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Title FATIGUE OF THE ALLOY COLUMBIUM (NIOBIUM) - ONE PERCENT ZIRCONIUM

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This paper is a study of the fatigue properties of the alloy columbium - one percent zirconium in reverse bending. This paper also is a design study of a satisfactory plate reverse bending fatigue specimen configuration for use with the General Electric PRB pneumatic testing machine. Selected tensile tests were also run on columbium - one percent zirconium to provide reference strength data with which to compare the results of the fatigue tests.

The fatigue tests showed the fatigue strength of columbium - one percent zirconium to be higher than that of pure columbium. No endurance limit was found for the alloy in the range of testing from 10^5 to 10^7 cycles of reverse bending. A successful reverse bending fatigue specimen was designed with a triangular constant stress region where failure occurred. Generous fillets prevented unacceptable fatigue failure in the machine grips.

FATIGUE OF THE ALLOY COLUMBIUM (NIOBIUM) - ONE PERCENT ZIRCONIUM

by

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FATIGUE OF THE ALLOY COLUMBIUM (NIOBIUM) - ONE PERCENT ZIRCONIUM

INTRODUCTION

Columbium

Columbium is the name generally used in the United States to refer to the metal of atomic number 41. Niobium was chosen as the international name by the Fifteen International Union of Chemistry Congress. It is one-third as abundant as copper in the earth's crust. It is a refractory metal with high strength at elevated temperatures and very good corrosion resistance when compared to other refractory metals. The metal is resistant to corrosion in most aqueous media and has a low cross-section for thermal neutrons. These properties are responsible for the increasing usage of columbium in the nuclear and high temperature structural fields. Coated columbium is used for high temperature skins of re-entry vehicles and rocket nozzle cases. It is also used as a structural material in high temperature reactor cores, and as jet engine turbine blades.

Fabrication is difficult due to its tendency to gall most tooling materials. Special lubricants and die materials are necessary to permit forming and machining of columbium. Difficulty in machining and high cost are major drawbacks to the use of the material.

Columbium and its alloys have been the focus of much research in recent years and many data are available as to its properties. Properties such as density, crystal structure, modulus of elasticity, melting point, creep, stress rupture strength, tensile strength and corrosion characteristics are well known for columbium and its commercial alloys.

Prior Work

Very little work has been done on the fatigue properties of columbium and none on the alloy columium-one percent zirconium. This lack of fatigue data has been a source of uncertainty in designs using columbium alloys subject to cyclical stresses. Such cyclic stresses occur in missile skins due to vibration and in nuclear cores due to fluid flow vibration. The only fatigue information available for columbium or its alloys is Begley's fatigue study of powder-metallurgy pure columbium sheet (Ref. 5). The results of his study, shown in Figure 1, are taken from the Defence Metals Information Center report on columbium and its alloys (9, p. 32).

Purpose

The purpose of this investigation is to determine the fatigue properties of the alloy columbium-one percent zirconium and the effect of alloying pure columbium on its fatigue characteristics, by comparison with prior work on pure columbium.

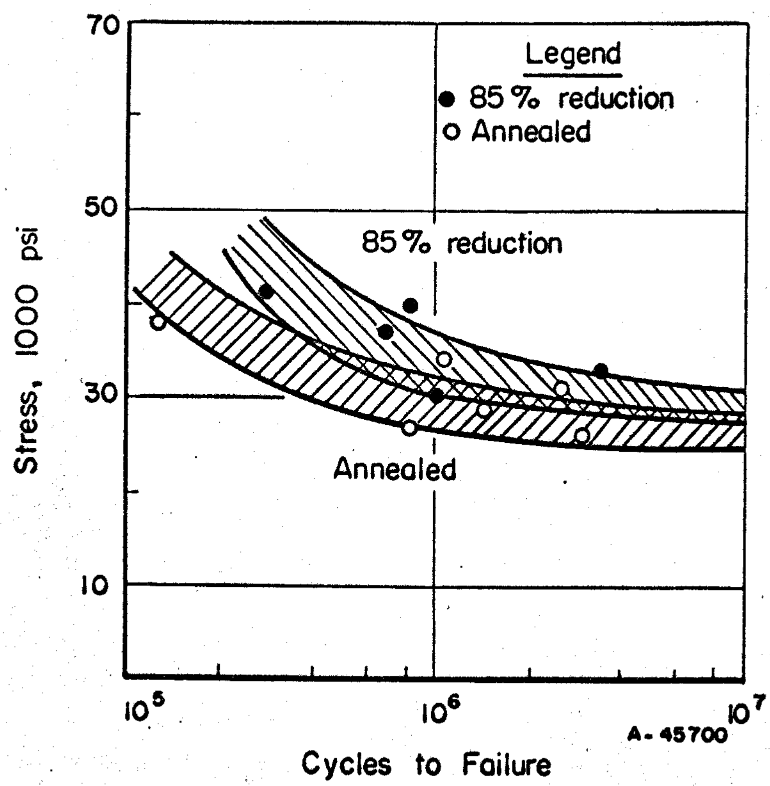


Figure 1. Fatigue characteristics of cold-worked and annealed powder-metallurgy columbium (9, p. 32).

This investigation, by its nature, also required the development of a satisfactory reverse bending fatigue specimen for use with the General Electric PRB pneumatic fatigue testing machine.

THEORY AND BACKGROUND

Metal fatigue failure is the process by which metals, subjected to cyclical stresses below the static strength of the metal fail by progressive fracture of a brittle nature. This failure of the metal occurs though the stress is far below its static strength. In modern design of highly stressed moving parts this type of failure will predominate and determine the service life of the part.

Fatigue strength is usually determined by the use of an S-N curve, which is a curve showing the number of cycles before failure at each maximum cyclical stress level. The nature of fatigue failure causes S-N curves to show a large amount of scatter, which indicates that fatigue strength can only be represented as a statistical function of the stress spectrum. Calculations based on fatigue data are not as reliable as standard static strength calculations and must be viewed as statistical probabilities.

When cyclical stress fluctuations are imposed on components such as shafts, springs or turbine blades, stresses far below the maximum static strength can lead to complete rupture. The metal withstands the stress for a time and appears unimpaired by visual observation, but at a later time microscopic cracks appear and spread during subsequent loadings (10, p. 40). Finally, after the crack has reached sufficient size, a sudden complete rupture may occur. Even in the most ductile of metals, the fracture resembles that of a brittle metal because there is no observable plastic distortion of the part as a whole.

