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Materials Research

THE ELEVATED TEMPERATURE TENSILE AND CREEP-RUPTURE PROPERTIES OF THE FS-82 COLUMBIUM ALLOY

R. L. Jones

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ENGINEERING DEPARTMENT

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FOREWORD

This report describes the experimental work performed under REA 111-9107 during the period 1 January 1960 to 31 December 1960 and includes some work carried out during the period 1 January 1961 to 31 March 1961 under REA 111-9205. REA 111-9205 is an extension of the work that started 1 January 1960 under REA 111-9107 entitled, "The Elevated Temperature Base Metal and Joint Properties of Refractory Metals." The refractory metals under consideration are columbium and molybdenum base alloys.

The alloy reported upon herein is the FS-82 columbium alloy (Cb+33Ta+0.7Zr). The effects of temperature on the tensile and creep-rupture properties were investigated. Since fabrication by welding can lead to considerable savings in weight and cost, the elevated temperature tensile and creep-rupture properties of fusion weldments were also investigated. Both electron beam and TIG fusion welds were included in the study.

Quite often the elevated temperature properties of metallic materials are found to be sensitive to thermal exposure time and strain rate. Thus, the effects of preheat time on tensile properties was evaluated; strain rate sensitivity will be investigated later.

Metallographic examination and microhardness surveys were performed prior to and after testing and the results were correlated with tensile and creep-rupture properties.

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THE ELEVATED TEMPERATURE TENSILE AND CREEP-RUPTURE PROPERTIES OF THE FS-82 COLUMBIUM ALLOY

R. L. Jones

ABSTRACT

Base metal and fusion weld tensile properties of the FS-82 columbium alloy (Cb+33Ta+0.7Zr) have been determined from ambient temperature to 2600°F. Base metal tensile properties were determined using two different heat-up periods prior to test: 30 seconds and 30 minutes. In general, the tensile properties determined using the short heat-up period were considerably lower than those found using the long heat-up period at temperatures below 1800°F, but much higher at higher temperatures. This behavior was related to strain aging at temperatures below 1800°F and recovery and/or recrystallization at temperatures above 1800°F. Creep-rupture properties of the base metal and fusion weldments were determined at 1800°, 2000° and 2200°F. Extrapolation techniques were employed to extend the data to cover temperatures, times and stresses not investigated in the experimental program. The creep behavior of the base metal was analyzed and the activation energy for creep was found to be 121,300 cal/mol. This was found to be in excellent agreement with the activation energy for self-diffusion of columbium (105,000 ± 3000 cal/mol) and/or the activation energy for self-diffusion of tantalum (110,000 cal/mol). This data strongly suggests that creep of the FS-82 columbium alloy is diffusion controlled above about 1800°F (0.45 Tm).

Electron beam fusion welds of FS-82 were found very ductile and free from contamination. Tungsten-arc shielded gas fusion welds (TIG welds) were contaminated slightly by gas pickup during welding but retained considerable ductility. Tensile weld efficiency of the TIG weldments decreased from 94 percent at ambient temperature to about 75 percent at 1800°F. Electron beam weld efficiencies were considerably lower; decreasing from 64 percent at ambient temperature to 54 percent at 1800°F. The rupture life of the fusion welds appear to be higher than the base metal rupture life at temperatures over 2000°F, but appears to be lower at lower temperatures. However, more data is required before this conclusion can be considered valid.

INTRODUCTION

Refractory metals have been under development for a great many years in the electronics and chemical processing industry. In general, however, the refractory metals had not been developed to the point that they could be considered for use for massive, structural applications until quite recently. A few years ago extensive programs were initiated on the metallurgy of molybdenum. More recently, a series of programs was started on columbium, and still more recently on tungsten and tantalum.

The airframe industry and the rocket motor industry constitute the major users of refractory metals at the present time. Re-entry glide vehicles are currently being designed to operate under conditions for which refractory metals are required. Sheet products are applied to the leading edge sections, and to the positions back on the underside of the lifting surface as well as to the vector controls. Rocket nozzles are also being fabricated from refractory metals. The most difficult mill product to make at present is sheet, though billets and forged parts cannot be considered to be simple production items. A government-sponsored refractory metals sheet rolling program was initiated recently in an attempt to hasten the development of quality refractory metal alloy sheet products.

Most advanced applications of refractory metals favor the use of sheet. The gages for structural applications range from 0.010 to about 0.100 in. Conventional sheet sizes, up to 36 in. x 96 in., are of interest. Quality requirements are high and gage tolerances must be somewhat better than ± 10 percent if drastic weight penalties are to be avoided.

There are two schools of thought with respect to the use of refractory metals on re-entry vehicles. It is not the purpose at present to discuss the various advantages and disadvantages of each; but only to mention the ideas. The "hot structure" school believes that the refractory metals should support the aerodynamic and thermal stresses developed during re-entry; while the "heat-shield" school believes that the function of the refractory metal should be as a heat shield backed by insulation, in order to permit the use of an efficient, low-temperature structural member. The design stresses expected for either application are not high, but in the hot-structure design use could be made of improved strength at elevated temperatures. Strength is not a significant factor in the heat-shield design, but methods of attachment of the skin to the insulation must be solved. This may necessitate a modest strength requirement.

Methods of construction of hot structures include conventional sheet and stringer. Corrugated sandwich construction is being considered for many of the skins. Weldability is very desirable as a weight-saving measure, although not mandatory, particularly in view of the thermal stresses that may make it impossible to have a large continuous structure.

The temperatures expected for hot-structure design range from about 2000° to 4000°F, depending on the trajectory during re-entry. Small errors in the re-entry angle of inclination will cause large variations in temperature. In heat-shield application, the refractory metal shield may operate at 4000°F or above. Coating systems to protect against oxidation and other corrosive environments may well prove to be the limiting factor in the use of these refractory metals, since strength requirements are moderate and well within the existing state of the art. Another factor of importance is the emissivity of the exposed coated surface. Emissivity should be at least 0.8 or above to provide efficient radiative cooling.

Efforts to develop refractory metal alloys have been very active in the past few years. Molybdenum is farthest along in development, but the rate of progress on columbium is so fast that it is expected to catch up with molybdenum within the next few years. The competition between molybdenum and columbium for the leading edge application is very spirited. The advantages of one appear to be offset by those of the other. Columbium and some of its alloys have the advantage of low-temperature ductility, relatively high oxidation resistance and weldability. However, their high temperature strength is considerably lower than the strong molybdenum alloys. Tungsten and tantalum are not competitive with lower-density molybdenum and columbium as sheet for the lower leading-edge temperatures (i.e., below 2500°F) now of major consideration, and activity is directed toward their use as forgings for solid-propellant rocket motors or for sheet application in rocket motors, such as flame shields in jetavators or flame skirts in liquid-propellant rockets.

Several excellent surveys of the mechanical properties, and forming processes for refractory metals can be found in the literature (1, 2, 3, 4). For purposes of comparison a few of the more important properties of the refractory metals will be considered here. An excellent survey of the machining, cutting, forming, welding, etc. properties of unalloyed molybdenum, columbium, tungsten and tantalum is given in reference 1 and is reproduced in Table I of this report. These properties are generally similar for the various alloys of the refractory metals with some exceptions.

Tungsten and tantalum should be considered for use as sheet for re-entry leading edge applications when the anticipated temperatures exceed 2500°F. Because of tantalum's superior sheet producibility, low-temperature ductility, weldability and formability (see Table I), it is probably the leading candidate.

Below 2500°F, where most of the present applications arise, tungsten and tantalum are not competitive with molybdenum or columbium and the choice is between these two materials. Tensile strength vs. temperature for some molybdenum and columbium base alloys are shown in Figure 1. According to this data, molybdenum alloys are the obvious choice since they are considerably stronger than the columbium base alloys. In most cases molybdenum alloys look better than weaker columbium alloys even on a density compensated basis. However, some of the stronger columbium alloys (F-48, for example) do compare favorably with the molybdenum base alloys on a density compensated basis. There are, however, many disadvantages associated with the use of molybdenum and its alloys (see Table I). Molybdenum and its alloys are brittle at room temperature; are difficult to weld; form a highly volatile oxide and oxidize catastrophically; and are extremely difficult to form. Until some of the major drawbacks are alleviated it appears wise to accept a slight weight penalty to gain high reliability, especially when the refractory metal is to be used on a manned recoverable-reusable vehicle. Pure columbium (see Table I) and some of its alloys do not suffer from any of these disadvantages. Furthermore, the appropriate use of welding in the construction of a columbium leading edge may make it competitive with the molybdenum leading edge, since mechanical joining (and its attendant weight penalty) is all but mandatory with molybdenum alloys.

The compositions and consolidation method of some of the alloys of columbium are shown as follows:

Alloy Composition	Consolidation Method
Cb-15W-5Mo-1Zr-0.05C(F48)	Arc-cast
Cb-15W-5Mo-1Zr-5Ti-0.05C(F-50)	Arc-cast
Cb-1Zr	Arc-cast
Cb-10Mo-10Ti(D31)	Arc-cast
Cb-20W-10Ti-6Mo(D41)	Arc-cast
Cb-15W-20Ta	Electron Beam Melt
Cb-20W-20Ta	Electron Beam Melt
Cb-33Ta-1Zr(FS-82)	Arc-cast

Of the alloys listed only two can be considered as being commercially available in reasonable sizes and shapes. These are the Du Pont alloy (D31) and the Fansteel alloy (FS-82). Although the Du Pont alloy is slightly stronger than the Fansteel alloy (see Figure 1) it is more difficult to weld and weldments are brittle. An alloy which can be readily welded to give sound ductile welds is necessary if this joining technique is to be used to secure reasonable weight savings with high reliability. Consequently, the Fansteel FS-82 alloy appears to be the best selection of a columbium alloy when all factors are taken into consideration.

If designs are to be made using refractory alloys such as Fansteel FS-82, mechanical and physical properties as a function of temperature are needed. Furthermore, the mechanical properties of weld joints are required for design of those components requiring construction by welding. Consequently, the primary purpose of the present program is to establish sufficient data to allow at least preliminary designs to be made using one of the refractory alloys, FS-82. To obtain this goal, properties of FS-82 fusion welded by various techniques will be established in order to select the most satisfactory welding process. Both tensile and creep-rupture properties of the base metal and of fusion weldments will be determined as a function of temperature.

LITERATURE SURVEY

Physical and Mechanical Properties

Some of the physical properties of the FS-82 alloy are given in Table II. As can be seen, very little actual data has been generated on this alloy and at present such physical constants as thermal expansion coefficients, specific heat and thermal conductivity must be estimated from data on pure columbium. The density is relatively high because of the high percentage of tantalum in the alloy.

Mechanical properties of the FS-82 alloy are presented in Tables III and IV. Data is sparse and scattered. Most available data covers tensile properties since these are readily obtained. As shown in the table, the actual values vary considerably from heat to heat and from investigator to investigator. This data represents almost all the mechanical property data generated on this alloy. Creep-rupture properties and properties of weldments and mechanical joints are particularly scarce. Ductility at room and elevated temperatures is good and the ductile to brittle transition temperature is below -70°F, but the actual temperature has not been definitely established.

A minimum in the tensile elongation occurs as the temperature is increased (see Table III). For one set of data the minimum occurs at about 1600°F, whereas it occurs at about 1000°F in the second set of data. A clear explanation of the factor or factors causing the decrease in tensile elongation has not been offered. However, it is possible that strain aging is a contributing factor.

Strain aging has been found in pure columbium in the temperature range from 300° to 1400°F and has been attributed to the presence of oxygen, nitrogen, hydrogen and/or carbon in solid solution (10, 11, 16). It is quite possible that strain aging in alloyed columbium may take place at higher temperatures, as has been found in other alloy systems, and thus account for the ductility minimum observed in the Cb-33Ta-.7Zr alloy.

Notched properties of the Cb-33Ta-.7Zr alloy at room temperature are given in Table IV. The material appears to be relatively insensitive to notches, and the notch/unnotch tensile ratio is about 1.0.

Joining

Columbium, in contrast to molybdenum, has excellent weldability. A wealth of knowledge has been accumulated on both resistance and inert arc fusion welding of unalloyed columbium (12). A lesser amount of information is available on the ductile columbium alloys such as the Cb-33Ta-.7Zr alloy.

Typical butt and "T" type fusion joints have been prepared from the Cb-33Ta-.7Zr alloy by several investigators. Generally the welds are made under gas atmosphere in an enclosed chamber. Some work has been carried out using backup and trailing gas shields only. Evaluation by bend tests show high ductility and little contamination by gaseous pickup. However, the strength of these joints has not been adequately evaluated. One test result (8) showed a room temperature joint efficiency of 92 percent for fusion welds. Although the fusion welds, as-welded, are generally quite ductile, there is some evidence that subsequent heat treatment at 1900°F causes the welds to become brittle (9). This has been attributed to precipitation of oxides or nitrides during the subsequent heat treatment.

Spot welding of the Cb-33Ta-.7Zr alloy has been carried out successfully. Spot-welding schedules and some tensile shear strength values are given in Table V. The spot welds are quite ductile.

Brazing has not been nearly as successful as welding. Palladium, nickel and chromium have been used as brazing alloys for the Cb-33Ta-.7Zr alloy without success. These alloys cause severe erosion of the base metal and produce a brittle joint. So far, only titanium has produced a strong ductile joint with little erosion of the base metal. Properties of the titanium joints have not been reported.

Coating

Although the present program does not deal with coatings, it is appropriate to review the state of the art of coatings for the Cb-33Ta-.7Zr alloy since they are most important in the final application of the material.

Coating development has progressed rapidly within the last two years and is due mainly to the work carried out at the General Electric Company (8, 9). A summary of the coatings and coating techniques employed by General Electric and some other companies is given in Table VI. The most promising coating techniques so far evaluated are chromium electroplate and aluminizing by hot drip or slurry dip. The chromium plate (2 mils thick) has been found to give protection against oxidation to the base metal for 1 hour at 2500°F. The aluminized coating appears a little better and gives protection for at least 2 hours at 2500°F. It should be recognized that the oxidation tests have been carried out under static conditions. Consequently, the coatings require further evaluation under conditions simulating re-entry conditions. Some preliminary tests of coated specimens under load show these coatings to be less protective, apparently because of the development of cracks through the coating.

EXPERIMENTAL PROCEDURES

Materials

One heat of the Cb-33Ta-.7Zr alloy (FS-82) sheet (.040 gage) was obtained from the Fansteel Metallurgical Company. According to the manufacturer, it was supplied in the stress relieved condition (1 hour at 1900°F). All testing was carried out on as-received material. Test coupon configurations for the testing described in the following sections are shown in Figures 1, 2 and 3 in the Appendix.

Tensile Testing

Some tensile tests were performed in the high heating rate, high strain rate testing unit at Convair-Pomona. The samples were heated by self-resistance and tests were carried out in an helium atmosphere. The strain rate to yield was between 0.003 and 0.005 in./in./minute and after yield it was increased to 0.02 - 0.04 in./in./minute. Because the samples were heated by self-resistance, elongation values are not reliable and were not obtained.

To obtain further mechanical property data, especially elongation values, additional tensile testing was carried out using the Instron testing machine. Testing was performed in a vacuum. Only ultimate tensile strength and tensile elongation were obtained from these tests because strain measurements were not possible with the present vacuum testing chamber. Crosshead speed was maintained at 0.05 in./min. during the test. This crosshead speed produced a strain rate of 0.004 in./in./min. up to about yield. A vacuum of 5×10^{-4} mm Hg was maintained throughout the test. Most tensile tests were sectioned after testing for structural examination and microhardness testing.

Creep-Rupture Testing

Creep-Rupture tests at temperatures up to 2200°F were carried out in a vacuum of 5×10^{-4} mm Hg or better. An extensometer system could not be attached to the specimen and elongation measurements were made by measuring the movement of the pull rods extending out of the vacuum chamber. Thus, it was not possible to obtain accurate measurements of strain on the reduced section of the test coupons.

However, by carefully measuring the elongation of the test sample and comparing it to the over-all elongation as measured from the pull rods it was possible to establish the following:

1. No measurable creep elongation occurred in the Mo-1/2 Ti alloy pull rods or grips.

2. An average gage length of 3.25 in. represented the length over which the majority of the creep deformation occurred.

Consequently, the creep rate and creep deformation data presented in this paper was derived from the elongation measurements taken from the pull rod system using an average gage length of 3.25 in. to obtain strain.

The creep-rupture specimens were sectioned for structural examination and microhardness testing after the creep-rupture test was completed.

Fusion Welding

Butt welds of the Cb-33Ta-.7Zr alloy sheet were made without filler wire by the TIG process. The welding equipment and welding schedule are as follows:

1. Butt weld columbium to columbium (0.040 to 0.040 gage)

2. Welding gas, high purity helium (CW) dew point < -80

Equipment: Miller a-c/d-c rectifier type welding machine
Longitudinal welding fixture, straight-line weld
Copper backup bar
Copper hold-down shoes
Trailing shield

Airco welding head

3/32 in., 2% thoriated tungsten

No. 10 copper cut

Welding schedule: 150 amps, 12 volts

Welding speed 20 in. per min.

Torch gas flow 45 CFM

Backup gas flow 11 CFM

Trailing shield gas flow 20 CFM

CW helium, dew point - < 80 or better

A few electron beam welds were prepared at 10,000 volts and 0.03 amps at a vacuum of 0.05 microns. However, the electron gun design was not completely suitable for welding columbium and a rather large weld resulted.

