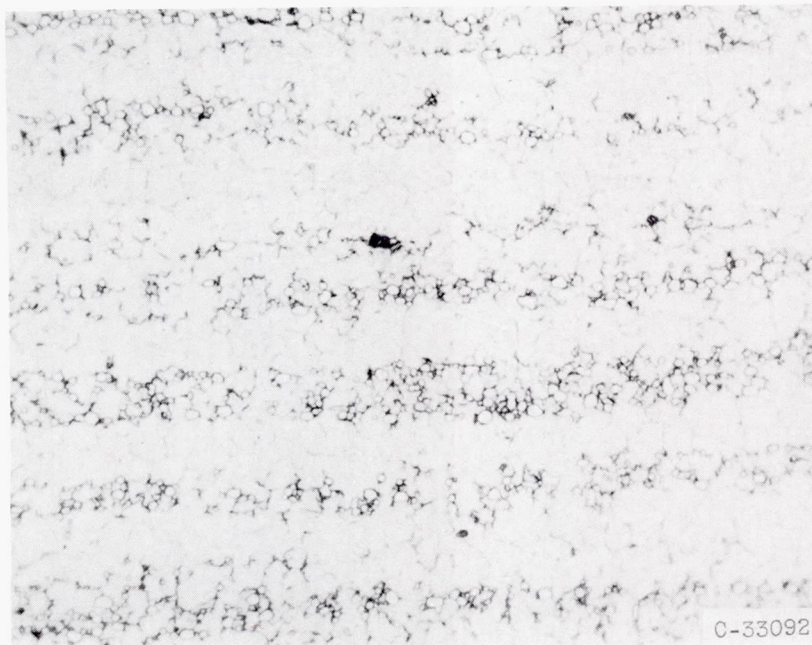
^bFurnace quenched to isothermal transformation temperature.

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2-20



Cross-sectional view



Longitudinal view (note presence of "stringers")

Figure 1. - Microstructure of wrought Stellite 21. Etchant, 5-percent aqua regia, electrolytic; X750.

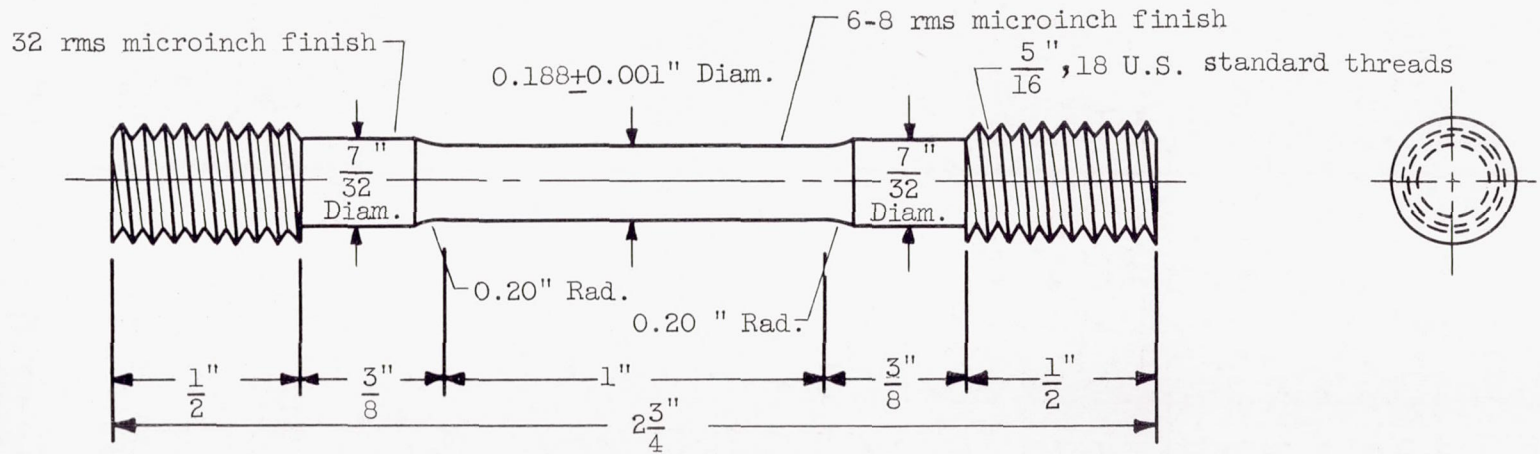
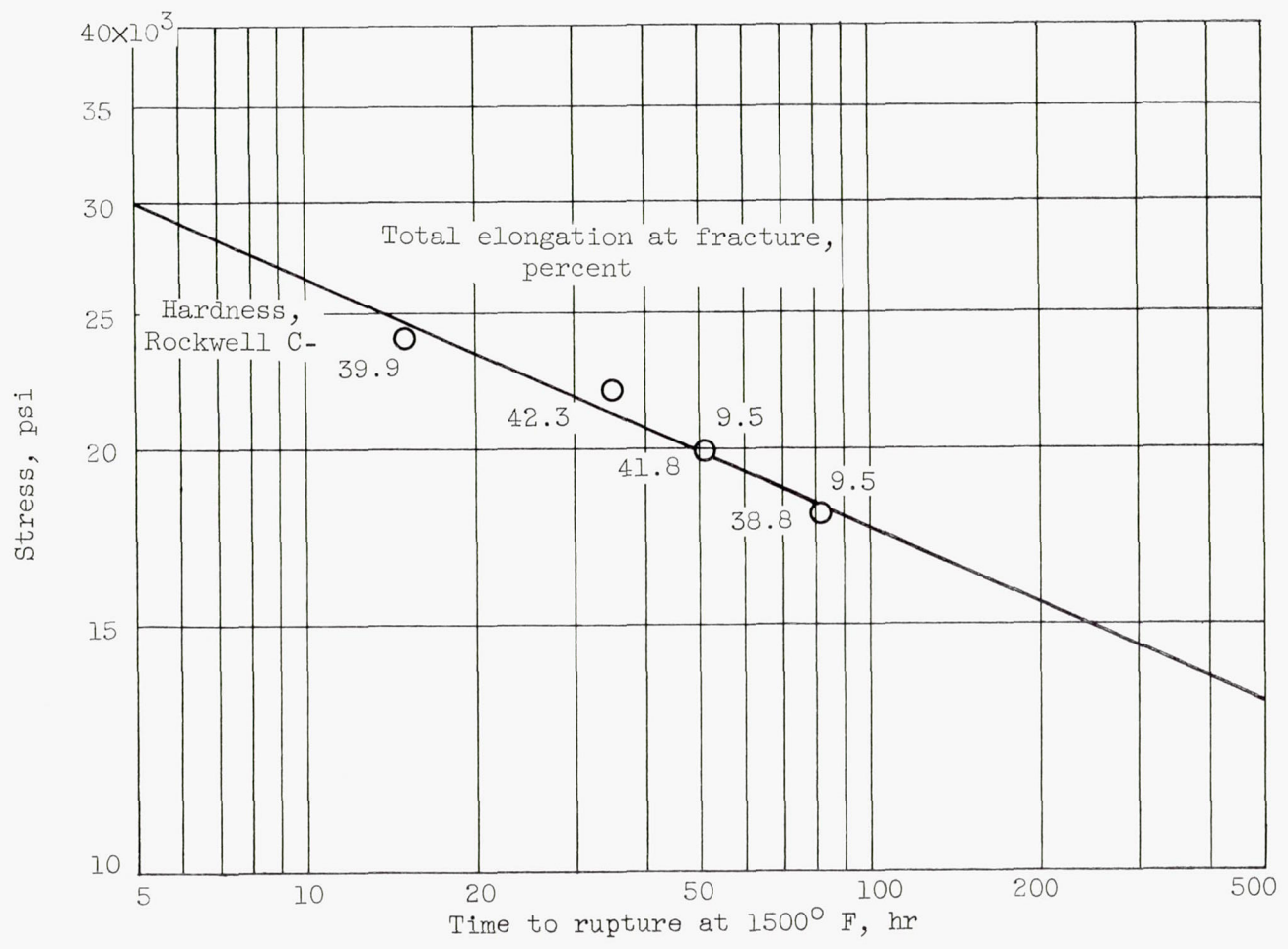
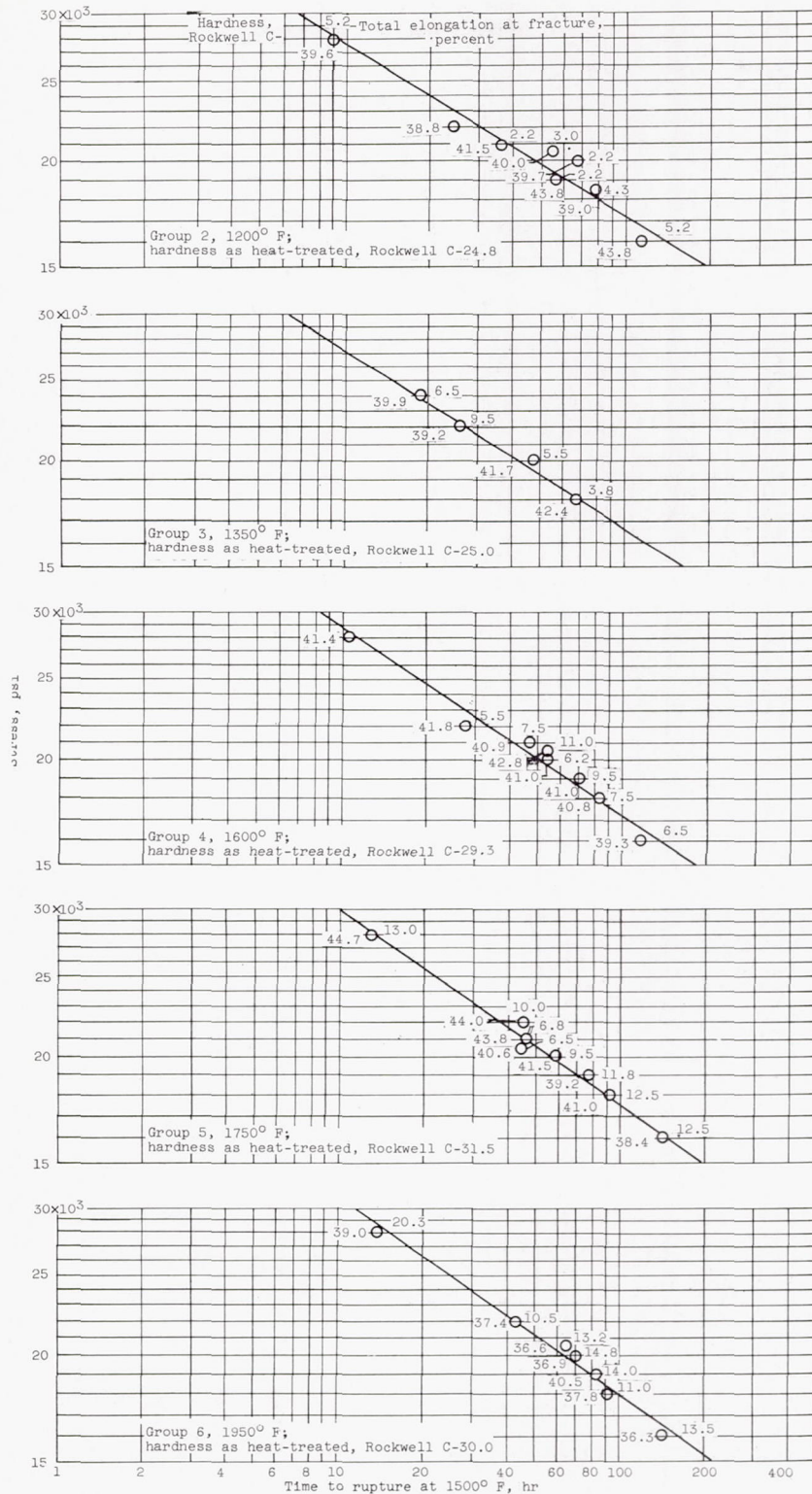


Figure 2. - Test specimen.



(a) Specimens of solution-treated wrought Stellite 21 (group 1).
Hardness as heat-treated, Rockwell C-26.1

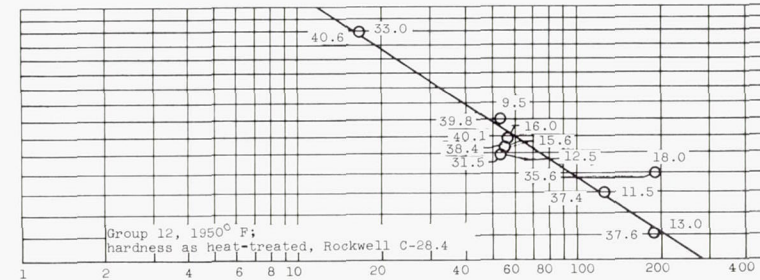
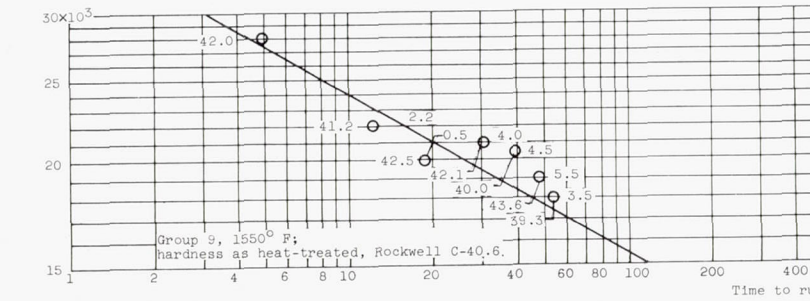
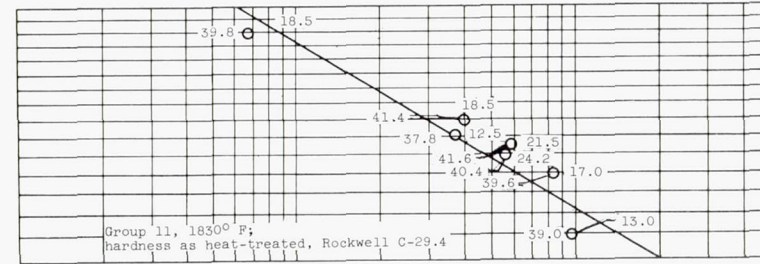
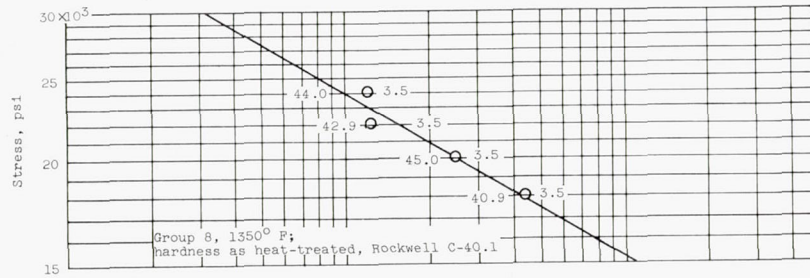
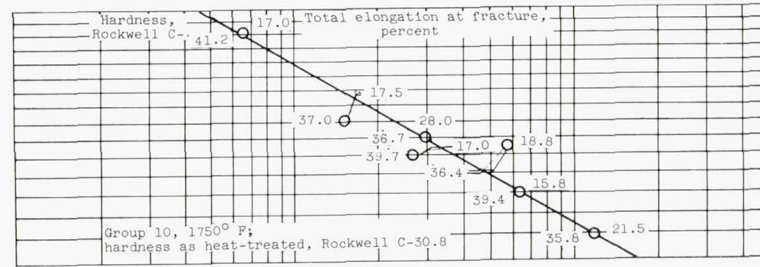
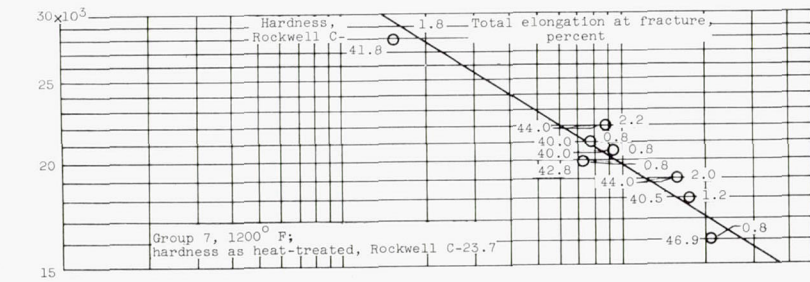
Figure 3. - Stress-rupture results.