About a century ago it was thought that the metal had "crystallized" or become embrittled due to the repeated loadings. Metallurgists have since shown that all solid metals are crystalline and that no extensive recrystallization or grain growth occurs as the result of repeated stressing. The explanation for the apparent difference in behavior under repeated loading lies in the nature of initial fatigue damage and in the accumulative or progressive nature of the damage developing under each repetition of loading. Visible fatigue cracks are a result of prior damage on a submicroscopic scale and the whole process of fatigue is more aptly referred to as progressive fracture (10, p. 41).

Metals are made up essentially of crystals (or grains), a crystal being a structure made up of an orderly arrangement of atoms. Examples of simple atomic arrangements are the face-centered cubic, as found in aluminum or copper, and the body-centered cubic, as found in α iron, tungsten, chromium, or molybdenum. In a metal crystal the basic unit is joined to similar neighbors and repeated throughout the extent of the crystal, resulting in certain lines and planes of atoms in the atomic structure. Usually the crystals or grains are randomly oriented; that is, the directions of the rows of atoms in different grains make different angles with the axis of the piece. As load is increased on a single crystal specimen of ductile metal, slip eventually occurs in a plane of maximum atomic density and in a direction in that plane of maximum linear atomic density. There are 12 possible slip possibilities in the face-centered cubic crystal which result from four planes of maximum atomic density and the three directions of maximum linear atomic density in each such plane. Slip may be visualized as a shearing action, like the sliding of one card over another in a deck of cards. Under a microscope one sees "slip bands" also called "slip lines" corresponding to the steps in the surface (10, p. 70). Slip occurs in response to maximum resolved shear stress, independently of normal stress acting on the slip plane. Most of the basic concepts of slip at the atomic level have been built upon dislocation theory which visualizes slip within a crystal lattice as a step-by-step displacement of a dislocation or mismatch in the lattice (10, p. 43) until it appears on the surface as a "slip band." Thus far the theory is not sufficiently advanced to deal successfully with polycrystalline metals and the multitude of foreign atoms present in real materials, or with the complexities introduced by reversal and repetition of stress. Therefore, for the purposes of this discussion, attention will be directed mainly to observations of microscopic structural changes and of accompanying phenomenological behavior.

In a polycrystalline metal the crystals are randomly oriented. Certain crystals in a zone of high stress will be oriented so that the maximum resolved shear stress and a slip direction will nearly coincide. For an axial tension specimen the maximum shear stress occurs on all planes tangent to a 45 degree cone on the tensile axis. These planes on which the maximum shear stress occurs intersect the specimen surface in angles varying from 0 to 45 degrees as measured from the normal to the tensile axis (10, p. 76).

When a specimen is loaded with a slowly increasing static stress, fine localized "slip bands" occur within a few crystals and then become more numerous until practically all crystals are greatly fragmented and deformed. In contrast to this, repeated loading at stresses below the nominal yield strength of the material localized these effects in a few crystal zones of high stress concentration which will exhibit a small number of very heavy "slip bands." These heavy "slip bands" can develop separations or "cracks" very early in the fatigue life of the specimen.

Although the fatigue failure may start on shear planes, it is well known that most fatigue fractures follow the normal stress direction. In an axial tension member the separated "slip bands" will lie between 0° and 45° as measured from the normal to the tensile stress. If these randomly oriented microcracks join together along various adjacent randomly oriented crystals, the general path will tend, on the average, to go in a normal direction. The micro-cracks will not connect smoothly, but will have "mismatch regions" at the boundaries where the cracks will not join. The normal tensile stress may be a factor in breaking through these obstructions. The larger crack will then be opened and closed as a result of the cyclic normal stress. This results in increased stress concentration at the end of the gross crack, which tends to propagate the crack in the normal direction. Fatigue cracking is transcrystalline in ductile materials at normal temperatures. In the final stages the stress concentration at the crack tip exceeds the strength of the metal and rupture occurs.

The fatigue life of a specimen is affected by many factors, such as its stress spectrum, atomic arrangement, crystal arrangement, phase distribution, temperature, surface condition, environment and many other factors.

Stress concentrations such as notches and holes have less effect on the fatigue strength of a specimen than would be expected from the static stress concentration factors. Fatigue life estimates based on static stress concentration factors are thus conservative in nature and the strength-reduction factors as determined by fatigue tests are usually of the order of 10 to 20 percent lower than the static strength-reduction factors (10, p. 15). Stress concentrations due to surface roughness have a marked effect on the fatigue life of metals because usually the highest stresses occur in the fibers at the surface of a specimen in tension or torsion. A rough surface, particularly one containing markings perpendicular to the principle tensile stress, will have a considerably lower fatigue life than a smooth surface of the same material.

Since compressive stresses have a shear component and fatigue damage is due initially to shear stress, fatigue failure in compression is to be expected. Metals fail by fatigue in compression, but the fatigue life is of the order of ten times that in tension. Plastic action around a stress concentration can cause local yielding which will result in local tensile stresses when the compressive load is removed. Compressive fatigue failure might, therefore, be attributed to tension, but when one considers that all commercial metals contain internal microscopic stress concentrations, compressive fatigue failure becomes possible.

When two specimens are tested under identical cyclical stress loads and additional different mean tensile stresses are superimposed on each specimen, the specimen with the highest mean stress will produce failure in the shortest time. A mean stress can be superimposed over a fluctuating stress either by service loads or residual stresses. If the residual stresses are compressive the fatigue life of a specimen will be improved. Compressive stresses induced in surface fibers by cold working or shot peening improve the fatigue life of metals.

Frequency of stress cycling has a negligible effect on fatigue life up to the range of 5000 cpm for iron and steel and 7000 cpm for copper. Beyond this range higher frequency tends to increase fatigue life by a small percent. The effect of frequency is less than one percent for copper up to 20, 000 cpm (10, p. 25).

Microstructure has an important influencing role in fatigue. As the size of the crystals in a metal increase, the endurance limit is decreased. In two phase alloys the distribution of the phases has an important effect on the fatigue strength. The nature of the relationship is not yet quantitatively understood.

Almost all wrought materials exhibit directional characteristics in their mechanical properties. Fatigue strength is also affected by directional characteristics; usually being higher in the longitudinal direction and lower in the transverse rolling direction.

Internal defects affect the fatigue strength, but as most high stresses occur at the surface they are only important when they occur in moderate and high stress regions.

Corrosion can accelerate fatigue failure and the combination of corrosion and fatigue can cause failure many times faster than fatigue alone. Even in a normal atmosphere many materials exhibit lower fatigue strengths than under vacuum. Usually the short duration of testing tends to reduce atmospheric corrosion effects even in fairly active metals.

Temperature is an important variable in fatigue strength. Fatigue strength usually decreases gradually with increasing temperature and strongly affects the influence of rest periods on the fatigue strength. Since creep and fatigue damage can occur simultaneously, the total elapsed time affects the fatigue behavior as well as the number of stress cycles. Increased temperature does not decrease the severity of a stress raiser. All metals show improved fatigue properties at low temperatures since the notch sensitivity in fatigue does not show the drop typical of static notch sensitivity.