The ductility of both type of welds was evaluated by bend tests around a 1T radius. Welds were sectioned for metallographic examination and microhardness testing.

RESULTS AND DISCUSSION

Base Metal Properties

Tensile Properties

The microstructure of the Cb-33Ta-.7Zr alloy in the stress-relieved condition is shown in Figure 2.

The tensile properties of this heat of Cb-33Ta-.7Zr alloy are given in Table VII. The results of a microhardness survey across the thickness of the sample after tensile testing are also presented. Even though a helium atmosphere was used, slight surface hardening occurred on many of the tests. However, as can be seen, this slight surface contamination did not markedly affect the tensile properties. When contamination was severe, as in the second test at 2400°F, some definite effect was noted.

This data is plotted in Figure 3. One set of tensile data from Table III (see reference 9) is included for comparison. Except for the room temperature properties, there is a considerable difference in tensile and yield strength at elevated temperatures. It is possible that this difference is due to heat to heat variation but it is more likely due to differences in testing procedure.

In the present study the sample was brought to temperature within 30 seconds and testing started immediately, whereas testing carried out in reference 9 was started after a 15-minute soak at temperature. Time to reach temperature was not mentioned. The higher properties established at the lower temperature could possibly be accounted for by strain aging that occurs during the heating and holding period. The considerably lower properties reported at temperatures in excess of 1600°F probably result from structural changes that occur with time at the elevated temperatures. For example, recrystallization is 50 percent complete in 1 hour at 2200°F. Some recrystallization must have occurred in the tests performed in reference 9 at temperatures over 2200°F. However, very little recrystallization was observed in the high-heating rate tensile samples tested in this program even at the highest testing temperature. In this test, maximum time at elevated temperatures did not exceed 3 minutes.

The results of the tensile tests carried out in the Instron testing machine lend considerable support to the explanation offered above. The tensile properties are tabulated in Table VIII and the data is plotted in Figure 4. The ultimate tensile strength curve established from the rapid heating tests (data in Table VII) is included for comparison. The strain rate employed in both tests were similar (see Tables VII and VIII). However, the heating time employed prior to testing was considerably different; testing was started after 30 seconds in the rapid heating tests, whereas 30 minutes was employed in the Instron tests. As shown in Figure 4, a marked difference in tensile properties was found. With a 30-second preheat period, the tensile strength dropped off with temperature in a continuous and normal manner. However, when a 30-minute preheat period was used, the tensile properties first decrease up to a temperature of 600°F and then increase to a maximum at a temperature of 1200°F. At temperatures about 1200°F the tensile strength decreases continuously with increasing temperature but at a faster rate than found for tests employing only 30 seconds of preheat time. At temperature over 1800°F, the tensile strength determined from the Instron tests was considerably lower than found from rapid heating tests. In fact, the ultimate tensile strength curve determined from the Instron tests is quite similar to that obtained from reference 9 (see Figure 3). Thus, there is little doubt that major differences in tensile properties can result solely from differences in testing technique at most all elevated temperatures.

As discussed previously, the high tensile properties that result from preheating 30 minutes in the temperature range of 600° to 1600°F are probably caused by an aging and/or a strain aging reaction. The lower properties observed at temperatures above 1800°F possibly result from recovery and/or recrystallization. Further work is needed to verify these points.

Consequently, it is extremely important to test this alloy under condition of temperature, time and strain rate, simulating actual use if realistic design allowables are to be specified. Advantage can be taken of the higher tensile properties at temperatures over 1800°F if application is of the very short time, one shot type. However, if reasonably long times are to be experienced (20 to 30 minutes) the lower tensile properties should be used.

Elongation decreases gradually with increasing temperature until a minimum is reached at about 1200°F. The minimum in elongation corresponds to the maximum in tensile strength (see Table VIII and Figure 4).

Some transverse tensile properties are given in Table VIII. At room temperature the tensile ultimate strength and yield strength in the transverse direction are higher than those in the longitudinal direction. Elongation is slightly lower. The transverse tensile strength and elongation are about 5 percent lower than the corresponding properties in the longitudinal direction at elevated temperatures.

The thermal expansion of the alloy as a function of temperature was obtained as a by-product of the data obtained from the tensile tests performed on the high heating rate, high strain rate testing machine. The total expansion from room temperature to several elevated temperatures is given in Table IX and plotted in Figure 5. The average coefficient of thermal expansion from room temperature (75°F) to 2600°F was found to be 4.56×10^{-6} in./in./°F.

Creep-Rupture Properties

The creep and rupture properties of stress relieved Cb-33Ta-.7Tr alloy are tabulated in Table X. The rupture life (time) as a function of temperature and stress is shown in Figure 6 and the 1%, 2% and 5% creep deformation strengths are plotted in Figure 7. The creep rate in second-stage creep is shown in Figure 8 as a function of temperature and stress.

With one exception, it is not possible to compare this data to others since it is the first of its kind to be determined and reported. However, rupture strengths at 2000°F can be compared and are as follows:

	PRESENT STUDY	REFERENCE 5	REFERENCE 8	REFERENCE 9
10 Hour Rupture Stress, psi	23,500	26,500	15,000	13,000
100 Hour Rupture Stress, psi	16,000	17,500	10,000	

The rupture strengths obtained in this study agree rather well with those reported in reference 5 but are considerably higher than those reported in references 8 and 9. It is not known whether this difference is due to testing technique or to an actual variation in the manufacturer's product.

The stress to yield 1, 2 and 5 percent creep deformation and the stress to yield rupture are plotted against a time-temperature parameter in Figure 9. The time-temperature parameter is that due to Larson and Miller and is as follows:

$$LMP = T\,(C + \log t)$$

where

LMP = Larson-Miller Parameter

T = absolute temperature, °R

t = time in hours

C = constant

Using a value of the constant "C" equal to 20 a reasonable correlation is obtained. This master curve can be used to predict stresses for rupture or for a given creep deformation under other conditions of temperature and stress by calculating the Larson-Miller parameter corresponding to these conditions or more simply by using the time-temperature curves at the top of the figure. Care should be taken when making such predictions that the temperature and times are not excessively out of the range from which the curves were established. In other words, predicting stresses from these curves is not always reliable, especially when conditions of time and temperature are quite different from those used to establish the curves.

The cost of running vacuum creep-rupture tests and the time consumed in the testing process is quite high. Because of this it is necessary to obtain as much information from the data as possible. To this end, the curves of creep rate, creep deformation strength and rupture strength in Figures 6, 7 and 8 were extrapolated to longer and shorter times or higher and lower creep rates. These data were then plotted against temperature and the properties at higher and lower temperature were determined from extrapolation of these curves. Such cross plotting and extrapolation of the original data resulted in additional data covering temperatures and times not investigated in the present work. The additional data, together with the original data, is plotted in Figures 10, 11 and 12. Actual and extrapolated data are indicated by solid and dashed lines. These data have been replotted in Figure 13 as stress vs. the Larson-Miller parameter and appear to show that extrapolation of the data in the original Larson-Miller plot (Figure 9) is reasonably safe.

Activation Energy for Creep

Many metallurgical processes have been shown to be thermally activated processes and as such their reactions obey the rate theory (13). The foundation of the present rate theory goes back historically to Arrhenius, who found that for many processes the specific reaction-rate constant K may be written as a function of temperature in the following way:

$$k = Ae^{-Q/RT}$$

k = reaction rate constant

A = constant depending on reaction

R = gas constant

T = absolute temperature

Q = energy or heat of activation of the reaction

Thus, ln k is a linear function of 1/T. The quantity $\frac{Q}{R}$ is the slope of the linear relation and A is determined from the intercept.

The activation energy for creep can be determined from continuous or differential tests. For example, a creep-rupture test represents a continuous test in which stress is held constant and thus:

$$\text{Creep rate } (\dot{e}) = f(te^{-Q/RT}) \qquad \text{Stress } (\sigma) = \text{constant}$$

If a series of tests are run at various temperatures and the same stress, an activation energy for creep can be determined.

Differential tests such as those proposed by Dorn and coworkers have proved quite valuable in activation energy studies. In this technique, a constant stress creep-rupture test is started and allowed to proceed into the second stage of creep. The temperature is then changed rapidly by 50° to 100°F. The creep rate prior to and after the temperature change is recorded and activation energy is calculated as follows:

$$Q = R \frac{\ln \dot{e}_2/\dot{e}_1}{\frac{1}{T1} - \frac{1}{T2}}$$

It has been found that for pure metals the activation energy for creep may vary with temperature and/or stress at temperatures less than about one-half the absolute melting temperature (0.5 Tm). Above 0.5 Tm, quite often the activation energy for creep (Qc) becomes equal to the activation energy for self-diffusion (Qd) and is independent of stress and strain. Consequently, creep of pure metals above about 0.5 Tm is a thermally activated process limited by the self-diffusion of the metal atoms. This is generally referred to as high-temperature creep and is thought to be controlled by dislocation climb.

Although the FS-82, columbium alloy is far from a pure metal, it is made up of two elements very similar in nature, i.e., columbium and tantalum. Furthermore, it is a solid solution alloy and, consequently, is single phase. In view of this, it would be interesting to examine the activation energy for creep of this alloy over the temperature and stress range covered in this study. Secondary creep rate as a function of temperature at three constant stresses was obtained from the data of Figures 8 and 11 and are shown in Table XI. This data is plotted as the ln creep rate ($\dot{e}$) vs. 1/T at three different stresses in Figure 14. The slope of the three straight lines yield quite similar activation energies: 121,000 $\frac{cal}{mol}$ at 4200 psi stress, 115,000 $\frac{cal}{mol}$ at 8000 psi stress and 128,000 $\frac{cal}{mol}$ at 40,000 psi stress. Thus, the activation energy for creep appears to be independent of stress having an average value of 121,300 $\frac{cal}{mol}$. Being independent of stress suggests Qc should equal Qd and that creep observed between 1800° and 2200°F is controlled by self-diffusion of columbium and/or tantalum atoms. This temperature range varies from 0.45 Tm to 0.53 Tm and is within the temperature range where high-temperature creep could be controlled by dislocation climb.

The activation energy for self-diffusion of columbium has been reported by Resnick and Castleman (14) to be 105,000 ± 3000 $\frac{cal}{mol}$. The activation energy for self-diffusion of tantalum was reported as 110,000 $\frac{cal}{mol}$ by Eager and Langmuir (15). Thus, the activation energy for creep of 121,300 $\frac{cal}{mol}$ found in the present study is in excellent agreement with the activation energy for self-diffusion of columbium and/or tantalum. It appears that the FS-82 single phase columbium-tantalum alloy acts very much like a pure metal and creep above about 0.5 Tm is controlled by self-diffusion of columbium and possibly tantalum atoms by dislocation climb.

Recrystallization Behavior

Although a specific program was not carried out to determine the recrystallization temperature of the stress relieved FS-82 alloy, an approximate recrystallization temperature was derived from metallographic examination of tested tensile and creep-rupture specimens. Certain of these specimens showed partial recrystallization as a result of high temperature exposure. The data are presented in Table XII. The percent recrystallization is plotted against a temperature-compensated-time parameter (Larson-Miller parameter) in Figure 15. From this plot it is possible to estimate the Larson-Miller parameter to yield 100% recrystallization and from this parameter a recrystallization temperature can be calculated. For example, a 1-hour recrystallization temperature of 2220°F was determined. Of course, it is possible that the recrystallization behavior is influenced by the applied stress. However, the recrystallization temperature determined in this study is in reasonably good agreement with the data reported in reference 9. Unstressed samples of FS-82 were found 50% recrystallized after 1 hour at 2200°F.

Weldment Properties

The microstructure and hardness of TIG and electron beam fusion welds are shown in Figures 16 and 17. As shown by the higher hardness of the weld nugget of the TIG fusion weld, some gas pickup occurred during welding (see Figure 16). Such was not the case in the electron beam fusion welds (see Figure 17). In both cases, a relatively high hardness was found to occur in the weld metal near the heat-affected zone. The explanation for the maximum in the hardness traverse is not obvious. It is quite possible that an accelerated aging phenomenon occurs in this area. Further work is required before the actual cause can be determined.

The slight contamination of the TIG fusion welds was evidenced also in the bend ductility of the welds. Bend ductility of the base metal, TIG fusion welds and electron beam fusion welds are presented in Table XIII. All samples were bent in a brake press around a 1T radius (see Figures 18 and 19). The base metal and electron beam fusion welds were bent 180° without cracking, but a few small cracks appeared at 100° in the

TIG fusion welds. As shown by the hardness and bend tests, the amount of contamination was not extremely serious and all welds were judged to have sufficient ductility to provide reliable engineering structures.

Tensile Properties

The tensile properties of electron beam and TIG fusion welds are presented in Table XIV and plotted against temperature in Figure 20. The ultimate tensile properties of the parent metal determined under identical testing condition are included for comparison (data taken from Table VIII). There is a slight indication in the data that a maximum in the tensile strength of TIG weldments occurs at 1200°F, but this is not nearly as marked as that found in the unwelded metal. Weld efficiency of TIG weldments decreased from 94 percent at room temperature to about 75 percent at 1800°F (see Table XIV).

The tensile strength and weld efficiency of electron beam weldments were much lower than TIG weldments. Weld efficiency was only 64 percent at room temperature and dropped to 54 percent at 1800°F. It is felt that the lower properties of electron beam weldments are directly related to the extremely large heat affected zone and grain size that resulted from welding. It is quite reasonable to expect that improved properties will result from improved welding procedure and electron gun design.

In general, fracture occurred in the heat-affected zone in both TIG and electron beam fusion weldments. Occasionally fracture occurred in the parent metal near the heat-affected zone or in the fusion weld.

Rupture Properties

The rupture strength of the fusion welds is given in Table X and plotted in Figure 21. Base metal data is included for comparison. At 1800°F TIG fusion weldments are considerably weaker than the base metal. At higher temperatures the TIG fusion weldments appear to be stronger than the base metal. Similarly, the electron beam fusion weldments were weaker than the base metal at 2,000°F but stronger at 2200°F. This behavior may be related to the difference in grain size between the base metal and the weldments. The grain size in the weld and heat affected zone is quite large in comparison to the grain size of the base metal (see Figures 16 and 17).

Most materials exhibit a temperature (sometimes called the equicohesive temperature*) below which fine grains are stronger in creep-rupture than course grains and above which the opposite is true. Consequently, the higher rupture strength of the fusion weldments above about 2000°F may be due to the larger grain size of the weldments.

However, the analysis is somewhat complicated by the atmospheric contamination of the creep-rupture coupons as evidenced by hardness measurements (see Table X). It is felt that the contamination occurred from a loss in vacuum after the sample fractured. Rupture of the sample resulted in a severe shock to the vacuum furnace and the hot pull rods move up into the rubber U-cup vacuum seal. The heated rubber seal became hardened and no longer sealed the system. Furthermore, except in one case, the vacuum was always found satisfactory when checked during the creep-rupture tests. Further evidence in support of this argument can be found in the one test in which it was observed that vacuum was lost during the testing period. This occurred in a base metal sample tested at 2200°F and 13,000 psi stress (see Table X). Contamination as measured by hardness was about as severe as that found in many of the fusion weld test coupons. However, the sample was strengthened to a much greater extent than that observed in the fusion weld samples. Thus, it is felt that the gas contamination of the fusion weld samples occurred after the test was completed and the effects observed are real. Further testing should verify this point.

CONCLUSIONS

The tensile properties of the columbium alloy, FS-82, have been determined from ambient temperature to 2600°F. Markedly different tensile properties were obtained when different heatup times were employed prior to the tensile test. The difference in properties was related to metallurgical phenomena such as strain aging, recovery and recrystallization.

Rupture strength, 1, 2 and 5 percent creep deformation strength and creep strength have been determined at 1800, 2000 and 2200°F. The data were extrapolated using appropriate techniques with excellent success.

The activation energy for creep was found to be 121,300 cal/mol and is in excellent agreement with the activation energy for self-diffusion of columbium and/or tantalum. Thus, above about 1800°F creep of this alloy is diffusion controlled.

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*The equicohesive temperature is really a range of temperature since it has been found to be stress dependent.

Tensile and rupture properties of TIG and electron beam fusion welds were determined. TIG fusion weld efficiencies decreased from 94 percent at ambient temperature to about 75 percent at 1800°F. Electron beam weld efficiencies were considerably lower, decreasing from 65 percent at ambient temperature to about 54 percent at 1800°F. The lower weld efficiency of the electron beam fusion weldments was related to their larger heat affected zone and grain size. Weldments were weaker than the parent metal in creep-rupture below about 2000°F but they appear to be stronger at higher temperatures.

The average coefficient of thermal expansion between room temperature and 2600°F was found to be 4.56×10^{-6} in./in./°F.

FUTURE WORK SUGGESTED BY THIS PROGRAM

The results of this program suggest several important areas for further work which should prove quite fruitful. These are discussed as follows in the estimated order of importance.

1. Elevated temperature tensile and creep-rupture properties should be determined on a second heat of FS-82 columbium alloy. This study will help determine product variability which at the present time is difficult to determine from published data because of the wide range of testing procedures employed.