(b) Specimens from heat-treatment groups 2 to 6 (solution-treated and isothermally transformed 2 hr at indicated temperatures).

Figure 3. - Continued. Stress-rupture results.

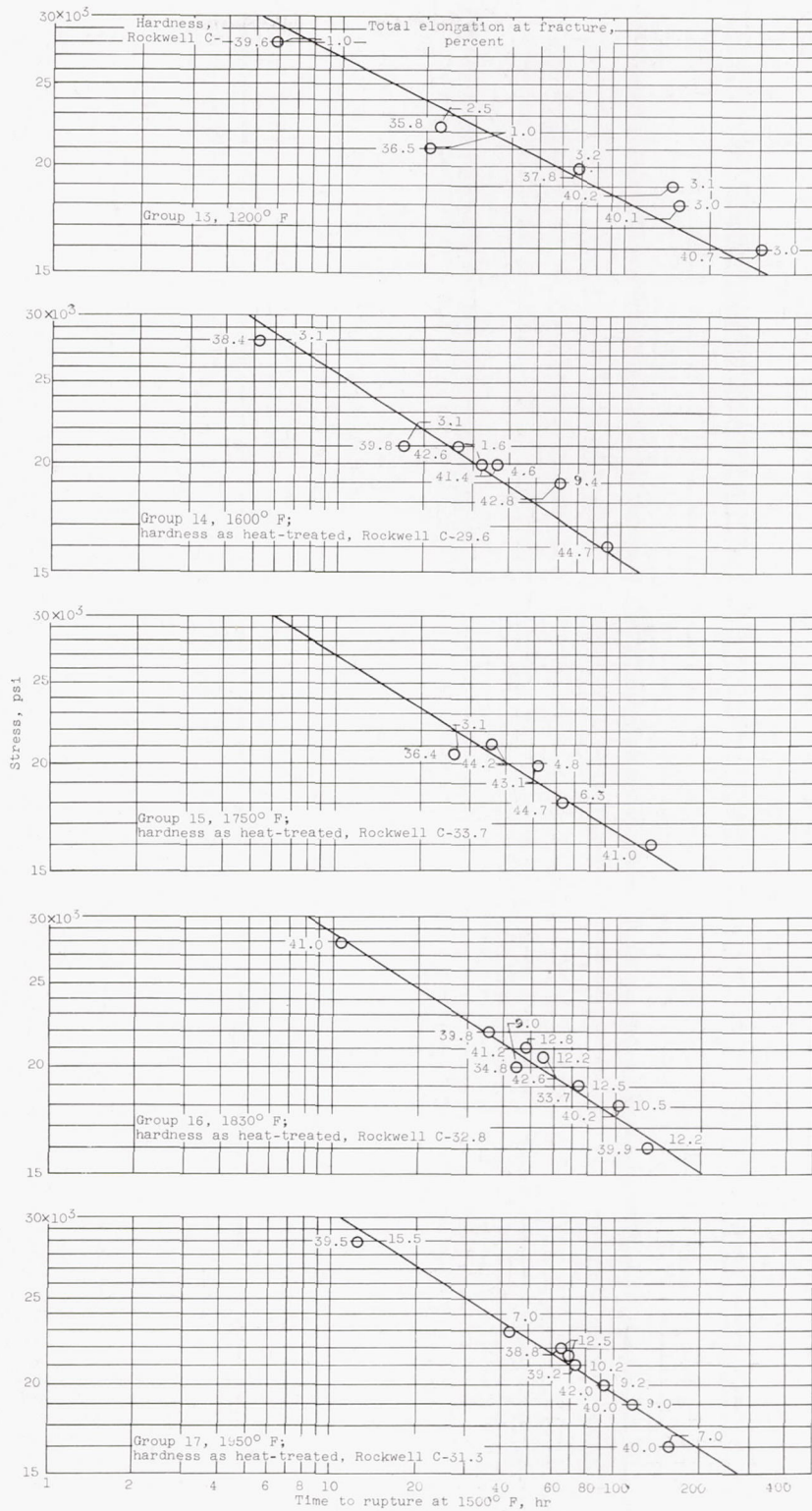
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(c) Specimens from heat-treatment groups 7 to 12 (solution-treated and isothermally transformed 72 hr at indicated temperatures).

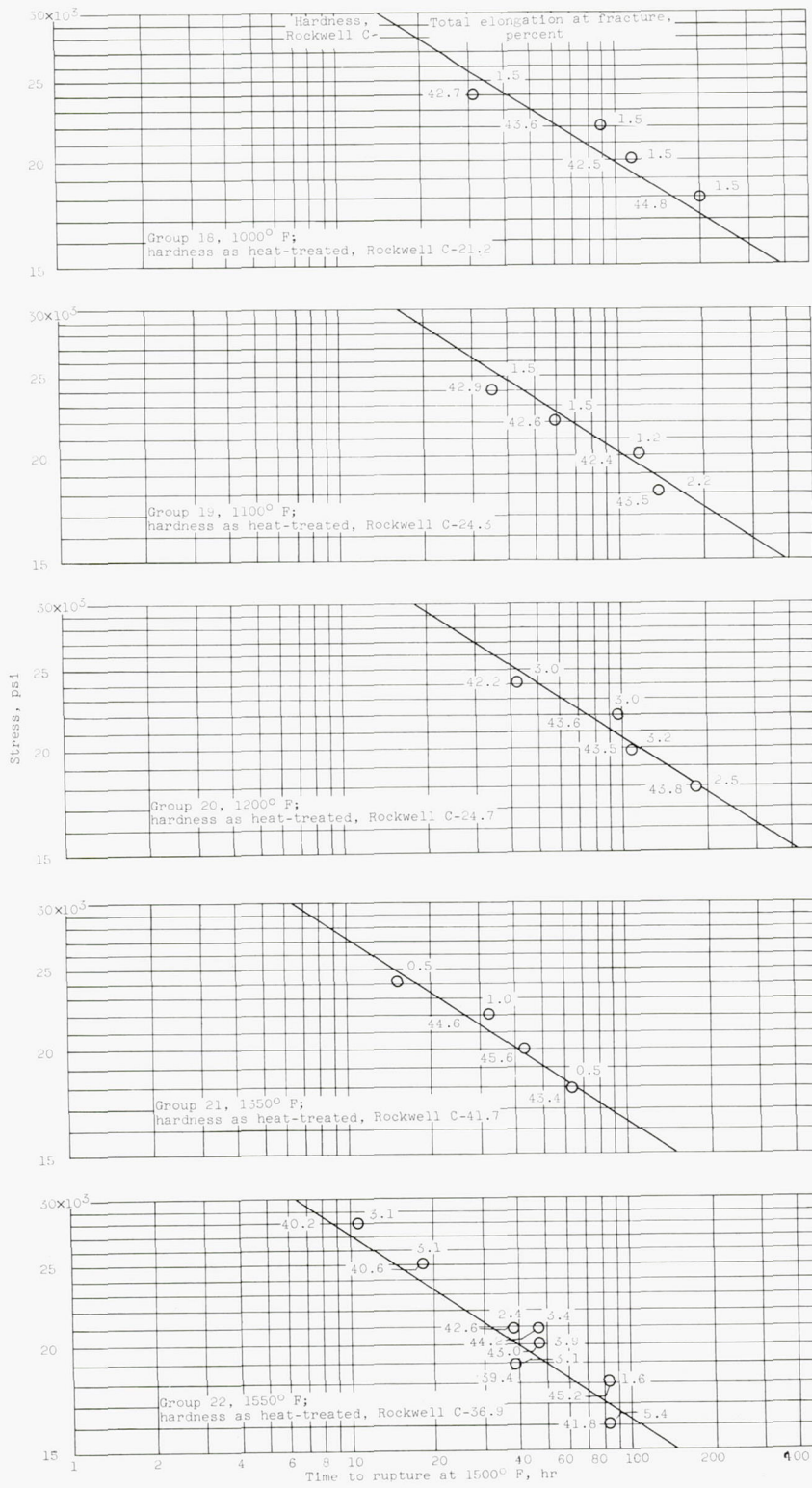
Figure 3. - Continued. Stress-rupture results.

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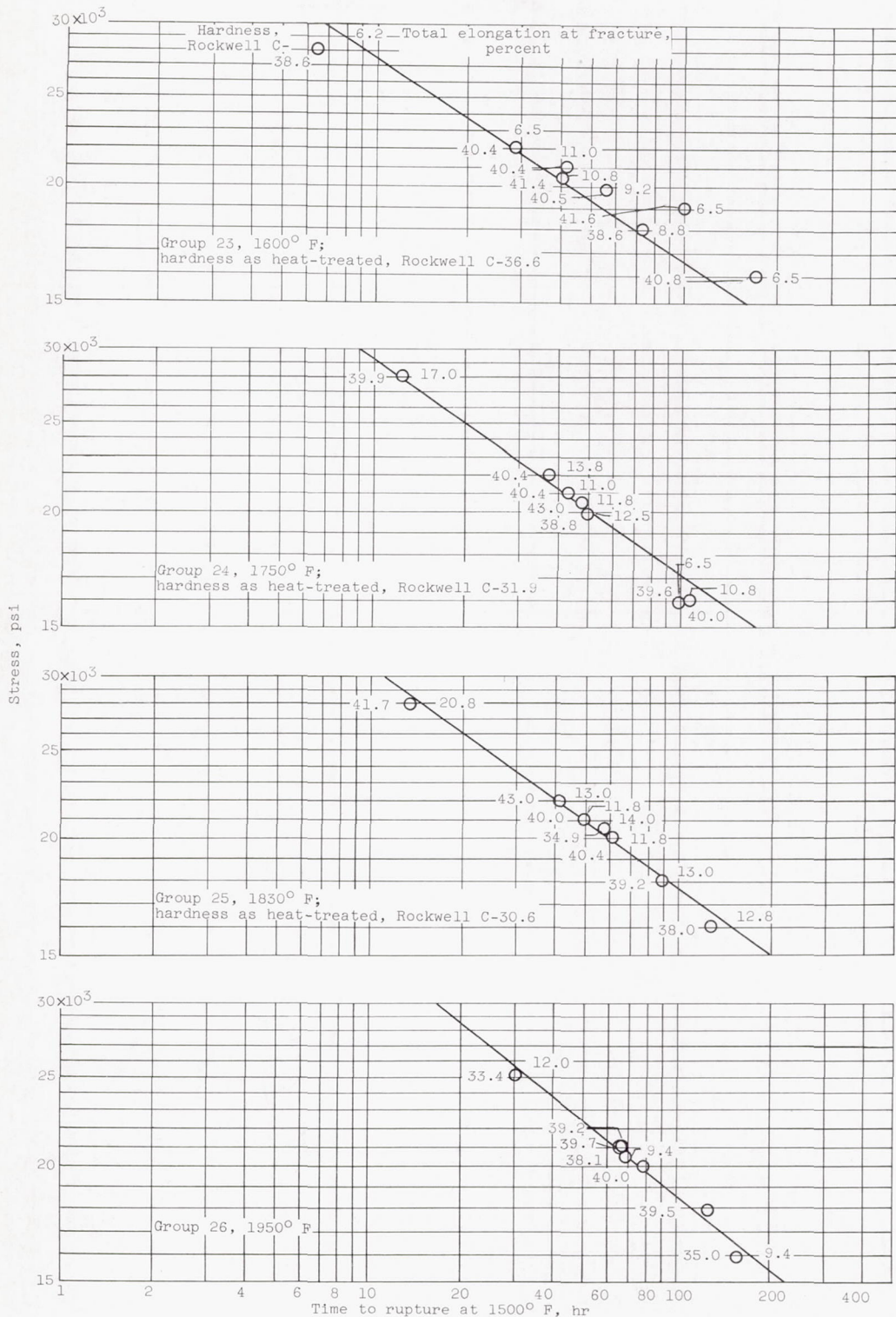
(d) Specimens from heat-treatment groups 13 to 17 (solution-treated and aged 2 hr at indicated temperatures).

Figure 3. - Continued. Stress-rupture results.



(e) Specimens from heat-treatment groups 18 to 26 (solution-treated and aged 72 hr at indicated temperatures).