Surface coatings on a metal may affect the fatigue life. The effect is complex in that the coating application may make itself felt in any or all of a number of different ways. Cleaning prior to coating may either smooth or roughen the surface. The application of coatings may induce tensile or compressive stresses in the base metal and in the coating. The basic fatigue strength of the coating is important since fatigue cracks formed in the coating may propagate into the base metal. The relative Young's modulus of the coating and base metal are important in determining the ratio of the stresses in the coating to those in the base metal. Layers formed on the surface of metal by changing the chemical composition may have a large effect on fatigue strength. Two factors are responsible for the effect of these surface changes. The first is the change of the fatigue strength of the surface layer and the second is the effect of residual stresses induced by surface changes and heat treatment. Nitriding and carburizing are examples of surface changes that build up high residual stresses. Nitriding builds up compressive residual stress which improves fatigue strength. Carburizing can build up either tensile or compressive residual stresses.

Some metals, such as mild steel, can withstand an infinitely large number of stress cycles provided the stress is below a limiting stress, the endurance limit (7, p. 339). Other metals, such as aluminum, exhibit no endurance limit, but have a continual decrease in fatigue strength with increasing stress cycles. The fatigue properties of a metal can be compared to the tensile properties by the use of the endurance ratio which is the ratio of the endurance limit to the ultimate tensile strength. For nearly all metals the endurance limit approaches the yield strength as an upper value, but a few cases exist in which it exceeds the yield strength (10, p. 29). For annealed metals that exhibit an endurance limit, the endurance ratio is usually of the order of 50 percent. When cold working or heat treating is used to increase the ultimate tensile strength of these metals the endurance ratio usually decreases because the endurance limit is not improved as greatly as the tensile properties.

Since many factors affect the fatigue strength of a metal, careful design and preparation of test specimens is necessary to determine the true fatigue strength of the metal. Poor specimen design, preparation and testing result in the determination only of the fatigue strength of the specimen itself.

Specimens should be designed to prevent stress concentration and polished to eliminate the effect of surface defects on the fatigue strength. Testing procedure should be carefully checked to determine if it affects the fatigue strength.

MATERIALS AND APPARATUS

Materials

Two different alloys of columbium were furnished by the Wah Chang Corporation in Albany, Oregon and were received in a shipment of four pounds of columbium-ten percent hafnium-one percent zirconium (Wah Chang C-103) alloy plate, hereafter referred to by the identification letter A, and another shipment of 18 pounds of columbium-one percent zirconium plate, hereafter referred to by the identification letter B. The columbium plates were furnished from production runs and their chemical compositions are given in Table 1.

The specimens of columbium alloy A were cut from 0. 25-inch plate which had been consolidated by electron beam melting, extruded to sheet bar, recrystallized one hour at 2200 F, cold rolled at 800 F to 0. 25-inches and then pickled and recrystallized again for one hour at 2200 F. Rectangular specimens were machined to conform to Figure 2. This was a preliminary specimen design for use as a cantilever beam in the General Electric pneumatic fatigue tester. The machining was controlled so as not to overheat the metal by too rapid removal of material. The finish machined specimens of columbium alloy A were polished in a longitudinal direction with silicon carbide metallographic polishing papers from 120 mesh to 600 mesh

Table 1. Nominal chemical composition of columbium alloy plate.

	A Alloy C-103 (percent)	B Alloy Columbium-One Percent Zirconium (percent)
Hafnium	9-11	< 0.01
Titanium	0.7-1.3	< 0.05
Zirconium	0.7	0.8-1.2
Oxygen	< 0.03	< 0.03
Nitrogen	< 0.03	< 0.03
Hydrogen	< 0.002	< 0.002
Carbon	< 0.01	< 0.01
Columbium	balance	balance

grit. The specimens were then polished by hand with 600 mesh alundum powder and finish polished with Linde fine abrasive powder of one-half micron diameter. Polishing removed all visible defects and longitudinal polishing assured that no diagonal defects would be present (3, p. 35).

The specimens of columbium alloy B were cut from 0.35-inch plate which had been consolidated by electron beam melting, extruded to sheet bar, recrystallized one hour at 2200 F, cold rolled at 800 F to 0.35-inches and then pickled and recrystallized again for one hour at 2200 F. All specimens were originally cut to conform to Figure 3.

The two specimens of alloy B which were tested in the configuration of Figure 3 failed at the grips. The remainder of the specimens were remachined to conform to Figure 4. The fatigue specimens shown in Figures 3 and 4 were designed to conform to the suggested configuration of cantilever plate fatigue specimens (4, p. 31). The tapered section of the specimen is a region of constant stress since the section modulus increases in the same proportion as the applied moment (12, p. 210). Generous fillets were provided in the specimens to avoid stress concentrations. The specimen of Figure 4 was chosen as the final specimen design because the increased width of the specimen at the grips of the machine reduced the stress concentration at the grips and all specimens of this design failed in the constant stress tapered section of the specimen. Specimen designs of Figures 2 and 3 caused fatigue failure to occur at the grips where stress concentrations and fretting caused the actual stresses to be indeterminate.

The machining and polishing, of the specimens of columbium alloy B, was controlled so as not to heat the metal by too rapid removal of metal. The finish machined specimens of columbium alloy B were polished in a longitudinal direction with silicon carbide metallographic polishing papers ranging in grit from 80 mesh to 600 mesh. The specimens were then hand polished with 600 mesh alundum powder and finish polished with Linde fine abrasive powder of one-half