2. The strain aging response of the FS-82 columbium alloy should be thoroughly studied. This study should include the effect of strain rate and soaking time on tensile strength and elongation in the temperature range from 600°F to 2000°F. The effects of interstitial element (carbon, nitrogen, oxygen and hydrogen) content should be studied. This program should be extended at a later time to include the effect of strain aging on the creep behavior of the alloy.

3. The possibility of weld embrittlement due to heating in the temperature range from 1900° to 2000°F should be studied. If weld embrittlement does occur means of eliminating or minimizing it should be studied.

4. Means of increasing high-temperature tensile weld efficiency should be studied.

5. The effect of oxidation protection coatings on base metal tensile and creep-rupture properties should be investigated. It is quite possible that such coating may markedly affect these properties.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the assistance of the personnel of several groups who actively participated in the experimental phase of this program. In particular, I would like to express my appreciation to Mr. Walter Wyman of the General Atomic Division of General Dynamics Corporation for preparing the electron beam fusion welds and to Mr. D. E. Arndt of the Applied Manufacturing Research Department of Convair-Astronautics for preparing the TIG fusion welds. The assistance of Mr. Robert E. Mihalco of Convair-Pomona who ran the high heating rate tensile tests is gratefully acknowledged. Special tanks go to Mr. G. Hill who carried out the vacuum creep-rupture tests and the vacuum tensile tests, and who assisted in many other ways on the over-all program.

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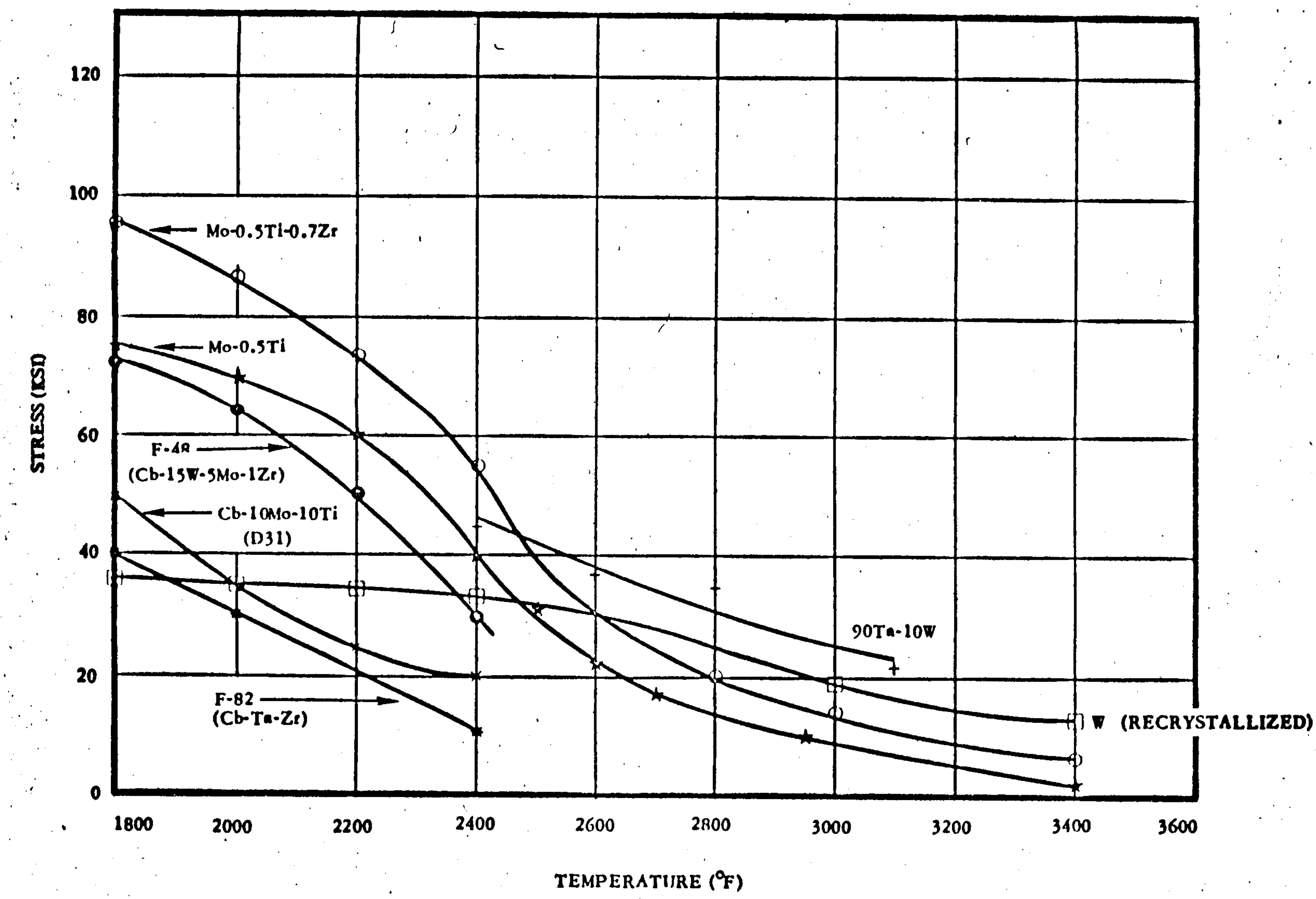


Figure 1. Ultimate tensile strength of several refractory metals and alloys.

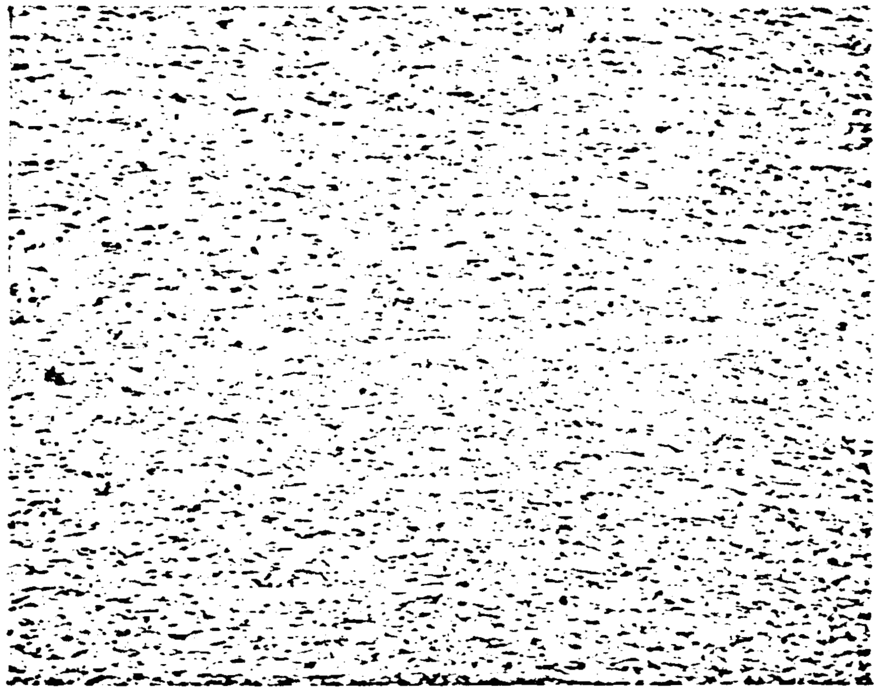


Figure 2. Microstructure of columbium FS-82 alloy as-received (stress relieved 1 hour at 1900°F), 100X.

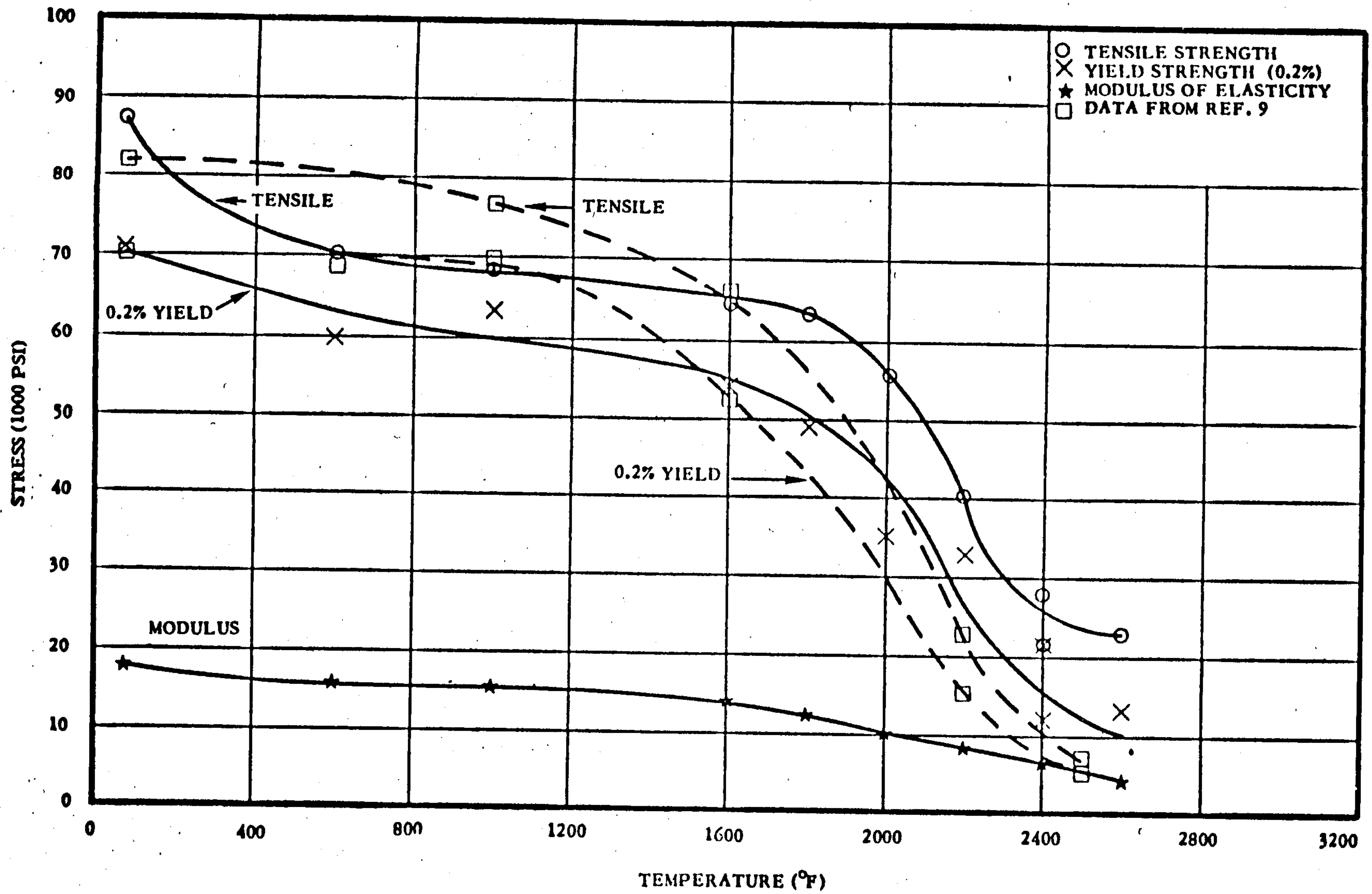


Figure 3. Tensile properties of FS-82 columbium alloy; heated to test temperature in 30 seconds by self-resistance; helium atmosphere.

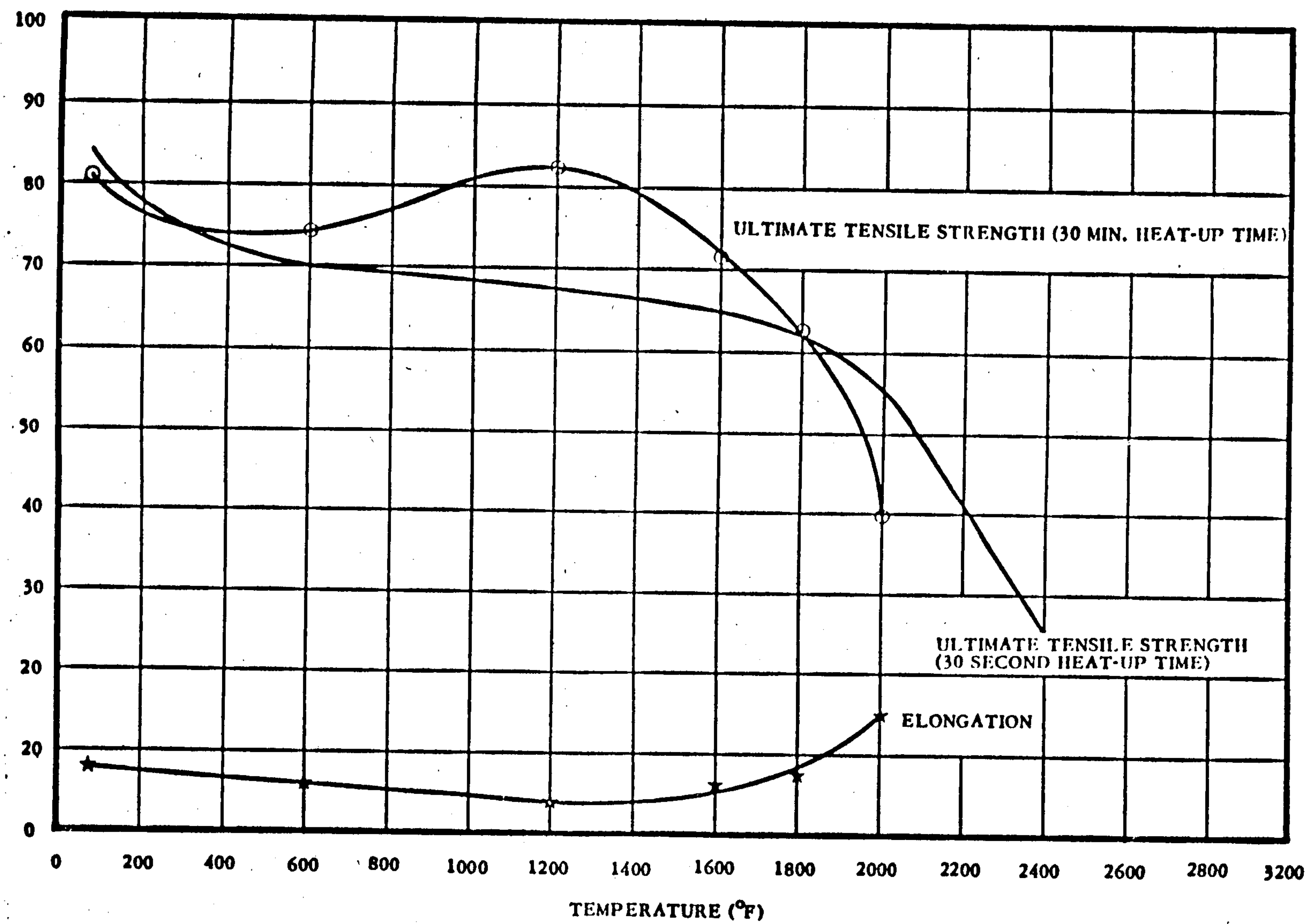


Figure 4. The effect of heating time prior to test on ultimate tensile properties of FS-82 Cb alloy.

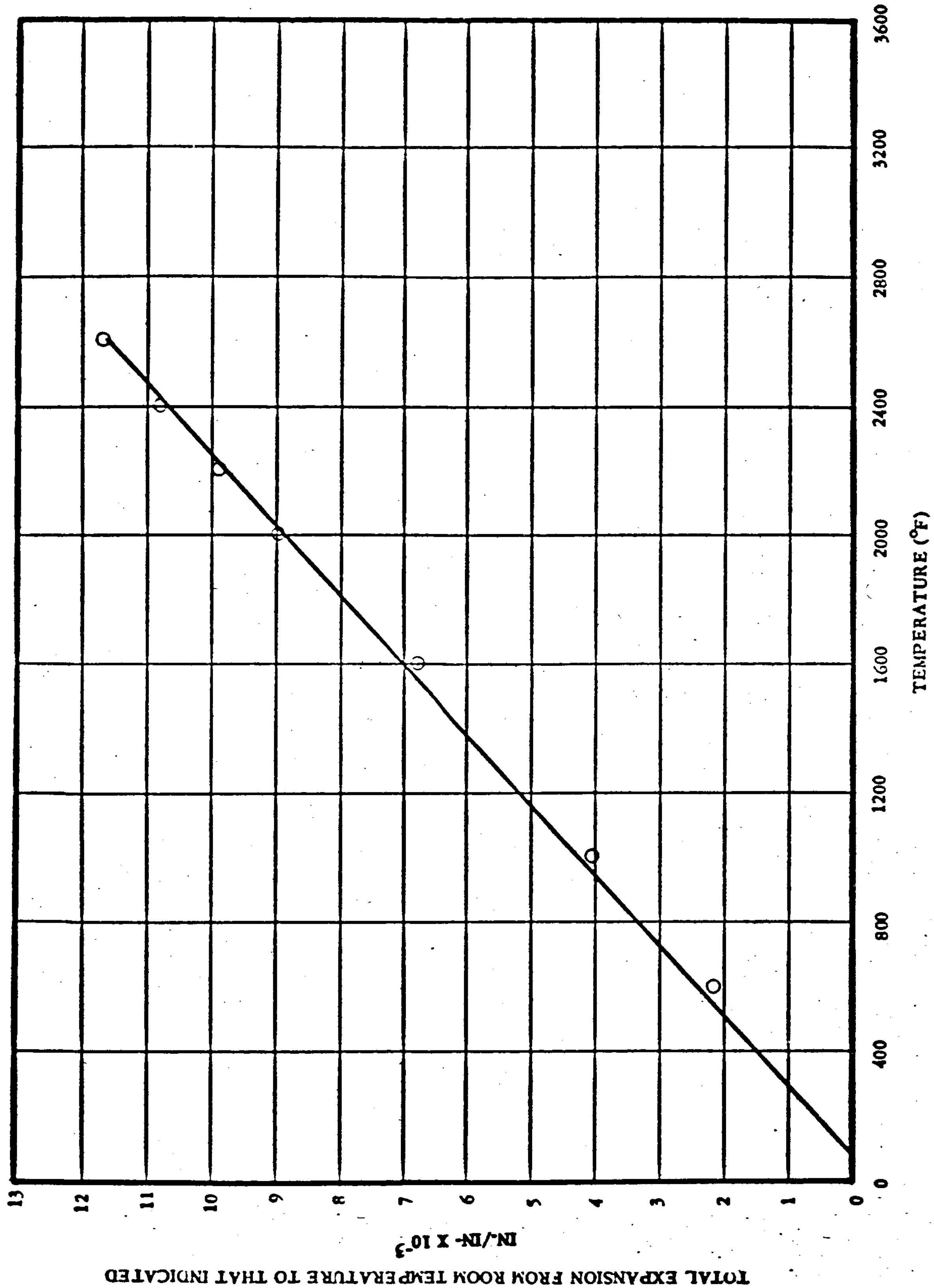


Figure 5. Thermal expansion of columbium FS-82 alloy.