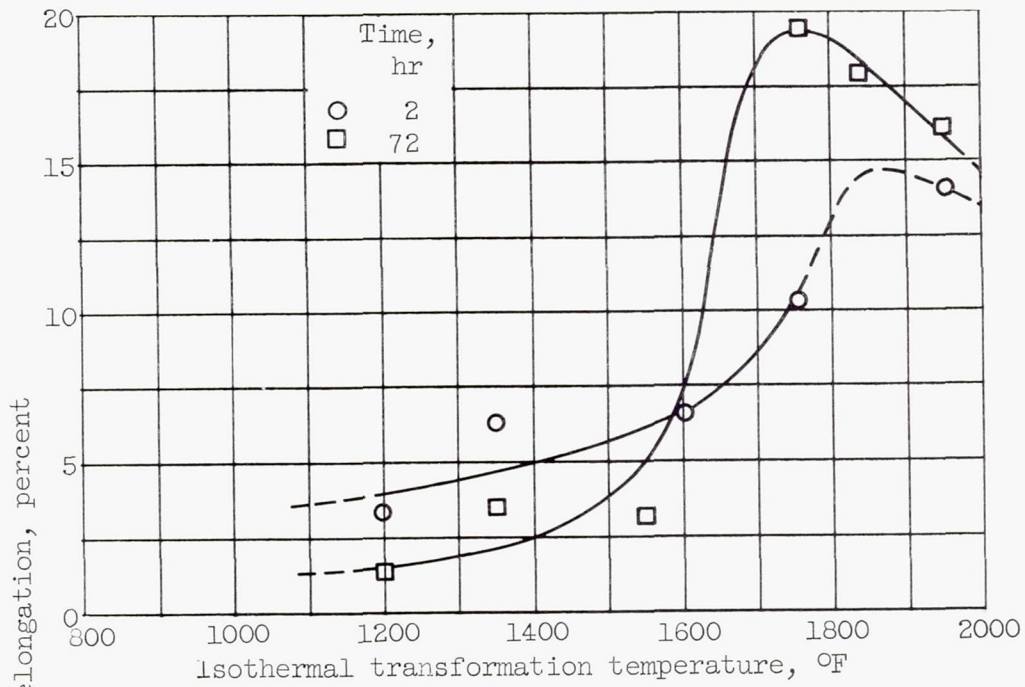
Figure 3. - Continued. Stress-rupture results.



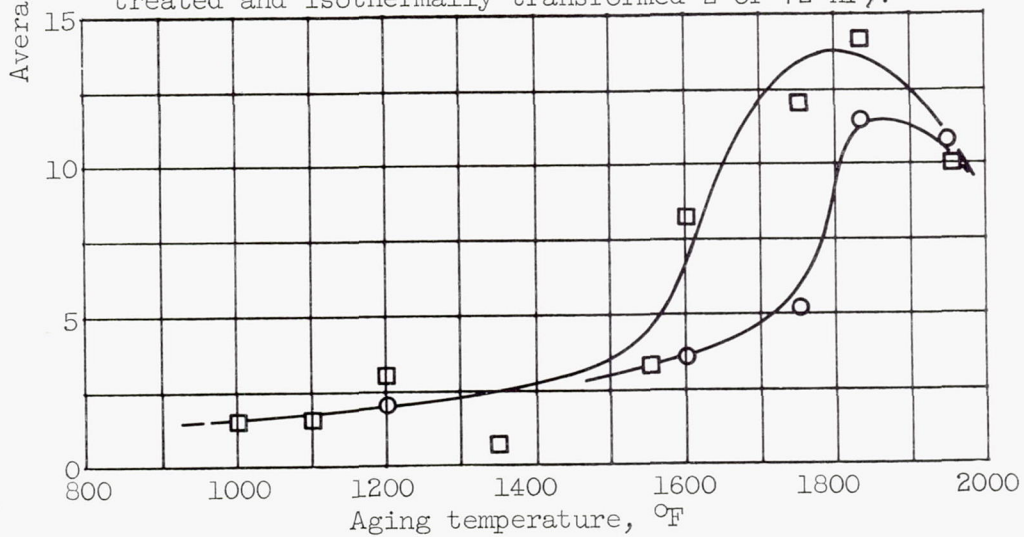
(e) Concluded. Specimens from heat-treatment groups 18 to 26 (solution-treated and aged 72 hr at indicated temperatures).

Figure 3. - Concluded. Stress-rupture results.

CJ-4 2810

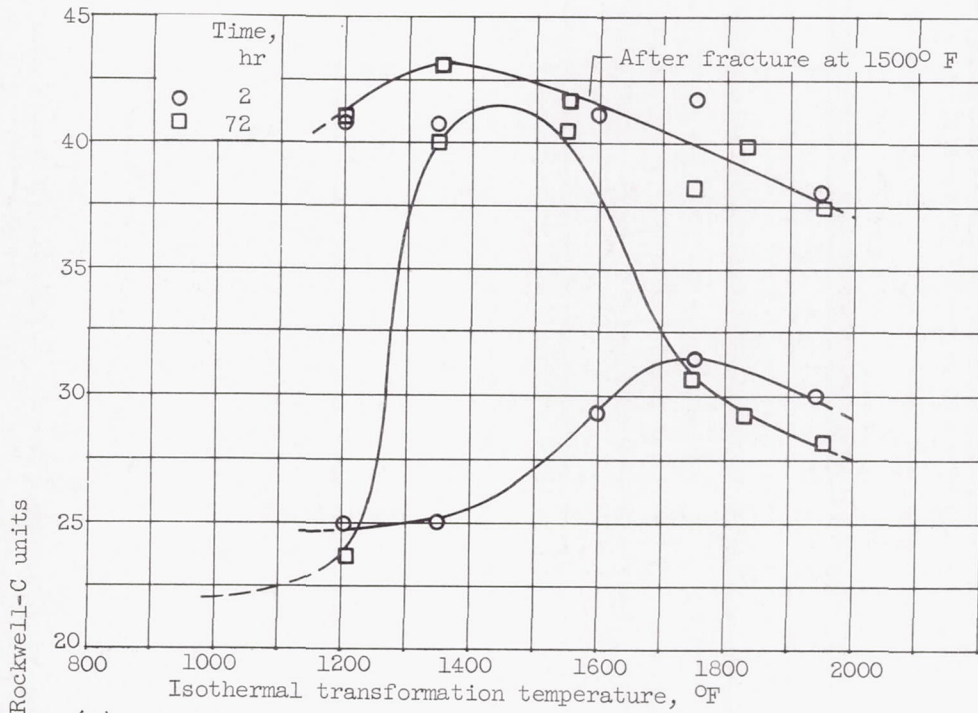


(a) Specimens from heat-treatment groups 2 to 12 (solution-treated and isothermally transformed 2 or 72 hr).

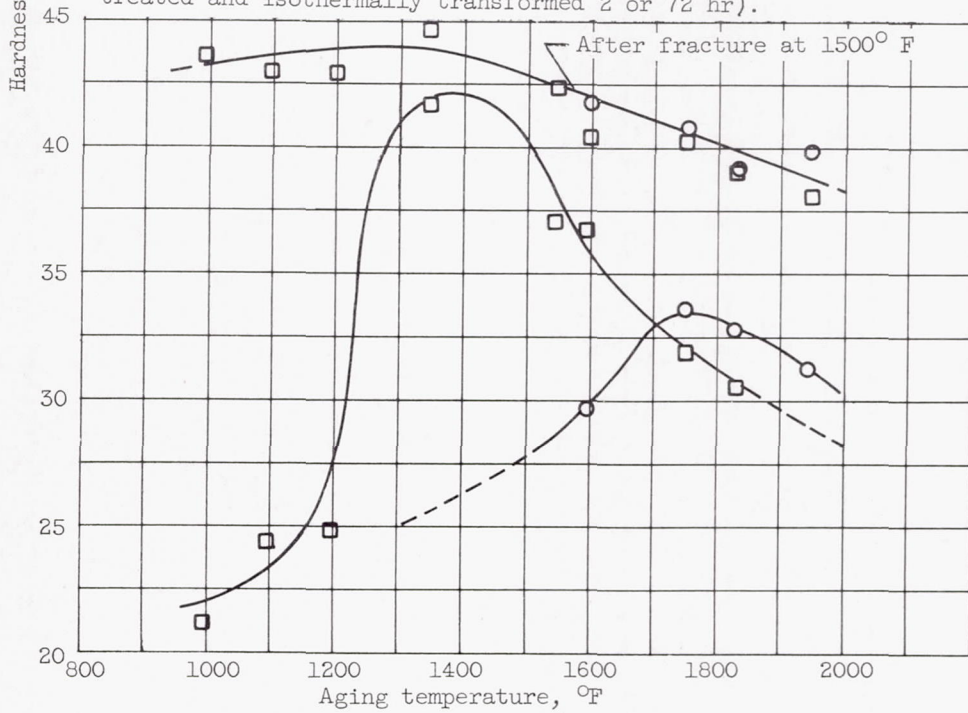


(b) Specimens from heat-treatment groups 13 to 26 (solution-treated and aged 2 or 72 hr).

Figure 4. - Elongation results.



(a) Specimens from heat-treatment groups 2 to 12 (solution-treated and isothermally transformed 2 or 72 hr).



(b) Specimens from heat-treatment groups 13 to 26 (solution-treated and aged 2 or 72 hr).

Figure 5. - Hardness results.

2810 CJ-4 back

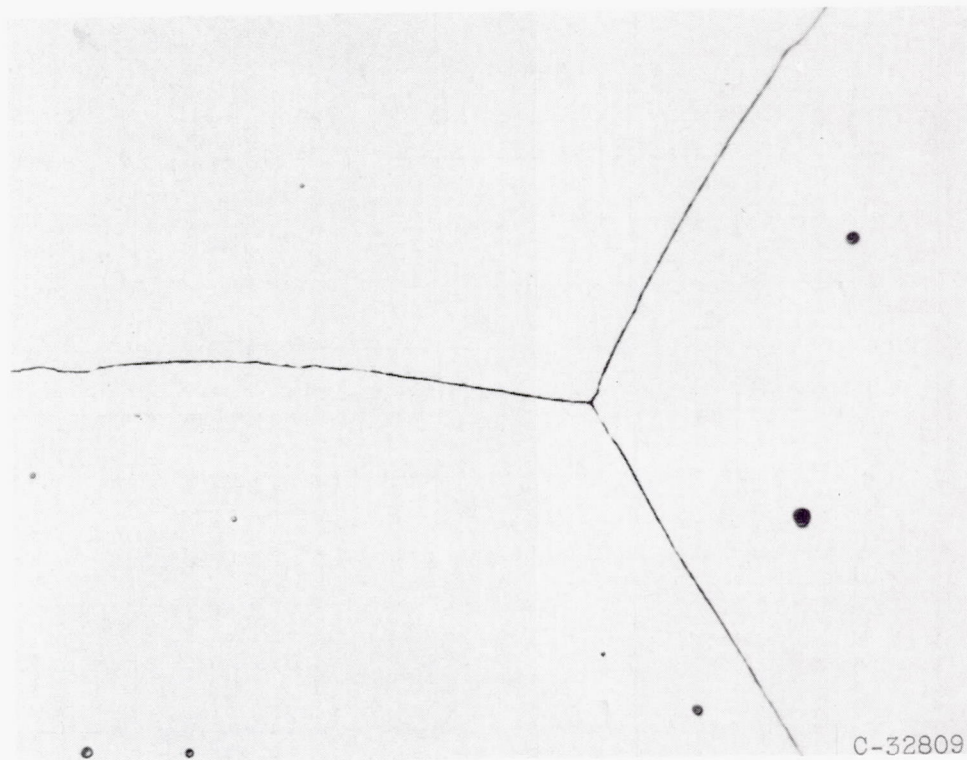
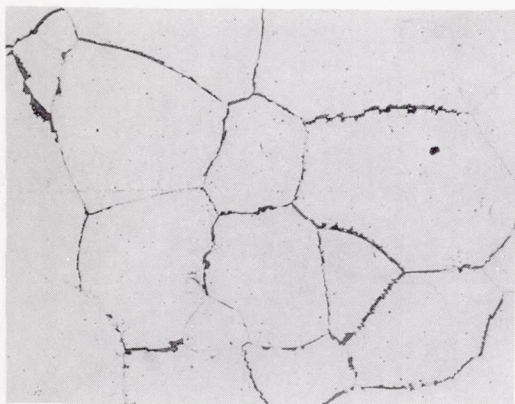


Figure 6. - Solution-treated structure. X750.

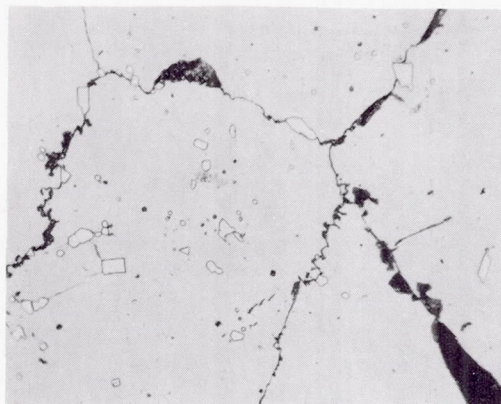
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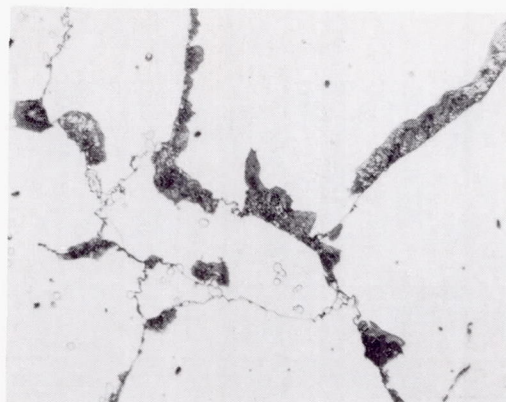
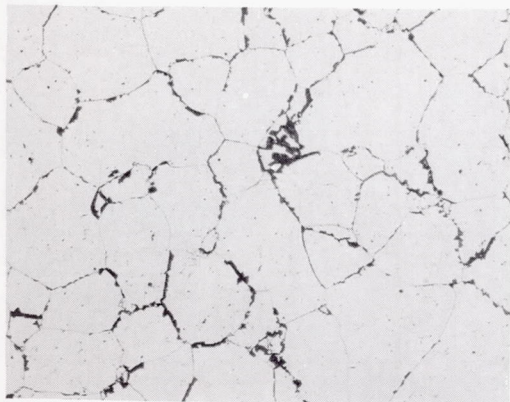
X100



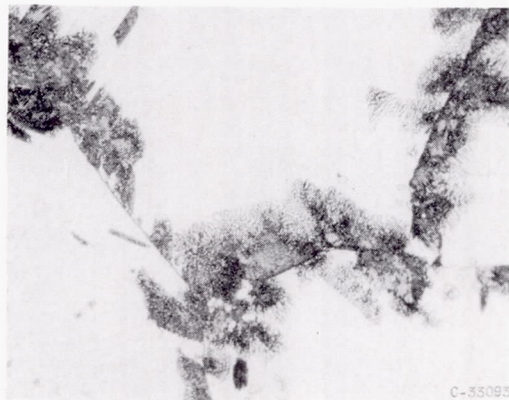
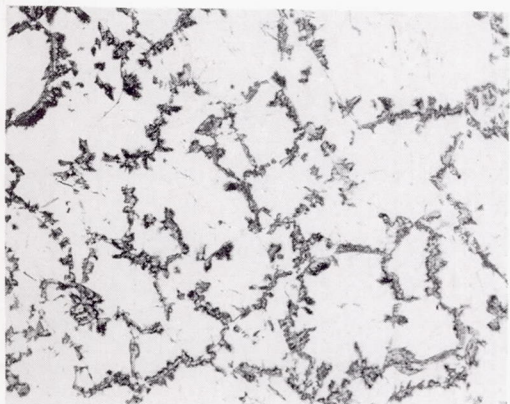
X750



1200° F



1350° F



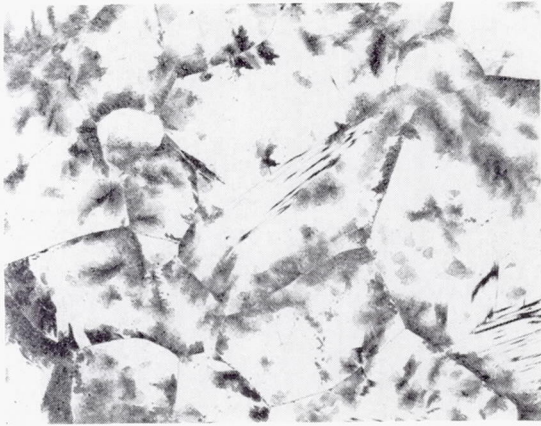
C-35093

1550° F

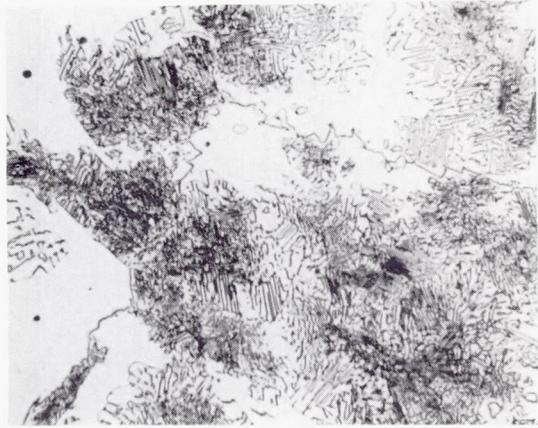
(a) Groups 2 to 6 (solution-treated and isothermally transformed 2 hr at indicated temperatures).