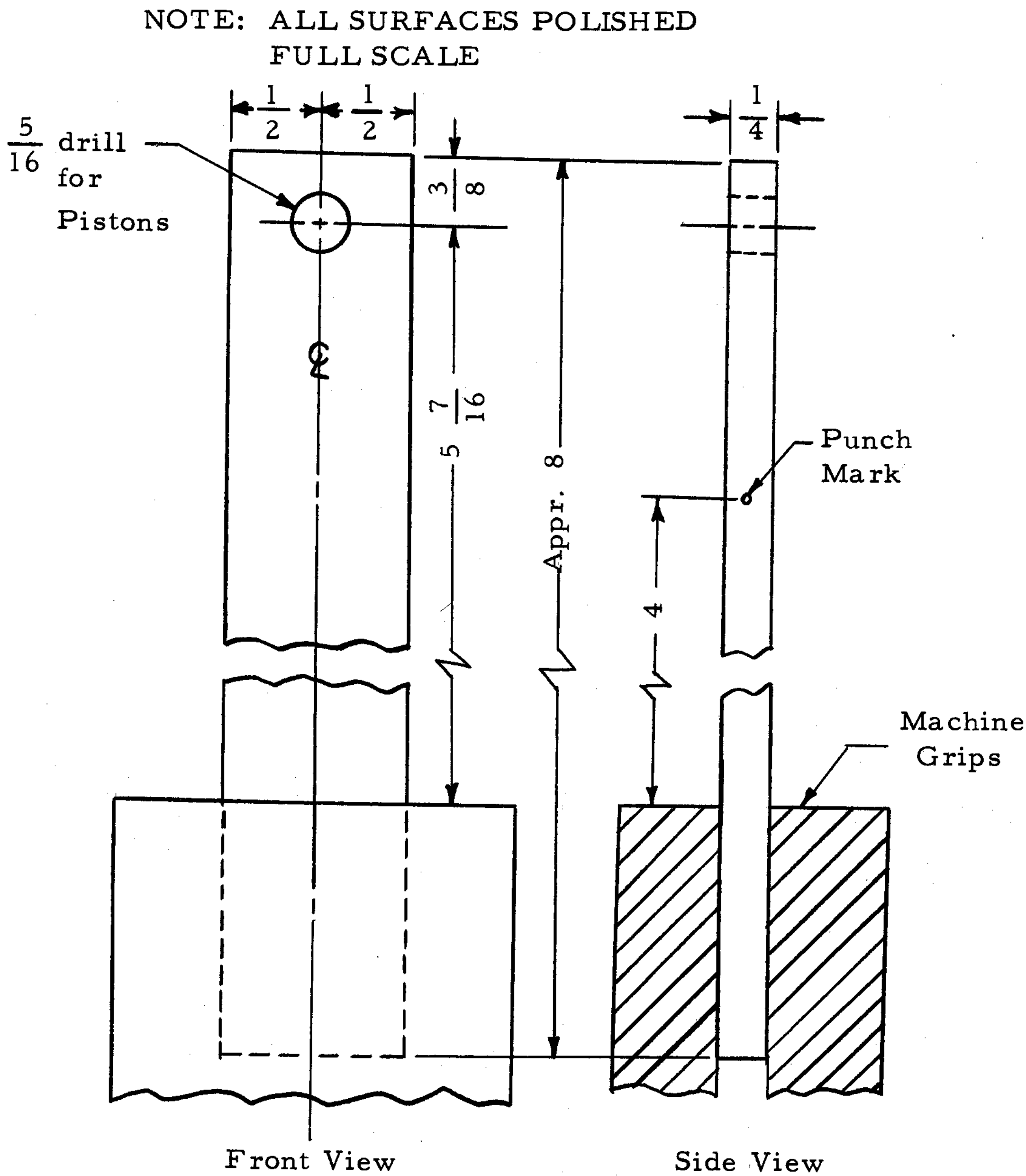


Figure 2. Rectangular cantilever fatigue specimen and specimen mounting.

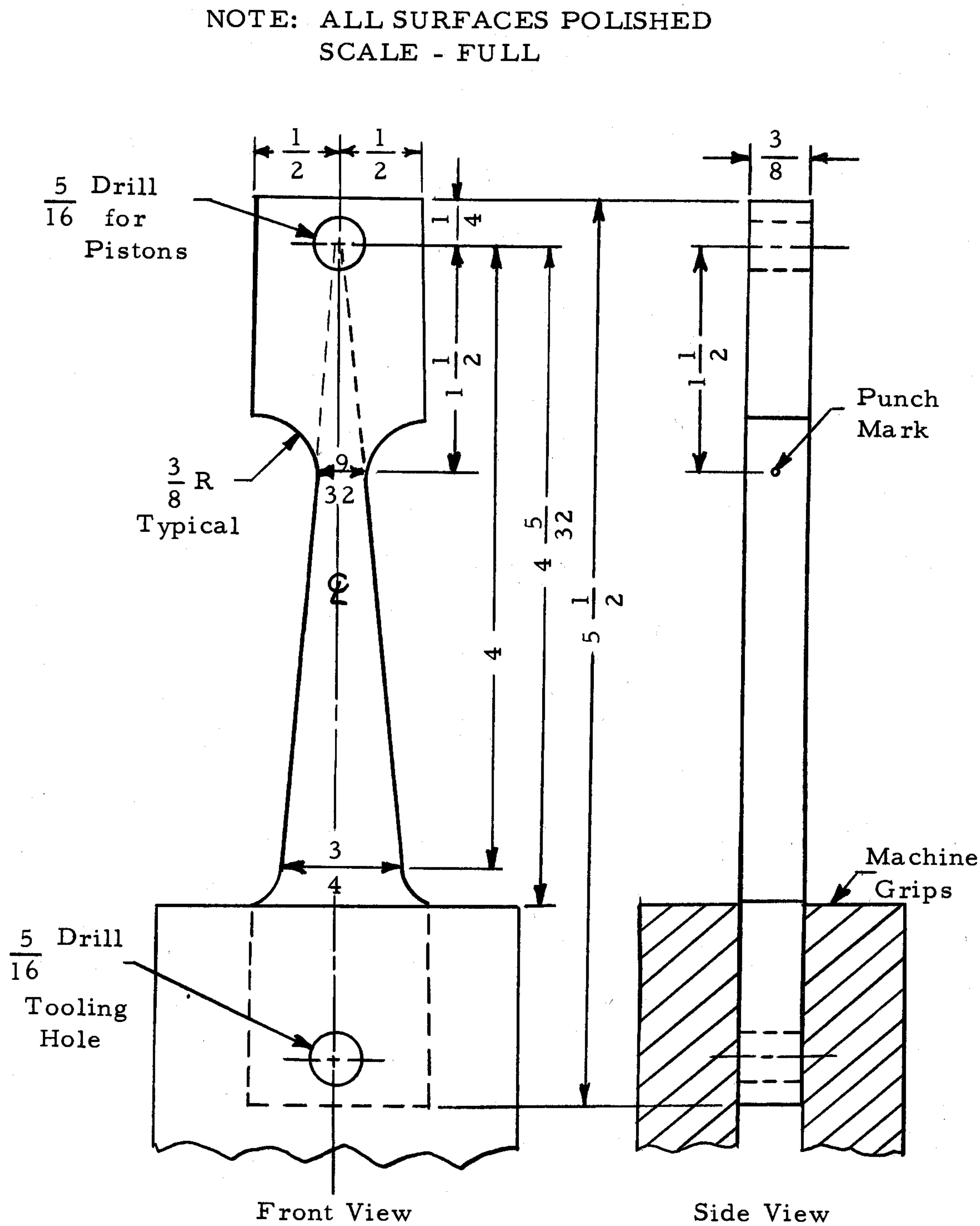


Figure 3. Constant stress cantilever fatigue specimen and mounting.

