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PROGRESS REPORT ON STRENGTH AND CREEP OF SPECIAL CERAMIC BODIES IN TENSION AT ELEVATED TEMPERATURES

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National Bureau of Standards

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ADVANCE RESTRICTED REPORT

PROGRESS REPORT ON
STRENGTH AND CREEP OF SPECIAL CERAMIC BODIES
IN TENSION AT ELEVATED TEMPERATURES

By R. F. Geller and M. D. Burdick

SUMMARY

This is a progress report of an investigation for the determination of the behavior of ceramic bodies in tension at elevated temperatures for the purpose of determining data essential to the design of ceramic blades for gas turbines. Observations made on six special ceramic bodies covered the determination of their tensile strength, and also their creep during prolonged loading, under stresses ranging from 3000 to 15,000 psi and at temperatures ranging from 1500° to 2230° F. These observations were supplemented by preliminary tests on a large number of bodies to observe their strength in bending at 1800° F, and also their resistance to thermal shock.

Preliminary tests in bending at 1800° F gave modulus-of-rupture values for commercial specimens ranging from 4900 to 18,000 psi; for specimens of bodies developed at the National Bureau of Standards, the values ranged from 6000 to 38,000 psi. Young's modulus of elasticity values ranged from 6×10^6 to 17×10^6 psi for the former group, and from 9×10^6 to 31×10^6 for the latter group.

Preliminary tests for resistance to thermal shock indicate that three of the compositions developed at the National Bureau of Standards, and three commercial compositions, can be quenched 10 times from a furnace held at 1700° F to room temperature in an air blast without loss in strength as determined by modulus of rupture in bending at room temperature.

Short-time tensile tests at 1800° F have given somewhat erratic results but tensile strengths in excess of 10,000 psi seem

assured. In the creep tests at 1500° F none of the bodies showed sufficient creep, at loads up to failure, to be measurable by the methods employed. Creep at 1800° F and at stresses from 11,000 to 13,000 psi was not excessive for several of the bodies tested. In evaluating these strengths consideration should be given the relative bulk densities of the ceramic materials as compared with those of metallic alloys.

INTRODUCTION

The resistance of ceramic (or nonmetallic) oxides to deformation at high temperatures is their most valuable characteristic as a potential material for gas turbine parts, especially rotor blades or buckets. (See reference 1.) In such blades, the principal stress is tensile and may reach values of several thousand pounds per square inch. Since the net efficiency of a gas turbine power plant increases with increase in the operating temperature, it follows that the higher the temperature at which a blade material will retain suitable resistance to operational stresses, the more attractive will it be for design purposes.

For these reasons, data on the strength and creep characteristics in tension of ceramic bodies is essential to their consideration in turbine design. However, a survey of the literature dealing with the mechanical strength of ceramics in pure tension at ordinary temperatures will show that little information is available. There is even less published information on the mechanical strength of ceramic bodies at elevated temperatures, and the volume of data for tests in tension applying specifically to porcelains is almost negligible. References 2, 3, 4, 5, 6, and 7, and the bibliographies contained in them, are a fairly comprehensive coverage of the literature. Published values deal mostly with strength and modulus of elasticity determined in bending because the preparation of the specimens, the test apparatus, and the procedure for bending tests, is comparatively simple. A tensile test method is described in the ASTM Standards for 1944. (See reference 8.) This method is suitable only for determinations at room temperature and could not be adapted readily to measurements of length changes.

Therefore the tests reported herein were undertaken to obtain additional data on ceramics under tension and at elevated temperatures. This investigation, conducted at the National Bureau of Standards, was sponsored by and conducted with the financial assistance of the National Advisory Committee for Aeronautics.

CERAMICS FOR HIGH TEMPERATURES - GENERAL

Conventional ceramic bodies, such as reported on in references 2 through 7, contain feldspar because it reacts with clay and silica as a flux at elevated temperatures, dissolving them in part to produce a more or less viscous liquid. In a porcelain body, this liquid fills the interstices between the crystalline phases of the mixture, if the ware is properly "fired," thus producing a nonporous mass. Upon cooling, the liquid becomes a matrix of glass upon which the strength of the ceramic is largely dependent. This glass, in common with all glasses, does not have a sharply defined fusion point and will soften and deform under stress at temperatures much lower than the temperatures at which the crystalline phases liquify. This will explain why all the published values on porcelains show that these conventional ceramics deformed at comparatively low temperatures and low stresses.

In 1940, the National Bureau of Standards began a series of investigations to develop glass-free ceramic bodies of the porcelain type which would have simple compositions favorable to high mechanical strength (particularly at elevated temperatures) and good resistance to thermal shock. The compositions studied were binary and ternary combinations of MgO, CaO, BaO, BeO, Al_2O_3, ThO_2, and ZrO_2 both as practically pure oxides and with minor additions of SrO, B_2O_3, SiO_2, TiO_2, or PbO (references 9 and 10).

Consequently, when the National Bureau of Standards was contacted by the National Advisory Committee for Aeronautics in the spring of 1944, regarding the possibilities of ceramics in jet propulsion and in gas turbine engines, there was available for consideration a number of promising ceramic bodies which showed resistance to crushing at ordinary temperatures, to thermal shock, and to deformation in bending at temperatures up to 1800° F, far in excess of corresponding properties for the conventional feldspar porcelains. Shapes can be made of these bodies by any of the established commercial methods for the fabrication of products from nonplastic materials. The compositions are so formulated that they can be matured in commercial kilns, and the products can be finish-ground after maturing in the same manner now employed for the grinding of articles such as spark-plug insulators.

However, before designating compositions to be used in fabricating tension test specimens, leading industrial ceramic concerns were requested to submit appropriate specimens which could be subjected to preliminary flexure tests at 1800° F, and also to thermal shock. The concerns cooperated by submitting bars approximately 6 inches long, and 1 by 1/4 inch in cross section, representing a total of 28 compositions. The preliminary tests of the commercial compositions, and also of compositions developed at the National Bureau of Standards, are given here briefly, together with the results.

FLEXURE TESTS AT 1800° F

The furnace in which these tests were made has been described (reference 11). The specimens were loaded at midpoint, across a span of about 5 inches. The furnace accommodated one specimen at a time and was heated by means of four Globar elements symmetrically spaced about the specimen. After the furnace and specimen had reached equilibrium at 1800° F, load was applied in increments of 7.5 pounds at 2-minute intervals. Deflection at midspan, for the calculation of Young's modulus, was observed by means of a gage reading to 0.0001 inch. The results for the most promising specimens are given in table 1.

RESISTANCE TO THERMAL SHOCK

The data from the thermal shock tests are limited. They were obtained on 34 specimens, representing 14 compositions, by heating the test pieces (in the form of bars) for 30 minutes in a furnace at 1500° F or 1700° F, and then removing them quickly to the air blast from a blower with a 4-inch orifice. The blower was located immediately in front of the furnace and the blast struck the specimens edgewise. After 15 minutes in the air blast, the specimens were returned to the furnace. These specimens which had not cracked during 10 such cycles were tested in bending at room temperature. The modulus of rupture values were compared with values obtained at room temperature on specimens which had not been quenched and the results are summarized in table 2.

TENSION TEST, SPECIMENS AND APPARATUS

In a tensile test, the alinement of the stressing members and the freedom from bend of the specimen must be as nearly perfect as it is humanly possible to make them. Any deviation from perfect alinement, or out-of-straightness of the specimen, or unevenly distributed stress, will introduce bending moments. In addition, every point of weakness within the specimen itself, or within the specimen assembly (such as sharp V-threads and fillets of relatively sharp curvature) forms a source of stress concentration which may result in rupture outside the specimen proper or at a stress not representative of the full strength of the material under test. It may be expected, therefore, that under optimum test conditions, with premature failures in points of weakness eliminated, the maximum observed tensile strength will be more nearly representative of the true tensile strength than is the mean value. It may be expected also that optimum test conditions, and full development of the tensile strength of a given body, will be attained only rarely.

The following compositions which were developed at the National Bureau of Standards have been fabricated into tension specimens:

NBS body designation	MgO (percent)	CaO (percent)	BeO (percent)	AlO_3 (percent)	ThO_2 (percent)	ZrO_2 (percent)	TiO_2 (percent)
151	14.0	--	43.3	--	--	42.7	--
163	7.2	--	26.8	--	--	66.0	--
353	19.6	--	20.3	--	--	60.1	--
358	9.8	--	10.2	--	--	80.0	--
4811	--	2.0	84.2	7.2	--	8.6	--
16021	--	--	90.0	4.3	5.7	--	2.0

All of the tension tests, for which values are given in this report, were conducted on specimens fabricated under contract by the cooperating industrial concerns. All were made by pressing in rubber molds with the exception of the specimen, reported on in figure 3, which had been made by the extrusion process.

The design of the tension specimens, and the necessary adapters for transferring the stress to them, could have been greatly simplified had it not been necessary to determine the extension, or creep. The requirement was that these determinations be made to 1 part in 100,000. For a 100-millimeter gage length, this involved measurements to 1 micron. Therefore, a 100-millimeter gage length was considered the minimum feasible for the work. Since values for modulus of rupture in bending, at 1800° F, in excess of 25,000 psi were observed in the preliminary work, it was estimated that corresponding values in tension might reach 15,000 psi. Therefore, to keep the required loads at a reasonable figure, the specimens were designed with a cross section of approximately 0.07 square inch along the gage length. The shape of the tension specimen is indicated in figure 1. They are round in cross section and screw into adpaters which are $8\frac{1}{8}$ inches long, $1\frac{3}{16}$ inches in diameter, and are made of Body 358.

The assembly of tension specimen and two adapters is of such length that the outer ends of the adapters protrude from the furnace. The adapters, in turn, screw into brass heads which connect with universal joints and, through these, to the loading system.

In 2 furnaces, the specimens can be held at 2400° F, or higher if necessary. In the remaining 10 furnaces, they can be held at a maximum of 1800° F. Each furnace is provided with suitable insulation and a combination of manual and automatic temperature controls permitting maintenance of a predetermined average air temperature over the entire gage length to within ±5° F. Two "peep holes" are provided at mid-height through which the gages can be observed.

The design and locations of the gages are shown in figure 1. These gages are similar in principle to those used at the National Bureau of Standards for measuring creep of metals. These gages, in turn, are a modified form of those described by Fellows, Cook, and Avery (reference 12). They were fabricated from platinum-rhodium tubing and wire. The wire is free to move within the tube, and the relative movements of the specimen at the points where the two parts of the gage are cemented to the specimen may be determined by observing reference lines on the wire and on the cutaway section of the tube. The measurements were made with an illuminated Gaertner extensometer-viewing device provided with a filar micrometer eyepiece. It is similar to the instrument described by John A. Bonnett and Dunlap J. McAdam, Jr. (See reference 13.) This instrument permits measurements to be made with a precision of 1 micron, and readings may be interpolated to 0.1 micron.

Initially, the metal frames supporting the furnaces and loading devices were placed directly upon the concrete floor. It soon became evident that vibrations transmitted from the building to the gages during most of the working day made observations of length changes very difficult and inaccurate. Cork pads 3/4 inch thick, and averaging about 1 square inch in area, for every $6\frac{1}{4}$ pounds of load, placed between the racks and the floor eliminated all but the major shocks resulting from the occasional operation of heavy machinery.

Tension Test Procedure

Two general procedures have been followed so far, and both may be described as "step testing." They are:

Method 1. - The specimen is heated gradually to 1500° F and a stress considered to be well below the tensile strength of the specimen is applied. After observing length changes for some predetermined time, the stress is increased. Length changes are observed again, whereupon another increment of stress is applied, and so on to rupture. It was customary to increase the stress in 1000-psi increments at about 200-hour intervals.

Method 2. - The original temperature and load conditions are essentially as described for Method 1. In this method, however, the load remains constant and the temperature is raised, usually in 100° F steps. In some cases, the specimen had not ruptured after prolonged holding at 1800° F. When this occurred in a furnace in which the temperature could not be raised safely above 1800° F, the test was continued by increasing the load in steps while maintaining the 1800° F temperature.

Two developments interfered with the completion of some of the tests made according to Methods 1 and 2. Failure might occur in the adapter, and the stress at time of failure consequently did not represent the strength of the specimen (see fig. 3) or, after prolonged use (usually over 2000 hours), the thermocouples might become inoperative and the test was discontinued (e.g., fig. 16).

Short-time tensile tests also were attempted. In these tests, the specimen temperature was raised to 1800° F and stress was then applied, at the rate of about 1200 psi per minute to failure, by flowing shot into a bucket at the end of the loading beam. As the data in table 3 show, the results were vitiated in seven out of the eight tests because failure occurred in one of the adapters, leaving the specimen intact.

Creep Test Results

The results, presented in the form of time-extension (elongation) curves in figures 2 through 20, are grouped according to body compositions. Some curves will show irregularities, or apparent negative creep. Many of those small length changes probably are not significant. A length change of 0.01 percent represents an actual change of only 10 microns, or about 0.0004 inch, over the 100-millimeter gage length. Deviations of not over 0.004 percent from a mean curve are considered to be within the over-all reproducibility of a length determination.

The "total extensions" shown in the figures include the elastic deformation which occurred upon application of the initial load. In some tests (fig. 2), this deformation may be greater than the subsequent creep which was observed while the specimen was being held under load at the elevated temperatures.

The tensile stress induced in a turbine blade by centrifugal force is directly proportional to the density of the blade material. Therefore, the density of the ceramic should be considered when comparing the strength of one ceramic with that of another, or with the strength of metallic alloys. The bulk density of a ceramic body depends upon its history. Table 4 summarizes the results of the creep tests, and gives the approximate maximum bulk densities for six compositions developed at the National Bureau of Standards.

Body 151. - Two tests have been completed on specimens of Body 151 and the results are shown in figures 2 and 3. Both tests were conducted at a constant temperature of 1500° F, with the stress increased in increments of 1000 psi. The two results are not comparable because: (a) one specimen (fig. 2) was pressed and the other specimen (fig. 3) was extruded; (b) one failure (fig. 3) occurred in the adapter and not in the specimen; and (c) the initial stress for one specimen (fig. 2) was 7000 psi, while the other (fig. 3) had been under test for $548\frac{1}{2}$ hours before 7000 psi was applied. The stress-temperature history of a specimen may have an effect on its strength, as will be pointed out in the discussion of other tests.

Body 163. - Neither of the two tests completed on specimens of Body 163 was productive of completely positive data. In the first test (fig. 4), the specimen apparently continued to contract very slightly at stress of 6000 psi or less, and then remained practically constant in length. The total length change, observed before the adapter broke, was only 0.0002 inch per inch, and apparently was negative.