Figure 7. - Microstructure of specimens after heat treatment.

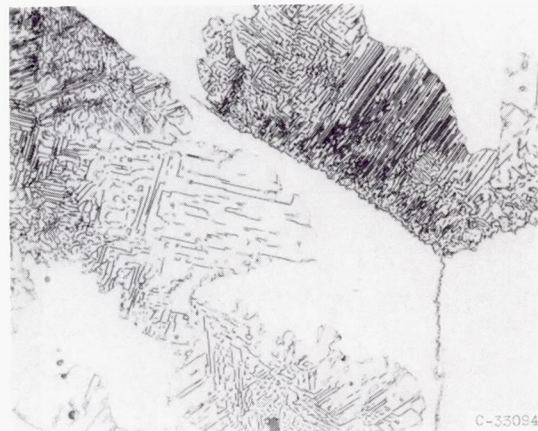
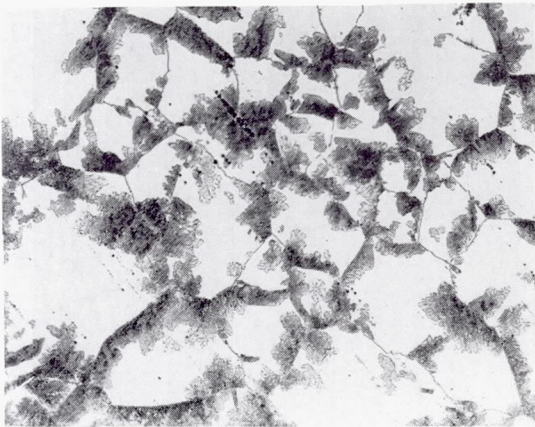
X100



X750



1750° F



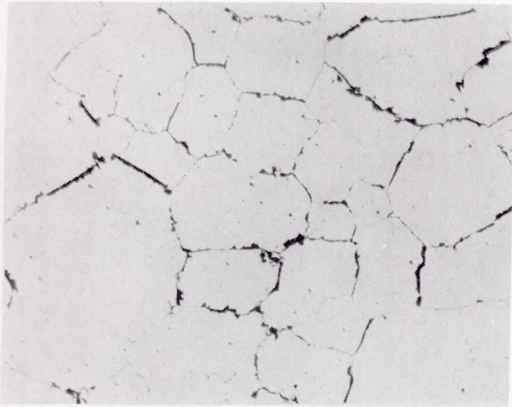
1950° F

(a) Concluded. Groups 2 to 6 (solution-treated and isothermally transformed 2 hr at indicated temperatures).

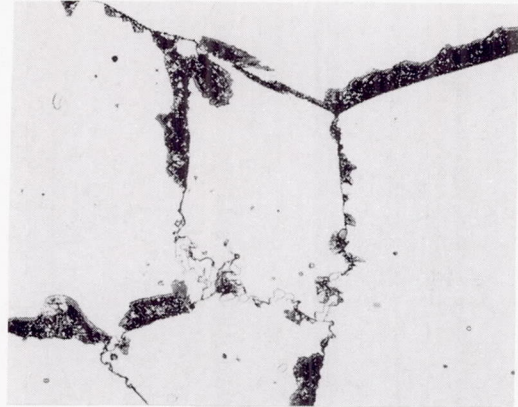
Figure 7. - Continued. Microstructure of specimens after heat treatment.

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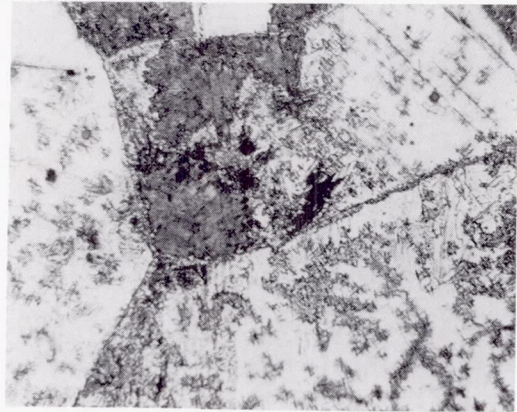
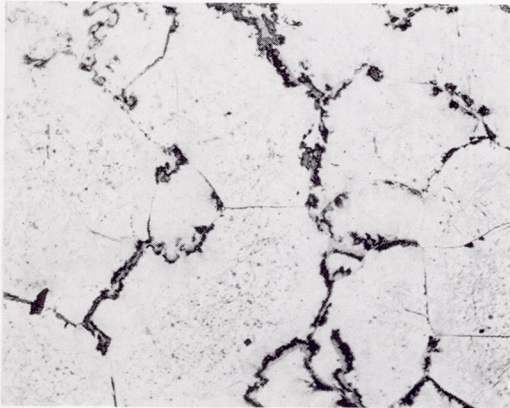
X100



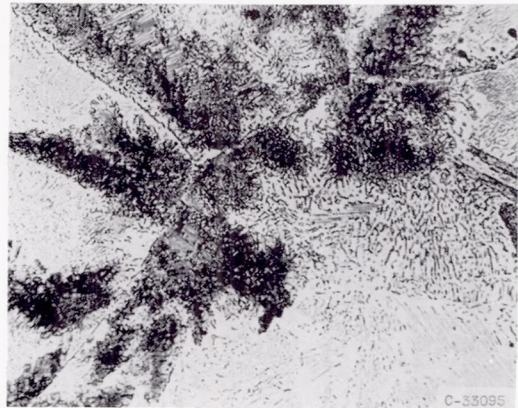
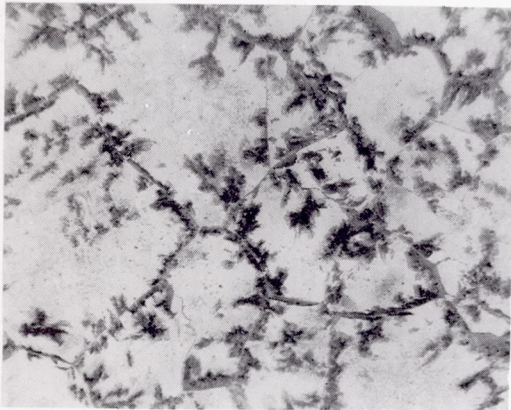
X750



1200° F



1350° F



1550° F

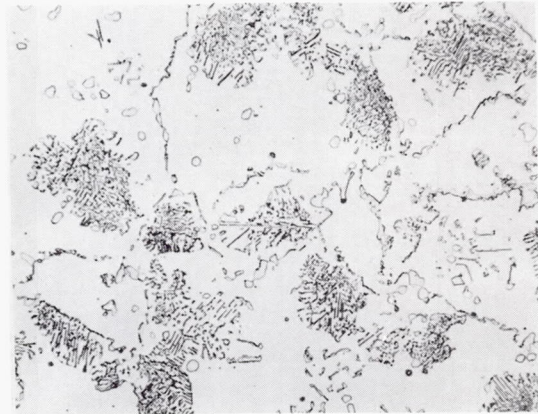
(b) Groups 7 to 12 (solution-treated and isothermally transformed 72 hr at indicated temperatures).

Figure 7. - Continued. Microstructure of specimens after heat treatment.

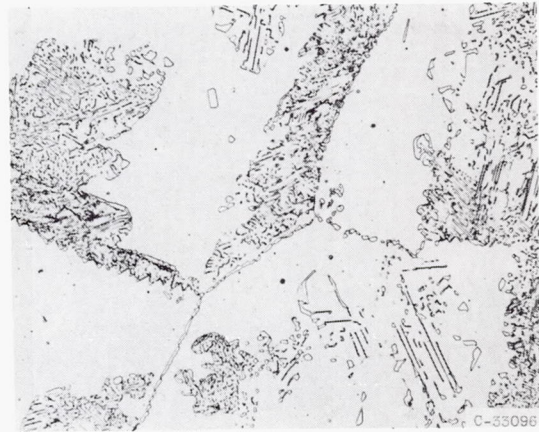
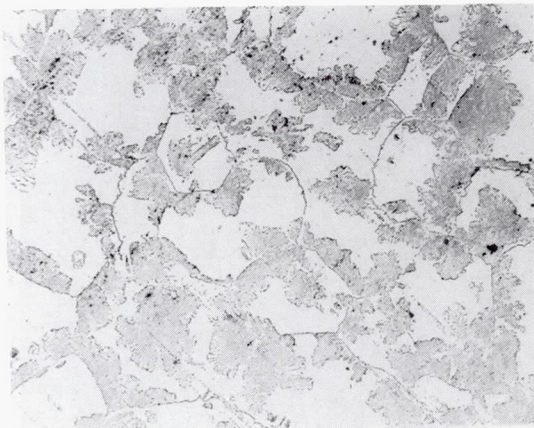
X100



X750



1750° F

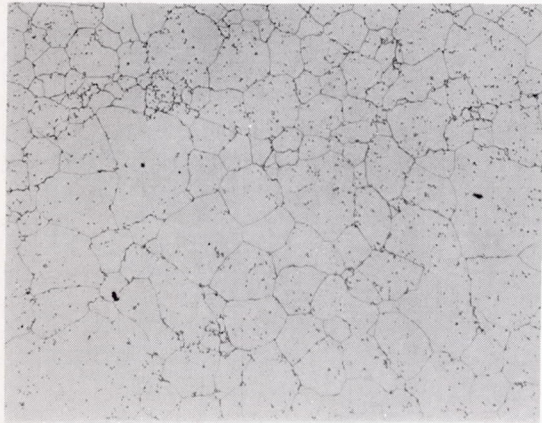


1950° F

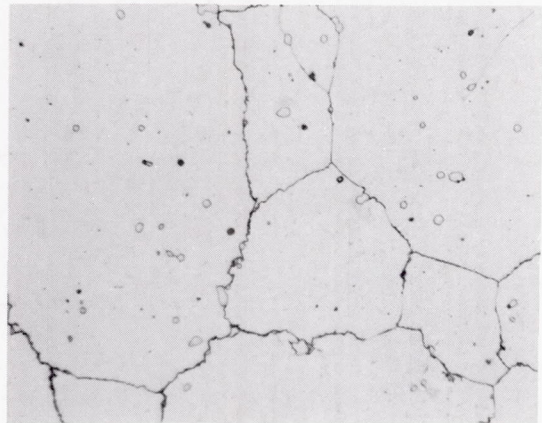
(b) Concluded. Groups 7 to 12 (solution-treated and isothermally transformed 72 hr at indicated temperatures).

Figure 7. - Continued. Microstructure of specimens after heat treatment.

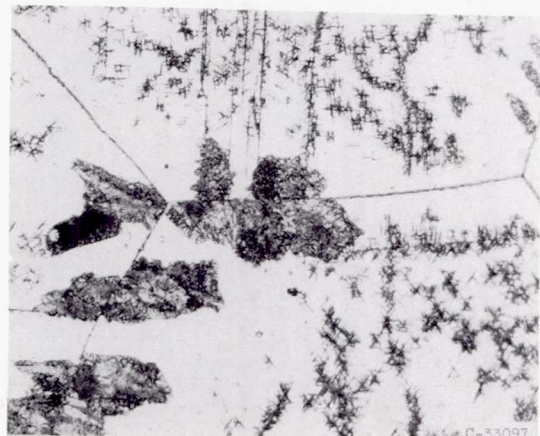
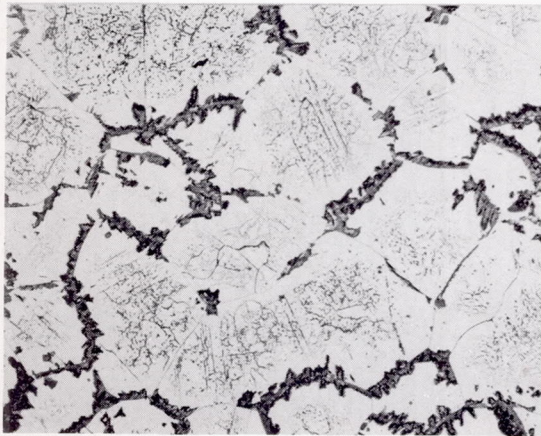
X100



X750



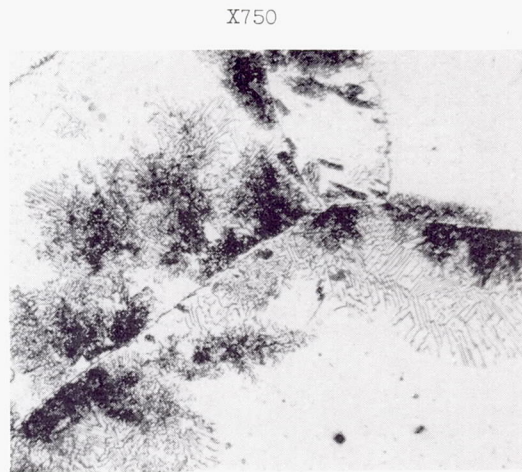
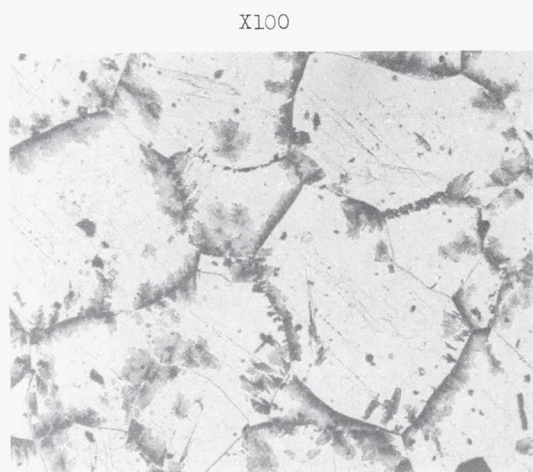
1200° F



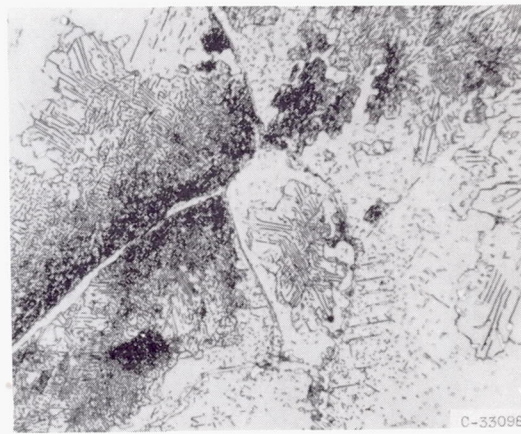
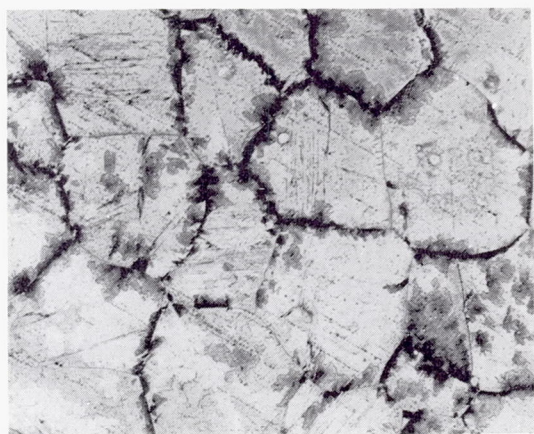
1550° F

(c) Groups 13 to 17 (solution-treated and aged 2 hr at indicated temperatures).