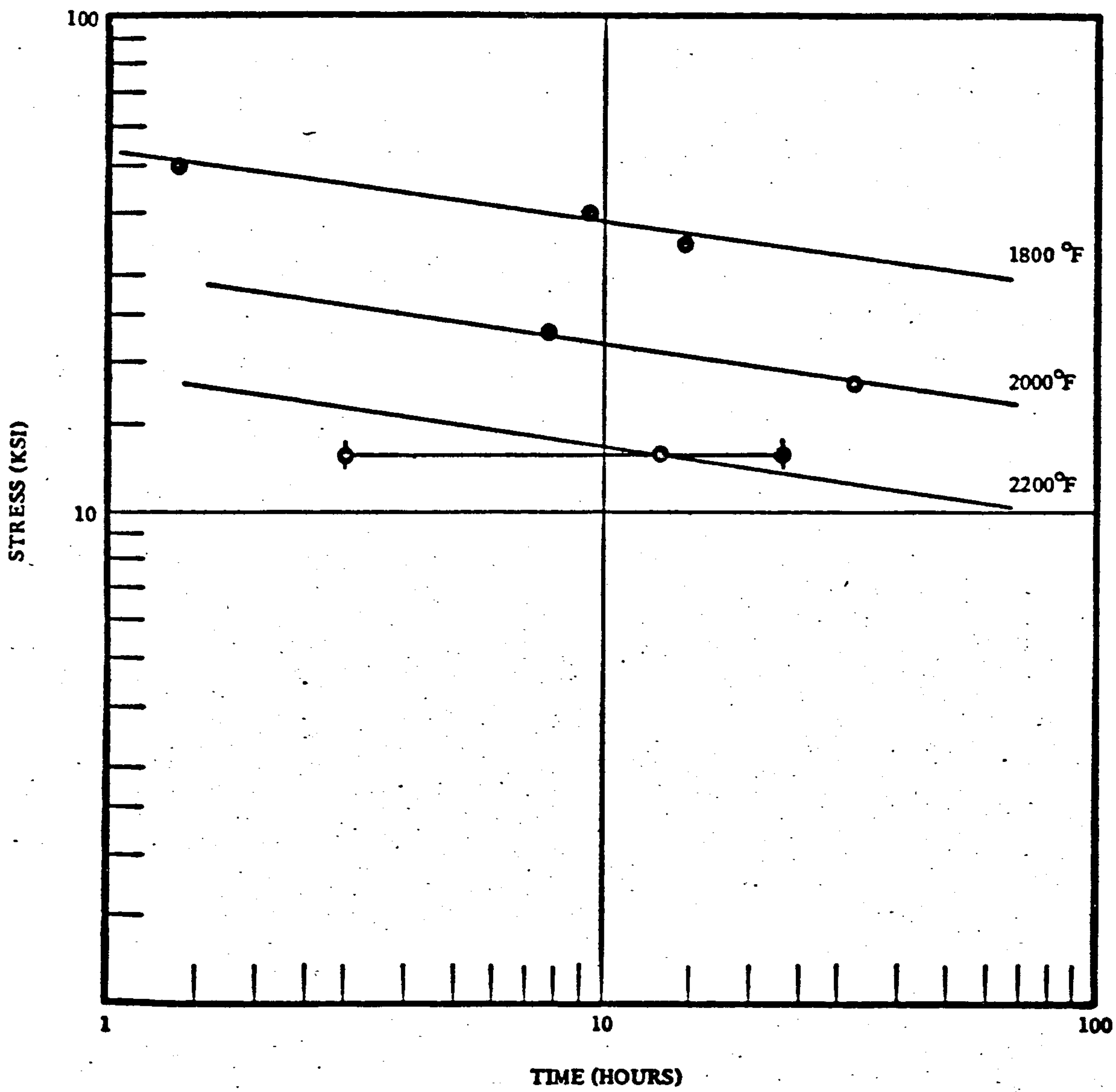


Figure 6. Rupture strength of columbium FS-82 alloy at 1800°, 2000° and 2200°F (stress relieved 1 hour at 1900°F).

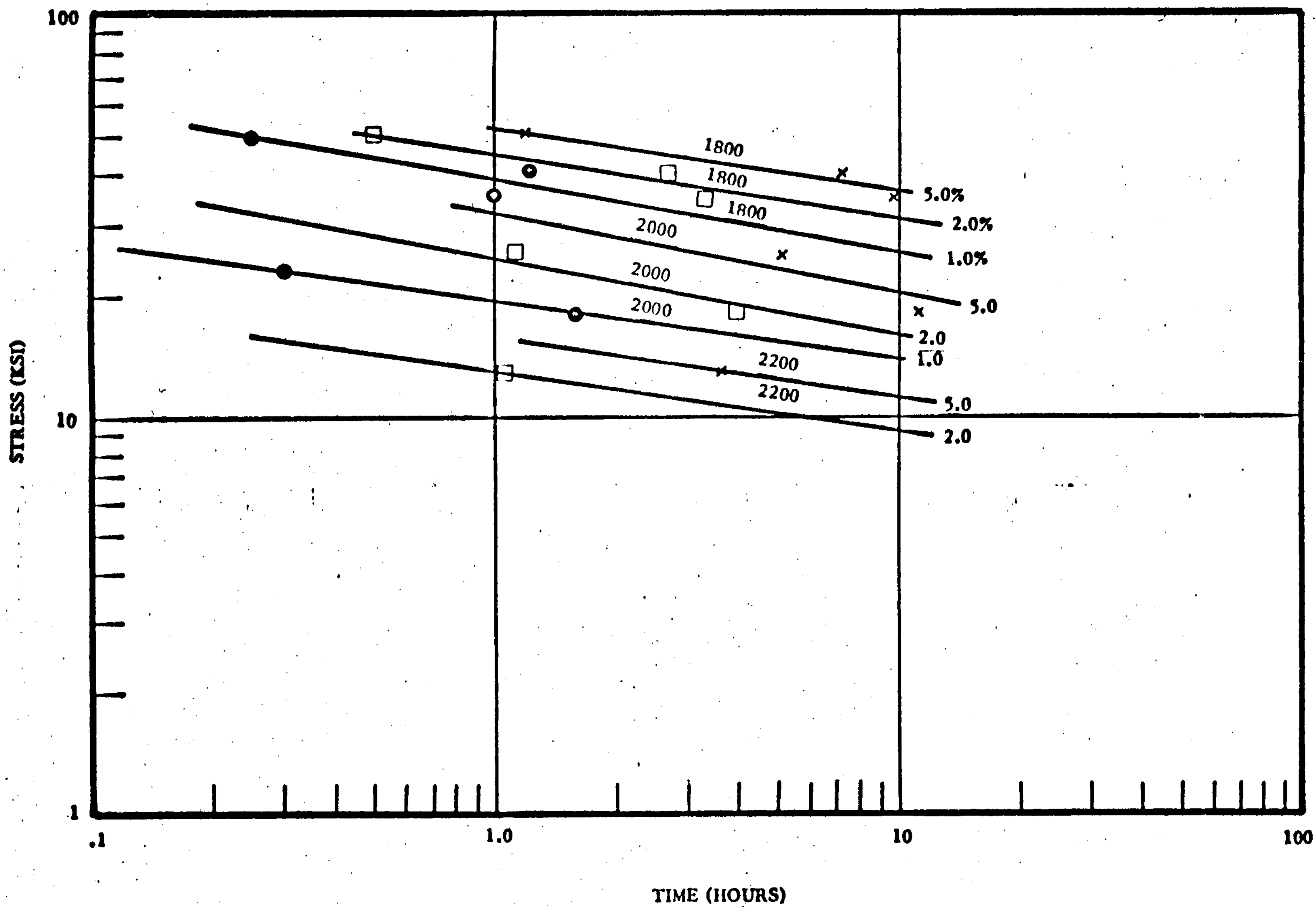


Figure 7. Creep deformation of columbium FS-82 alloy at 1800°, 2000°, and 2200°F (stress relieved 1 hour at 1900°F).

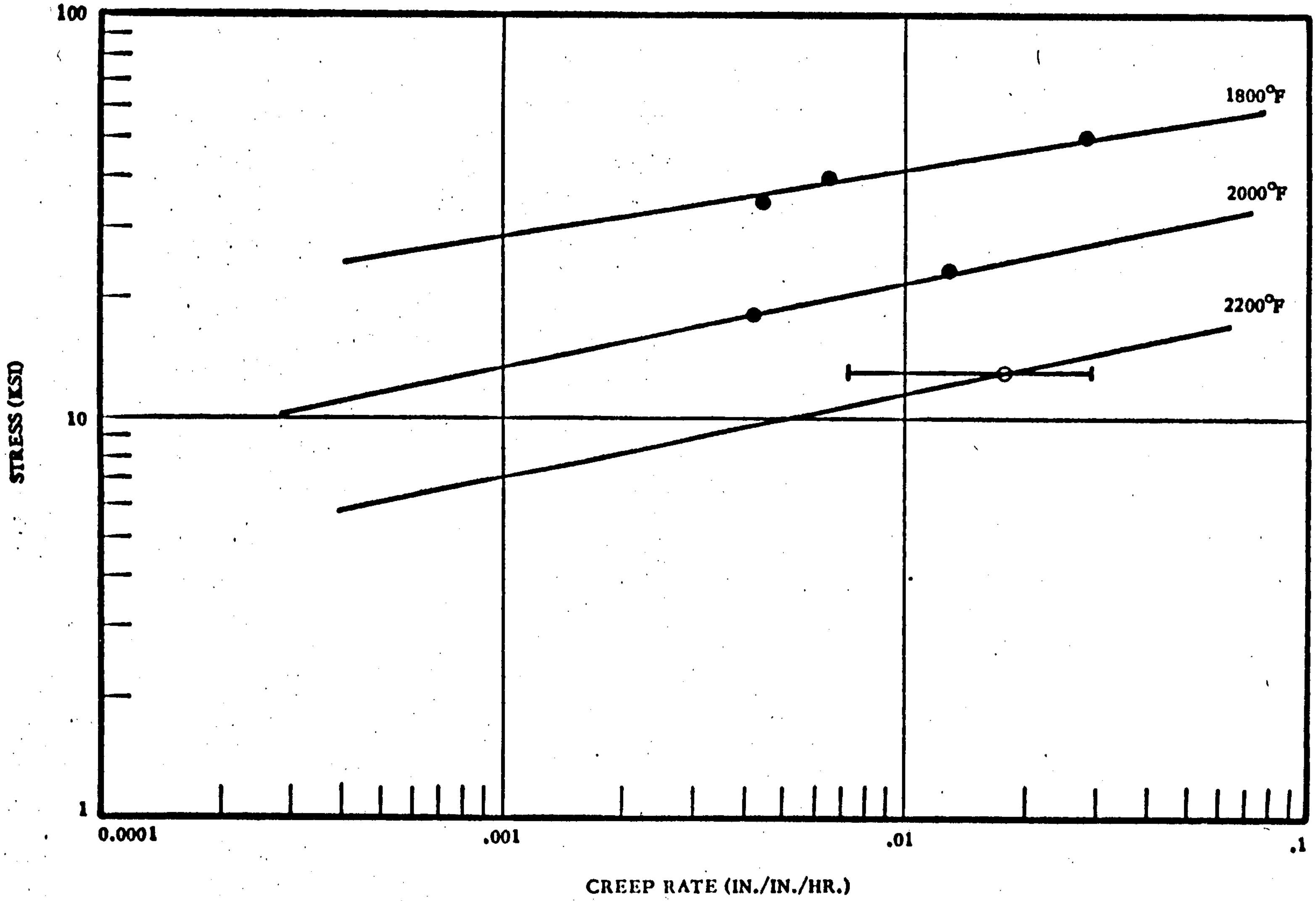


Figure 8. Secondary creep rate of columbium FS-82 alloy at 1800°, 2000° and 2200°F (stress relieved 1 hour at 1900°F).

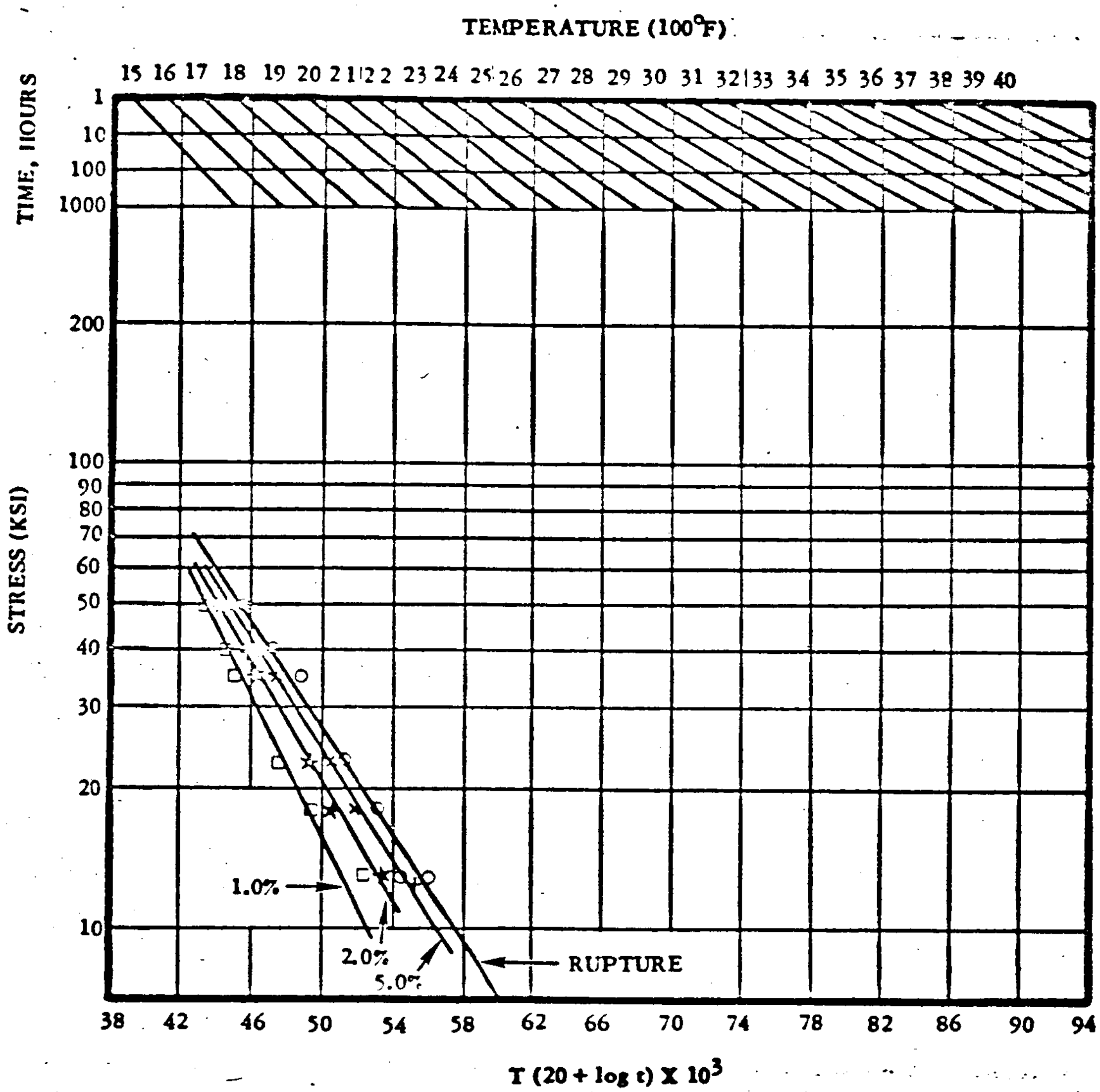


Figure 9. Creep deformation and rupture strength of columbium FS-82 alloy as a function of a time-temperature compensated parameter. Tested in vacuum at 5×10^{-4} mm Hg.