In the second test (fig. 5), the temperature was 1700° F for 2180 hours, during which time elongations of definitely measurable magnitude occurred under stresses of from 8000 to 12,000 psi, with the maximum creep rate of 1.2×10^{-6} inch per inch per hour while at 11,000 psi. Curiously, the creep rate was very low (av. 0.5×10^{-6} in. per in. per hr) under stresses of 13,000, 14,000, and 15,000 psi.

Body 353. - Of the four tests (figs. 6, 7, 8, and 9) conducted on this body, three were completed satisfactorily in that the specimens ruptured at a point within the gage length.

The first test (fig. 6) shows clearly measurable creep, under the constant load of 4000 psi, when the holding temperature was 1800° F, and very high creep rates when the temperatures were 1900° and 1950° F. It is interesting to note that, when the temperature was then dropped back to 1800° F, the creep rate was only about one-tenth of the rate observed during the first holding at 1800° F; in fact, even when the stress had been increased to 7000 psi the creep rate was only one-half of the first value. This effect of the stress-temperature history of a specimen was referred to earlier in connection with Body 151.

In the test for which results are shown in figure 7, the load was held constant at 6800 psi, and the temperature increased until the limit of the heating coils (1800° F) was reached. The load was then removed to observe elastic recovery which, as may be seen from the curves in figure 7A, amounted to about 0.025 percent during the first few minutes and increased to about 0.035 percent during the succeeding 22 hours. This was approximately 35 percent of the total extension up to the time the load was removed. When the stress of 6800 psi was again applied the immediate, or elastic, extension (see curves in fig. 7B) was about 0.025 percent. During the remainder of the test, the temperature was maintained at 1800° F while the stress was increased in steps to 12,000 psi and rupture. At no time was the creep rate excessive.

The results in figure 8 show no significantly measurable creep during any of the treatments. In fact, the total elongation during the entire test was only 0.0003 inch per inch. It will be noted that the "rate of creep per hour" is negative in most cases even though the "total extension" was increasing. The explanation for this lies in the fact that a certain amount of elastic deformation would occur upon application of a 1000-psi increment of stress. Upon holding, the specimen then appeared to undergo "negative creep" which, however, was in most cases not as great as the initial positive elastic deformation.

Figure 9 shows an appreciable initial deformation (0.021 percent) upon application of the 4000-psi stress at 1700° F, but throughout the test the creep rate was very low. For reasons given previously in the text, it was necessary to install cork pads under the furnace racks. Since some jarring of the loading system was unavoidable during the raising and lowering of the racks, for the installation of the cork pads, the rupture upon reapplication of the load probably meant that the specimen assembly had been injured.

Body 358. - Five tests were made on four specimens of Body 358 and the results are shown in figures 10 through 14. Results in figures 10 and 13 are for the same specimen.

The curves in figure 10 resemble those in figure 6 (Body 353) as regards the much higher creep rates at 1900° and 1950° F, compared to the rates at and below 1800° F. These higher rates for Body 358 were, however, only about one-half those for Body 353.

The curves in figure 10A, and figure 11A, show the effects of 4000 and 7000-psi stress at temperatures up to 1800° F. The higher stress more than doubled the creep rates. The elastic recovery is much like that observed in figure 7, and the creep rate upon reapplication of the 7000-psi stress (fig. 11B) also is not significantly different from the creep rate observed during the first holding at 1800° F. Note the contrast with the results in figure 6, in which intermediate holding at a higher temperature had greatly reduced the creep rate during the second holding at 1800° F. Also worthy of note in figure 11A and 11C is the fact that the creep was practically the same at 10,500-psi stress and 1800° F as it was at 7000-psi stress and 1700° F, and that it is almost ten times the creep rate observed in figure 12 at 1500° F, even with a stress of 12,000 psi.

In the latter test (fig. 12), the total deformation during the 1104 hours of the test was only 0.00006 inch per inch, but 13,000 psi probably represents the actual tensile strength of that particular specimen. This practical freedom from creep, up to failure at 1500° F, is shown also in figure 13. The failure was considered premature.

The effect of increasing the stress, while maintaining a constant temperature of 1700° F, is shown by the data in figure 14. The high deformation indicated during the first 26 hours at 7000 psi is probably due to some error in the observations.

Body 4811. - Results of three tests completed on this body are shown in figures 15, 16, and 17. The first test was carried out by holding the stress constant (4000 psi) while the temperature was raised in steps. This body, made of a high-BeO composition, showed satisfactory resistance to creep at 1900° F, and even at 2050° F the average creep was lower than for the high ZrO_2 composition (Body 358, fig. 10) at 1900° F. At 2100° F, however, the deformation became excessive, and rupture probably would have occurred in comparatively short time even if the temperature had not gone to 2230° F.

A comparison of results obtained with Body 4811 and Body 358 may be obtained from figures 16 and 11. The values show Body 4811 to be the stronger, and the most resistant to creep, at 1800° F. There is no explanation from the data for the comparatively large extension of Body 4811 during the first 120 hours at 1800° F and 7000 psi. (See fig. 16B.)

The curves in figure 17 show an extremely small extension for Body 4811 at 1500° F, and indicate a tensile strength of about 15,000 psi at this temperature.

Body 16021. - Results of creep tests on this body, which has a high-BeO content similar to Body 4811, are shown in figures 18, 19, and 20.

One specimen (fig. 18) broke near the center of the gage length after only $39\frac{1}{2}$ hours at 7000 psi and 1800° F. Moreover, the creep rate of about 9×10^{-6} inch per inch per hour indicates this composition to be weaker than Body 4811. (See fig. 16.)

This is substantiated by the comparable results in figures 17 and 19. In the latter test, the resistance to creep of Body 16021 was, in general, only very slightly less than that of Body 4811, but the specimen ruptured after 51 hours at 13,000 psi and 1500° F. Body 4811 (fig. 17), by comparison, when subjected to the same test conditions for $170\frac{1}{8}$ hours (and having had a similar stress-temperature history) showed an average creep rate of only 0.01×10^{-6} inch per inch per hour and required a stress of 15,000 psi to break it.

Also, results for Body 16021 and Body 358 at 1500° F may be compared by means of the curves in figures 12 and 19. They indicate nearly equal strength, but somewhat greater resistance to creep for the high-ZrO_2 Body 358. The data in figures 14 and 20 furnish a comparison between these two bodies at 1700° F and again indicated Body 358 to be slightly more resistant to creep.

Short-Time Tensile Tests

Although this part of the study has not been productive of satisfactory data, it has been instructive. (See table 3.) The true reason for the high percentage of failures which occurred in the adapters during short-time tensile tests is not known. It is possible that rapid application of stress may have accentuated some weaknesses in the adapters which would have had an opportunity to "iron out" under sustained loads at the elevated temperatures of the creep tests.

SUMMARY OF RESULTS

A series of preliminary tests were made to select the most promising compositions from a number of test specimens submitted by commercial concerns, and other specimens representing bodies developed at the National Bureau of Standards.

Modulus of rupture values obtained by center loading, at 1800° F, ranged from 4900 to 18,000 psi for commercial bodies, and from 6000 to 38,000 psi for bodies developed at the National Bureau of Standards. Six of the latter have been made into specimens for testing in tension and results are given in this report.

Some preliminary tests of resistance to thermal shock also were made. The results indicate that three of the commercial compositions and three of those developed at the National Bureau of Standards, can be quenched 10 times from 1700° F to room temperature in an air blast without significant loss in mechanical strength.

Six bodies developed at the National Bureau of Standards were then selected for the creep tests. The following are their laboratory designations and approximate bulk densities:

Body 151,	density 3.8	Body 358,	density 4.9
Body 163,	density 4.4	Body 4811,	density 3.0
Body 353,	density 4.4	Body 16021,	density 3.0

These densities should be considered in the evaluation of a composition. The factor obtained when strength in tension is divided by density is suggested as the measure of resistance to stress. This is especially desirable when comparing ceramics with metallic alloys.

In this progress report, the results are, for the most part, based on a single specimen for a given set of conditions. Consequently, they do not justify definite conclusions or generalizations. The data available at this time indicate the following:

Body 151. - The results show that Body 151 is capable of withstanding at least 13,000 psi in tension at 1500° F for a prolonged period of time. The total length change during the entire time of the tests (exclusive of the elastic deformation upon application of the initial load) was too small to be determined with certainty by the test method used.

Body 163. - It appears that this composition could withstand a tensile stress of at least 15,000 psi at 1700° F without creep in excess of about 1×10^{-6} inch per inch per hour.

Body 353. - The tests indicate that this body can resist a tensile stress of 13,000 psi at 1700° F, or 11,000 psi at 1800°F, without excessive creep but that even 4000 psi at 1900° F might be too severe. However, one specimen did sustain a stress of 4000 psi at 1900° and 1950° F for a total of about 335 hours without failure.

Body 358. - In general, high creep rates may be expected from this body at and above 1900° F, but at 1700° F a stress of 13,000 psi produced an average elongation of only 0.5×10^{-6} inch per inch per hour.

Body 4811. - The results show an extremely small extension for this body at 1500° F, and indicate an ultimate strength in tension of about 15,000 psi at this temperature. In general, Body 4811 shows satisfactory resistance to creep and rupture under a stress of 4000 psi at 2050° F, 13,000 psi at 1800° F, or 14,000 psi at 1500° F.

Body 16021. - This body showed negligible creep at 1500° F for all stresses up to rupture (13,000 psi). At 1700° F the creep was, in general, very small for stresses of 12,000 psi or less, but at 1800° F a stress of 7000 psi was sufficient to cause appreciable deformation.

The short-time tensile tests, which were made in an endeavor to develop stress-rupture-time curves, were not productive of satisfactory data, but the results are instructive. It may be taken as axiomatic that any test of mechanical strength cannot magnify the inherent strength of a specimen but may, and is very apt to, fail in developing the full resistance to failure because of any one of many unfavorable test conditions. Therefore, in exploratory tests such as these, the maximum strengths determined for a series of specimens, and not a mean value, should be considered as more truly depicting the potentialities of the composition under test. For this reason, the results of tests for specimen 104 in table 3 are of especial interest. The exceptionally high strength shown by specimen 104 may be the result of a very good assembly alinement and load application, or the result of its stress-temperature history, or both. The stress-temperature history is shown by the curves in figure 16.

The data indicate that a stress-rupture-time curve for a ceramic body may not be obtainable because the strength at a given temperature, or the creep behavior at a given stress-temperature condition, may be varied to considerable degree by its stress-temperature history.

In summarizing the tests, it might be of value to select the three best bodies of the six tested. The selection may be made on the basis of tensile strength or of resistance to creep at elevated temperatures.

The best, on the basis of tensile strength, appear to be Bodies 4811, 358, and 16021 in the order given. Strengths in excess of 10,000 psi at 1800° F seem assured.

The best, on the basis of resistance to creep at 1800° F and above, appear to be Bodies 4811 and 358. At 1500° F none of the bodies show sufficient creep, at loads up to failure, to be measurable by the methods used. At 1900° F and above, the bodies tested showed much less resistance to creep than they did at 1700° and 1800° F.

National Bureau of Standards,
Washington, D. C., April 4, 1946.

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TABLE 1.- STRENGTH IN BENDING AT 1800° F

[Stress applied in 7.5-pound increments at 2-minute intervals.]

Body designation[1]	Span (in.)	Cross section (in.)	Modulus of rupture (10^3 psi)	Young's modulus of elasticity (10^6 psi)
1089	5.5	0.738 x 0.249	6.7	(2)
2454	5.5	.756 x .257	8.7	7.8
2673	5.5	.752 x .216	13.8	17.4
2693	5.5	.745 x .213	5.6	8.6
9535-16	5.5	.747 x .210	5.6	(2)
2676-16	6	.743 x .212	6.0	(2)
2676-30	6	.737 x .248	7.5	7.0
4142-16	6	.745 x .210	8.2	6.8
4142-30	6	.744 x .215	7.1	6.7
4147-16	6	.742 x .208	6.3	(2)
4147-30	6	.746 x .214	7.9	7.4
35-9	6	.752 x .241	12.4	6.4
3081	6	.768 x .243	10.4	6.4
2933	6	.785 x .230	4.9	(2)
3262[3]	5	.748 x .184	18.0	15.0
1019	6	.727 x .231	(2)	(2)
3239	6	.717 x .232	17.5	12.7
B1-99	4.5	.600 x .260	11.2	18.6
B1-99	4.5	.573 x .220	15.9	23.0
B1-99	4.5	.573 x .221	14.5	23.6
B1241	4.5	.592 x .225	14.5	22.8
B1241	4.5	.595 x .243	12.2	19.0
B1241T	4.5	.605 x .278	15.3	14.2
B1241T	4.5	.594 x .214	13.0	14.6
B61201	4.5	.603 x .265	6.0	9.2
B6781	5	.572 x .222	6.9	14.0
B6781	5	.569 x .196	7.3	15.2
B3401	4.5	.596 x .183	10.1	21.9
B151	4.5	.596 x .186	22.3	20.0
B163	5	.628 x .224	30.5	18.5
B163	5	.624 x .241	24.9	16.8
B163	4.5	.612 x .181	29.9	24.3
B163	4.5	.622 x .183	31.9	21.1
B353	4.5	.607 x .212	31.8	16.9
B358	4.75	.546 x .225	26.9	20.0
B358	4.75	.547 x .242	25.3	19.0
B358	4.75	.634 x .183	25.1	19.7
B358	5	.573 x .244	38.0	18.2
B4811C	4.5	.585 x .204	14.6	20.5
B4811C	4.5	.577 x .197	13.0	20.7
B16021T	4.5	.575 x .181	16.0	29.3
B16021T	4.5	.572 x .165	17.5	30.6
B16021T	4.5	.582 x .262	16.7	19.3

[1] The letter B preceding the body number designates a composition developed at the National Bureau of Standards; all others are from commercial concerns.

[2] Plastic flow.

[3] 3.1 percent water absorption by 5-hour boil method.

NOTE — Tests performed by A. S. Creamer of the National Bureau of Standards.

TABLE 2.- RELATIVE RESISTANCE TO THERMAL SHOCK

(Specimens quenched 10 times from temperature indicated to air blast; tests by A. S. Creamer.)