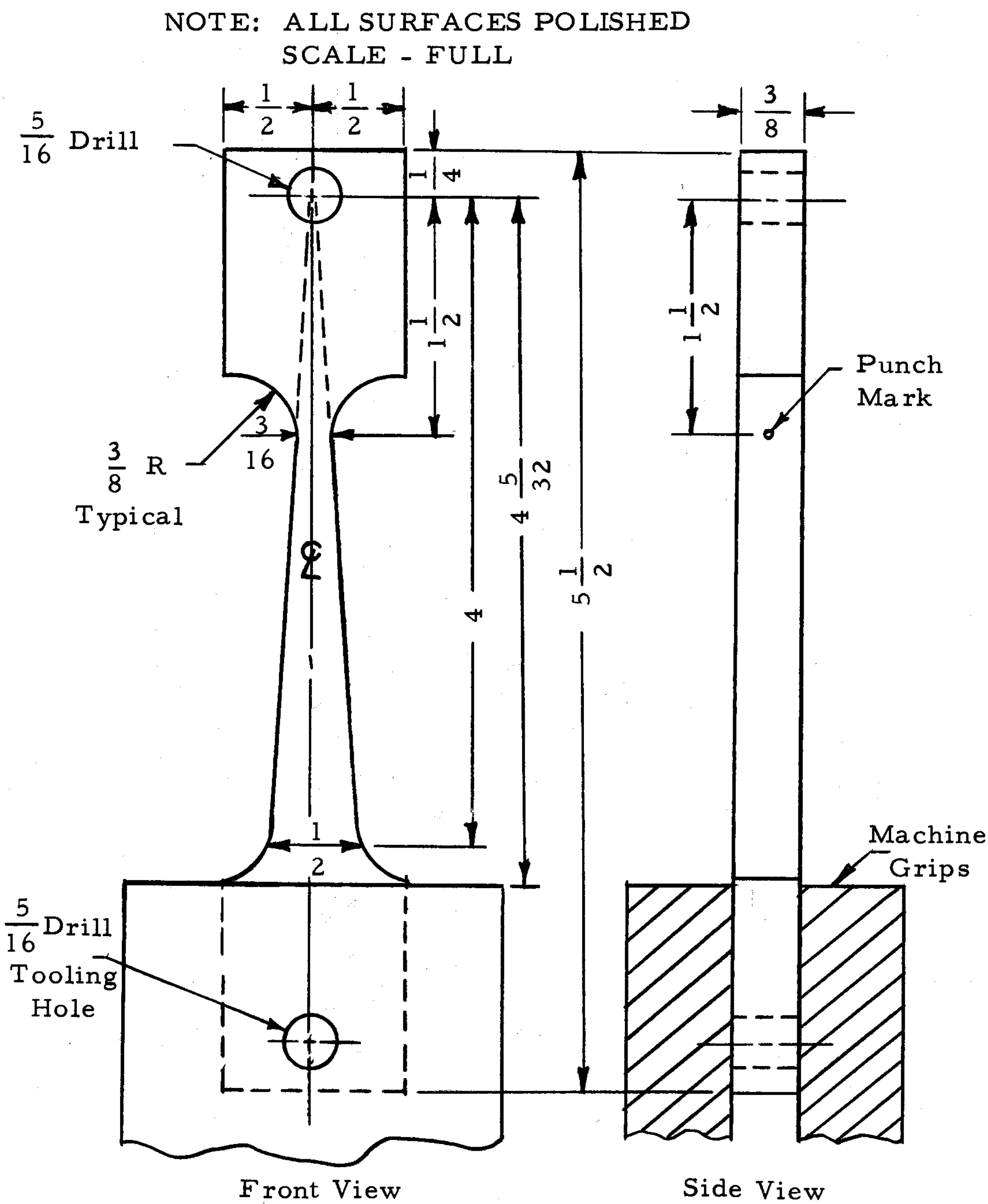
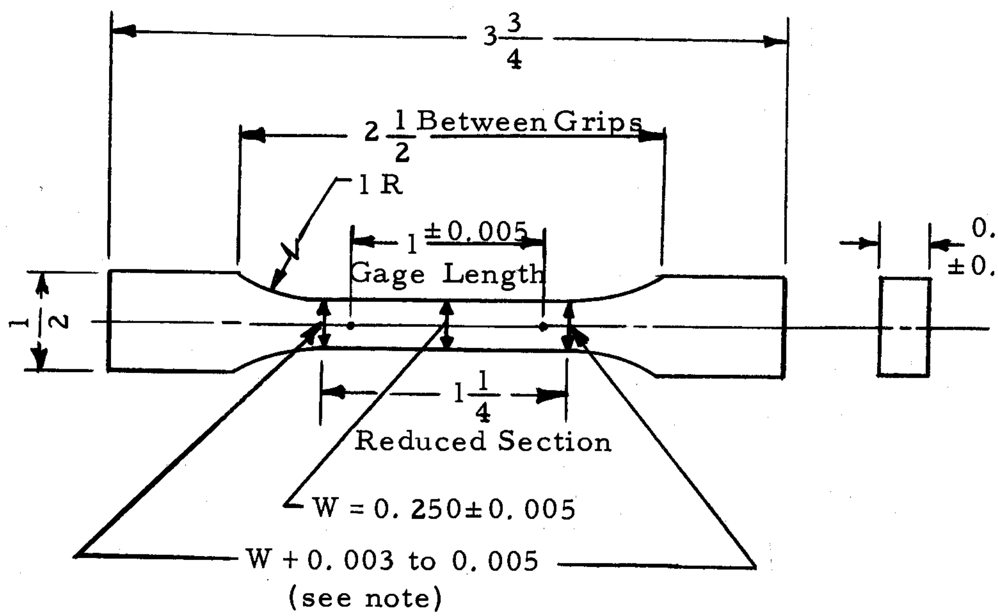


Figure 4. Constant stress cantilever fatigue specimen and mounting.



NOTE: GRADUAL TAPER FROM ENDS OF REDUCED SECTION TO MIDDLE.

Figure 5. Rectangular tension test specimen with one-inch gage length.

micron diameter.

All specimens of columbium alloys A and B were cut in the rolling direction and all polishing was done in a longitudinal or rolling direction on the specimens. This procedure eliminates transverse scratches and defects which greatly affect fatigue strength and cause excessive scatter of test results (4, p. 35).