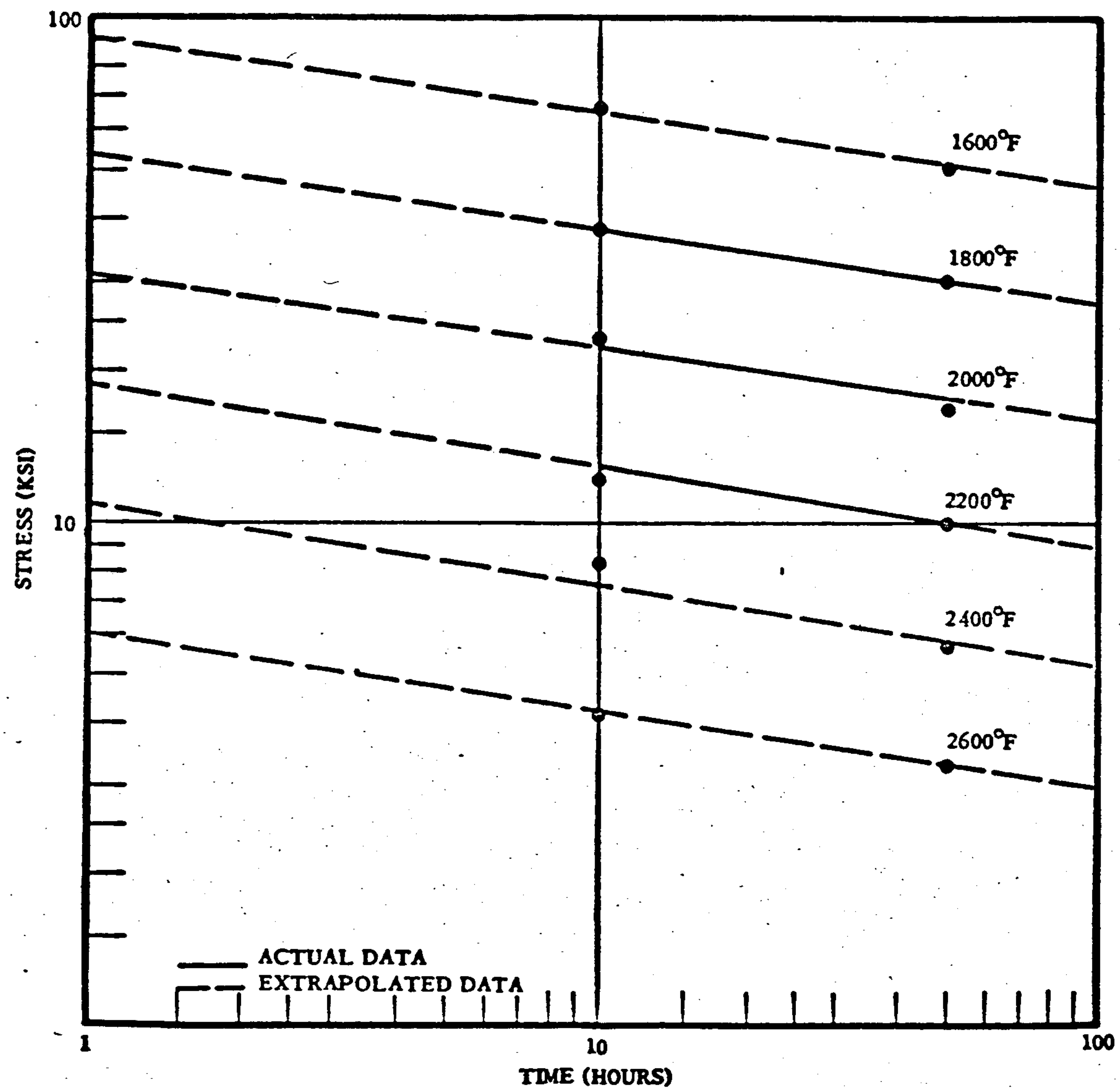


Figure 10. Extrapolated rupture strength of columbium FS-82 alloy.

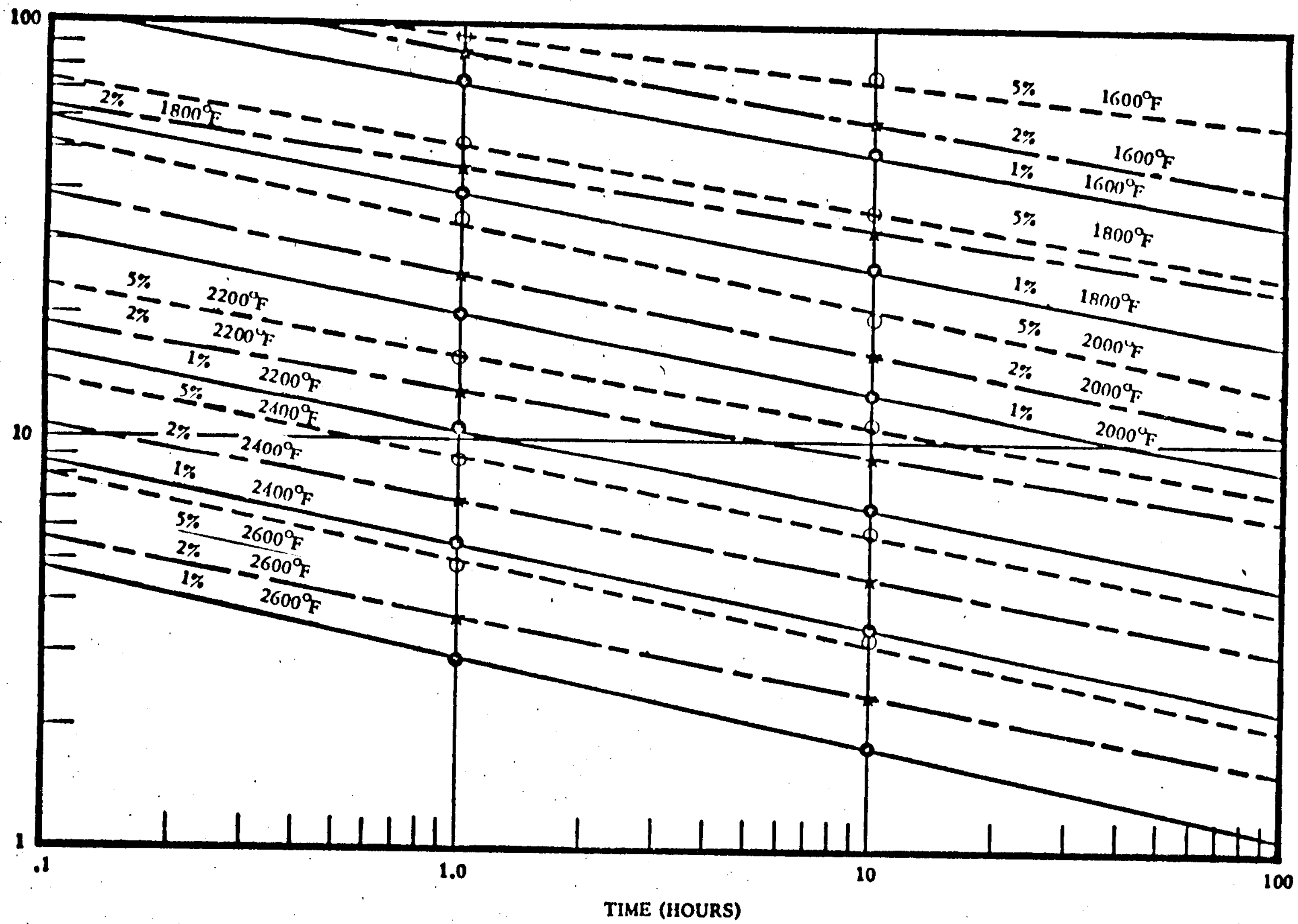


Figure 11. Extrapolated creep deformation strength of columbium FS-82 alloy.

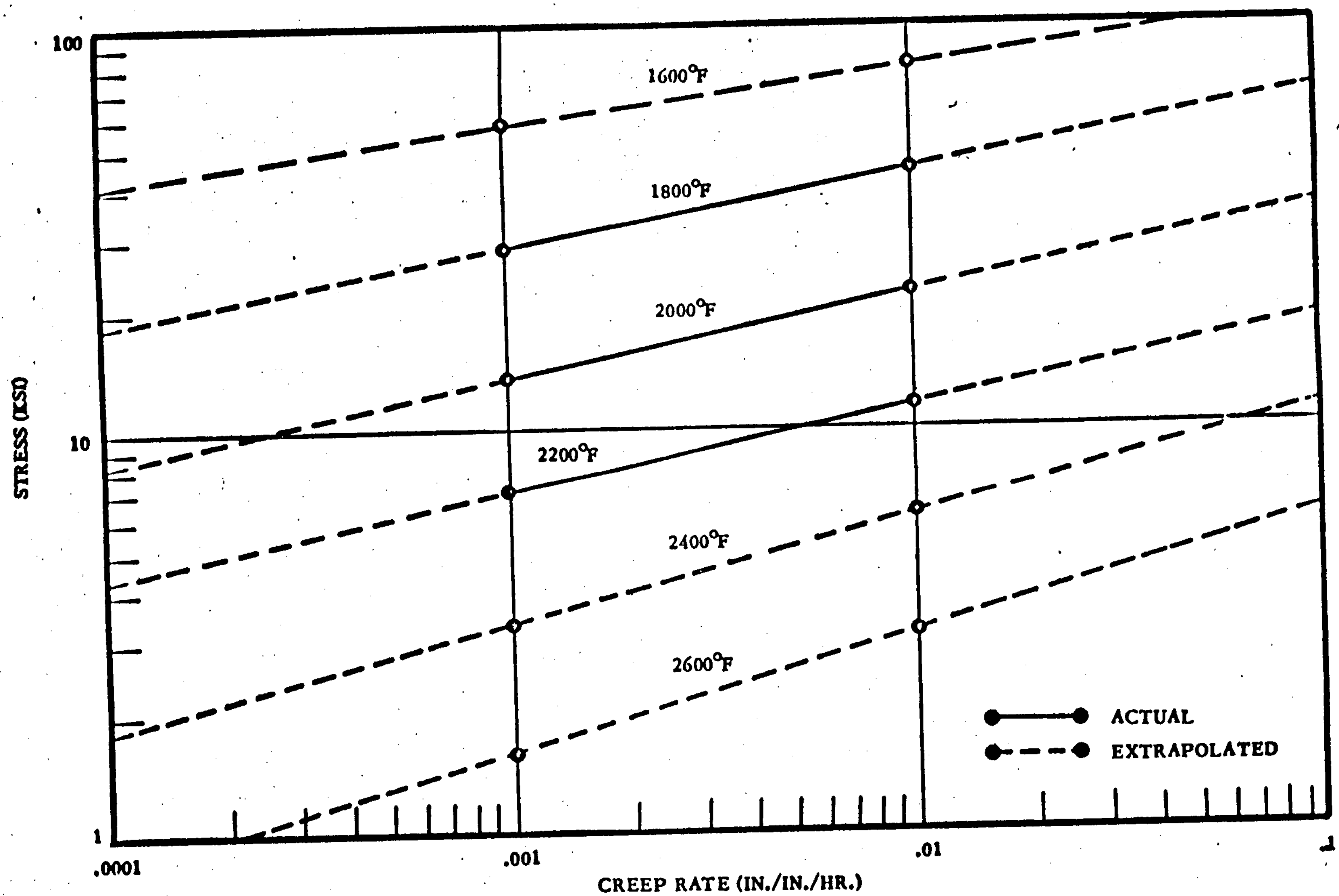


Figure 12. Extrapolated creep rate vs. temperature for columbium FS-82 alloy.

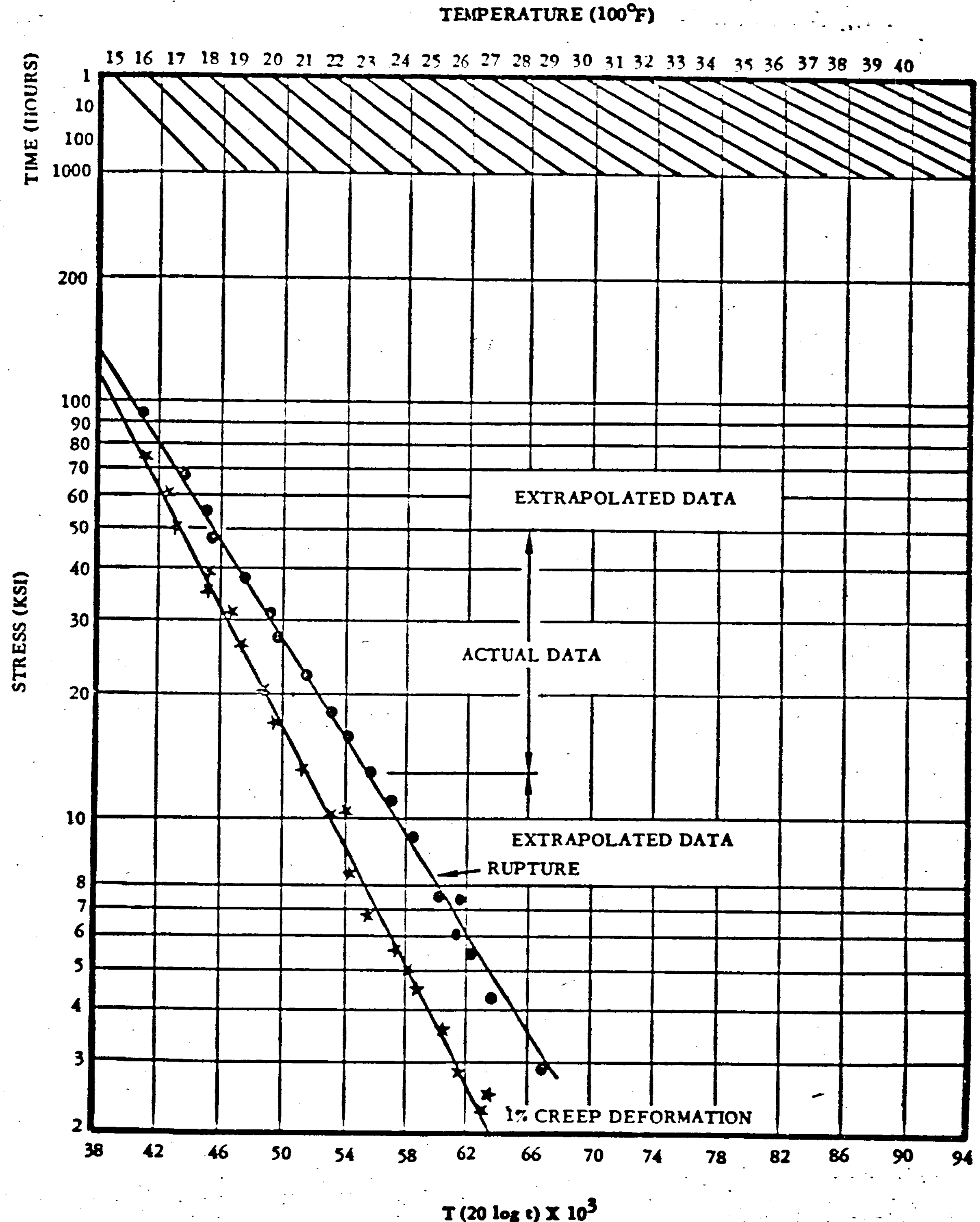


Figure 13. Creep deformation and rupture strengths of columbium FS-82 alloy. Larson-Miller plot, based on extrapolated data.