Body designation[1]	Quenching test - furnace temperature (° F)	Modulus of rupture at room temperature -	
		Specimens quenched (10^3 psi)	Specimens not quenched (10^3 psi)
3239	1500	18.5	18.8
3239	1500	19.0	19.6
3239	1500	19.0	19.1
3239	1700	15.3	----
3239	1700	15.6	----
3270	1700	24.8	21.2
3270	1700	21.0	23.1
3273	1700	21.0	18.4
3273	1700	24.3	20.8
2454	1700	39.0	36.0
2454	1700	29.8	34.9
2454	1700	3.9	----
2454	1700	43.7	----
2673	1500	31.1	28.2
2673	1500	31.1	34.6
2673	1700	5.3	----
2673	1700	(2)	----
2673	1700	10.6	----
2673	1700	22.5	----
2673	1700	26.6	----
2673	1700	6.6	----
B1-99	1700	3.5	15.9
B1-99	1700	15.1	14.5
B163	1700	(2)	36.6
B163	1700	(2)	40.5
B174	1700	(2)	28.2
B174	1700	(2)	24.5
B353	1500	29.2	24.9
B353	1500	29.2	27.6
B353	1500	32.0	----
B353	1700	(2)	----
B353	1700	(2)	----
B358	1700	(2)	22.7
B358	1700	(2)	27.2
B4811C	1700	23.6	24.1
B4811C	1700	24.6	26.3
B16021T	1700	20.8	21.9
B16021T	1700	21.6	18.5

[1] See footnote 1, table 1.

[2] Cracked before 10 quenches were completed.

TABLE 3.- SHORT-TIME TENSILE TESTS AT 1800° F

Specimen No.	Body No.	Max. stress: (psi)	Rupture occurred in:
90	163	6,400	Adapter
91	163	9,980	Do.
92	163	5,870	Do.
100	4811	4,610	Do.
102	4811	11,750	Do.
103	4811	10,060	Specimen
104	4811	18,990	Adapter
104	4811	18,370	Do.

TABLE 4.-CREEP TESTS IN TENSION AT ELEVATED TEMPERATURES

NBS Body No.	Approximate bulk density	Total time of test (hr)	Total extension[1] (percent)	Maximum rate of creep[2] (percent x10^{-4}/hr)	Duration of test at maximum temperature and stress (hr)	Maximum temp. (°F)	Maximum stress[3] (psi)
151	3.8	900	0.027	--	29	1500	12,000
151	3.8	1650	.012	0.27	92	1500	[4]13,000
163	4.4	1417	.022	--	38	1500	[4]12,000
163	4.4	2198	.216	1.25	164	1700	[5]15,000
353	4.4	1554	.029	.26	1	1500	13,000
353	4.4	1438	.064	.56	137	1700	[6]13,000
353	4.4	1591	.510	--	141	1800	7,000
353	4.4	2141	.184	1.3	8	1800	12,000
358	4.9	668	.015	--	0.1	1500	[6]10,000
358	4.9	1104	.006	.35	0.2	1500	13,000
358	4.9	1268	.103	2.08	91	1700	14,000
358	4.9	2238	.423	2.68	93	1800	[5]10,500
358	4.9	1028	.373	9.12	219	1950	[6]4,000
4811	3.0	1627	.021	.39	0.25	1500	15,000
4811	3.0	2589	.230	2.36	117	1800	[5]14,000
4811	3.0	2094	.339	24.1	.75	2230	4,000
16021	3.0	1247	.026	--	51	1500	[6]13,000
16021	3.0	1406	.152	2.51	44	1700	13,000
16021	3.0	929	.047	9.53	39	1800	7,000

[1] Includes elastic deformations, and creep, over a 100 mm gage length.

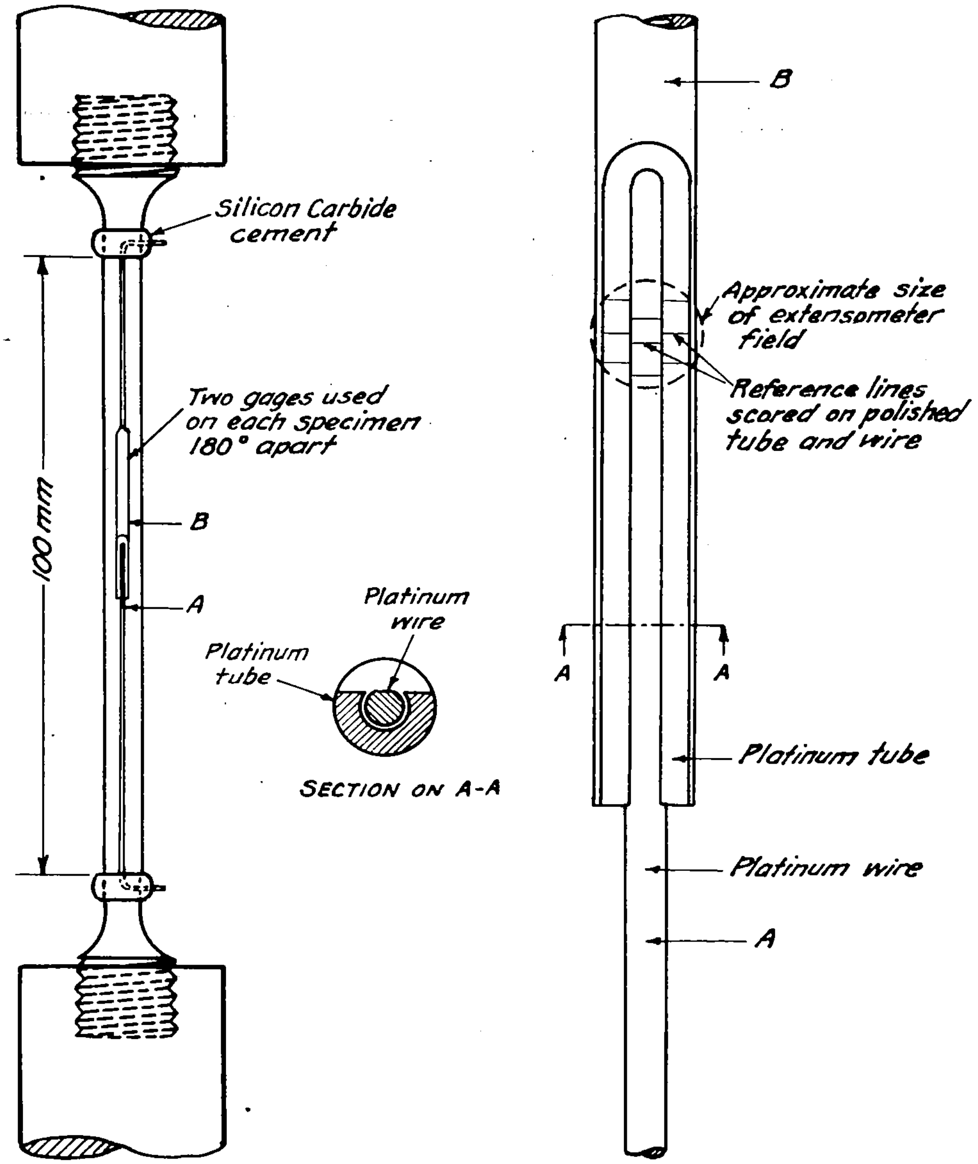
[2] Based on creep only.

[3] Stress at time of failure. Failure within gage length of specimen unless noted otherwise.

[4] Failure occurred in the adapter, leaving the specimen intact.

[5] Test discontinued; specimen still intact.

[6] Failure occurred in the threaded head of the tension specimen.



ASSEMBLED ADAPTORS, SPECIMEN AND GAGE

EXTENSOMETER GAGE

Figure 1.- Assembly of tension specimen with the ceramic adapters and the gage for observing length changes, and an enlarged view of the gage.

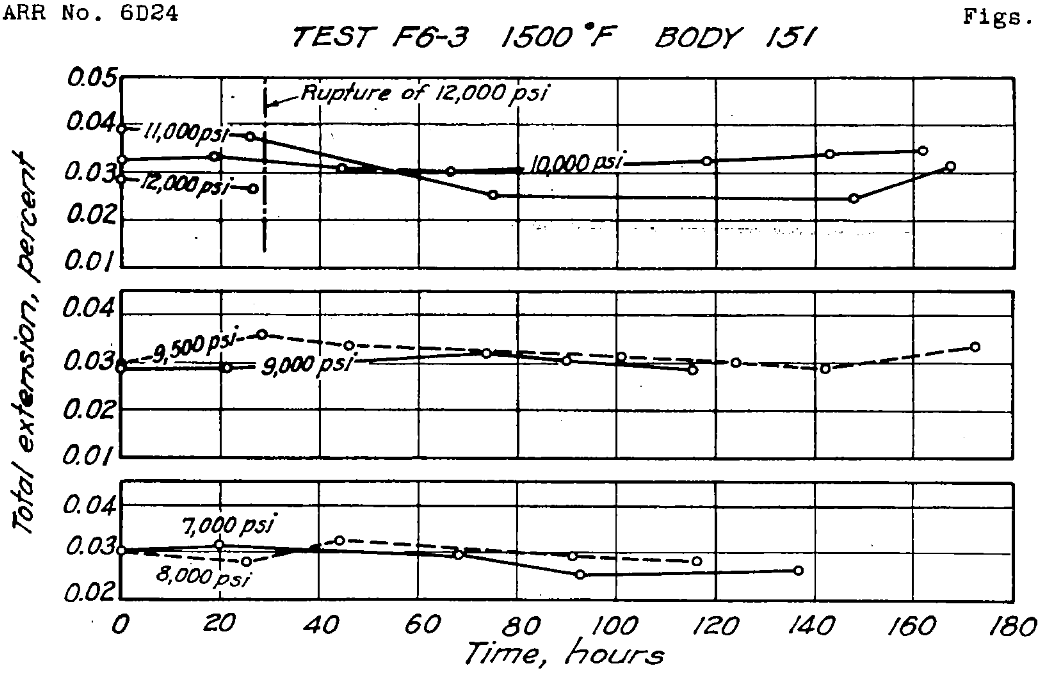


Figure 2.- Time-extension curves for a specimen of body 151 at 1500°F and supporting various loads applied in the order of increasing stress. Rupture occurred in the top fillet of the specimen after 29 hours at 12,000 psi. Total time of test was 900 hours. The creep was not measurable.

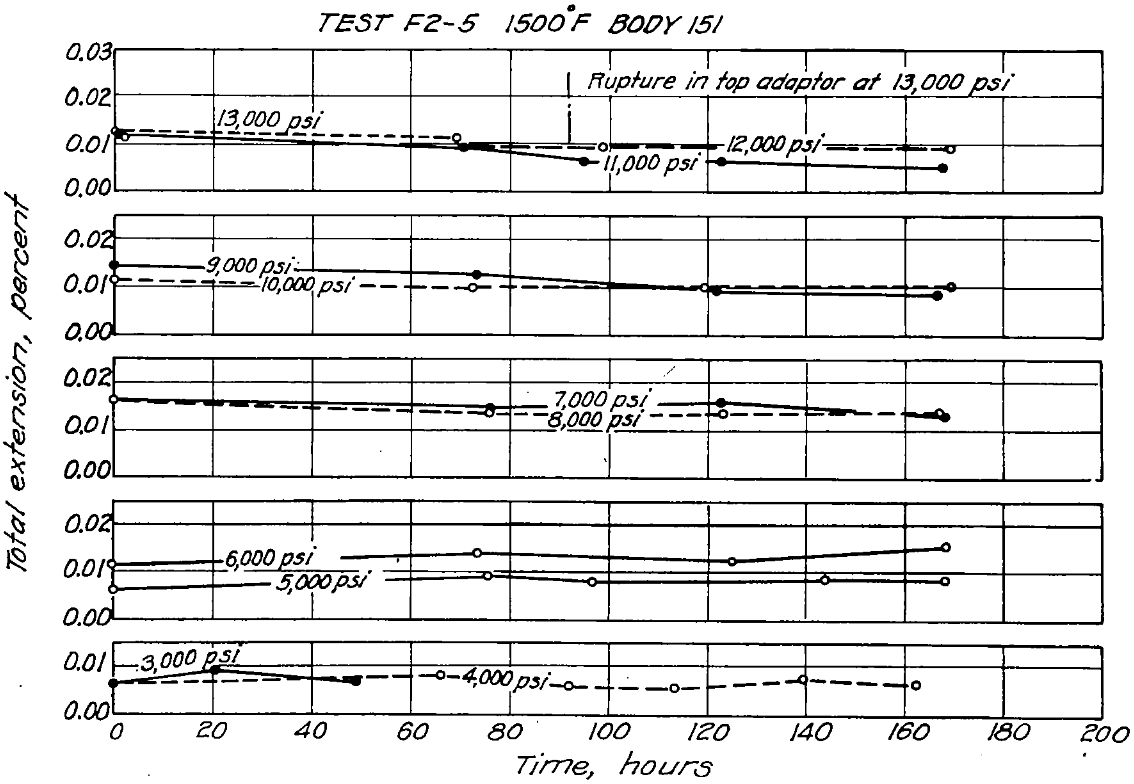
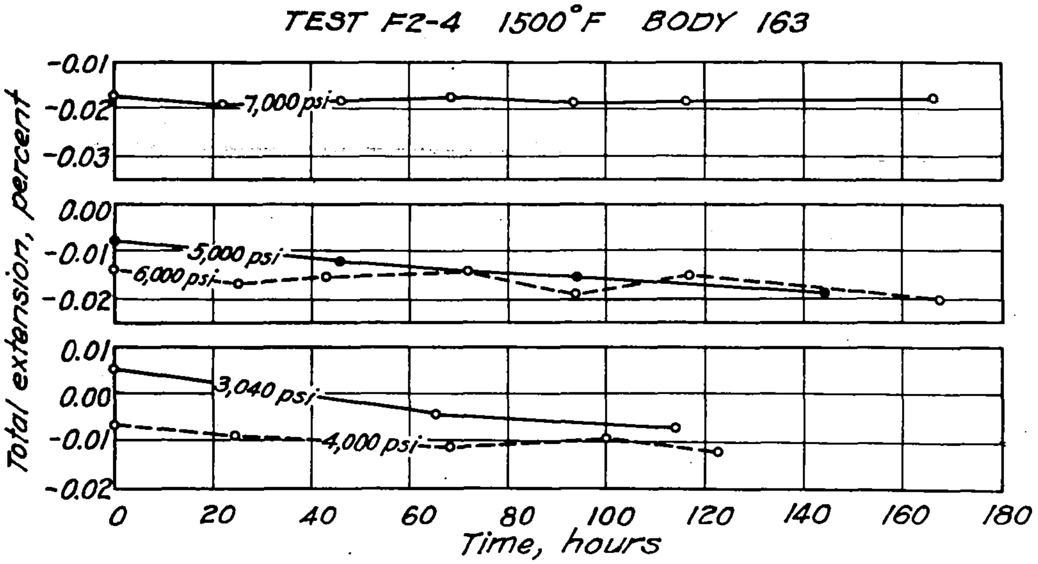
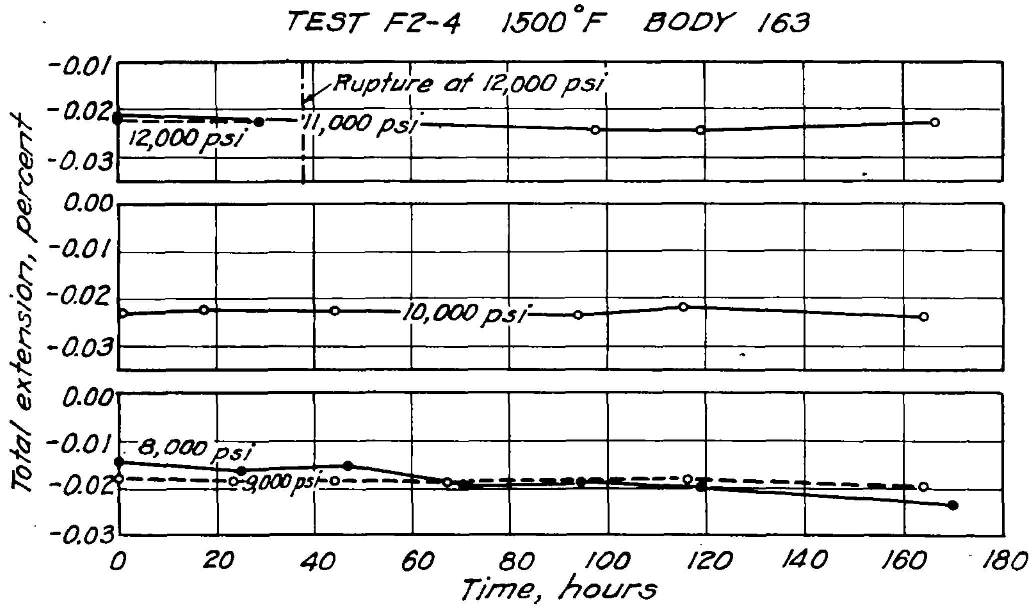


Figure 3.- Time-extension curves for a specimen of body 151 supporting various loads at 1500°F. The curves were obtained in the order of increasing stress. Rupture occurred in the top adapter, outside of the furnace, after 92 hours at 13,000 psi. Total time of test was 1650 hours and the maximum positive creep-rate was 0.27 x 10^{-6} in. per in. per hour.