Figure 7. - Continued. Microstructure of specimens after heat treatment.



1750° F



1950° F

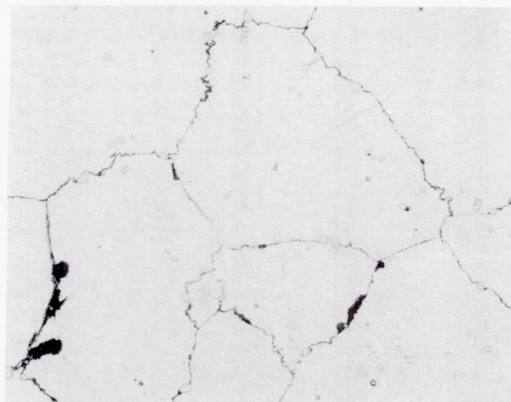
(c) Concluded. Groups 13 to 17 (solution-treated and aged 2 hr at indicated temperatures)

Figure 7. - Continued. Microstructure of specimens after heat treatment.

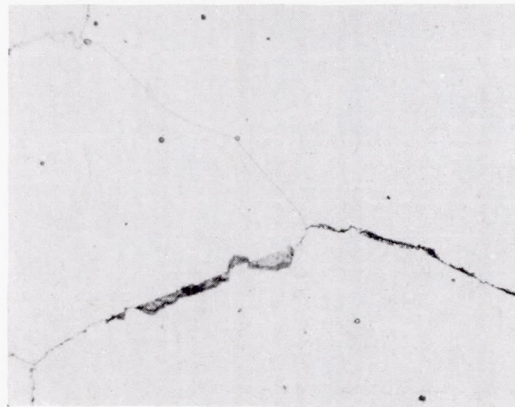
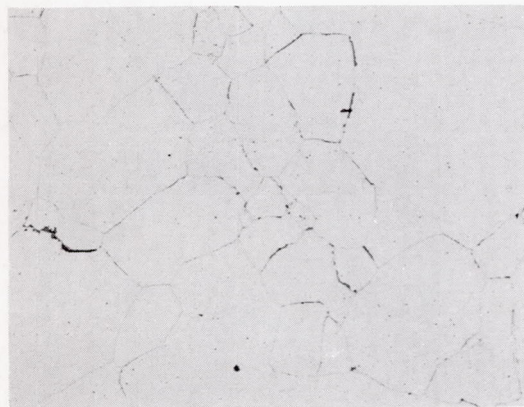
X100



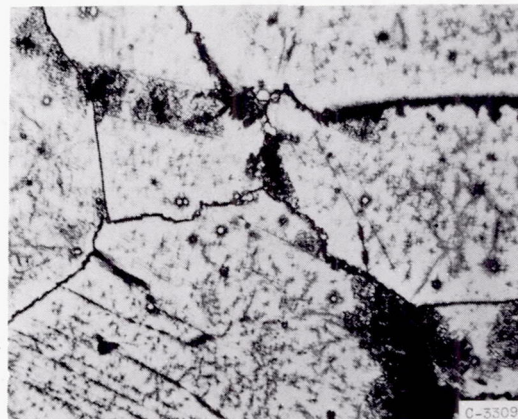
X750



1000° F



1200° F



1350° F

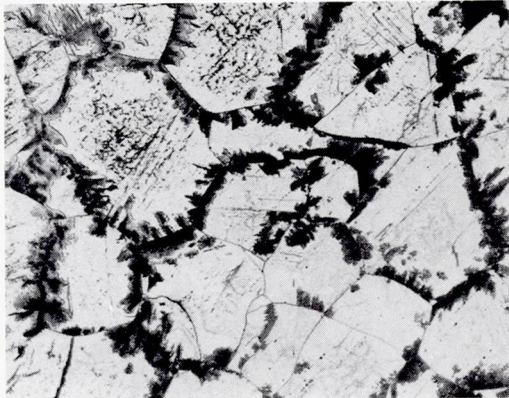
(d) Groups 18 to 26 (solution-treated and aged 72 hr at indicated temperatures).

Figure 7. - Continued. Microstructure of specimens after heat treatment.

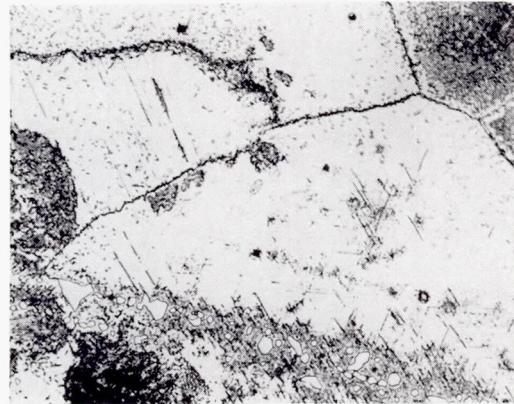
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2810

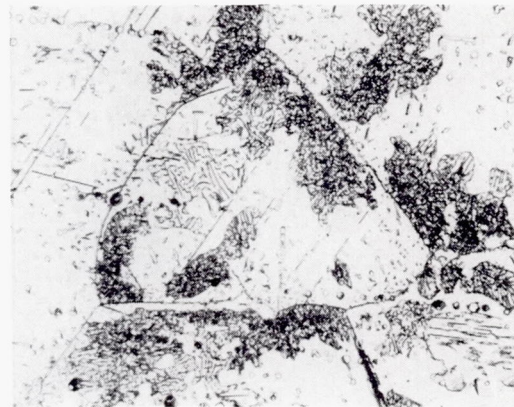
X100



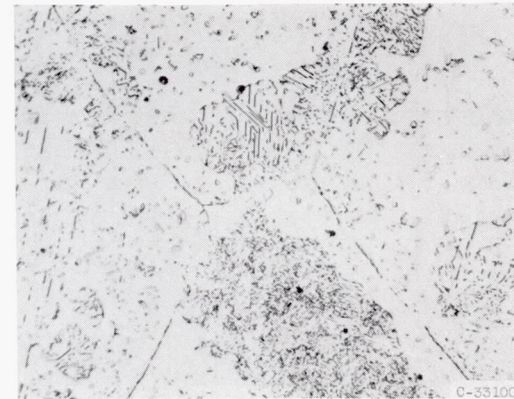
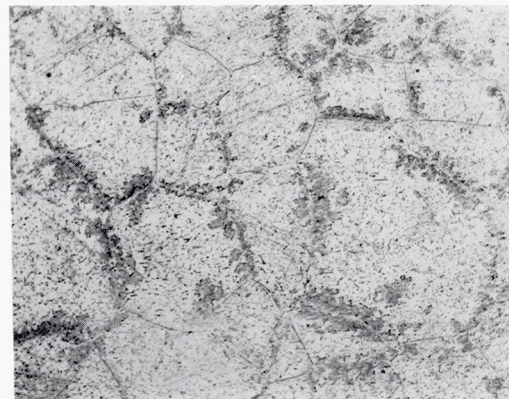
X750



1550° F



1750° F



1950° F

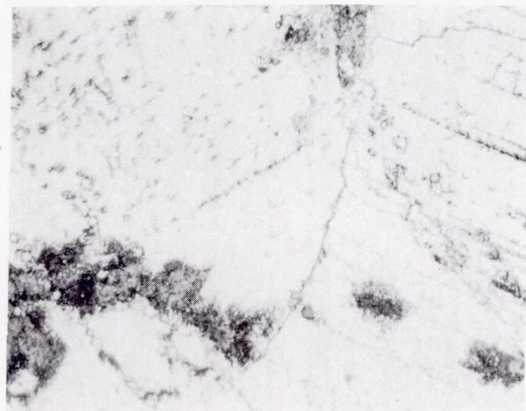
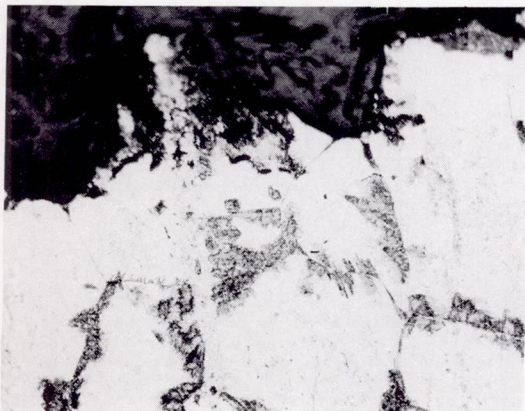
(d) Concluded. Groups 18 to 26 (solution-treated and aged 72 hr at indicated temperatures).

Figure 7. - Concluded. Microstructure of specimens after heat treatment.

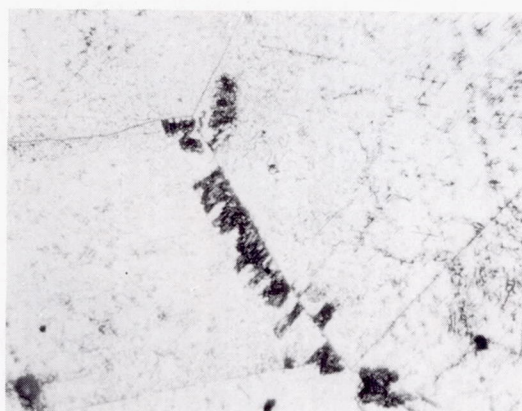
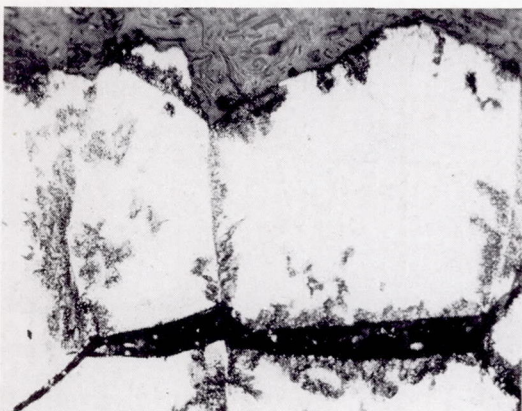
2810
0782

X250

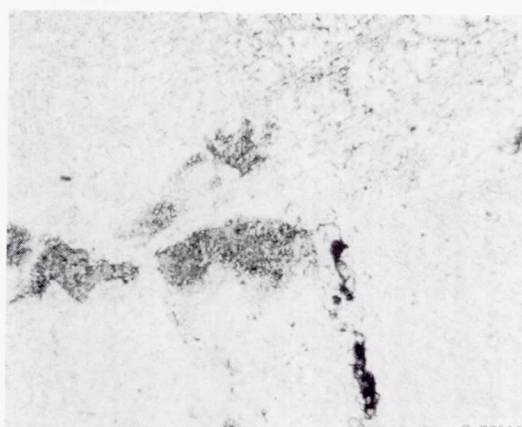
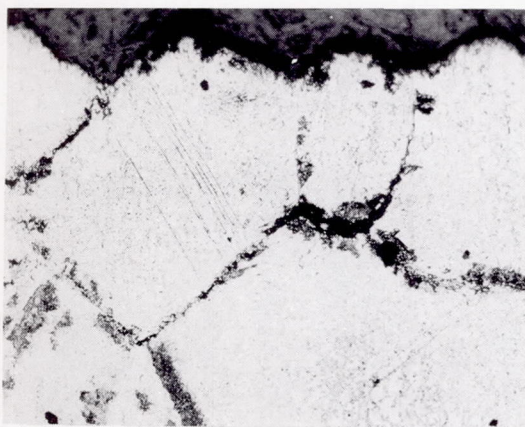
X750



Fractured after 9.1 hr at 28,000 psi



Fractured after 36.2 hr at 21,000 psi



C-33110

Fractured after 114.6 hr at 16,000 psi

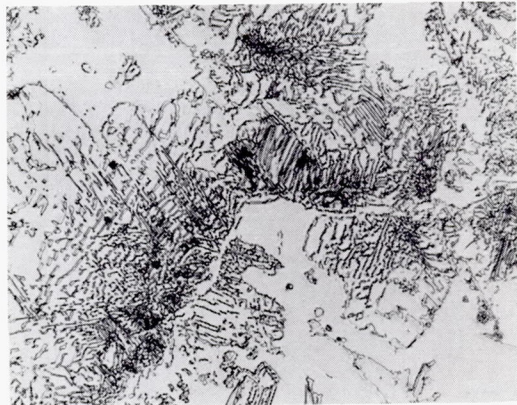
(a) Isothermally transformed 2 hours at 1200° F.