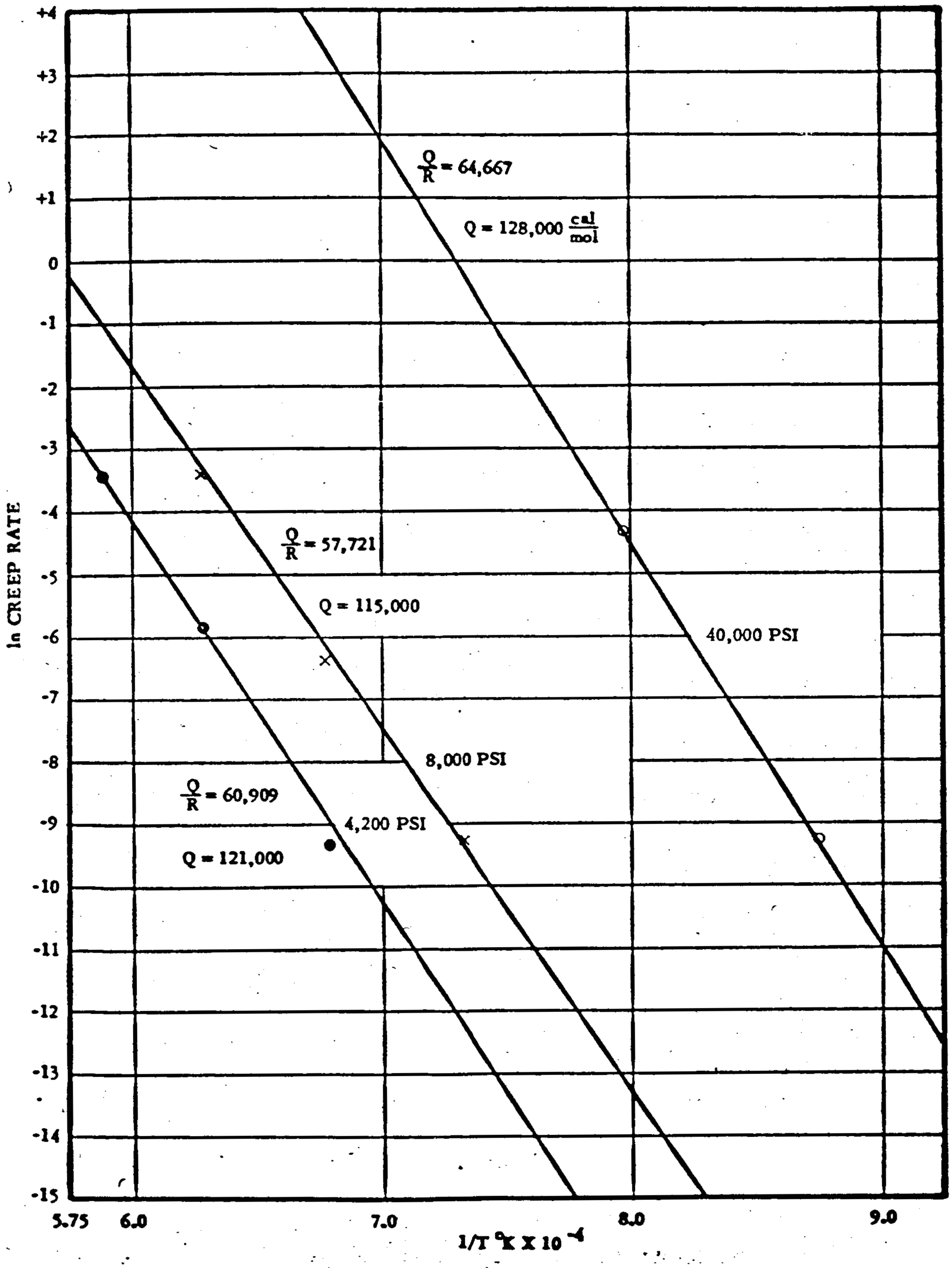


Figure 14. Activation energy for creep of columbium FS-82 alloy.

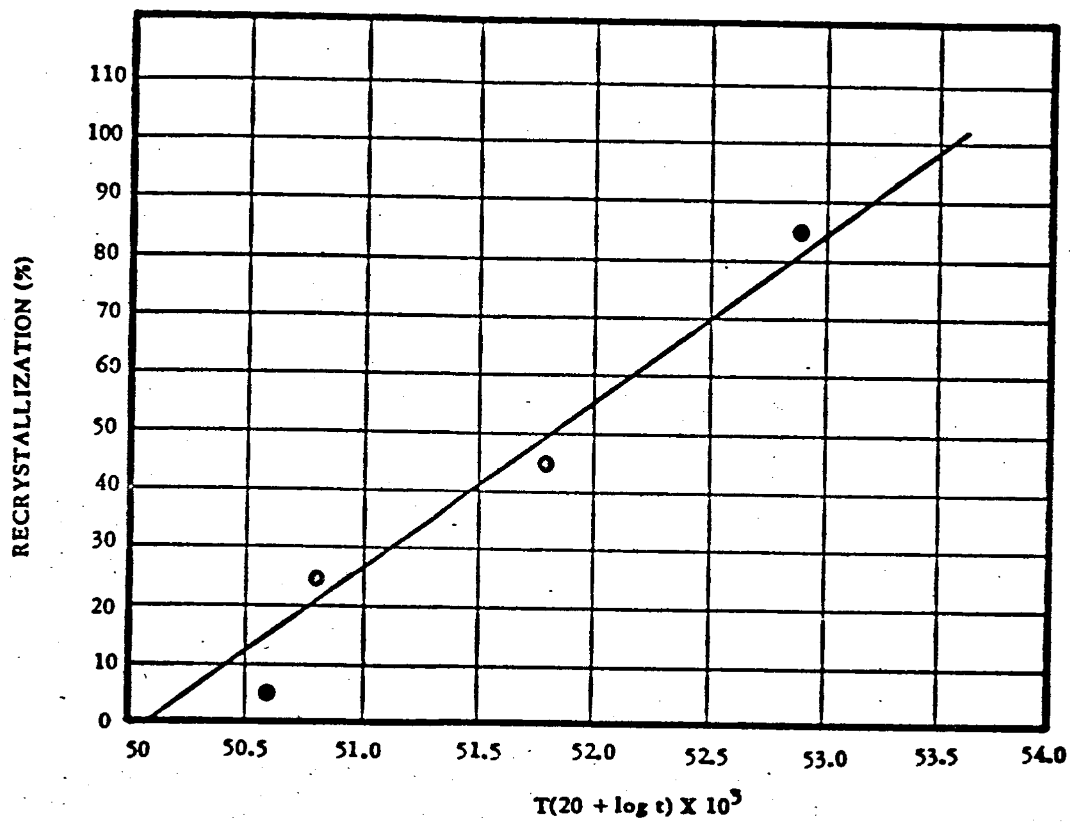


Figure 15. Recrystallization behavior of columbium FS-82 alloy (stress relieved 1 hour at 1900°F).

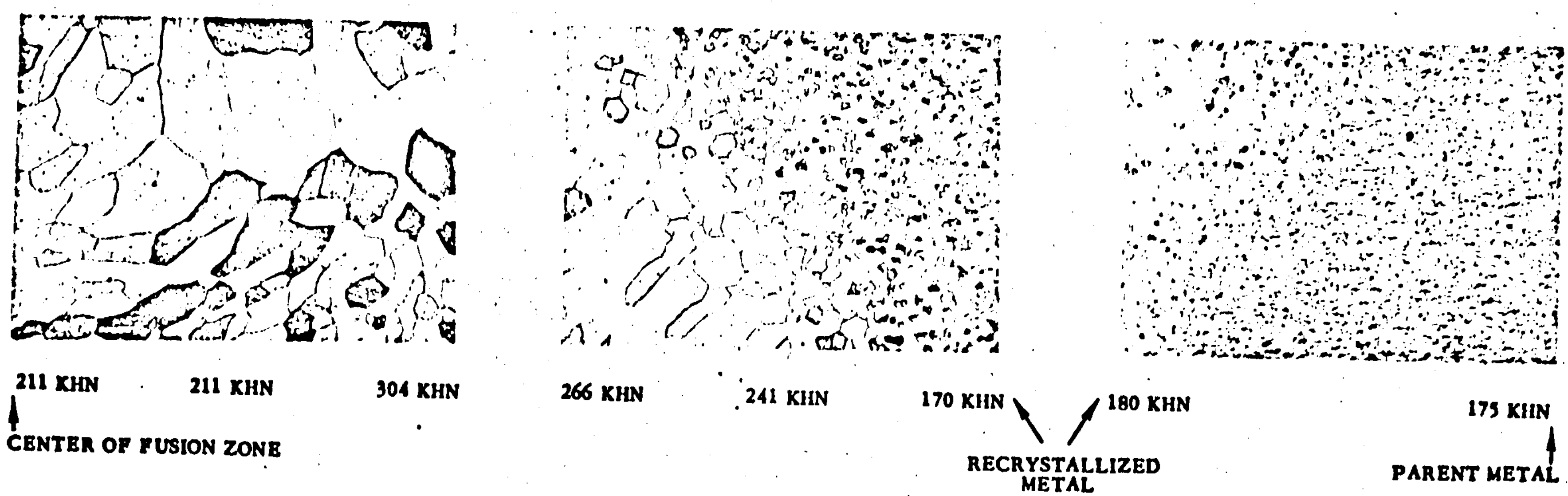


Figure 16. Microstructure of TIG fusion weld and corresponding microhardness measurements (Knoop, 1.0 Kg load); 100X.

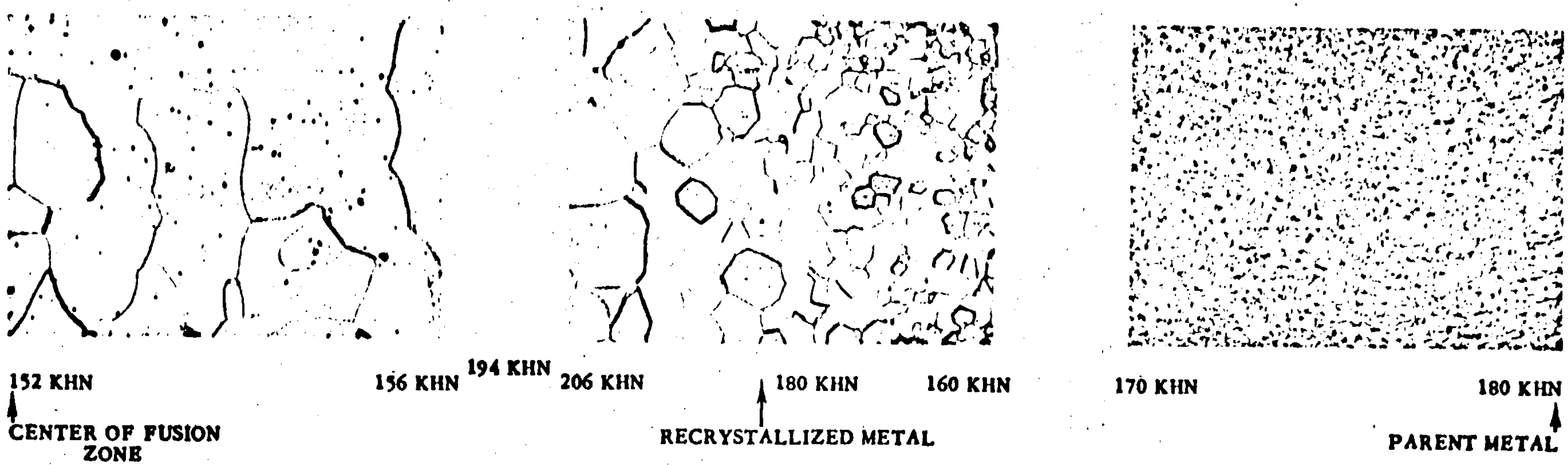


Figure 17. Microstructure of electron beam fusion weld and corresponding microhardness measurements (Knoop, 1.0 Kg load); 100X.

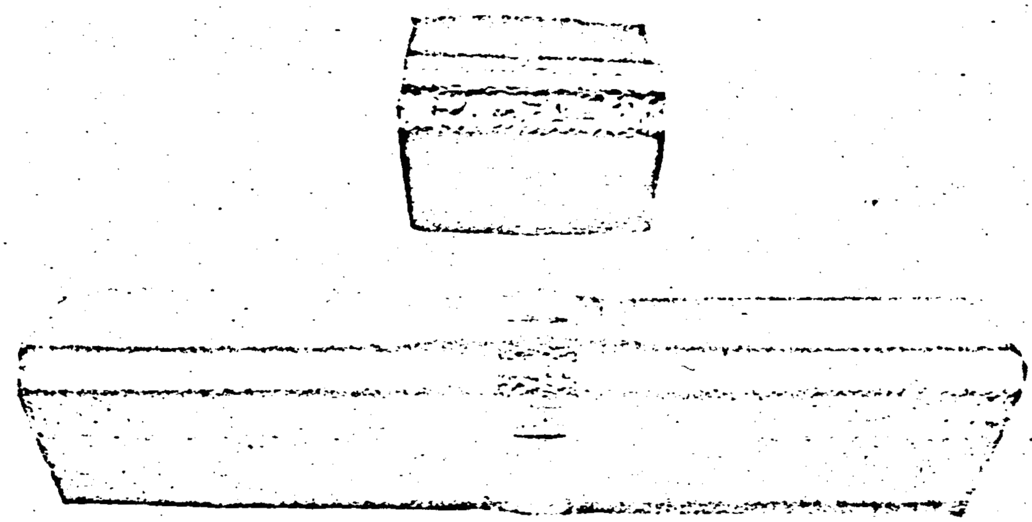
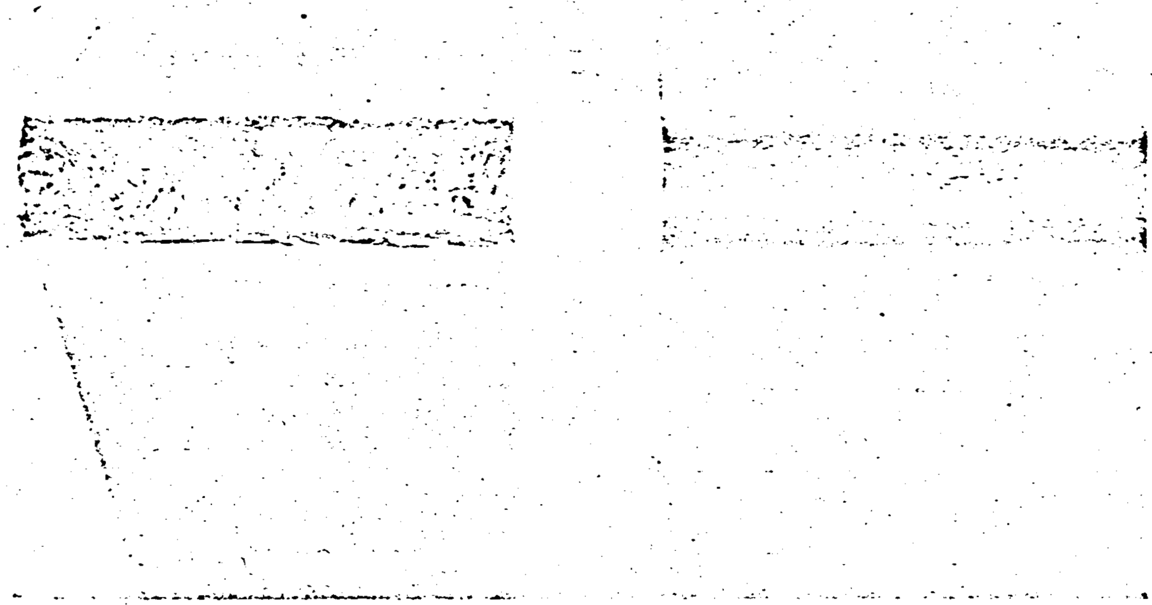


Figure 18. 140° bend of TIG fusion welded columbium FS-82 alloy; 1T radius bend; 3X.



WELDMENT BASE METAL

Figure 19. 140° bend of electron beam fusion welded columbium FS-82 alloy; 1T radius bend; 3X.

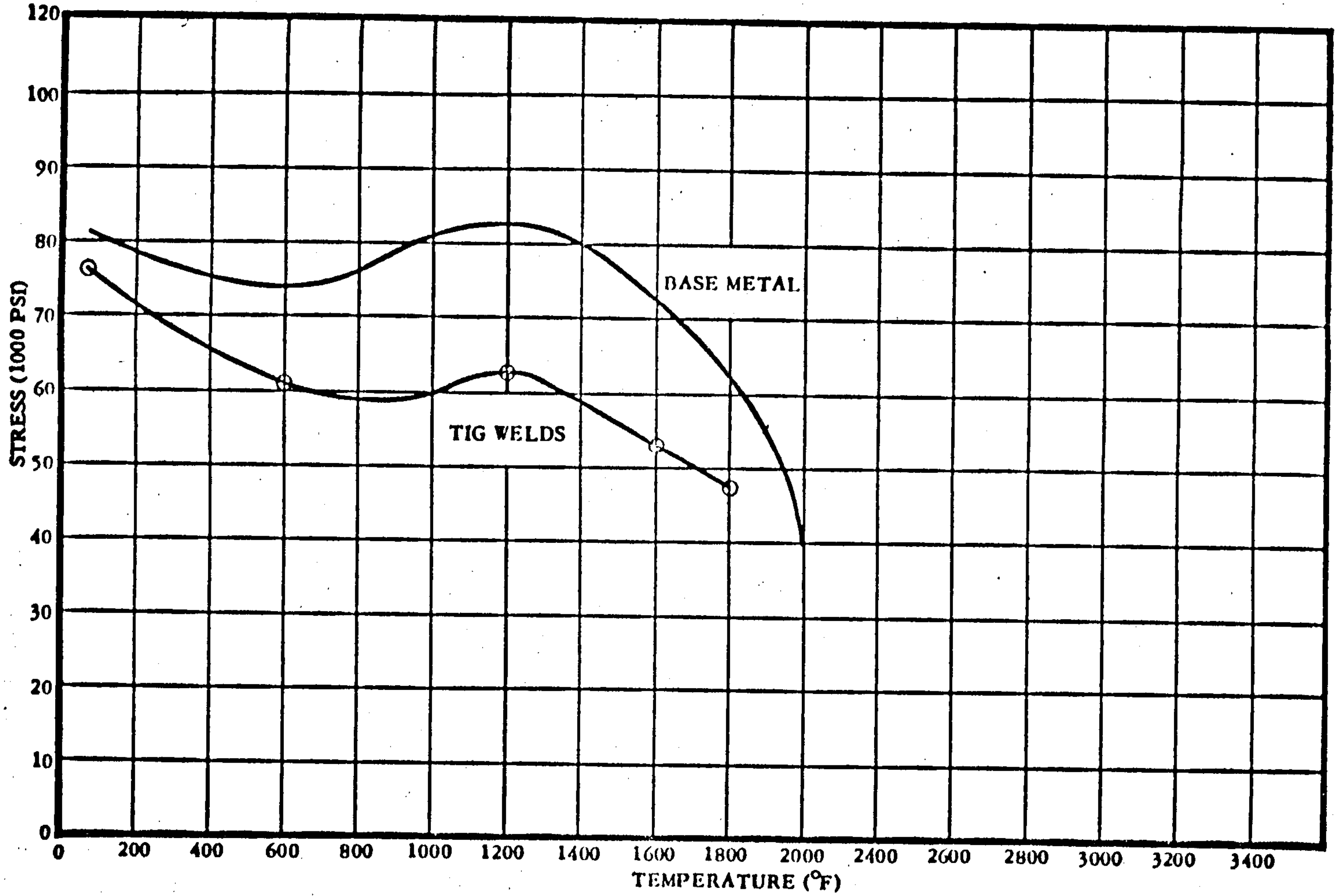


Figure 20. **Tensile properties of columbium FS-82 alloy fusion welds, heated to temperature in 30 minutes.**

Table IV. Notched-Unnotched Tensile Properties of FS-82[a]

ROOM TEMPERATURE			
UNNOTCHED TENSILE STRENGTH Ksi	K_t	NOTCHED TENSILE STRENGTH	RATIO NTS/UNTS
	5.7	81.6	1.00
81.6	5.7	82.1	1.01
	7.3	81.9	1.00
	8.2[b]	77.0	0.94

a. Data from reference 9.

b. K_t differed from one side of specimen to the other.