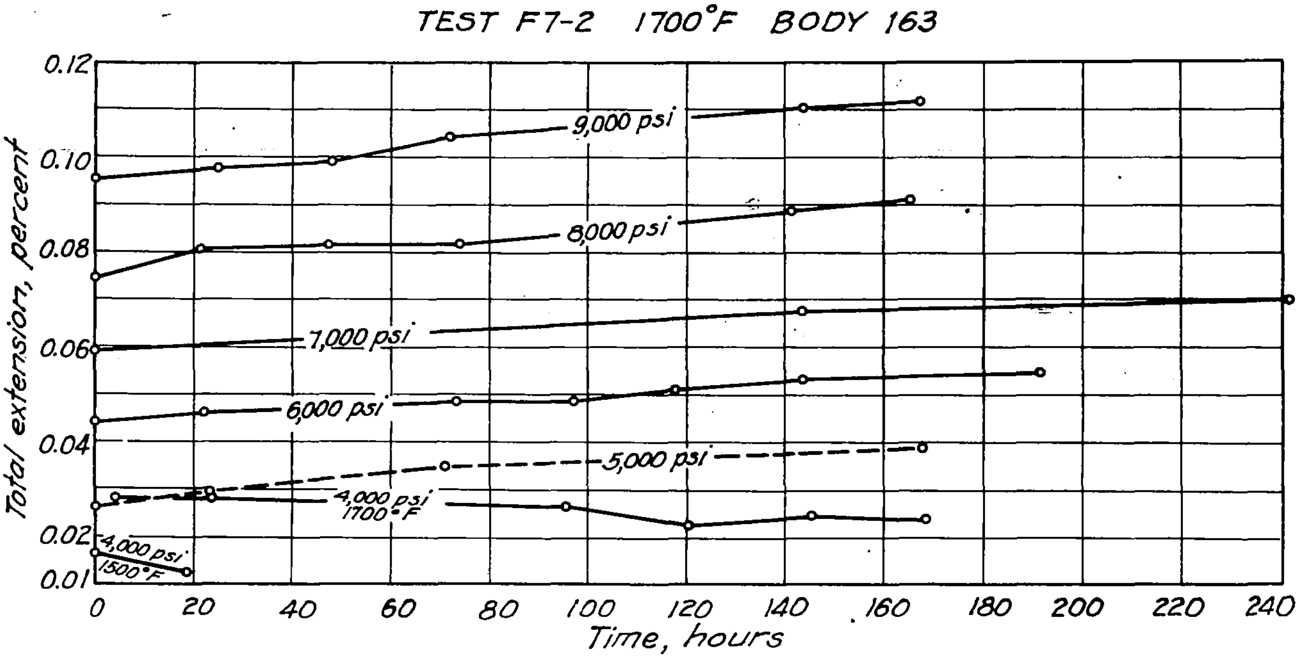


(a) Stresses between 3040 and 7000 psi at 1500°F.

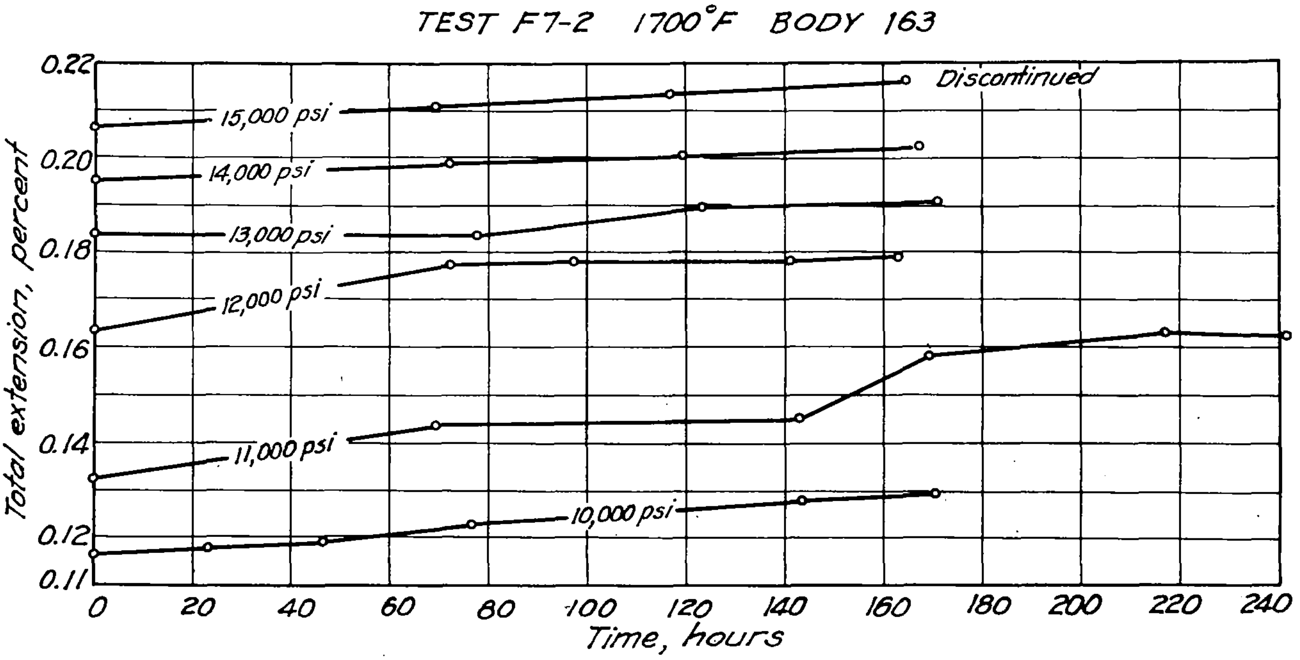


(b) Stresses between 8000 and 12,000 psi at 1500°F

Figure 4.- Time-extension curves for a specimen of body 163 subjected to various stresses. Rupture occurred in the top adapter after 38 hours at 12,000 psi. Total time of test was 1417 hours. The creep was not measurable.

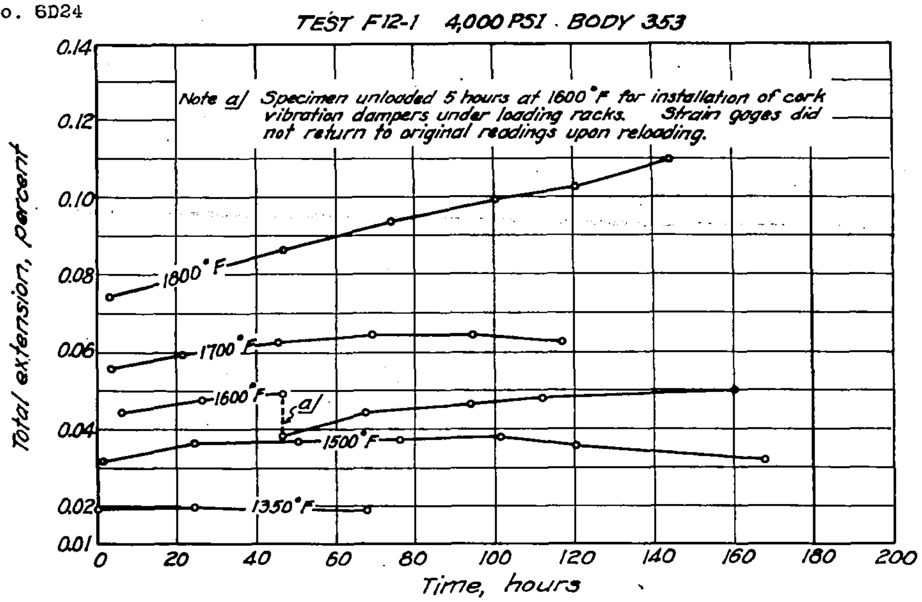


(a) Stresses between 4000 and 9000 psi at 1700°F.

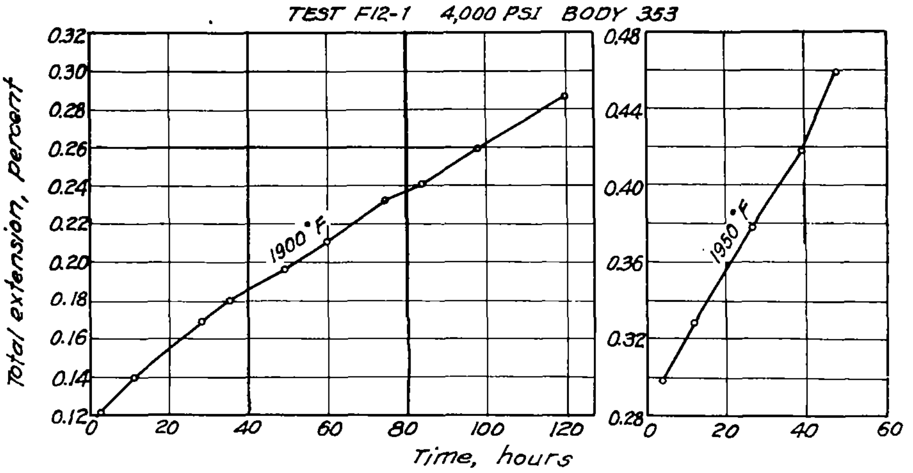


(b) Stresses between 10,000 and 15,000 psi at 1700°F.

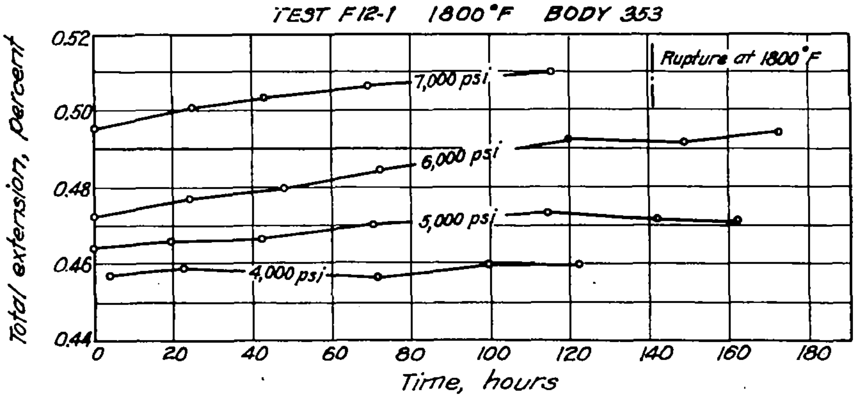
Figure 5.- Time-extension curves for a specimen of body 163 supporting various loads. A short preliminary treatment was allowed at 1500°F and 4000 psi. The test was discontinued after 2198-1/2 hours, and before rupture, when the base-metal thermocouples became unreliable. Maximum creep-rate was 1.25 x 10^{-6} in. per in. per hour.



(a) Temperatures increased in increments from 1350 to 1800°F.

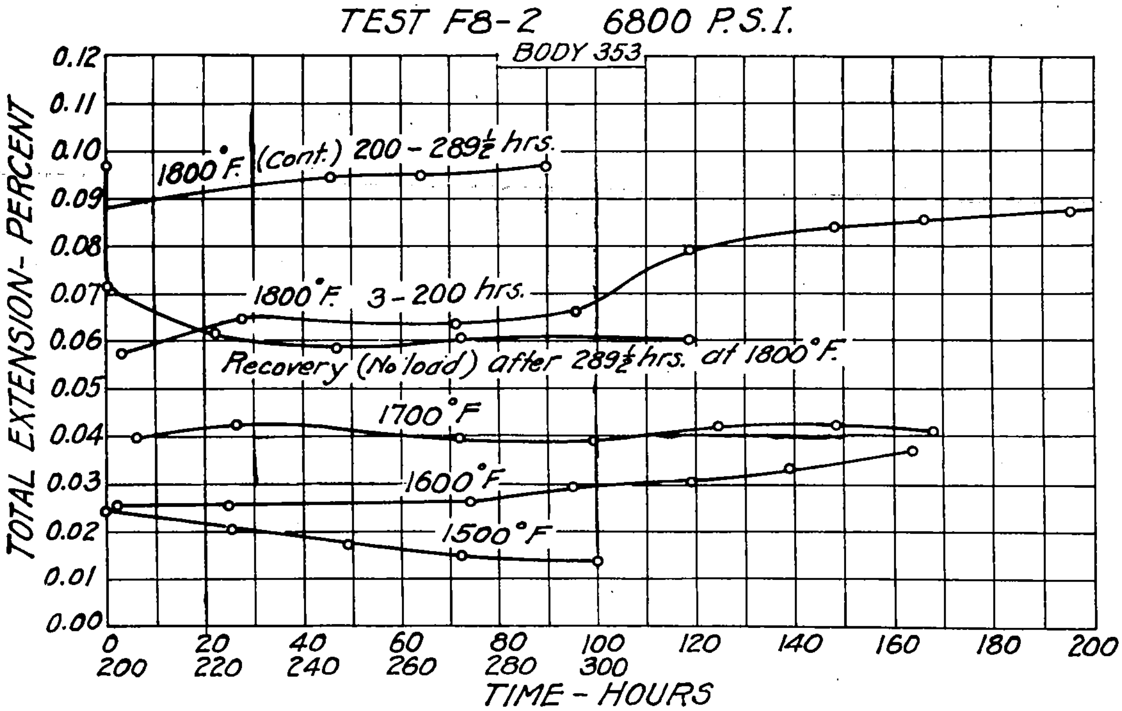


(b) Temperatures of 1900 and 1950°F.