Rectangular tension test specimens with a one-inch gage length were machined to conform to Figure 5. The tension specimens were prepared according to ASTM E8-54 T with modifications to use a one-inch gage length, since sufficient columbium alloy B plate material was not available to produce a standard two-inch gage length tension specimen (2, p. 185). The specimens were cut in a transverse rolling direction because only in this direction was sufficient metal available to make specimens of adequate size for testing.

Apparatus

The General Electric pneumatic fatigue testing machine type PRB used in this study is shown in Figure 6. It is particularly well adapted to testing of plate specimens.

The fatigue specimen is clamped into the specimen holder, and one end is left free so that the specimen becomes a cantilever beam. The free end of the specimen has two pistons attached which fit into the air nozzles on the two ends of the air column as shown in Figure 7.

The cantilevered specimen has a natural frequency of vibration to which the resonant air column is adjusted by varying its length. The air column can be adjusted by sliding a "trombone" shaped tubular member in or out and by changing to any one of seven different "trombones." The frequency of the resonant air column can be varied from 50 cycles per second to 300 cycles per second. The specimen must be designed so that its natural frequency, as mounted in the machine, falls in this testing range. When the resonant frequency of the air column is adjusted to the natural frequency of the specimen, the maximum displacement of vibration of the specimen is obtained. Pressure impulses from the two opposed nozzles that are 180 degrees out of phase cause the specimen to vibrate (6, p. 3).

The pneumatic fatigue machine is equipped with a constant pressure regulator on the air supply which can be adjusted to supply air at a constant pressure of from 0 to 20 pounds per square inch gage. The air supply valve can be adjusted to give the desired amplitude of vibration of the fatigue specimen. Once the air supply valve is set, the amplitude remains essentially constant. However, if the properties of the specimen change during the test, readjustment may be made to keep the displacement constant.

Amplitude of deflection of the specimen during vibration was measured through a telescope mounted on the machine which had a scale calibrated in thousandths of an inch. The frequency of the

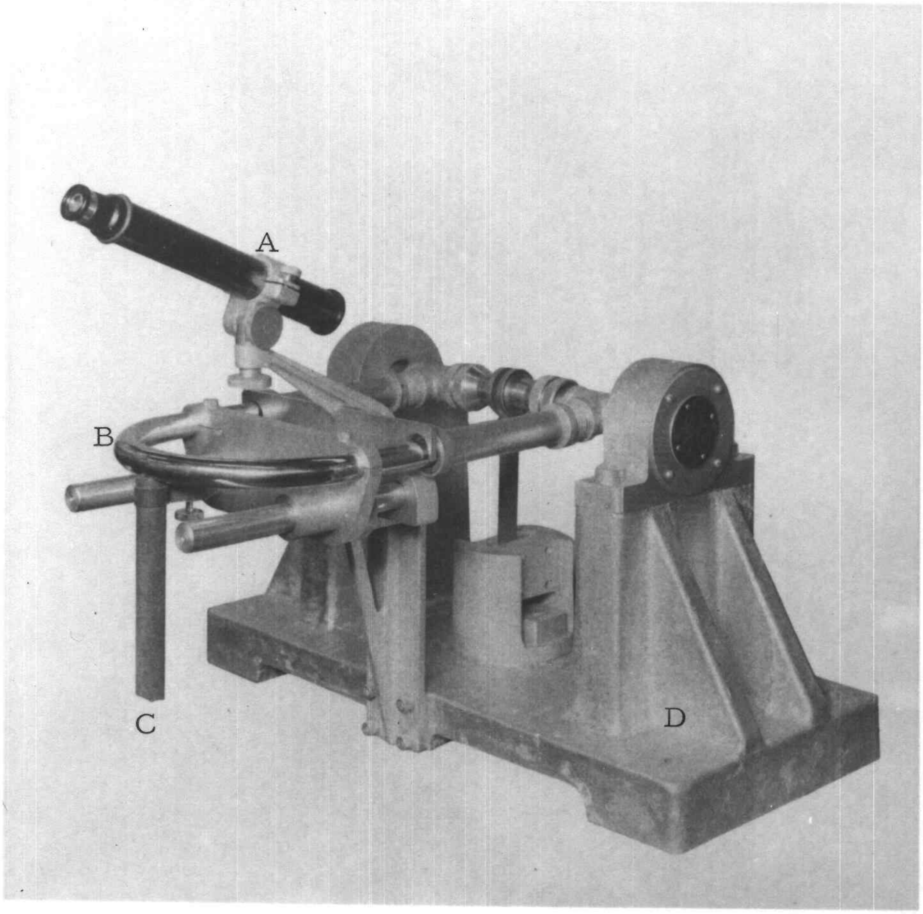


Figure 6. General Electric pneumatic fatigue machine, type PRB.

A - Telescope
B - Trombone
C - Air
D - Rigid Base

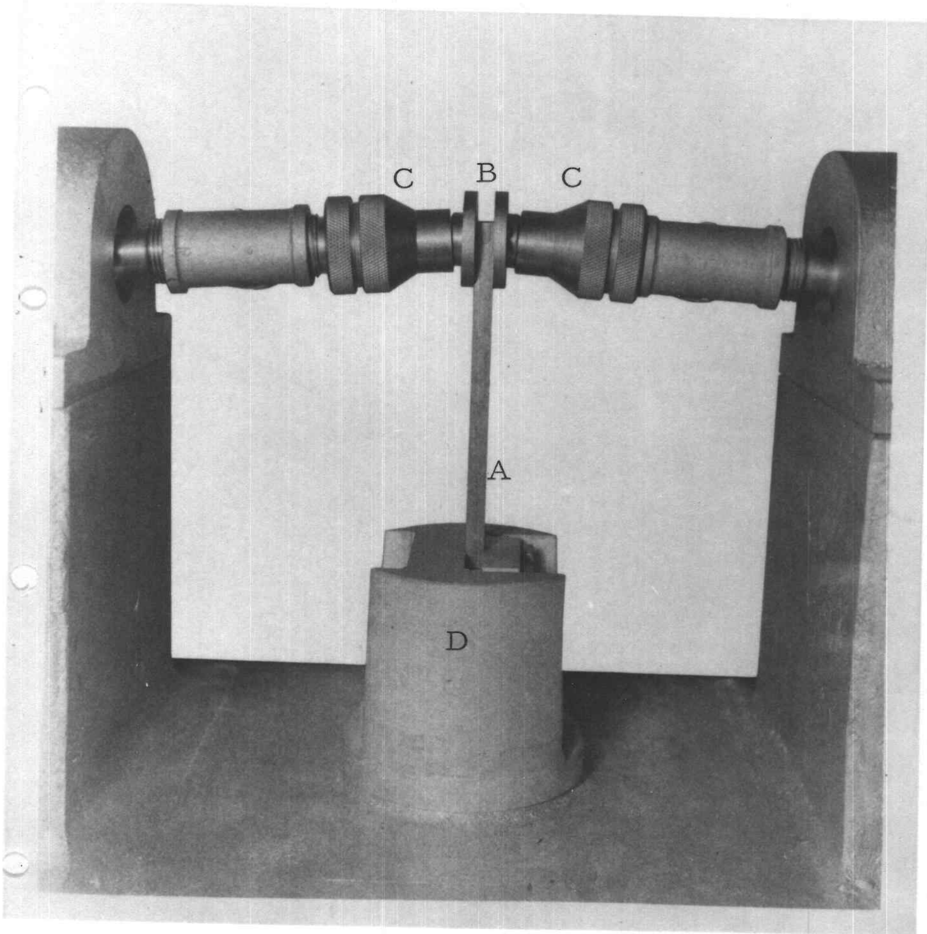


Figure 7. General Electric Type PRB pneumatic fatigue machine view showing specimen mounted with pistons attached.