Table 6. Applicability of Surface Protection on Columbium Alloys (From General Electric Co., Reference 8)

	REQUIRES FURTHER DEVELOPMENT	LIMITATIONS ON SHAPE	LIMITATIONS ON SHEET THICKNESS	PERMITS INSPECTION	PERMITS REPAIR	PERMITS JOINING	PERMITS MATERIALS RECOVERY	PROVIDES REASONABLE DUCTILITY	GOOD BASE FOR OTHER COATS
Electrodeposition									
Electroplated Cr	?	?		x	x	x	x	x	x
Other electro plates	?	?		x	x	x	x	x	x
Composites (Vitro)	x	x					x	?	x
CEM (platecraft)	x	x					x	?	x
Diffusion coatings									
Al-Si dip	x		?		?	?		?	?
Vapor deposition									
Chromium, etc. (alloyed res.)		x			?	?	x	x	x
S-2 (fansteel)		x			?	?			
Al_2O_3-dense (GE Co.)	x	x							
Sprayed									
System 400 (GE Co.) glass-sealed alumina	x	x	x			?			

x = yes

Table VIII. **Tensile Properties of FS-82** Columbium Base Metal[1] **(Heated to Temperature in 30 Min.)**

TEST DIRECTION	TEMPERATURE (°F)	ULTIMATE TENSILE STRENGTH (Ksi)	0.2% OFFSET YIELD STRENGTH (Ksi)	ELONGATION IN 2 INCHES (%)	ELASTIC MODULES (X10^6)
Longitudinal	75	82.8	69.7	-	16.3
Longitudinal	75	78.5	61.1	7.8	16.8
Ave		80.7	65.4	7.8	16.5
Transverse	75	85.8	72.5	6.7	15.9
Longitudinal	600	68.4		6.3	
Longitudinal	600	75.9		7.0	
Longitudinal	600	77.7		5.8	
Ave		74.0		6.3	
Longitudinal	1,200	77.1		3.0	
Longitudinal	1,200	80.6		4.5	
Longitudinal	1,200	84.1		2.8	
		82.4		3.7	
Transverse	1,200	77.1		3.0	
Longitudinal	1,600	72.2		6.8	
Longitudinal	1,600	70.9		5.5	
Ave		71.5		6.1	

Table VIII. Continued

TEST DIRECTION	TEMPERATURE (°F)	ULTIMATE TENSILE STRENGTH (Ksi)	0.2% OFFSET YIELD STRENGTH (Ksi)	ELONGATION IN 2 INCHES (%)	ELASTIC MODULES ($X10^6$)
Transverse	1,600	70.4		3.5	
Transverse	1,600	66.1		5.5	
Ave		68.3		4.5	
Longitudinal	1,800	65.5		5.0	
Longitudinal	1,800	59.6		9.0	
Ave		62.5		7.0	
Longitudinal	2,000	39.5		15	

1. All testing carried out in Instron tester - Vacuum $5x10^{-4}$ mm Hg.

Table IX. Thermal Expansion of the Columbium Alloy FS-82 (Stress Relieved 1 Hr. at 1,900°F)

TEMPERATURE (°F)	TOTAL EXPANSION 75°F TO INDICATED TEMPERATURE (IN./IN.)
600°F	0.00218
1,000°F	0.00405
1,600°F	0.00680
2,000°F	0.00895
2,200°F	0.00990
2,400°F	0.01080
2,600°F	0.01170

Average coefficient of thermal expansion from RT to 2,600°F = 4.56×10^{-6} in./in./°F

Table XI. Activation Energy Data

TEMPERATURE (°F & °C)	CREEP RATE AT CONSTANT STRESS OF 4,200 (psi)	8,000 (psi)	40,000 (psi)	$1/T \times 10^{-4}$ (°R)	(°K)
1,600 (871.1)			0.0001	4.85	8.739
1,800 (982.2)			0.0062	4.42	7.966
2,000 (1093.3)		0.0001		4.07	7.320
2,200 (1204.4)	0.0001	0.0018		3.76	6.768
2,400 (1315.6)	0.003	0.032		3.50	6.294
2,600 (1426.7)	0.034			3.27	5.883

Table XII. Recrystallization Behavior of FS-82 (From Creep Rupture and Heat Treated Specimens)

TEMP.	TIME AT TEMP. (HRS.)	RECRYSTALLIZED (%)	$T(20+\log t)10^3$
1,800°F	226	5	50.6
2,000°F	4.2	25	50.8
2,000°F	10.9	45	51.8
2,000°F	34.2	80	52.9

Table XIII. Bend Ductility of Columbium FS-82 Alloy Base Metal and Fusion Welds (1T Bend Radius)

SAMPLE	BEND	BEND ANGLE
Base Metal as received	⊥ to longitudinal sheet direction	180° - No cracking
E. B. Weld	Along weld direction	180° - No cracking
TIG Weld	Along weld direction	100° - A few cracks appeared
TIG Weld	Across weld direction	100° - A few cracks appeared

Table 14. Tensile Properties of FS-82 Columbium Fusion Weldments[1] (Heated to Temperature in 30 Min)

MATERIAL	TEMPERATURE (°F)	ULTIMATE TENSILE STRENGTH (KSI)	ELONGATION IN 2 INCHES (%)	POSITION OF FRACTURE	WELD EFFICIENCY (%)
Tig weld	75	72.3	---	heat affected zone	
Tig weld	75	77.7	---	heat affected zone	
ave		76.1			94.3
Tig weld	600	58.9	3.2	heat affected zone	
Tig weld	600	65.9	3.0	heat affected zone	
Tig weld	600	57.7	4.6	parent metal	
Tig weld	600	62.1	3.1	heat affected zone	
ave		61.1	3.5		82.5
Tig weld	1,200	64.1	5.3	weld	
Tig weld	1,200	63.9	3.7	heat affected zone	
Tig weld	1,200	60.8	3.7	parent metal	
		62.9	4.2		76.3
Tig weld	1,600	55.2	3.0	heat affected zone	
Tig weld	1,600	51.0	4.5	heat affected zone	
		53.1	3.8		74.3
Tig weld	1,800	45.6	5.5	heat affected zone	
Tig weld	1,800	48.3	5.3	heat affected zone	
Tig weld	1,800	47.7	5.0	heat affected zone	
		47.2	5.3		75.5
Electron beam weld	75	51.6	---	heat affected zone	64.0
Electron beam weld	1,600	46.1	5.3	weld	64.5
Electron beam weld	1,800	36.5	4.7	heat affected zone	53.5

1. All testing carried out in Instron Tester-Vacuum 5×10^{-4} mm Hg.

APPENDIX

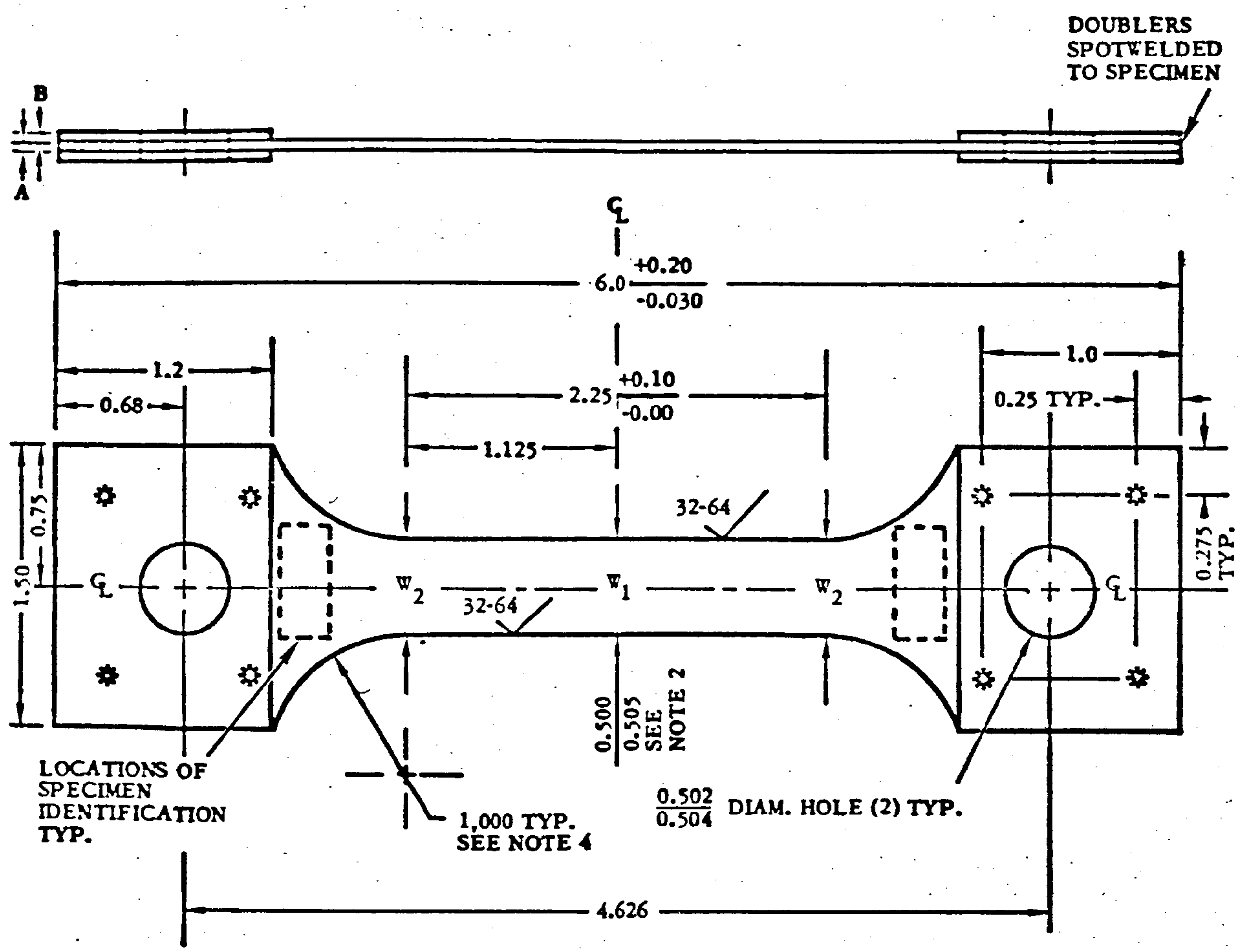


Figure A-1. Test coupon for vacuum creep rupture tests.

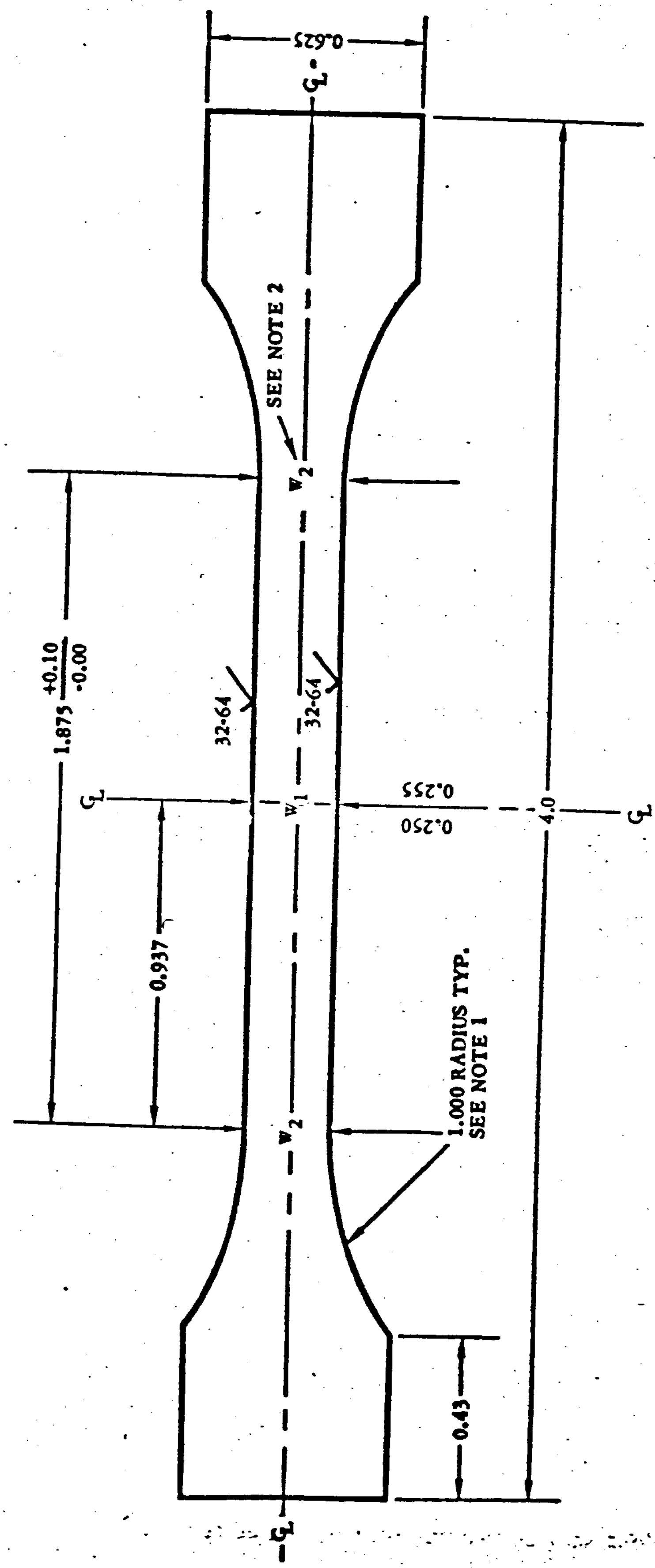


Figure A-2. Test coupon for Instron vacuum tensile test of sheet material.

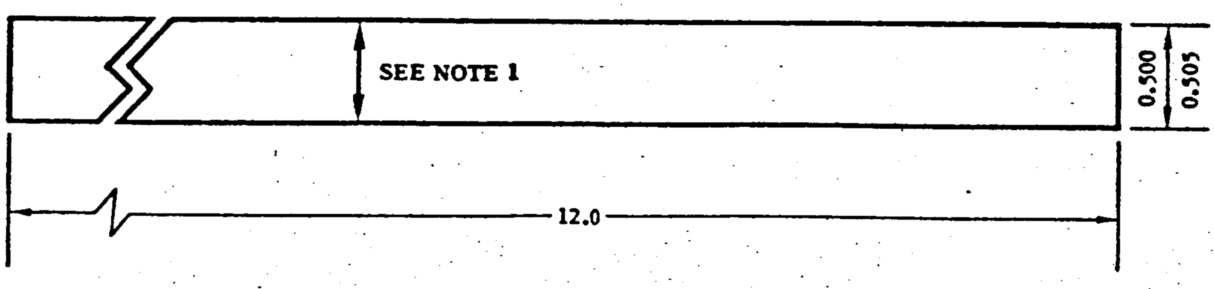


Figure A-3. Test coupon for resistance heated tensile test of sheet materials.