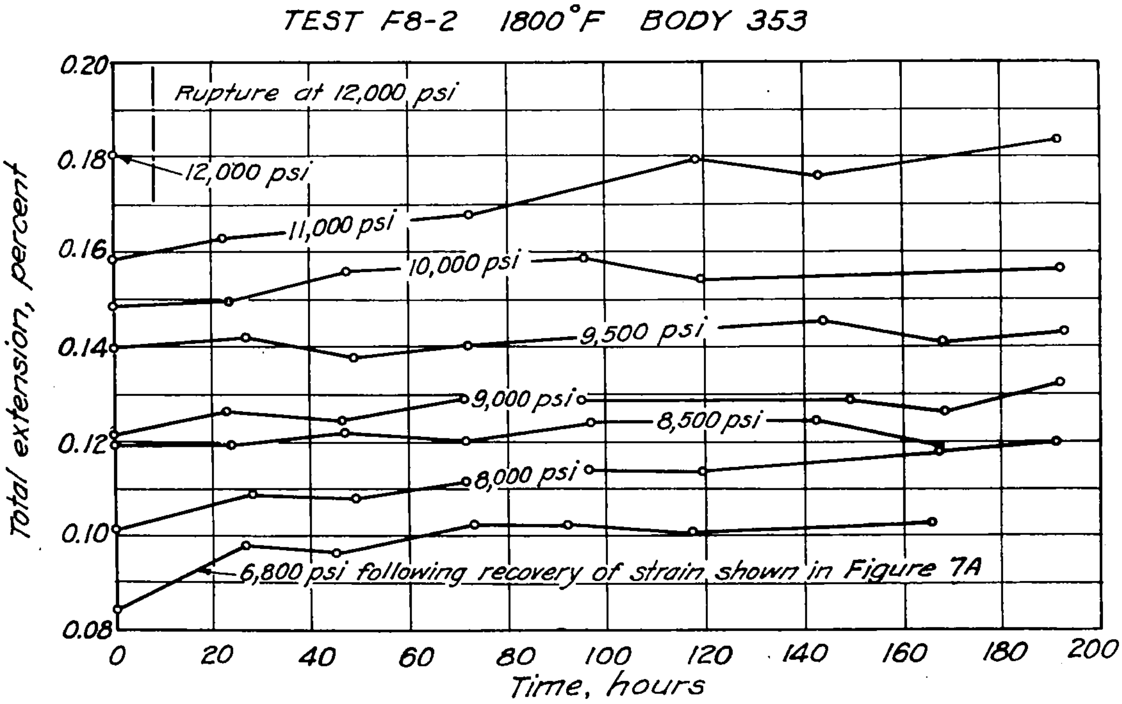


(c) Following the treatments outlined in (a) and (b), the temperature was reduced to 1800°F and the curves in this figure show extensions for the specimen supporting various loads at 1800°F.

Figure 6.- Time-extension curves for a specimen of body 353 held at a constant stress of 4000 psi. The curves in figure 6(c) were obtained in the order of increasing stress. Rupture occurred near the center of the gage length. The total time of the test was 1591 hours. Maximum creep-rate (18.2×10^{-6} in. per in. per hour) was observed at 1950°F.



(a) Constant stress of 6800 psi in the order of increasing temperatures between 1500 and 1800°F. The recovery curve (at no load and 1800°F) was obtained after 289-1/2 hours at 6800 psi and 1800°F.



(b) The curves were obtained at a constant temperature of 1800°F in the order of increasing stress.

Figure 7.- Time-extension curves for a specimen of body 353. Rupture occurred within the gage length. Total time of test was 2141 hours and the maximum creep of 1.37 x 10^{-6} in. per in. per hour was observed before the "recovery curve" was obtained.

TEST F10-1 1500°F BODY 353

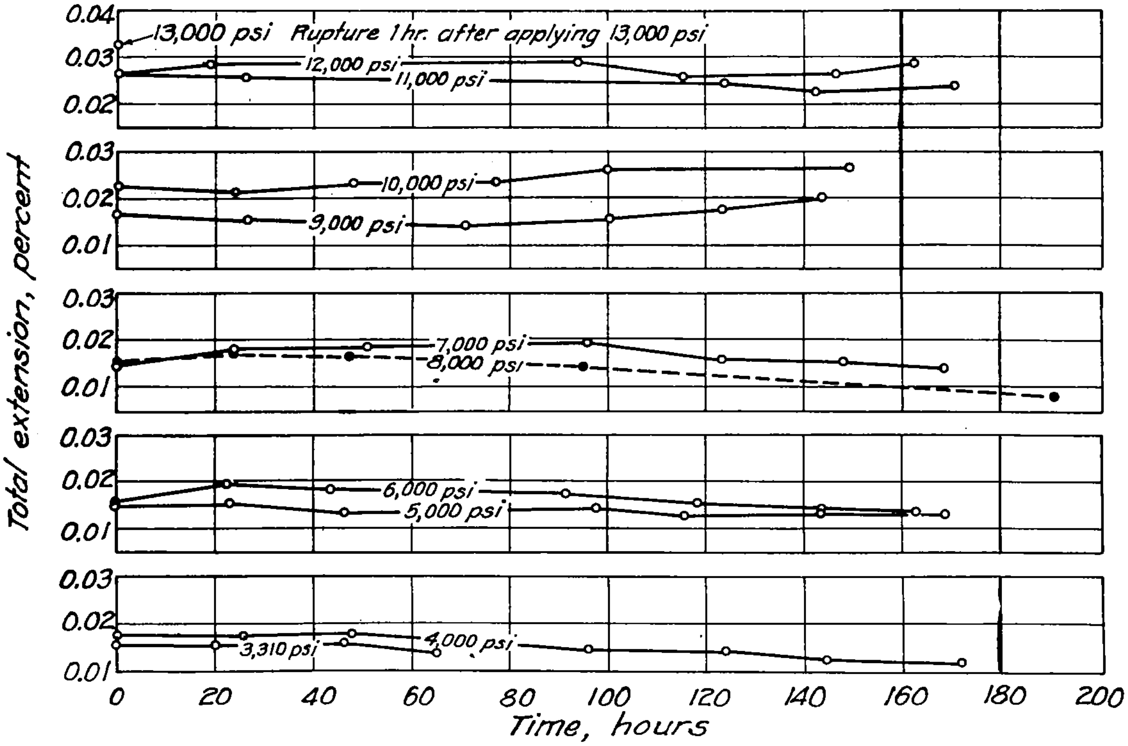


Figure 8.- Time-extension curves for a specimen of body 353 supporting various loads at 1500°F. Loads were applied in the order of increasing stress. Rupture occurred near the center of the gage length. Total time of test was 1554 hours and maximum creep rate was 0.26 x 10^{-6} in. per in. per hour, observed while under the 9000 and 10,000 psi stress.

TEST F10-2 1700°F BODY 353

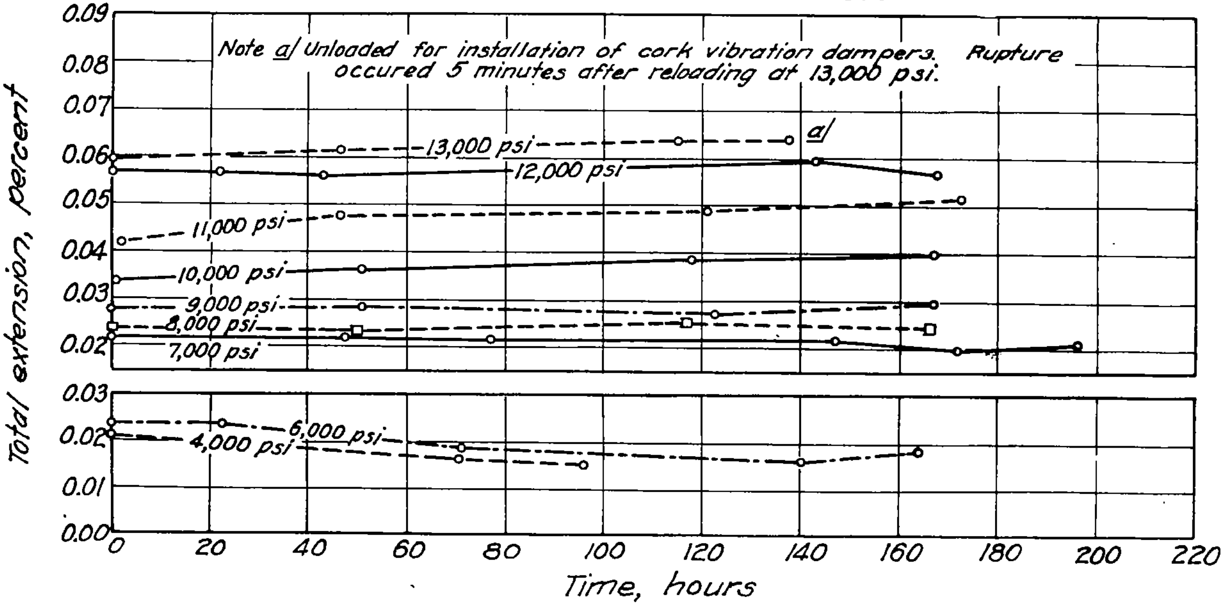
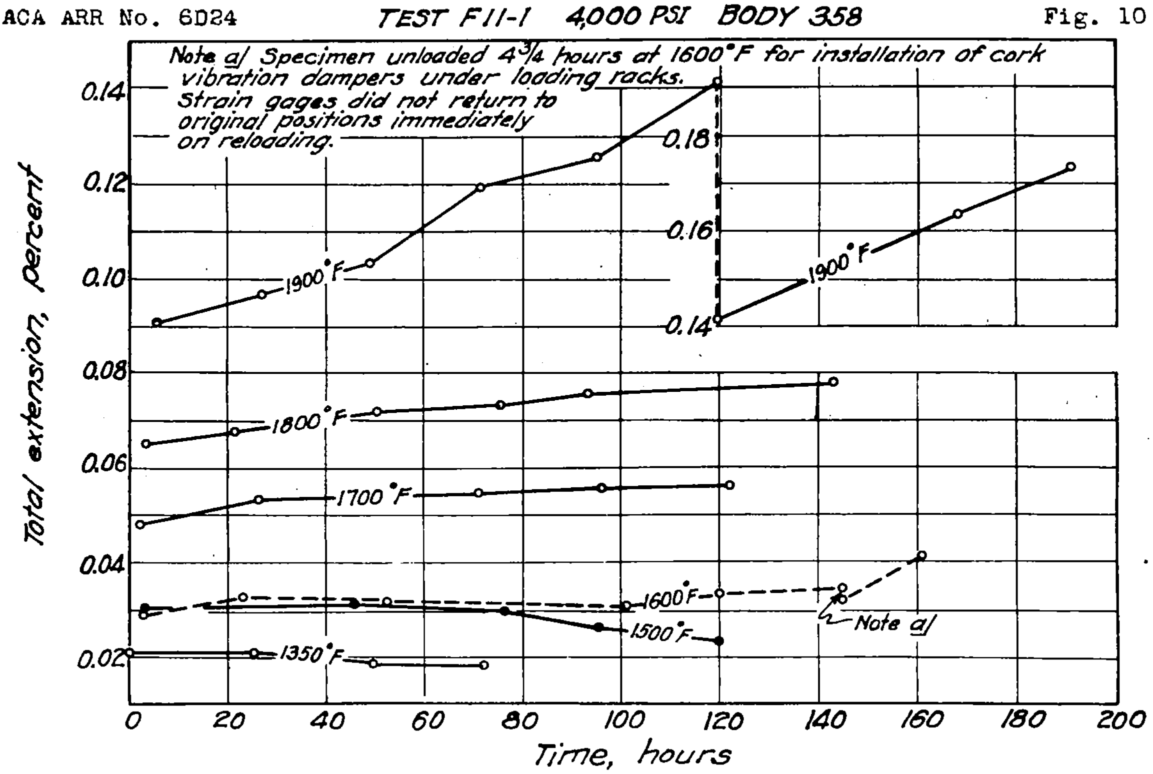
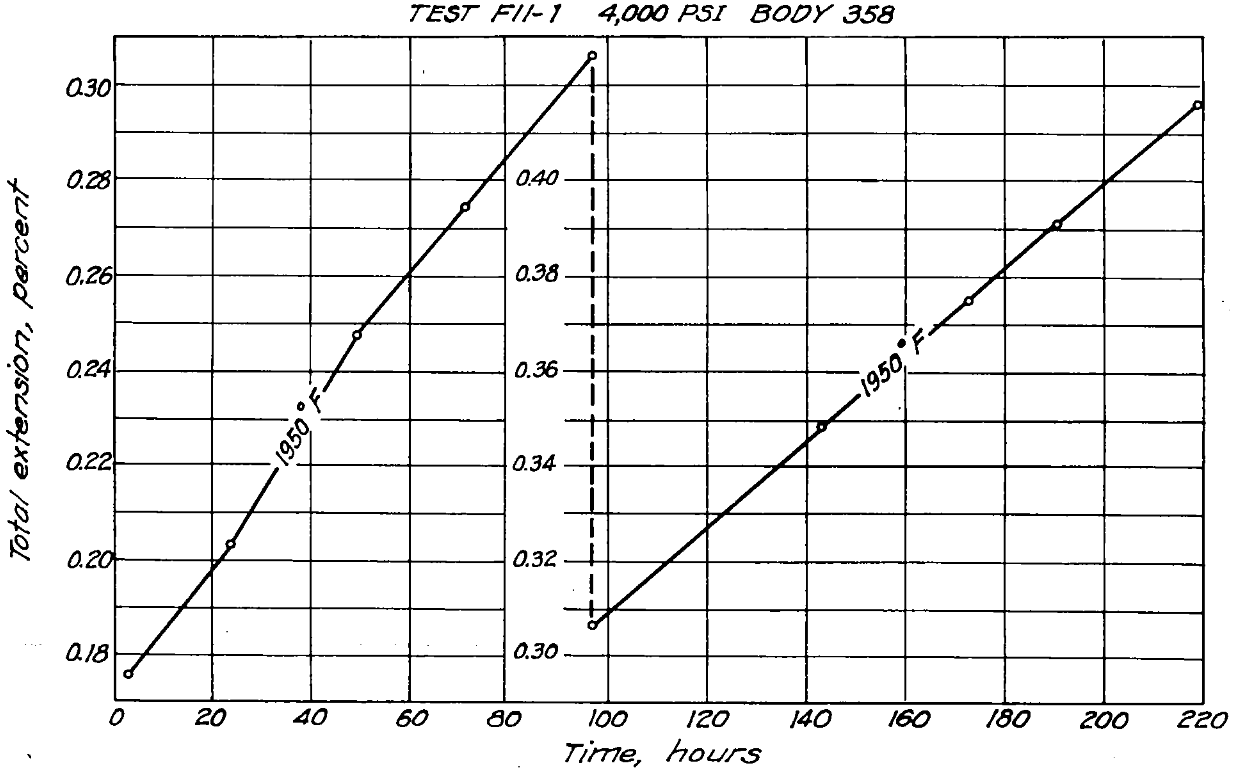


Figure 9.- Time-extension curves for a specimen of body 353 under stresses increased from 4000 to 13,000 psi at 1700°F. After 137-1/2 hours at 1700°F and 13,000 psi, the load was removed for 5 hours to install cork vibration dampers under loading racks. Rupture occurred, in the threaded portion of the specimen, 5 minutes after the load of 13,000 psi was reapplied. Total time of test was 1438 hours and a maximum creep of 0.56 x 10^{-6} in. per in. per hour was observed.