A - Specimen
B - Pistons
C - Nozzles
D - Grips

Figure 8. General Electric PRB pneumatic fatigue testing machine with CD Recorder and Strobotac.

A - CD Recorder
B - G. E. PRB Fatigue Testing Machine
C - Strobotac

specimen was determined by the use of a "Strobotac" which had a built-in calibration device and could be used to measure frequencies from 700 to 14,000 revolutions per minute. The "Strobotac" used was manufactured by the General Radio Company and operated on the principle of persistance of vision. A flashing light in the "Strobotac" appeared to stop the motion of the specimen when the frequency of the light was the same as that of the specimen.

The General Electric pneumatic fatigue testing machine was equipped with a General Electric CD recorder which could record amplitude and frequency on the same chart by use of a selector switch (Figure 8). The recorder must be calibrated for amplitude or frequency by use of the telescope for amplitude or "Strobotac" for frequency . The calibration was good only for a narrow range, and the recorder had to be recalibrated for each change of amplitude or frequency (6, p. 4). The chart had a speed of six inches per hour and was four inches wide.

Compressed air was supplied by a Gardner 20 cubic feet per minute reciprocating air compressor. The compressor air supply had a cut off at 100 pounds per square inch gage.

The frequency of the rectangular cantilever specimens of alloy A was determined by the use of the following equation:

$$f = \frac{1}{2\pi} \sqrt{\frac{3\,EIg}{L^3\,(W + 0.236\,w)}}\,, \qquad (1)$$

where

f = frequency in cycles per second,

E = Young's modulus in pounds per square inch,

I = moment of inertia of the cross-sectional area in inches^4,

g = gravitational constant in $\frac{lb_m\text{-ft}}{lb_f\text{-sec}^2}$,

L = free length of specimen in inches,

W = weight of pistons in pounds,

w = weight of free length of specimen in pounds,

and

0.236 = constant developed by Rayleigh's method to provide for effect of distributed weight of rectangular cantilever beam (13, p. 27).

The frequency of the triangular cantilever specimens of alloy B was determined by the use of the following equation:

$$f = \frac{1}{2\pi} \sqrt{\frac{2EI_o g}{L^3 (W + \frac{1}{15} w)}} , \qquad (2)$$

where

f = frequency in cycles per second,

E = Young's modulus in pounds per square inch,

I_o = moment of inertia of the cross-section of the base of the triangular specimen in inches^4,

g = gravitational constant $\frac{lb_m\text{-}ft}{lb_f\text{-}sec^2}$,

L = free length of specimen in inches,

W = weight of pistons and excess metal on end of specimen in pounds,

w = weight of imaginary triangle formed by tapered sides of specimen in pounds,

and

$\frac{1}{15}$ = constant developed by Rayleigh's method (13, p. 27) to provide for the effect of the distributed weight of a triangular cantilever beam.

Equation (1) and (2) were used to estimate the specimen frequencies so that the final specimen dimensions would yield a natural frequency within the machine testing range. The thickness of material available and length of the specimen's cantilever end limited the maximum frequency attainable.

The tension tests were carried out on a 60, 000 pound capacity Baldwin Tate-Emery hydraulic testing machine. For this test a model P-3M extensometer of the averaging type with one-inch gage length and a model MA-1 automatic microformer stress-strain recorder were used.

PROCEDURE

Preliminary Work

The initial phases of the fatigue testing were primarily an attempt at finding a satisfactory fatigue specimen design and a testing technique that would produce reliable fatigue strength data.

Four specimens were prepared from columbium alloy A in the rectangular cantilever configuration. One specimen was left in the as-machined condition and the other three were polished.

In order to have a fiduciary mark from which to measure amplitude the four samples were punch marked on the neutral axis at a distance four inches from the edge of the machine grips (Figure 2). The amplitude of the specimens was measured at the punch mark with the use of a telescope provided with a scale in thousandths of an inch. The frequency of the specimens was determined by the use of the "Strobotac." Frequency was recorded at the beginning of the run and near the end of the run. Any deviation in frequency throughout the run was compensated by averaging the initial and final frequencies. The automatic recorder was used as a timing device by setting it on amplitude and reading the breaking time of the specimen from the sharp drop in amplitude on the chart when failure occurred. Amplitude was checked periodically during each run but showed no drift.

The cycles endured by a specimen were calculated by multiplying specimen frequency by the time as indicated on the recorder chart. Maximum specimen stress was calculated using the following equations:

$$\sigma_{max} = (y_{max}) \frac{3Et}{2L^2} \quad (3)$$

$$y_{max} = (y) \frac{2L^3}{3Lx^2 - x^3} \quad \text{(11, p. 18).} \quad (4)$$

where

σ_{max} = maximum stress in outer fibers in pounds per square inch,

y_{max} = deflection at free end of cantilever beam in inches,

y = deflection (amplitude) at punch mark in inches,

L = free length of cantilever beam,

E = Young's modulus in pounds per square inch,

t = thickness of specimen plate in inches,

x = distance to punch mark from grip - four inches.

Since no more columbium alloy A was available from the Wah Chang Corporation, further testing was conducted with specimens machined from columbium alloy B. All specimens of columbium B were machined to conform to Figure 3 in order to provide a constant stress test specimen and avoid breakage in the grips as occurred on all specimens of columbium alloy A.

Two specimens conformed to Figure 3 were polished and fatigue tested. Both broke at the machine grips, so all remaining specimens of columbium alloy B were machined to conform with Figure 4 which has a smaller cross-section and thus lower stress at the grips. This specimen design was utilized in determining the actual fatigue strength of the alloy columbium-one percent zirconium.

Final Test Procedure

All specimens of columbium alloy B in the configuration of Figure 4 were polished and tested according to the following procedure.