Figure 8. - Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

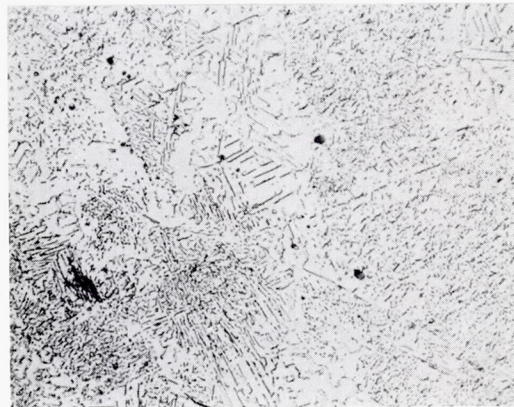
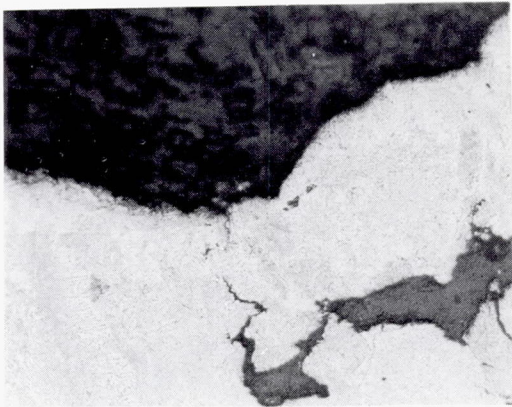
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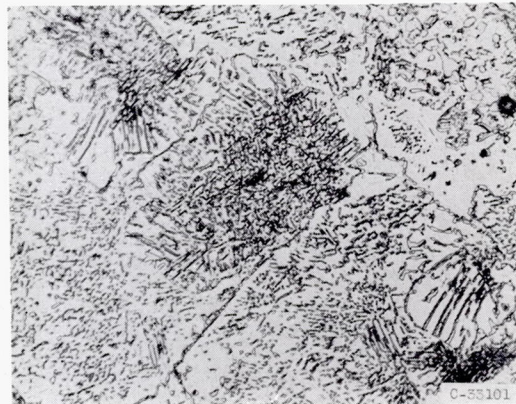
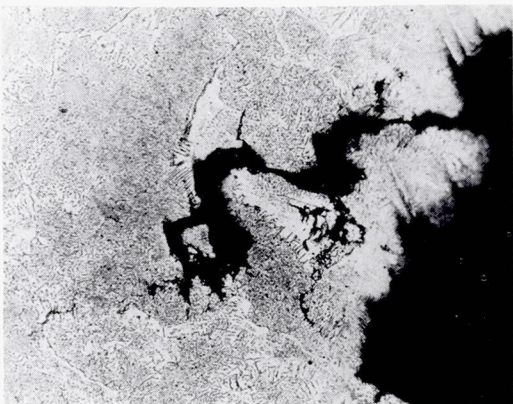
X750



Fractured after 13.8 hr at 28,000 psi



Fractured after 65.5 hr at 20,500 psi



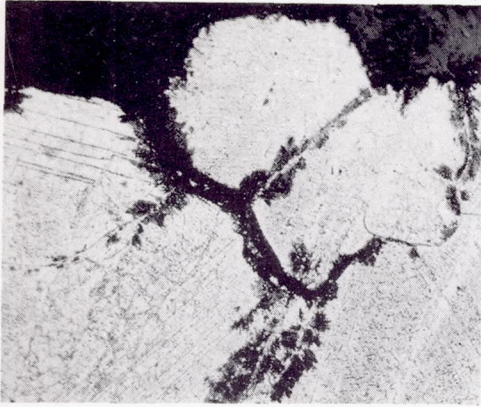
Fractured after 144.1 hr at 16,000 psi

(b) Isothermally transformed 2 hours at 1950° F.

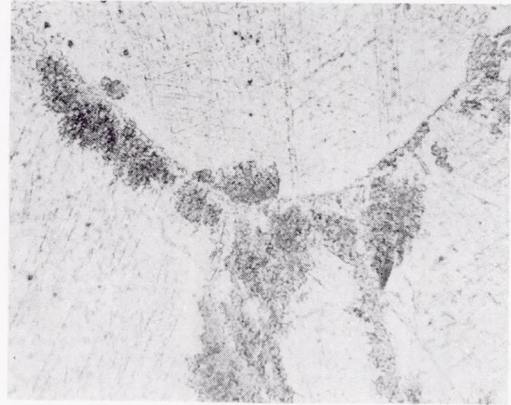
Figure 8. - Continued. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

2810

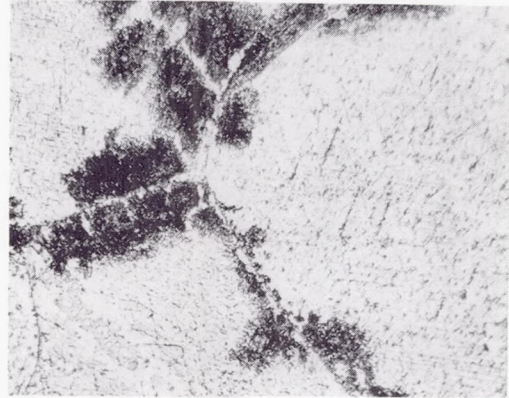
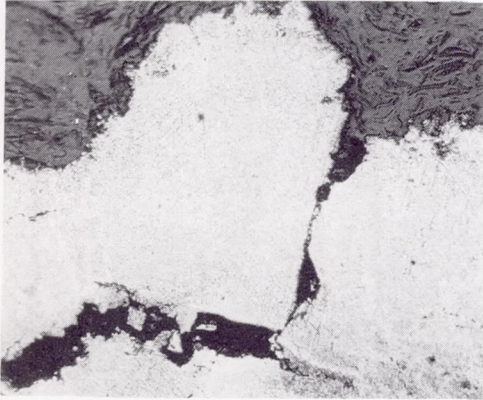
X250



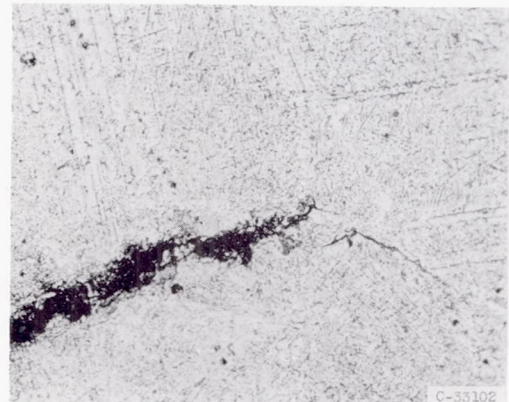
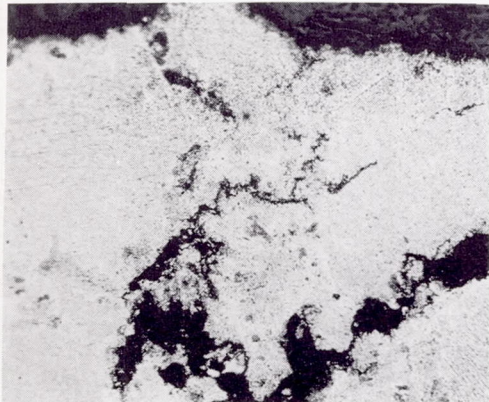
X750



Fractured after 15.2 hr at 28,000 psi



Fractured after 76.8 hr at 21,000 psi



Fractured after 207.9 hr at 16,000 psi

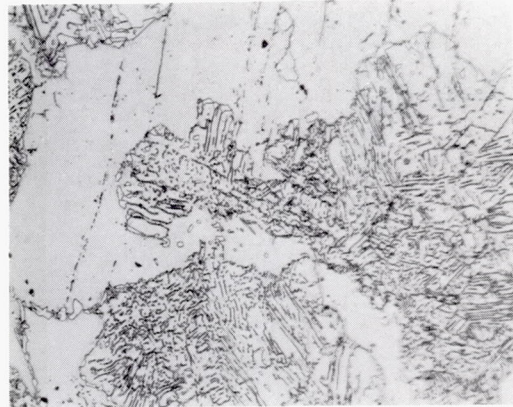
(c) Isothermally transformed 72 hours at 1200° F.

Figure 8. - Continued. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

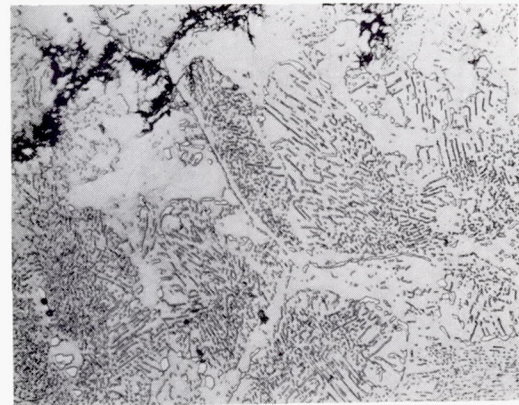
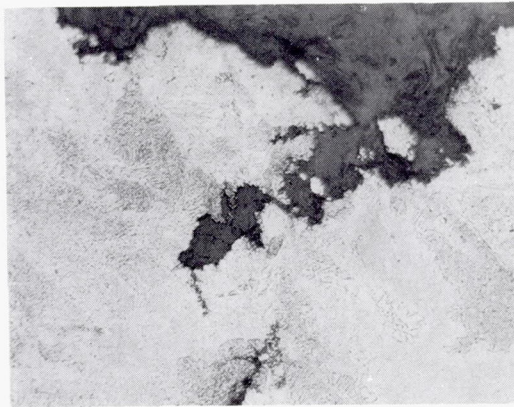
X250



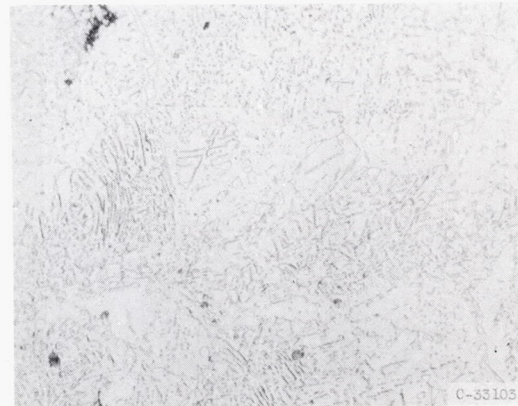
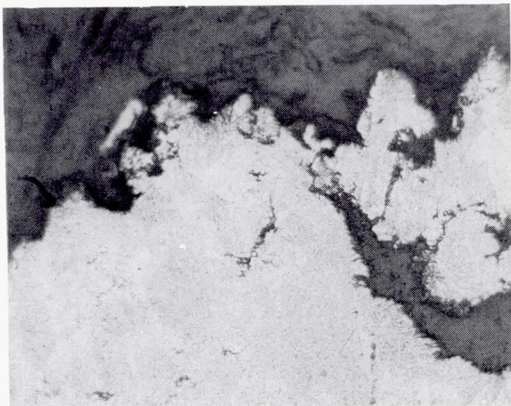
X750



Fractured after 16.6 hr at 28,000 psi



Fractured after 56.5 hr at 21,000 psi

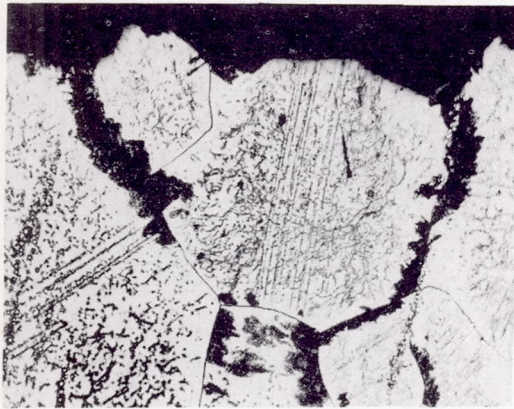


Fractured after 187.7 hr at 16,000 psi

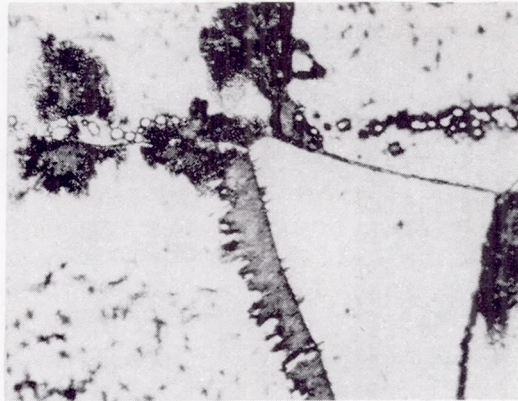
(d) Isothermally transformed 72 hours at 1950° F.

Figure 8. - Continued. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

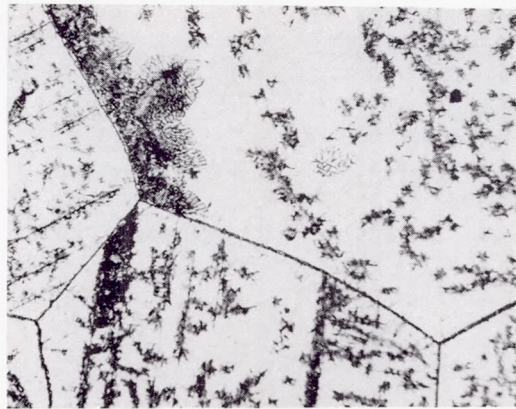
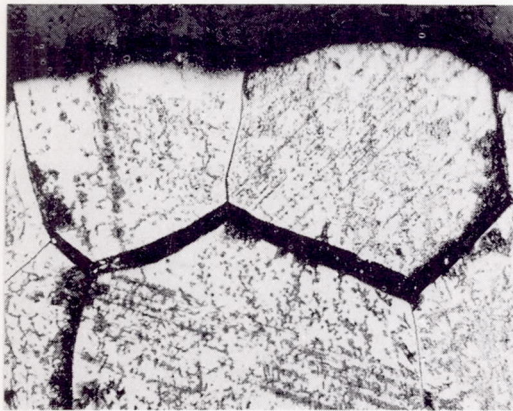
X250



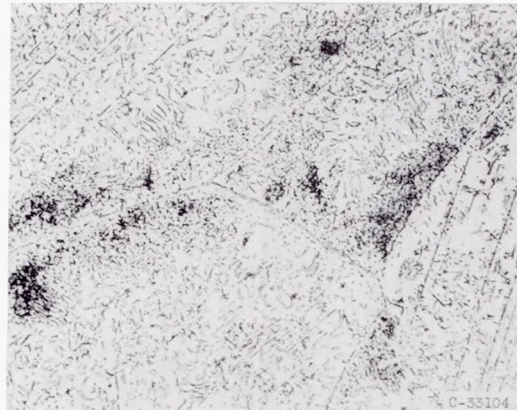
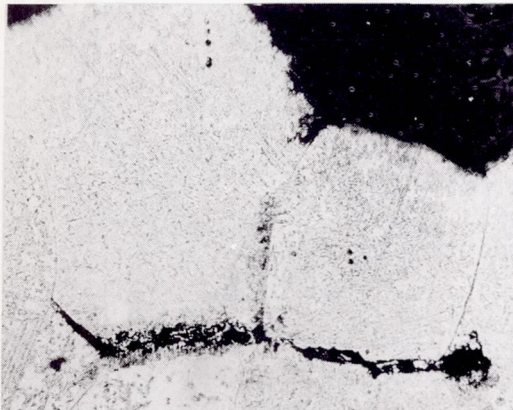
X750



Fractured after 5.9 hr at 28,000 psi



Fractured after 20.8 hr at 21,000 psi

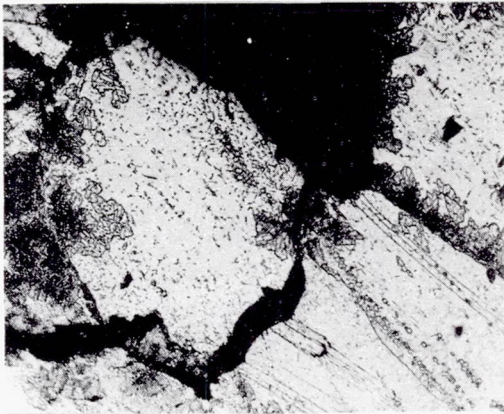


Fractured after 308.2 hr at 16,000 psi

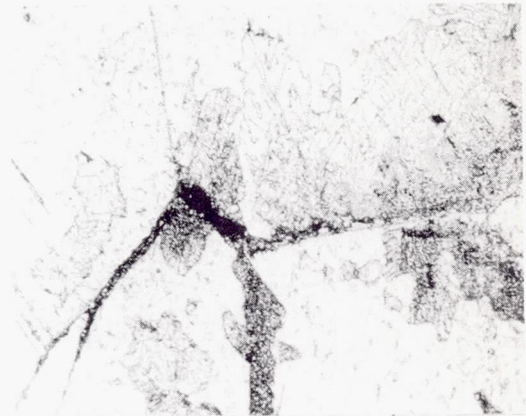
(e) Aged 2 hours at 1200° F.