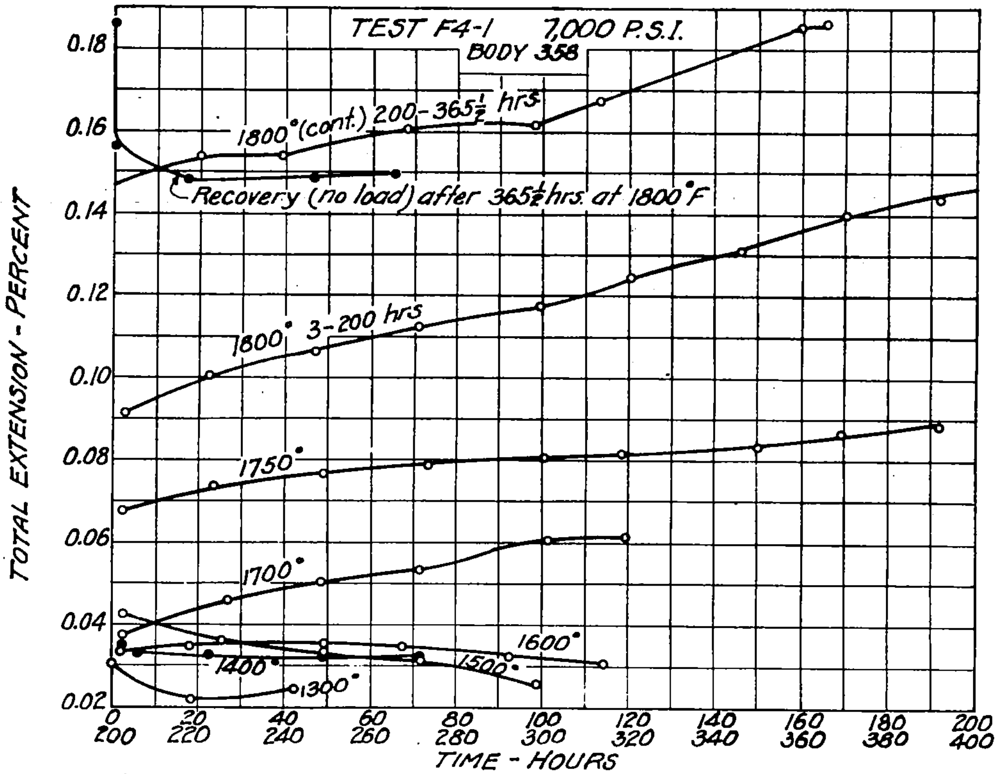


(a) Temperatures ranging from 1350 to 1900°F. The curves were obtained in the order of increasing temperatures.

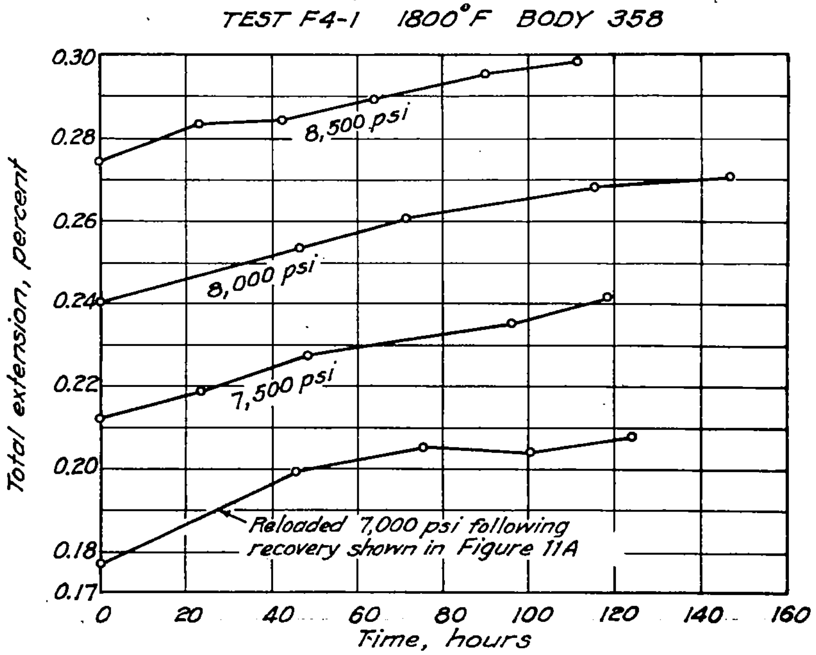


(b) Curve showing extension during 220 hours at 1950°F.

Figure 10.- Time-extension curves for a specimen of body 358 under a constant stress of 4000 psi. The test was discontinued before rupture and subsequently treated as shown in figure 13. Total time of test was 1023 hours and the maximum creep, 9.12×10^{-6} in. per in. per hour, occurred during the test at 1950°F.

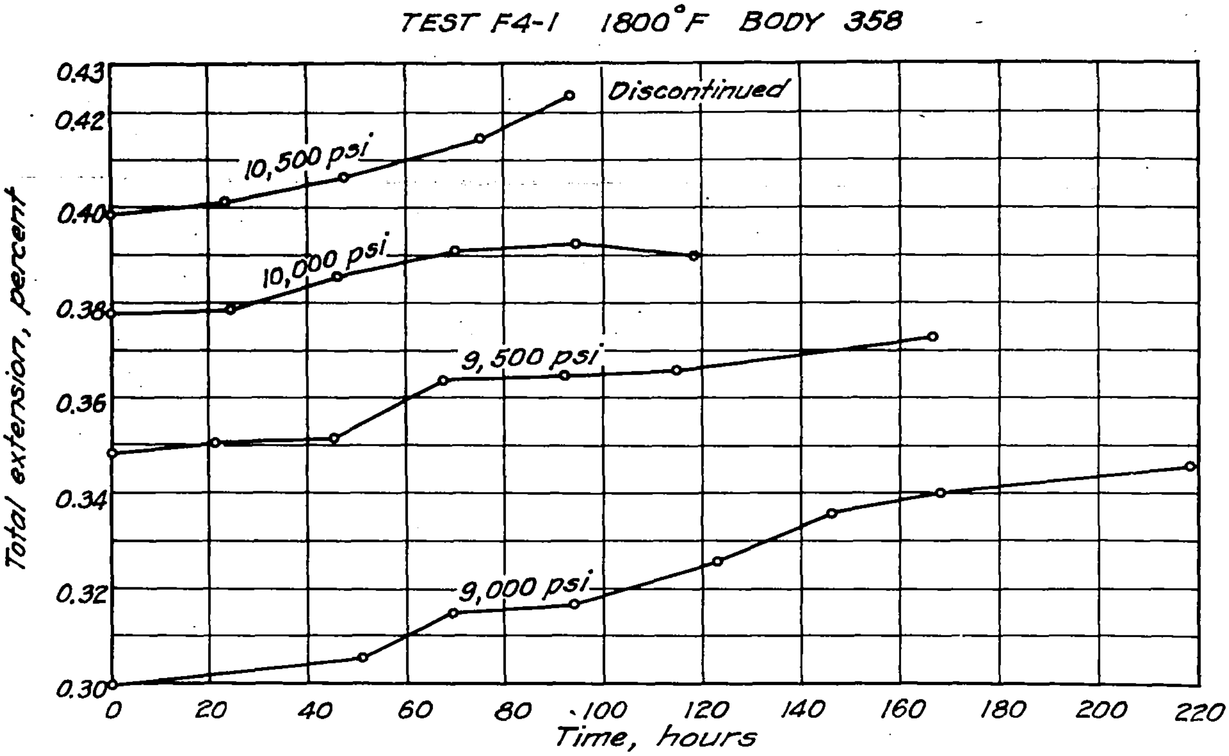


(a) Constant stress of 7000 psi in the order of increasing temperatures between 1300 and 1800°F. The recovery curve at no load and 1800°F was obtained after 365-1/2 hours at 1800°F and 7000 psi.



(b) Curves obtained at a constant temperature of 1800°F and increasing the stress in increments from 7000 to 8500 psi.

Figure 11 (a to c).- Time-extension curves for a specimen of body 358. The test was discontinued after 2238 hours when the thermocouples became unreliable. The specimen broke under load while cooling. The maximum creep (2.68×10^{-6} in. per in. per hour) was practically identical during the first holding at 1800°F and 7000 psi (figure 11a) and during the final holding at 1800°F and 10,500 psi.



(c) These curves were obtained at a constant temperature of 1800°F and continuing the increase in stress from 9000 to 10,500 psi.

Figure 11.- Concluded.

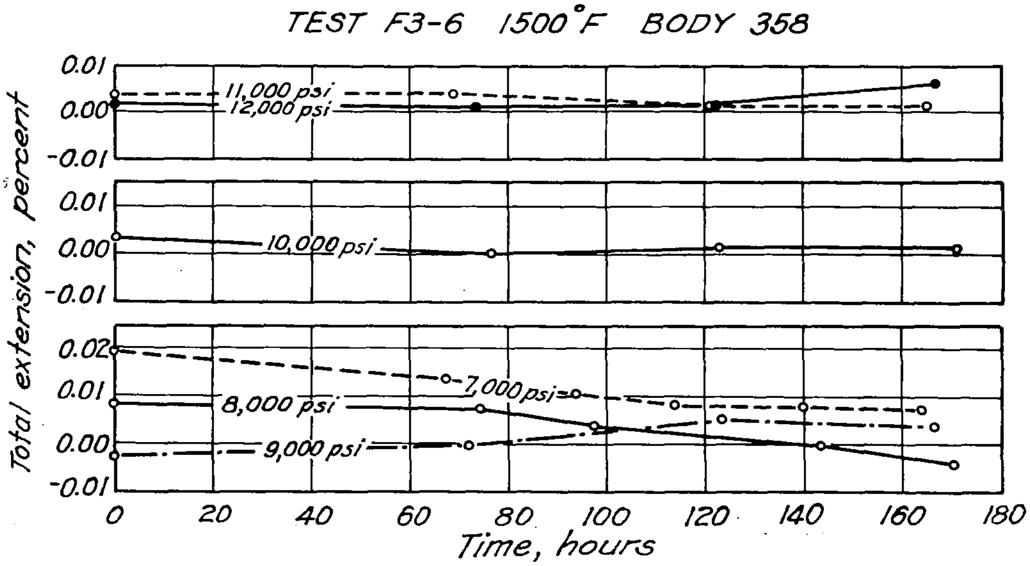


Figure 12.- Time-extension curves for a specimen of body 358 tested with various loads, in the order of increasing stress from 7000 to 13,000 psi at 1500°F. Rupture occurred in the bottom fillet of the specimen 10 minutes after applying 13,000 psi. Total time of test was 1104 hours and the maximum creep of 0.35 x 10^{-6} in. per in. per hour was observed while the stress was 9000 psi.

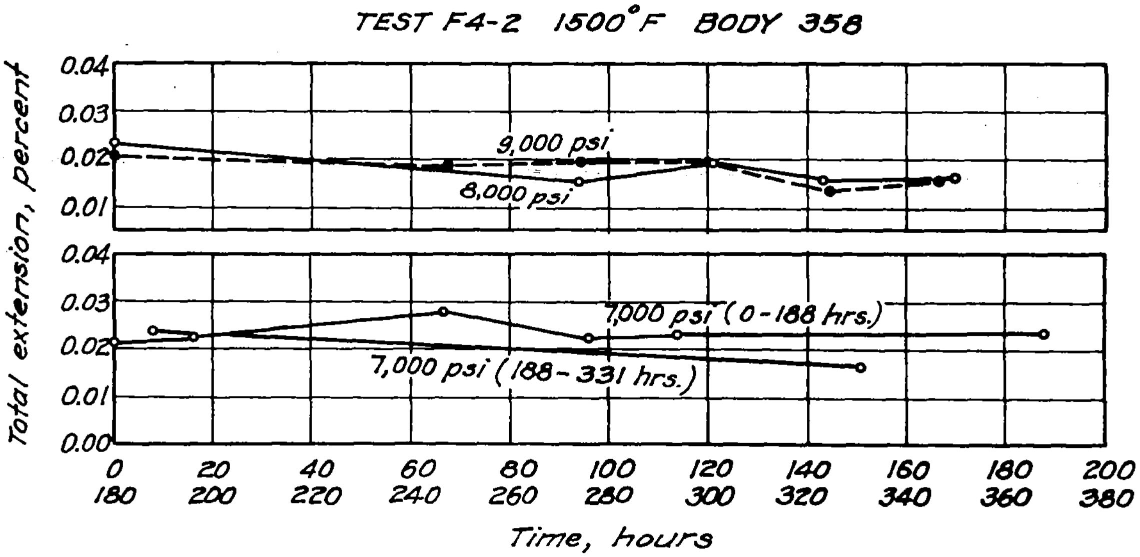


Figure 13.- Time-extension curves for a specimen of body 358 supporting stresses increased from 7000 to 10,000 psi at 1500°F. This specimen had been treated previously as shown in figure 10. Rupture occurred in the threaded portion of the specimen 5 minutes after stressing at 10,000 psi. Total time of test was 668 hours and the creep was negligible.