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Table I. Properties of Unalloyed M…

METALS	MACHINING	CUTTING
Molybdenum	Lathe turning, planing, milling, boring, drilling and grinding may be readily accomplished through the use of proper coolants & machining speeds. It is similar to steel of Rc 30 in machinability. In general, however, operations should be conducted at a slower rate.	Sawing can be accomplis… cial equipment. Shearin… er heating for various … Cutting rates are compar… used in cutting A-286 or …
Columbium	Lathe turning, planing, milling, and boring may be performed with high speed tools if light cuts are taken and a suitable lubricant used. Cb has a tendency to gall during machining. Drilling & grinding are also readily accomplished.	Sawing tends to flow Cb … gum up. Shearing easily … tools are sharp. May be …
Tungsten	Tool machining of tungsten very difficult. Grinding is satisfactory.	Tungsten can be cut best … cutoff wheels. Saw cutti… difficult. Shearing mus…
Tantalum	Lathe turning, planing, drilling, milling and boring can be performed without particular difficulty, providing its tendency to gall and tear is recognized. In lathe operation high cutting speeds using high-speed tools are preferred.	Sawing tends to flow Ta … gum up. Shearing easily … are sharp. May be done …

	FORMABILITY	FORGING AND EXTRUDING
ed on commer- requires prop- icknesses. ıble with those 9-9DL.	Cold forming should generally be avoided. In bending a slow rate of deformation should be observed. In general must be formed at temp.	Swaging & hammer forging can be done by conventional methods, but not much deformation is possible in one heat due to rapid cooling. Mo. is more difficult to forge than Ti. Forging temp. is betweeen 2,200 & 2,450°F. Mo. extruding must be done at high temperatures & speeds.
th saw and lone if lone at R. T.	Bending of Cb is as easy as bending of copper & aluminum. It does not work-harden rapidly & can be cold rolled & formed readily.	Die & hand forgings, rolled rings & extrusions have been successfully produced. Forging temp. is below 1,200°F. The forging of Cb is comparable to that of Ti.
rith abrasive ; possible but e done hot.	Hot forming necessary. Heavy sections may require almost a red heat for forming.	Extrusion is entirely feasible. Forging may be accomplished at temperatures in the range 2,400-2,800°F.
th saw and lone if tools t R. T.	Bending and twisting can be conducted at R. T. and in some cases using a soapy lubricant. Heavy sections may be more readily formed by heating, not to exceed 500°F.	Ta may be forged at room temp. It is difficult to extrude due to work hardening. The forging and machining process compares to that of Inconel.

Table I. Continued

METALS	MECHANICAL JOINTS	BRAZING
Molybdenum	Mechanical joints are the easiest joints to make with Mo. Readily accomplished by most common techniques. Hot riveting is generally done with Mo. rivets.	Brazing of molybdenum for low tempera- ture service is readily accomplished with available alloys. For service above 2,000°F special alloys are needed which must be tailored to the particular time- temp. conditions.
Columbium	Due to extreme ductility of Cb, mechanical joints are readily accomplished by most common techniques.	Very little work on brazing columbium for high temp. service has been done. Development work is needed for these applications.
Tungsten	Mechanical joints are the easiest joints to make with W. Readily accomplished by most common techniques. Hot riveting is generally done with Mo. rivets.	Brazing of tungsten for low temp. service is readily accomplished with available alloys. For service above 2,000°F special alloys are needed which must be tailored to the particular time-temp. conditions.
Tantalum	Due to extreme ductility of Ta, mechanical joints are readily accomplished by most common techniques.	

nued

	WELDING	OXIDATION RESISTANCE
pera- d with e which me-	Fusion welding has been done successfully utilizing stringent controls. Spot & seam welding is difficult to attain, owing to the general brittleness.	Very poor oxidation resistance above 1,000° F in oxidizing atmospheres except for very short time service. In slow moving air at 1,800° F the surface of Mo. will recede at the rate of 0.02 to 0.05 in./hr.
um ie. se	Welding may be conducted in chamber or air. It is necessary that Cb be extremely well cleaned on the surface. Welding of Cb is conducted in the same manner as for Ta. Cb is much easier to weld than Mo. Cb welding is similar to that of welding Ti.	Columbium oxidizes at about 1/10 the rate of Mo. The oxide is nonvolatile & melts at about 2,700° F. Cb is hardened by inward diffusion of oxygen from the scale.
h e	Fusion welding not possible for W structural applications.	Tungsten oxidizes at about one fifth the rate of Mo. in the range 2,000-2,500° F. It forms a volatile oxide at about 2,200° F.
	Welding must be done in the absence of all reactive gases. Spot and seam welding can be done. Seam welding is usually done under water to prevent oxidation of the metal. Helium or argon inert-gas arc welding processes are satisfactory.	While Ta has good corrosion resistance, like other refractory metals it needs high temp. protection against oxidation. At high temp., Ta is a little more oxidation resistant than Mo.

METALS	COATINGS	
Molybdenum	A large amount of work has been done on coatings & claddings for Mo. Excellent coatings have been developed for static oxidation protection up to 2,000-2,200°F. Coatings for cyclic service must be selected after a full consideration of the conditions involved.	Ingot Billet Bar Wire Sheet Plate Forging Tubing
Columbium	Coating of columbiumwill profit from coating studies on Mo. Here, again, coatings must be developed to fit the particular application under consideration.	Ingot Billet Bar Wire Sheet Plate Tubing
Tungsten	Little work has been done in the coating of W. Previous work on Mo. should be of aid to W.	Ingot* Billet Bar Wire Sheet
Tantalum	Ceramic coatings, alumina, zirconia. The method of coating that proved to be most practical was the flame ceramics process, which is only good for short times.	Ingot Billet Bar Wire Sheet Plate Tubing

*All powder metallurgy products.

le I. Continued

AVAILABLE FORMS AND SIZES	COST		
12 in. dia. 2,500 lb.	Powder form		
6 in. dia. 1,800 lb.	Powder	$ 3	to $ 4/lb.
4-1/2 in. dia. 250 lb.	Bar Stock	$ 7	to $ 12/lb.
0.001 in. x 3,000 ft.	Plate	$ 8	to $ 20/lb.
0.062 in. x 36 in. x 120 in.	Sheet	$10	to $ 40/lb.
0.002 in. x 14 in. x 72 in.	Wire	$10	to $600/lb.
1-1/2 in. x 8 in. x 30 in.	Foil	$25	to $200/lb.
1/2 in. x 12 in. x 36 in.	Arc cast form		
180 lb. per piece	Extrusion	$ 9.60	to $ 12/lb.
6 in. dia. x 5 ft. and various walls	Bar Stock	$12	to $ 17/lb.
	Forging Billet	$10	to $ 12/lb.
	Plate	$18	to $ 35/lb.
	Sheet	$35	to $ 65/lb.
	Foil	$25	to $200/lb.
	Wire	$20	to $600/lb.
1-1/2 in. sq x 24 in.	High-purity powder	$30	to $ 50/lb.
Same	Sheet	$75	to $110/lb.
To 1 in. x 5 ft.	Wire	$75	to $100/lb.
0.001 in. x 3,000 ft.	Foil	$75	to $175/lb.
0.001 in. x 2 in. x 6 in. (min.)			
1/4 in. x 8 in. x 24 in.			
1/4 in. x 5 in. x 48 in.			
Various sizes			
	Powder	$ 3	to $ 4/lb.
Special order	Bar Stock	$10	to $ 11/lb.
1/8 - 1 in. RD	Wire	$10	to $300/lb.
to 0.3 mils	Sheet	$10	to $100/lb.
0.062 in. x 12 x 36			
0.005 in. x 3 x 12			
1-1/2 in. sq. x 24 in.	Tantalum powder	$40	to $ 60/lb.
	Sheet	$50	to $ 68/lb.
to 1 in. x 5 ft.	Bar	$58	to $ 65/lb.
0.001 in. x 3,000 ft.	Wire	$65	to $ 83/lb.
0.001 in. x 2 in. x 6 in. (Min.)	Strip	$59	to $ 70/lb.
1/4 in. x 8 in. x 24 in.	Forging	$45	to $ 65/lb.
1/4 in. x 5 in. x 48 in.			
Various sizes			

Table III. Mechani...

COLUMB...

TEMPERATURE °F	ULTIMATE TENSILE STRENGTH, Ksi	YIELD STRENGTH 0.2% OFFSET	ELONGATI... %
RT	70.0[a]		11.0
2,000	44.7[a]	40.3[a]	
2,400	11.6[a]		19.0

a. Reference 5. Tested on Argon. Stress relieved condition, 0.040 sheet

b. Reference 5. Tested on Argon. Stress relieved condition, swaged rod

RT[c]	62.5	56.0	7.5
800	61.8	61.5	6.2
1,200	61.7	57.5	5.0
1,600	54.1	51.5	3.7
1,800	42.5	16.3	7.5
2,000	28.6	16.2	15.0
2,200	19.9	13.9	21.2

c. All data from reference 8. Tested in Vacuum. Stress relieved condition, 0.020 sheet

RT[d]	81.6	69.7	11.3
1,000	77.9	69.2	3.4
1,600	64.7	52.1	7.1
2,200	22.1	15.9	28.0
2,500	11.9	10.9	53.0
2,000[e]			

...Y

MODULES OF ELASTICITY X10^6	STRESS TO RUPTURE IN 10 HRS.	100 HRS.	500 HRS.	DUCTILE TO BRITTLE TRANSITION TEMPERATURE
				Slow bend (IT) < 70°F Tensile (.005 in./in./min.) < - 70°F
	26,500[b]	17,500[b]	13,500[b]	From reference 7 and reference 9
	15,000	10,000	8,600	
16.5				
16.0				
	13,000			

Table III. Mec[illegible]

TEMPERATURE °F	ULTIMATE TENSILE STRENGTH, Ksi	YIELD STRENGTH 0.2% OFFSET	ELONG[illegible] %
			COLU[illegible]
RT	70.0[a]		11.0
2,000	44.7[a]	40.3[a]	
2,400	11.6[a]		19.0
a. Reference 5. Tested on Argon. Stress relieved condition, 0.040 sheet			
b. Reference 5. Tested on Argon. Stress relieved condition, swaged rod			
RT[c]	62.5	56.0	7.5
800	61.8	61.5	6.2
1,200	61.7	57.5	5.0
1,600	54.1	51.5	3.7
1,800	42.5	16.3	7.5
2,000	28.6	16.2	15.0
2,200	19.9	13.9	21.2
c. All data from reference 8. Tested in Vacuum. Stress relieved condition, 0.020 sheet			
RT[d]	81.6	69.7	11.3
1,000	77.9	69.2	3.4
1,600	64.7	52.1	7.1
2,200	22.1	15.9	28.0
2,500	11.9	10.9	53.0
2,000[e]			

:al Properties of FS-82

IUM ALLOY

'ION	MODULES OF ELASTICITY $X10^6$	STRESS TO RUPTURE IN 10 HRS.	100 HRS.	500 HRS.	DUCTILE TO BRITTLE TRANSITION TEMPERATURE
					Slow bend (IT) < 70°F Tensile (.005 in./in./min.) < - 70°F
		26,500[b]	17,500[b]	13,500[b]	From reference 7 and reference 9
		15,000	10,000	8,600	
	16.5				
	16.0				
		13,000			

Table III. C...

COLUMBIU...

TEMPERATURE °F	ULTIMATE TENSILE STRENGTH, Ksi	YIELD STRENGTH 0.2% OFFSET	ELONGATION %
d. All data from reference 9. Tested in Vacuum. Stress relieved condition, 0.020 sheet			
e. Rupture data from reference 9. Tested in Vacuum. Stress relieved condition, 0.070 sheet			
RT[f]			
2,500			
f. From reference 9. Tested in vacuum. Stress relieved condition, 0.020 sheet			
RT Bare			
RT Cr Plated			

ntinued

ALLOY

MODULES OF ELASTICITY X10^6	STRESS TO RUPTURE IN 10 HRS.	100 HRS.	500 HRS.	DUCTILE TO BRITTLE TRANSITION TEMPERATURE
				Shear strength, Ksi 74.8 7.9
				Riveted joint ultimate strength, Ksi 81.0 74.0

Table V. Spot-Welding Schedule For Cb-33Ta-0.7Zr (FS-[illegible])

SHEET THICKNESS COMBINATION, (IN.)	ELECTRODE TYPE	ELECTRODE DIAMETER (IN.)	WELD FORCE (LB.)	WELD CURRENT (AMP.)	(C
0.010-0.025	M28	3/32	150	6,100	
	M28	3/32	150	6,100	
	M100	3/32	250	7,770	
0.025-0.025	M28	3/32	2,200	25,500	
0.010-0.010-0.025	M100	3/32	250	8,880	
0.010-0.070	M100	1/8	450	9,100	
0.070-0.070	M100	1/4	2,200	27,100	

2) Alloy (From General Electric, Reference 8)

HEAT TIME (CYCLES)	UP-SLOPE TIME (CYCLES)	DOWN-SLOPE TIME (CYCLES)	HOLD TIME (CYCLES)	TENSILE SHEAR STRENGTH, (LB.)
6	2	3	60	150
3	0	0	60	-
2	1	0	60	-
4	2	0	60	900-1,000
2	1	0	60	195
6	2	0	3	-
6	2	2	3	2,700

Table VII. Tensile Properties[a] of The Columbium Alloy

TEST TEMP. °F	ULTIMATE TENSILE STRENGTH (Ksi)[b]	0.2% OFFSET YIELD STRENGTH (Ksi)[c]
75	83.7	67.7
75	90.7	72.7
Ave	87.2	70.2
600	69.0	57.4
600	71.2	61.9
Ave	70.1	59.7
1,000	71.6	61.3
1,000	66.0	65.0
Ave	68.8	63.1
1,600	65.3	54.0
1,600	62.9	54.0
Ave	64.1	54.0
1,800	63.2	49.0
2,000	60.5	30.7
2,000	50.0	39.5
Ave	55.3	35.1
2,200	40.5	34.0
2,200	41.2	31.9
Ave	40.8	33.0
2,400	21.8	11.9
2,400	28.0	21.9
2,600	23.4	12.6
2,600	22.6	13.8
Ave	23.0	13.2

a - Tests carried out in flowing argon atmosphere at elevated temperature. All tests are longitudinal.
b - Strain rate to yield 0.003 - 0.005 in./in./min.
c - Strain rate after yield to fracture 0.02 - 0.04 in./in./min.

'S-82 (Stress Relieved 1 Hr. at 1,900°F)

MODULES OF ELASTICITY ($X10^6$ psi)	KNOOP HARDNESS SURVEY ACROSS THICKNESS AFTER TESTING (1.0 Kg LOAD) EDGE		CENTER		EDGE
18.8	180	184	182	186	183
16.4	183	181	186	180	181
17.6					
16.1	191	200	200	201	193
14.9	193	196	197	198	184
15.5					
16.1	171	196	194	190	194
14.4	191	196	197	196	158
15.3					
13.7	198	196	193	189	197
14.2	306	193	186	191	292
14.0					
12.2	261	197	197	191	372
7.5	367	196	205	236	416
12.1	232	185	197	188	290
9.8					
9.7	311	181	175	176	284
7.9	331	182	186	184	362
8.8					
8.1	214	160	154	162	194
7.0	479	253	211	333	512
2.6	512	320	261	362	500
5.7	305	177	166	162	281
4.1					

Table X. Rupture and Creep Properties of Columbium FS-

MATERIAL	TEMPERATURE (°F)	STRESS (psi)	TIME TO FRACTURE (HR.)	CREEP RATE (IN./IN./HR.)	ELONGA (%)
As received base metal	1,800	50,000	1.4	0.0280	3.0
As received base metal	1,800	40,000	9.3	0.0064	12.5
As received base metal	1,800	35,000	14.8	0.0044	16.8
As received base metal	2,000	23,000	7.7	0.0128	18.8
As received base metal	2,000	18,000	32.2	0.0042	26.0
As received base metal	2,200	13,000	14.5	0.0072	27.0
As received base metal	2,200	13,000	2.9	0.0290	17.5
As received base metal	2,200*	13,000	74	-	20.0
As received	2,200	13,000	23.2	0.0022	-
TIG fusion welds	1,800	40,000	0.08		<1.0
TIG fusion welds	1,800	35,000	9.9		2.7
TIG fusion welds	2,000	25,000	8.9		2.5
TIG fusion welds	2,000	25,000	2.2		17.0

* Vacuum lost during test.

-82 (Tested in Vacuum @ 0.5μ).

ATION	TIME TO REACH GIVEN DEFORMATION HR. (0.5	1.0	2.0	5.0)	KNOOP HARDNESS SURVEY ACROSS THICKNESS AFTER TESTING 1.0 Kg LOAD (EDGE)		(CENTER)		(EDGE)
	-	0.25	0.5	1.2	184	214	197	200	208
	0.6	1.2	2.7	7.2	192	188	192	188	173
	0.6	1.0	3.3	9.8	144	174	168	173	174
	-	0.3	1.1	5.1	150	154	151	156	147
	0.6	1.6	4.0	11.0	122	141	139	140	123
	-	0.5	1.6	5.9	158	157	175	160	151
	-	-	0.5	1.5	128	123	124	124	146
	-	-	-	-	351	378	356	383	351
	-	0.8	2.8	13.4					
	Position of fracture								
	Heat affected zone				291	253	232	244	290
	Heat affected zone				206	239	234	226	228
	Heat affected zone				427	400	387	427	428
	Through base metal				210	183	184	190	182

MATERIAL	TEMPERATURE (°F)	STRESS (psi)	TIME TO FRACTURE (HR.)	CREEP (IN./IN.
T1G fusion welds	2,200	13,000	36.9	
Electron beam	2,000	25,000	0.6	
Fusion welds	2,200	13,000	25.8	

X. Continued

ELONGATION (%)	TIME TO REACH GIVEN DEFORMATION HR. (0.5 1.0 2.0 5.0)	KNOOP HARDNESS SURVEY ACROSS THICKNESS AFTER TESTING 1.0 Kg LOAD (EDGE)		(CENTER)		(EDGE)
6.5	Through weld	369	383	385	350	359
2.5	Through weld	310	225	152	261	345
2.5	Through base metal	372	376	369	362	374