Figure 8. - Continued. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

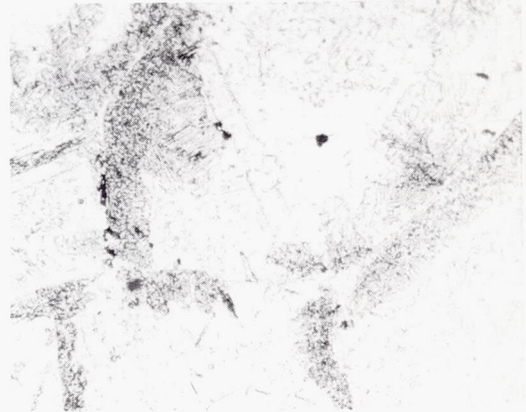
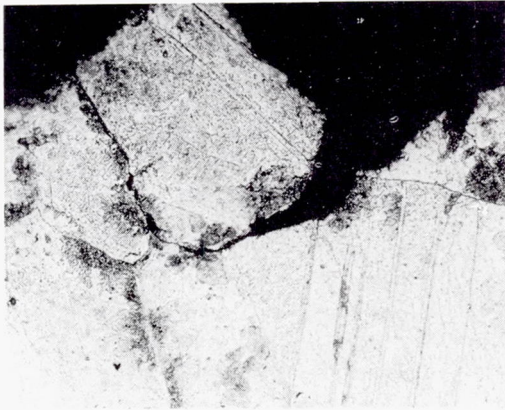
X250



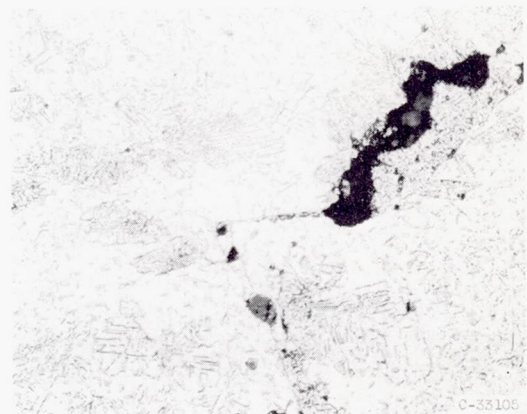
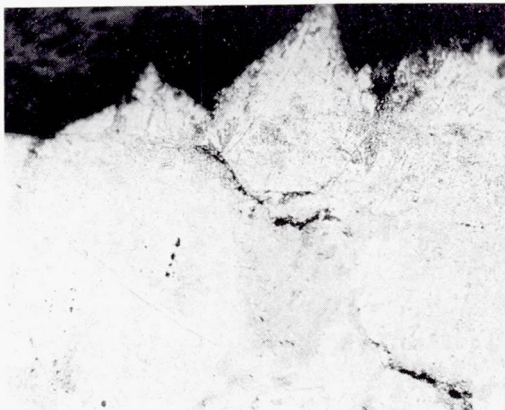
X750



Fractured after 12.5 hr at 28,000 psi



Fractured after 65.2 hr at 21,000 psi

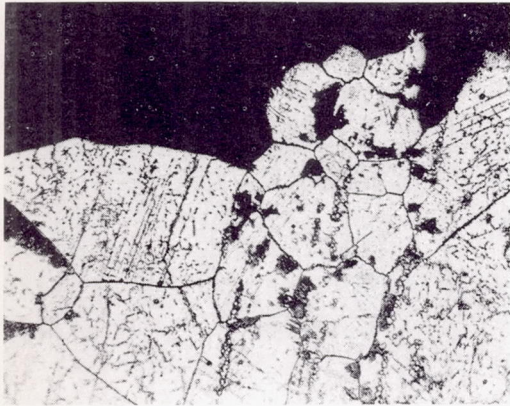


Fractured after 157.4 hr at 16,000 psi

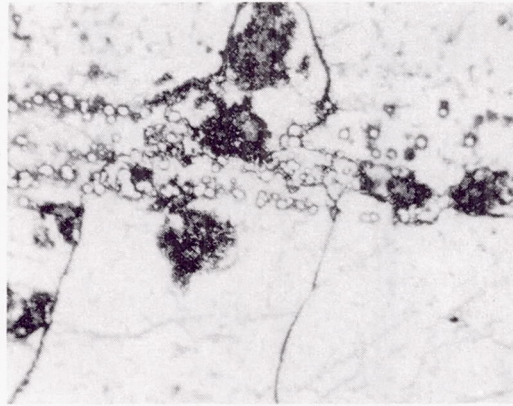
(f) Aged 2 hours at 1950° F.

Figure 8. - Continued. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

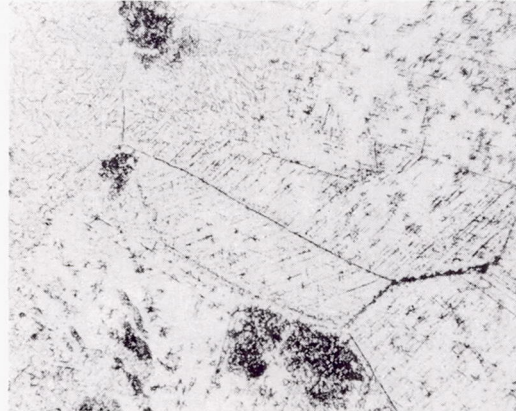
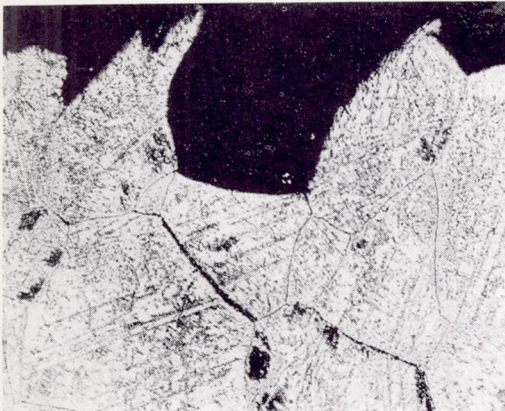
X250



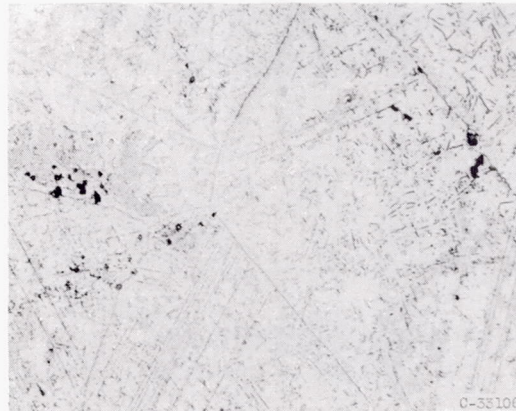
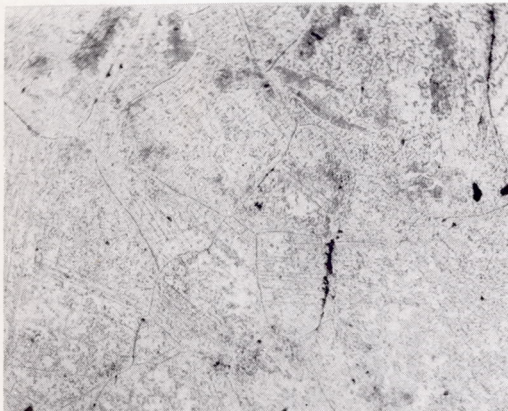
X750



Fractured after 2.5 hr at 28,000 psi



Fractured after 99.9 hr at 21,000 psi

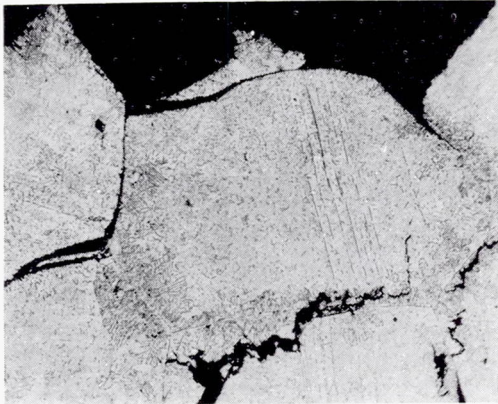


Fractured after 316.3 hr at 16,000 psi

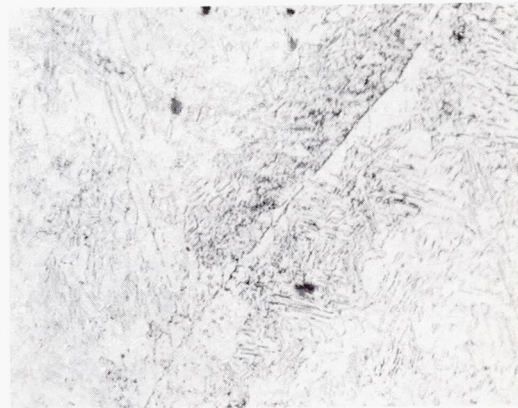
(g) Aged 90 hours at 1200° F.

Figure 8. - Continued. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

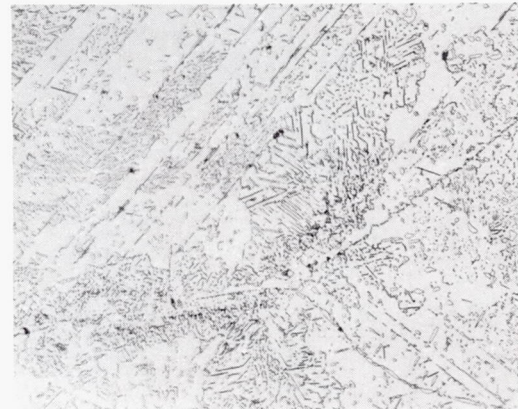
X250



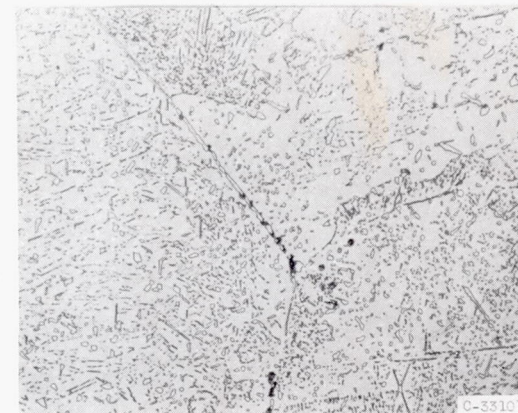
X750



Fractured after 30.1 hr at 25,000 psi



Fractured after 67.1 hr at 21,000 psi



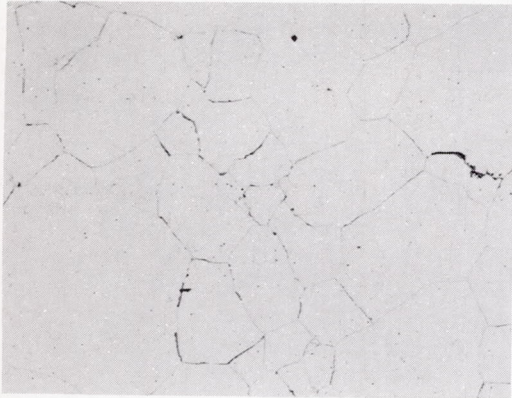
Fractured after 159.2 hr at 16,000 psi

(h) Aged 72 hours at 1950° F.

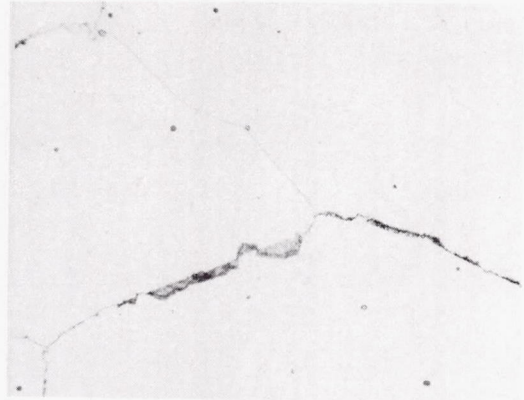
Figure 8. - Concluded. Microstructure of solution-treated wrought Stellite 21 after fracture in stress-rupture at 1500° F.