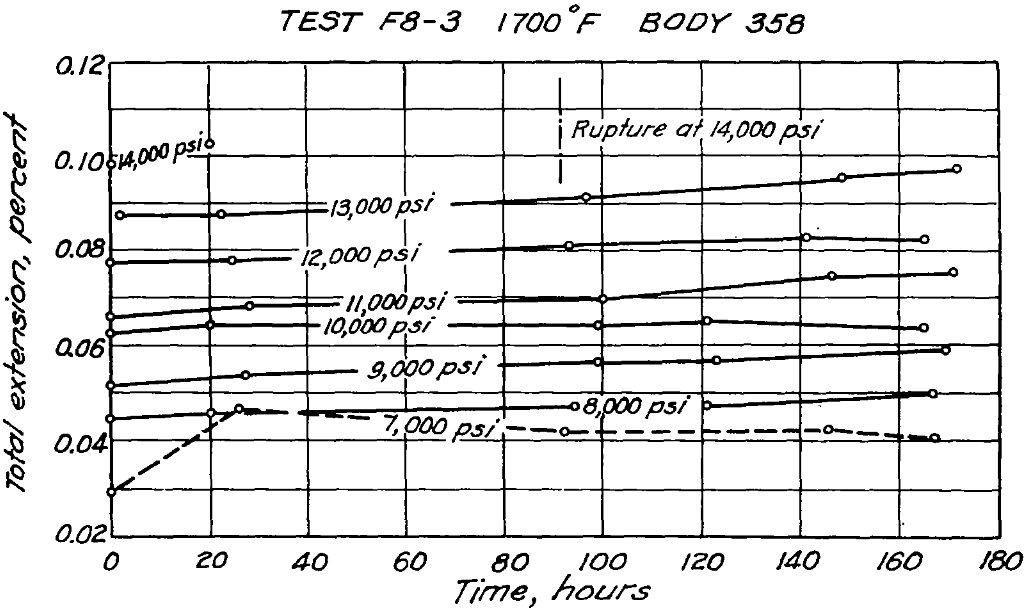
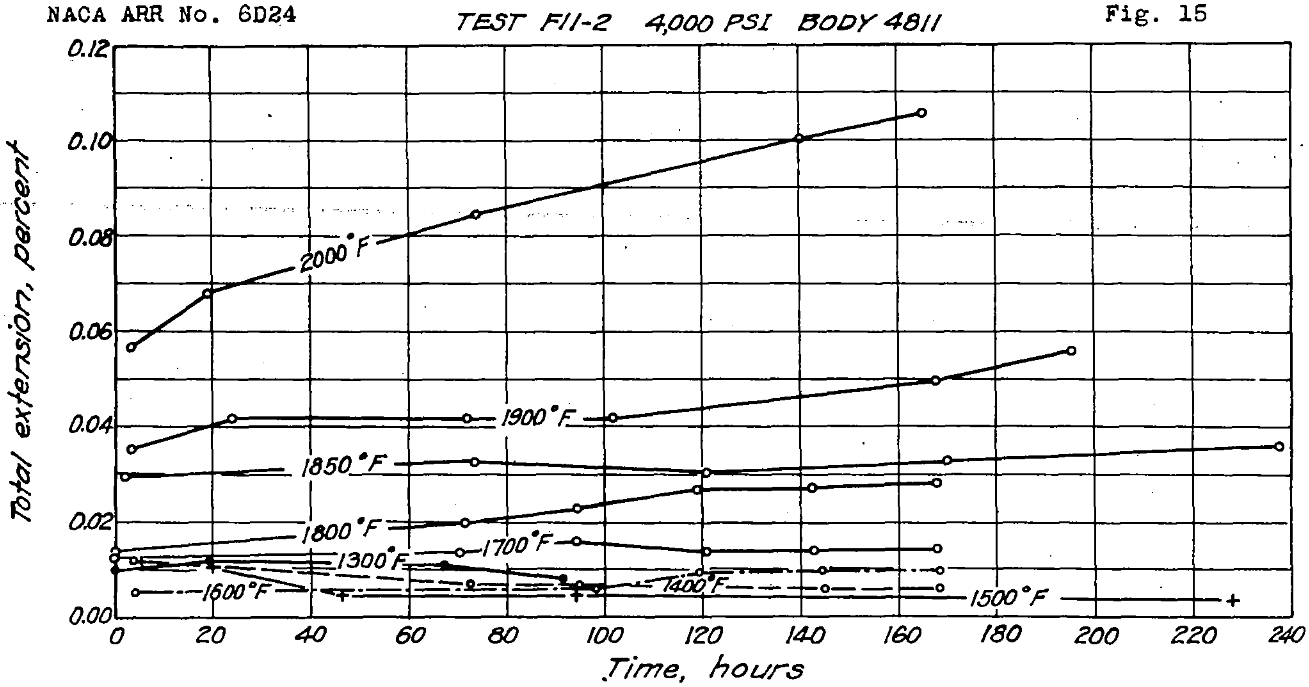
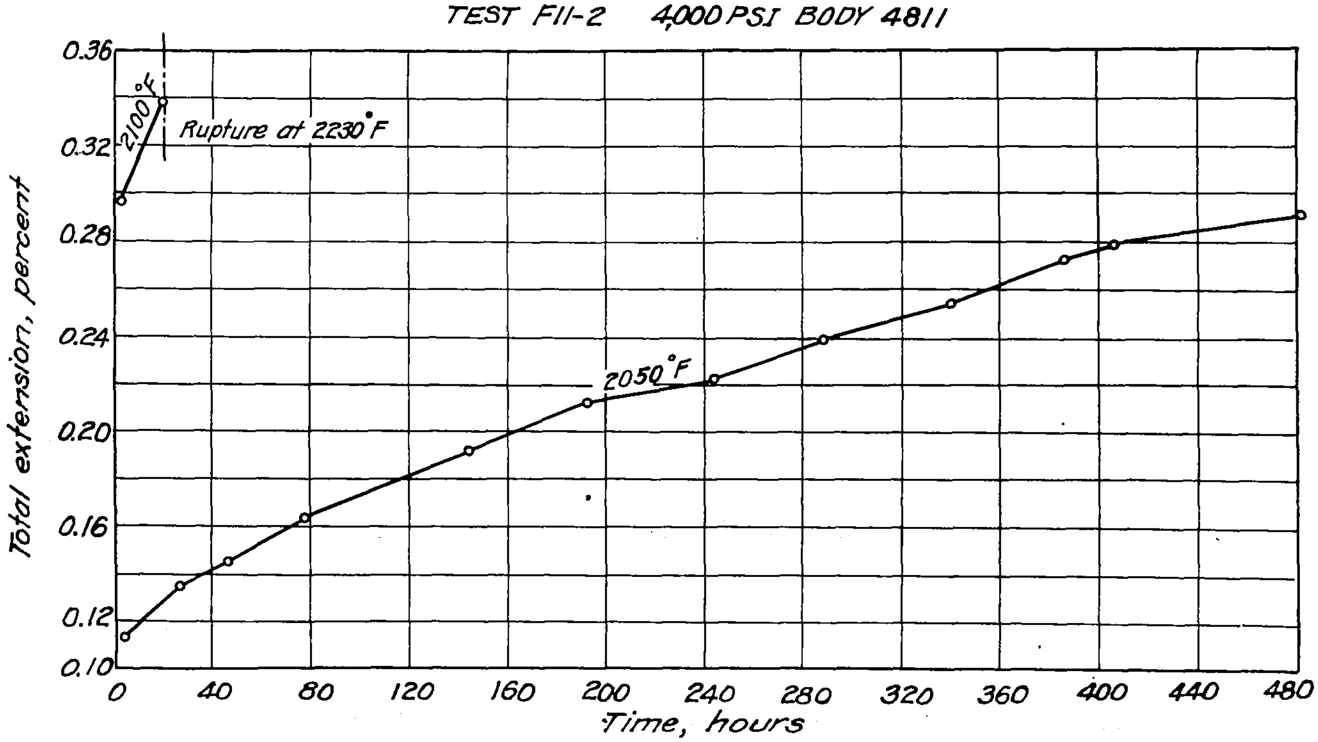


Figure 14.- Time-extension curves for a specimen of body 358 stressed at 7000 to 14,000 psi while being heated at 1700°F. The curves were obtained in the order of increasing stress, and rupture occurred in the gage length after 91-1/2 hours at 14,000 psi. Total time of test was 1268 hours. The maximum creep-rate was 2.08×10^{-6} in. per in. per hour with the stress at 14,000 psi.

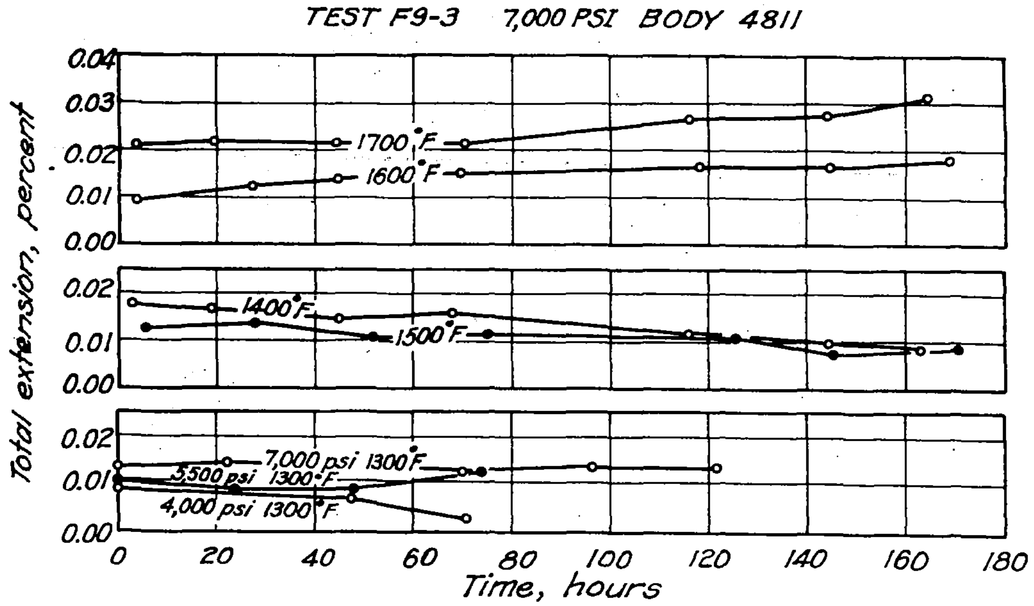


(a) The curves were obtained in the order of increasing temperatures from 1300 to 2000°F.

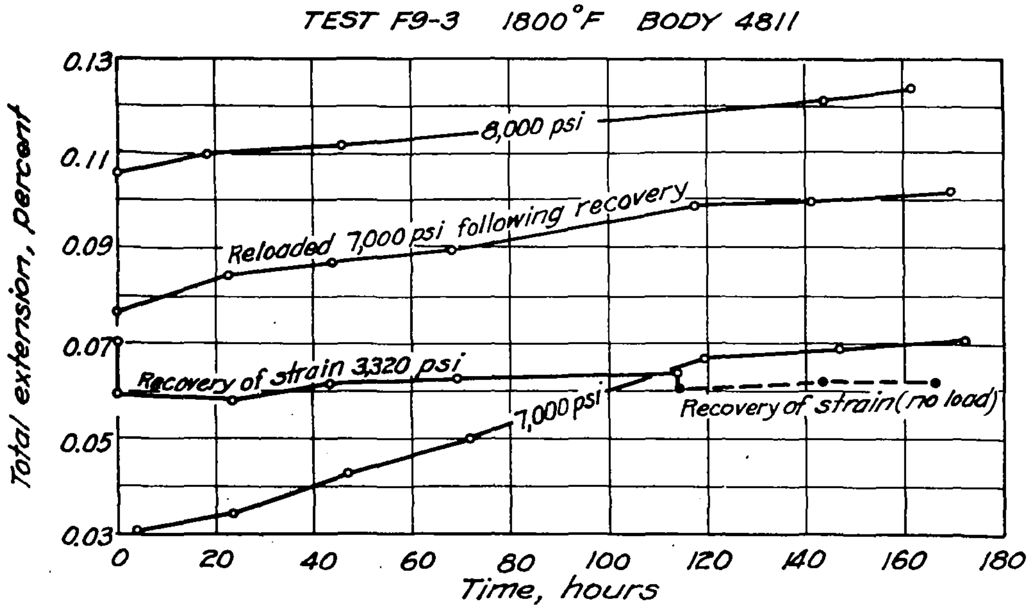


(b) The curves show length changes for temperatures of 2050 and 2100°F.

Figure 15.- Time-extension curves for a specimen of body 4811 at a constant stress of 4000 psi. Immediately after the last recorded extension measurement at 2100°F (19-1/2 hours), the temperature control instrument became inoperative. The temperature rose to about 2230°F and rupture occurred within the gage length of the specimen after 45 minutes. Total time of test was 2094 hours. The creep-rate at 2100°F was relatively very high (24.1×10^{-6} in. per in. per hour).

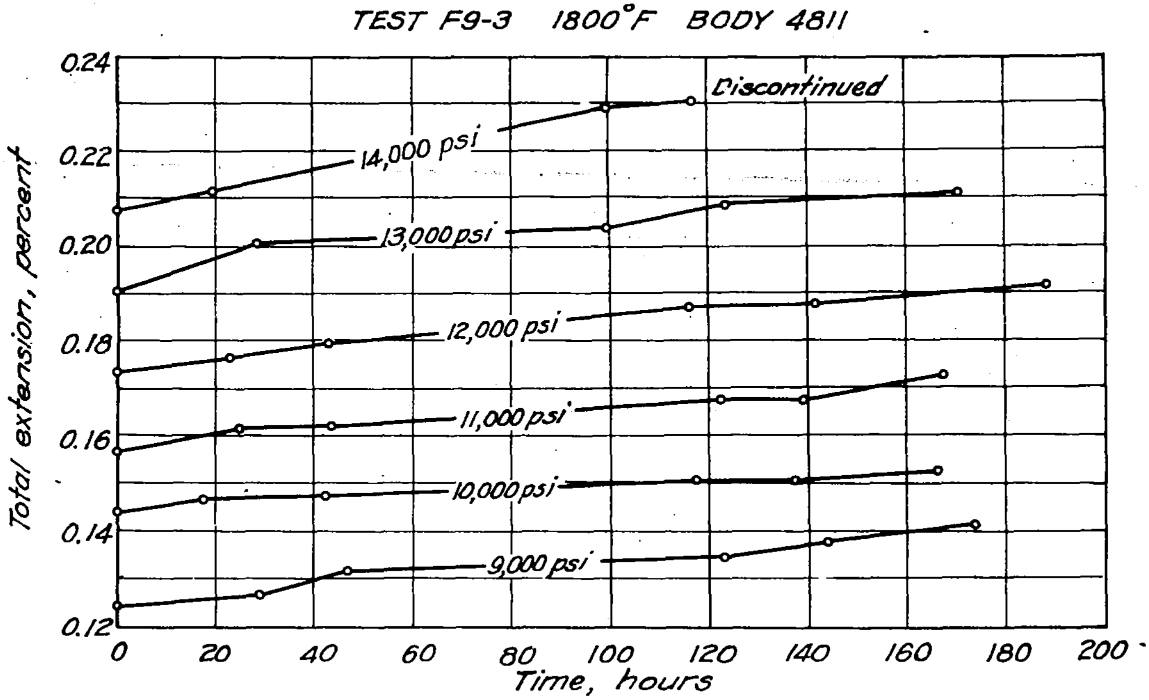


(a) Two preliminary stresses of 4000 and 5500 psi were applied at 1300°F The remaining curves are at a constant stress of 7000 psi and increasing temperature in 100°F increments.



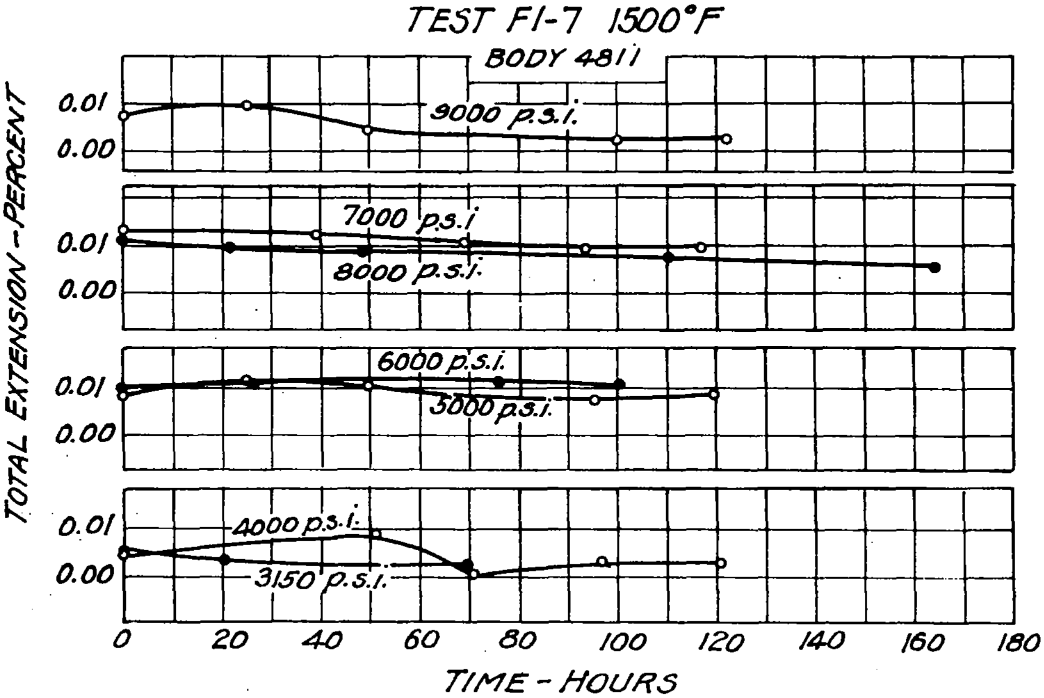
(b) These curves were obtained at a constant temperature of 1800°F and in the order of increasing stress. The recovery-of-strain curves at 3320 psi, and also at no load, were obtained between the two treatments at 7000 psi and 1800°F.

Figure 16 (a to c).- Time-extension curves for a specimen of body 4811.



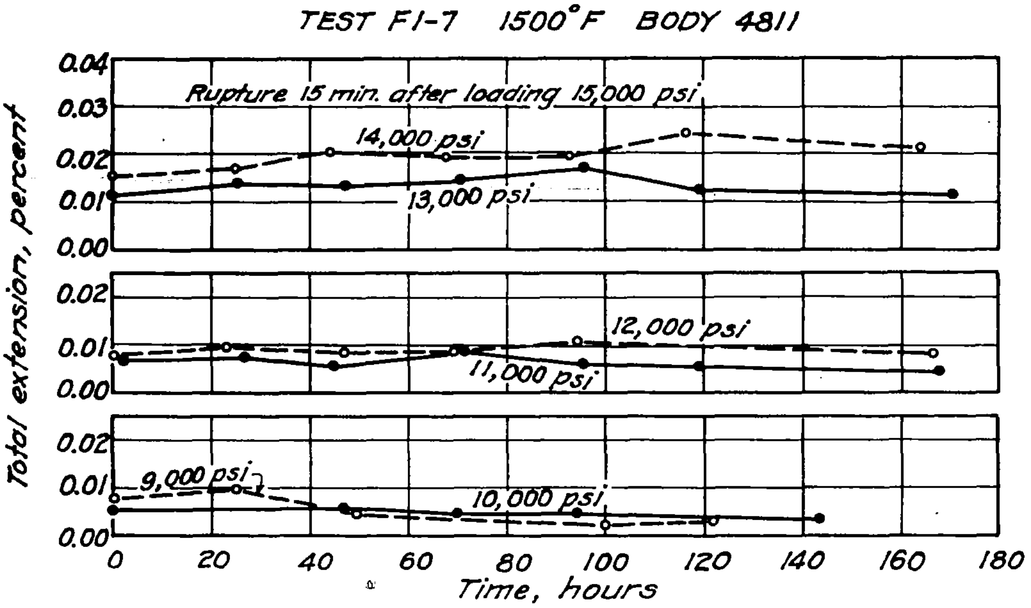
(c) These curves were obtained at a constant temperature of 1800°F in the order of increasing stress between 9000 and 14,000 psi. The test was discontinued when the base-metal thermocouples became unreliable after a total test time of 2589 hours. Maximum creep of 2.36×10^{-6} in. per in. per hour was observed during the initial holding at 7000 psi and 1800°F (figure 16b).

Figure 16.- Concluded.



(a) The loads were applied, in the order of increasing stress, from 3150 to 9000 psi.

Figure 17 (a,b).- Time-extension curves for a specimen of body 4811 supporting various loads at 1500°F.



(b) Curves for stress of 9000 to 14,000 psi. Rupture occurred at the fillet, outside of the gage length of the specimen, after 15 minutes under a stress of 15,000 psi. Total time of test was 1626 hours and the maximum creep-rate of 0.39 x 10^{-6} in. per in. per hour was obtained while the stress was 14,000 psi.

Figure 17.- Concluded.

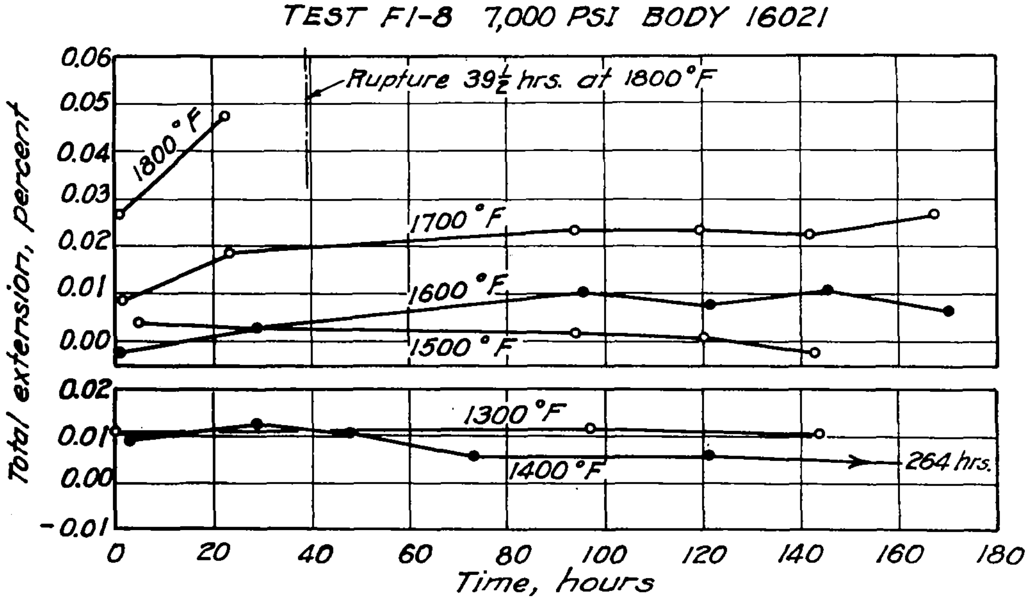


Figure 18.- Time-extension curves for a specimen of body 16021 at a constant stress of 7000 psi. The temperatures were increased from 1300 to 1800°F. Rupture occurred near the center of the gage length after 39-1/2 hours at 1800°F. Total time of test was 929 hours and the maximum creep-rate (9.53 x 10^{-6} in. per in. per hour) was recorded while the temperature was at 1800°F.

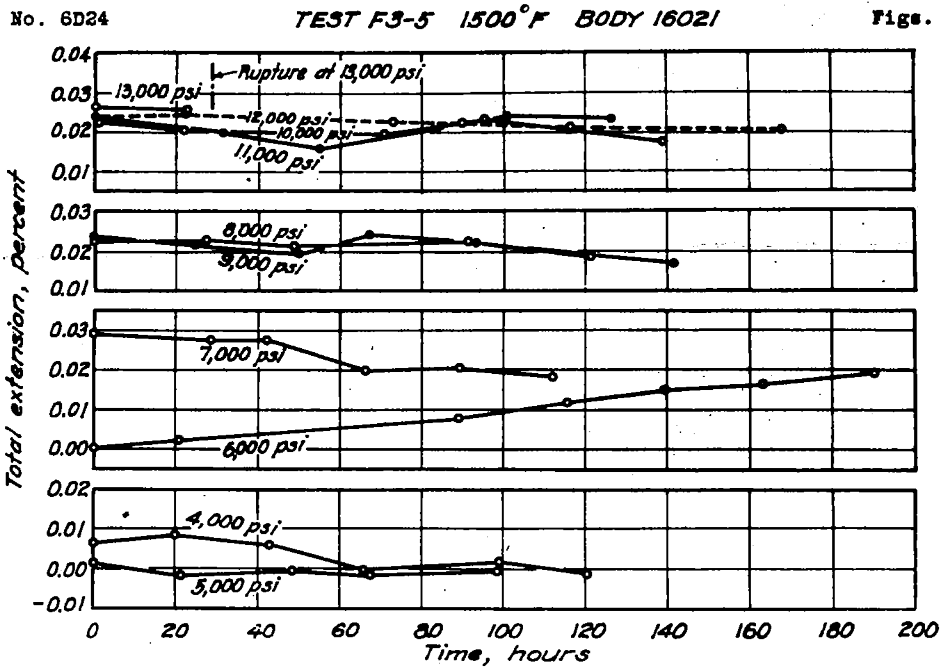


Figure 19.- Time-extension curves for a specimen of body 16021 supporting various loads at 1500°F. The curves were obtained in the order of increasing stress from 4000 to 13,000 psi. Rupture occurred in the threaded portion of the specimen after 29 hours at 13,000 psi. The creep-rate was maximum (0.01 x 10^{-6} in. per in. per hour) when the stress was 6000 psi.

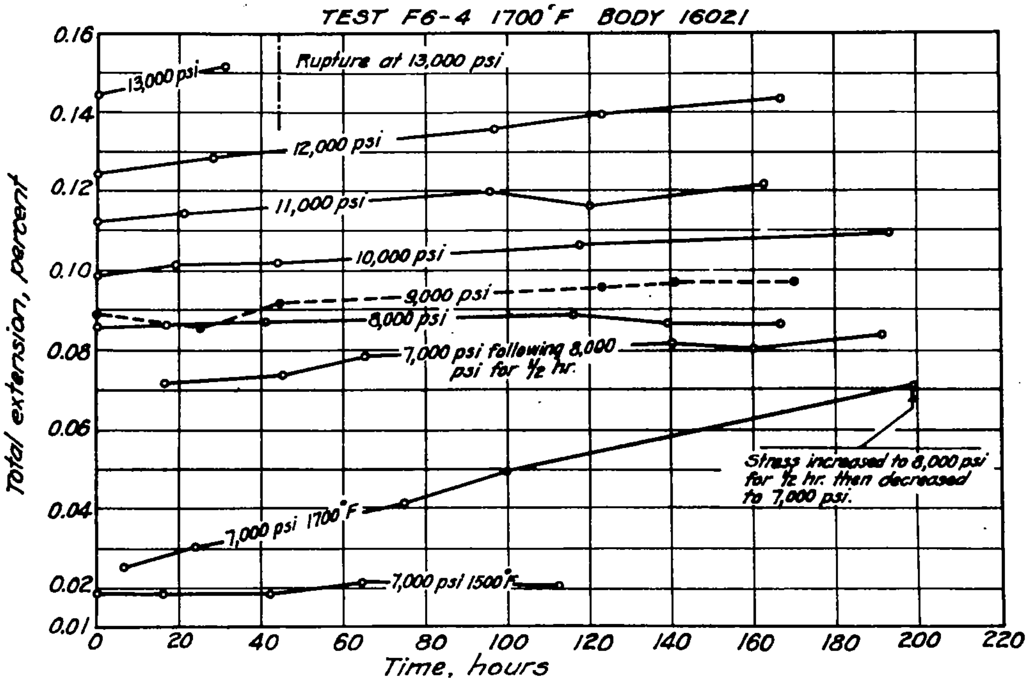


Figure 20.- Time-extension curves for a specimen of body 16021 supporting various loads at 1700°F after a preliminary test of 1500°F. The curves were obtained in the order of increasing stress. Note that a stress of 8000 psi was applied for 1/2 hour between the two tests at 7000 psi. The curves show the marked decrease in rate-of-extension at 7000 psi after this 8000 psi treatment. Rupture occurred in the gage length after 44-1/2 hours at 13,000 psi and 1700°F. Total time of test was 1406 hours and maximum creep (2.51 x 10^{-6} in. per in. per hour) occurred under 13,000 psi stress. However, the creep during the initial holding at 7000 psi was not much lower (2.36 x 10^{-6} in. per in. per hour).

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ABSTRACT

Investigation was made to determine the behavior of ceramic bodies in tension at high temperatures. Observations were made on six ceramic bodies covering the determination of their tensile strength and creep during prolonged stresses ranging from 3000 to 15,000 psi and at temperatures ranging from 1500° to 2230°F. Bending tests at 1800°F gave modulus of rupture values for commercial specimens which ranged from 4900 to 18,000 psi while the values of the bodies developed at the Bureau of Standards ranged from 6000 to 38,000 psi. Creep was not measurable.

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