2810

X100

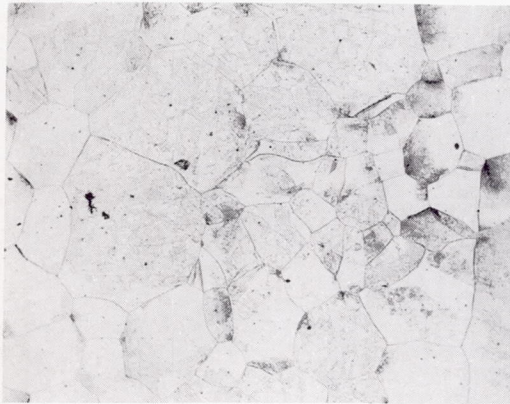


X750

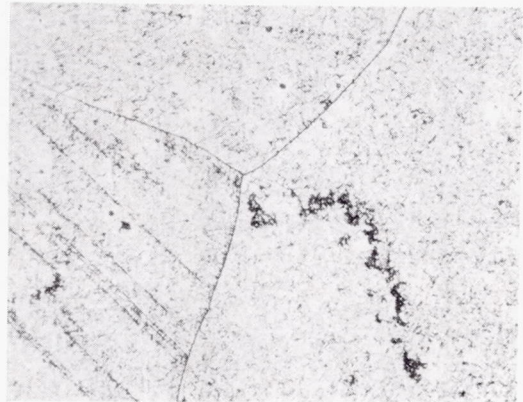


Solution-treated and aged 72 hr at 1200° F

X100



X750

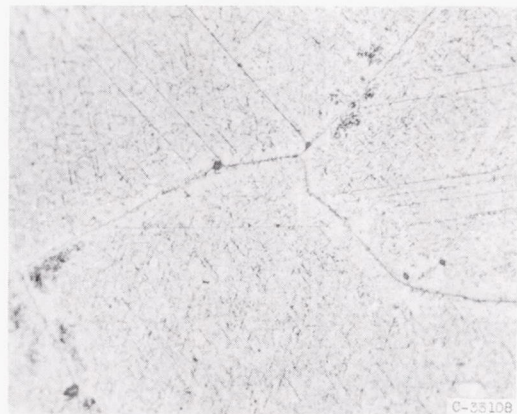


Solution-treated and "double-aged" 72 hr at 1200° F plus 24 hr at 1500° F

X250



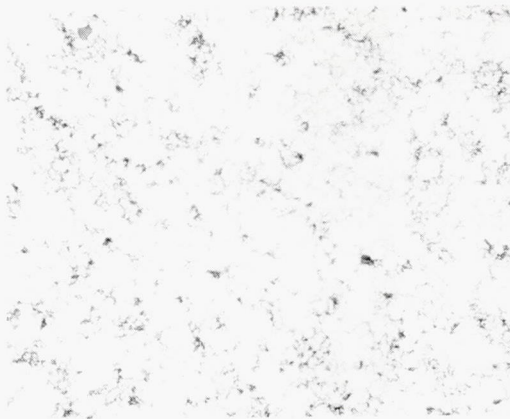
X750



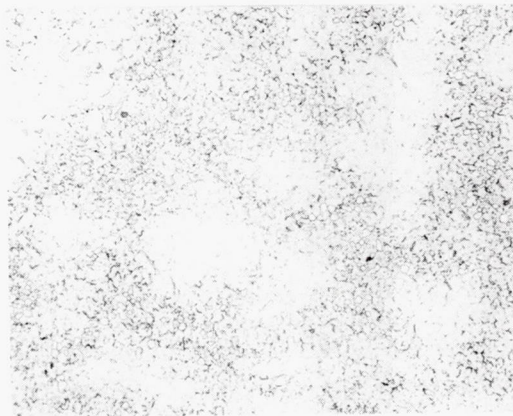
Solution-treated and "double-aged"; fractured after 168.6 hr at 21,000 psi

Figure 9. - Effect of "double-aging" treatment on microstructure of solution-treated wrought Stellite 21.

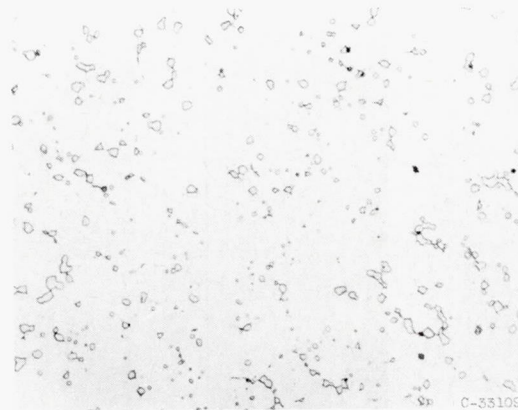
X750



1200° F



1500° F



1950° F

Figure 10. - Effect of aging (72 hr) at various temperatures on microstructure of wrought Stellite 21.

2810

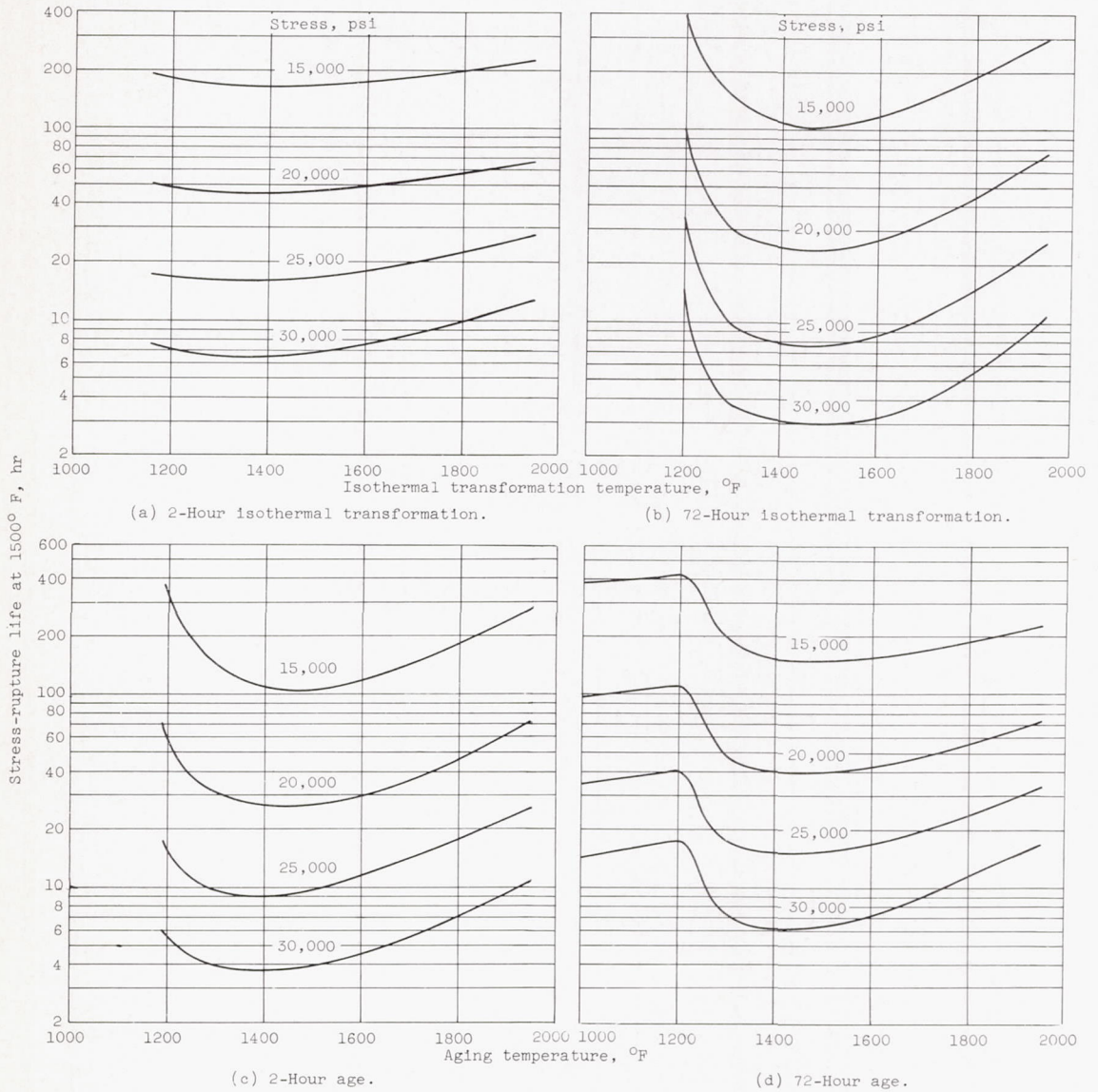


Figure 11. - Effect of transformation temperature on stress-rupture life for indicated heat treatments.

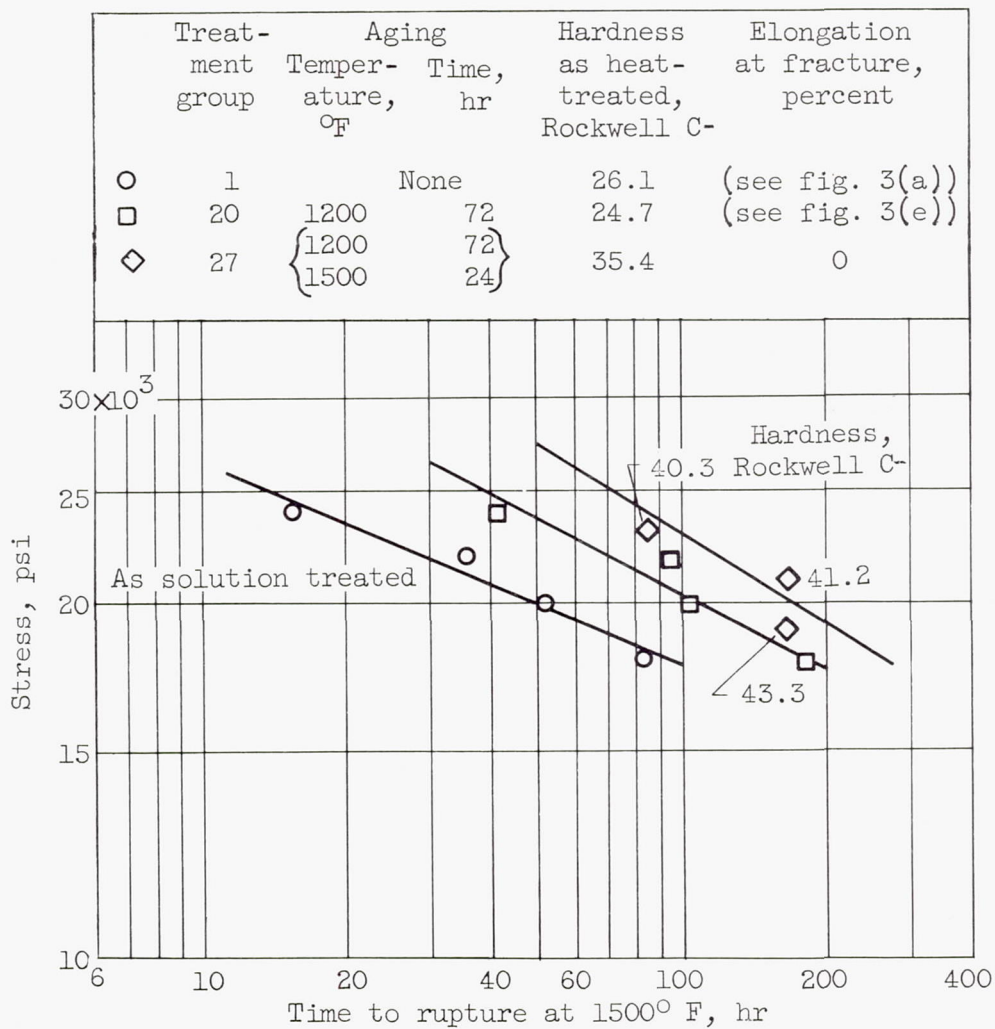


Figure 12. - Effect of aging on stress-rupture life of solution-treated wrought Stellite 21.

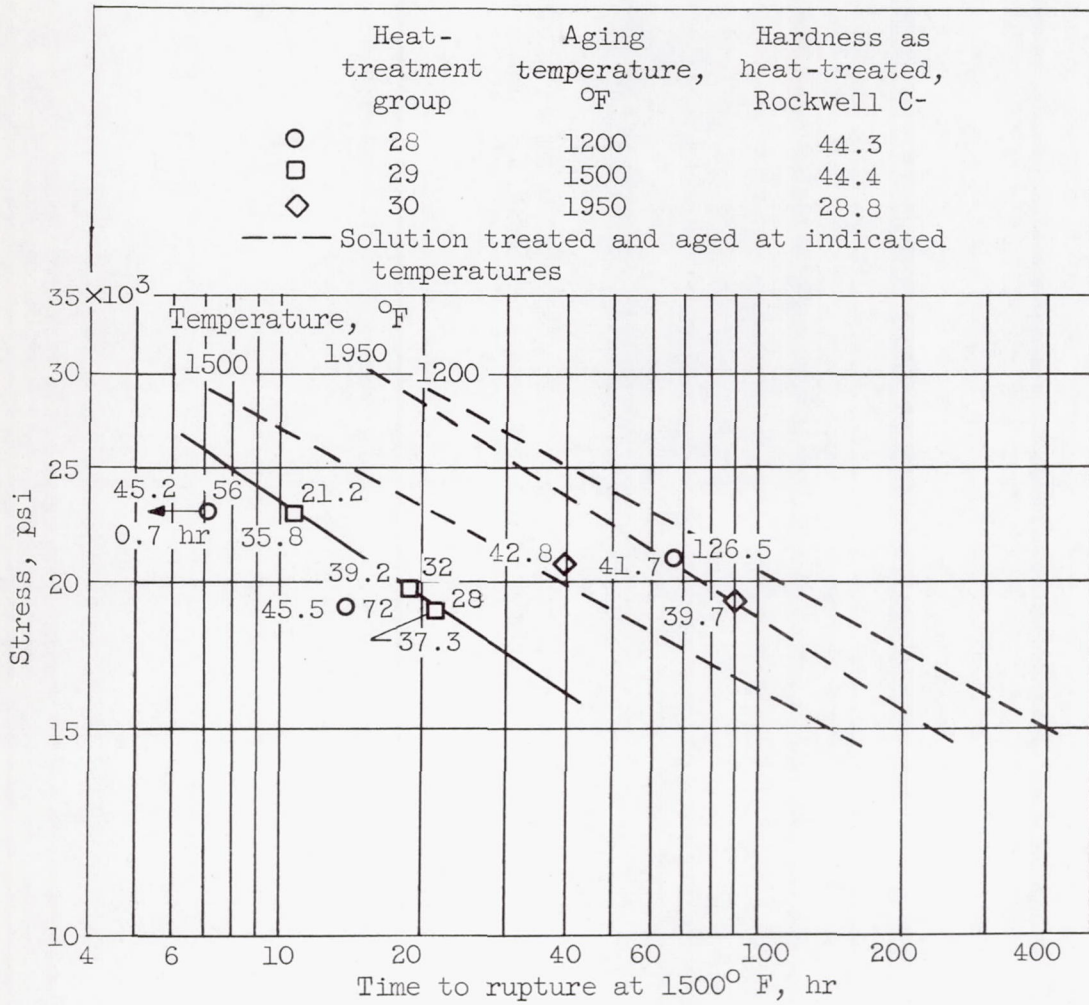


Figure 13. - Comparison of stress-rupture lives of specimens aged 72 hours with and without prior solution treatment. (Data points are plotted only for aging without solution treatment; Rockwell hardness values are to left, and total elongation at fracture (percent) to right of points.)

2810