The specimens were oiled in the grips to prevent fretting corrosion and each specimen was punch marked on the neutral axis 1.5 inches from the axis of the attached pistons. The air column was adjusted to the specimen frequency while the specimen vibrated at a very low displacement. The automatic recorder was again used as a timing device for each run by setting it to record amplitude of vibration and observing the sudden drop on the chart when the specimen broke. The telescope was focused on the punch mark and the specimen was brought up to the desired amplitude by adjustment of the air supply valve. Care was taken not to exceed the desired amplitude, as this would reduce the fatigue life of the specimen. The "Strobotac" was used to record the initial frequency and to periodically check for frequency change during the run. Amplitude was checked periodically to maintain the desired stress in the sample.

After each sample was broken it was given a number and marked. This was done after the specimen was run to avoid scratching the polished specimen before testing. Each specimen was then carefully measured to determine all the dimensions necessary to calculate the stress in the constant stress tapered test section.

The number of stress cycles was determined by multiplying the frequency of the specimen by the time as determined from the recorder chart.

The stress was calculated using formulas for constant stress triangular cantilever beams. In the triangular section of the beam, the bending moment increases in the same proportion as the section modulus, and since the stress is equal to the bending moment divided by the section modulus, it remains constant in the outer fibers of the triangular section of the specimen. This is only true if the apex of the triangle is at the point of application of the force (Figure 4).

For small deflections the stress can be taken to be equal to

$$\sigma_{max} = \frac{\delta E t}{L^2} \qquad (12, p. 210) \qquad (5)$$

where

σ_{max} = maximum stress in outer fibers in pounds per square inch,

δ = deflection (amplitude) at punch mark in inches,

E = Young's modulus in pounds per square inch,

t = specimen thickness in inches,

L = length of specimen from punch mark to the base of the triangular section in inches.

RESULTS

Tensile Tests

The results of the tension tests on columbium-one percent zirconium are given in Table 5 of the appendix and Table 2 gives a summary of the results. The stress-strain curves of the two transverse tensile specimens tested are given in Figure 9. The strain gage was removed from the specimens beyond the yield strength and only the load recorded automatically thereafter until failure.

The tensile test data from specimen B-1 are probably in error because a possible load beyond the yield strength may have been applied while setting up the specimen in the machine grips. During the set up of specimen B-1, the heads of the machine were moving at high speed and the grips clamped down on the specimen and it sustained an unknown load.

The tensile strengths of the specimens were taken in the transverse rolling direction because sufficient material was not available for longitudinal specimens. The high ductility and low tensile strength of the columbium-one percent zirconium alloy indicate the fully recrystallized condition.

Transverse tensile strength data for columbium-one percent zirconium was not available, but pure columbium shows a negligible

Note: Strain gage removed beyond proportional limit.

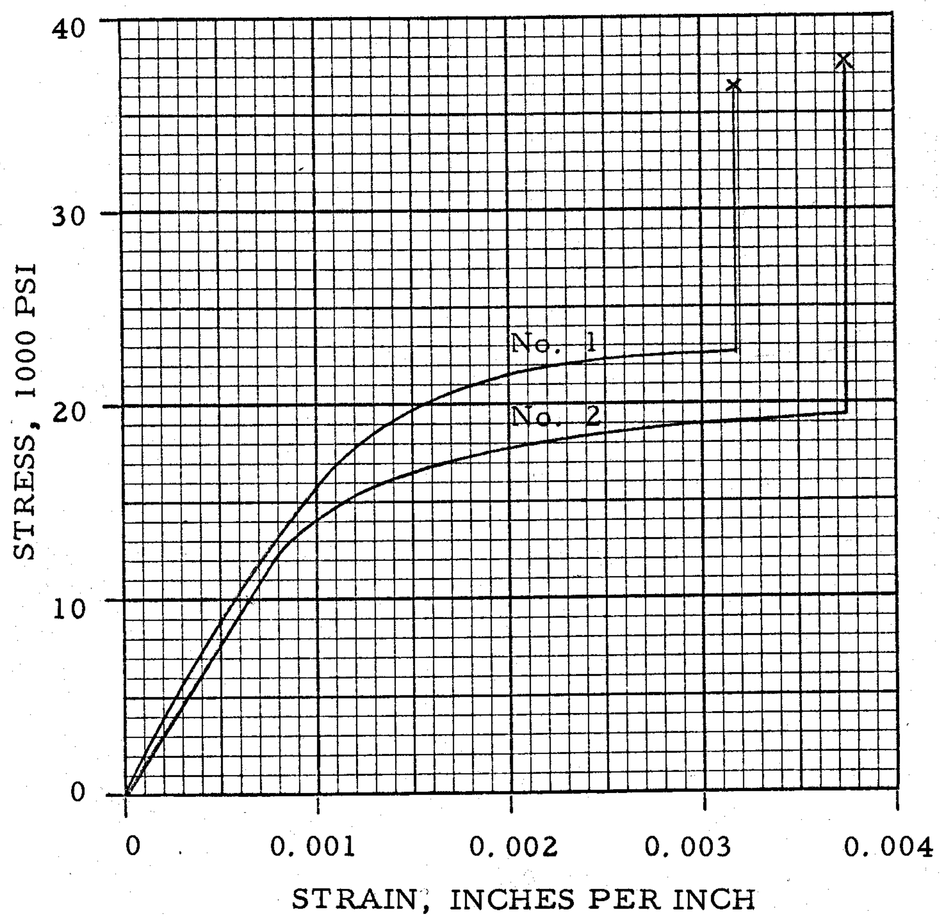


Figure 9. Tensile properties of recrystallized columbium - one percent zirconium in the transverse rolling direction.

Table 2. Tensile properties of recrystallized columbium-one percent zirconium (transverse to rolling direction).

Ultimate Tensile Strength	37, 150 pounds per square inch
Yield Tensile Strength 0. 2 percent offset	20, 900 pounds per square inch
Modulus of Elasticity	14.7×10^6 pounds per square inch
Elongation in One Inch Gage Length	49. 5 percent
Reduction in Area	95. 4 percent

reduction in tensile strength in the transverse direction when in a fully recrystallized condition (8, p. 406). The transverse tensile test results are probably quite close to the actual longitudinal tensile properties of columbium-one percent zirconium, but slightly lower.

Fatigue Tests

The results of the fatigue tests are given in Tables 3 and 4 of the appendix and the S-N (stress-cycles) curves plotted from them are shown in Figures 10 and 11.

The fatigue strength curves are plotted on semi-logarithmic paper with the number of cycles plotted on the logarithmic abcissa, and the maximum fiber stress plotted on the linear ordinate coordinate axis. The cycles are plotted on a logarithmic scale because this gives the clearest representation of the variation of cycles with

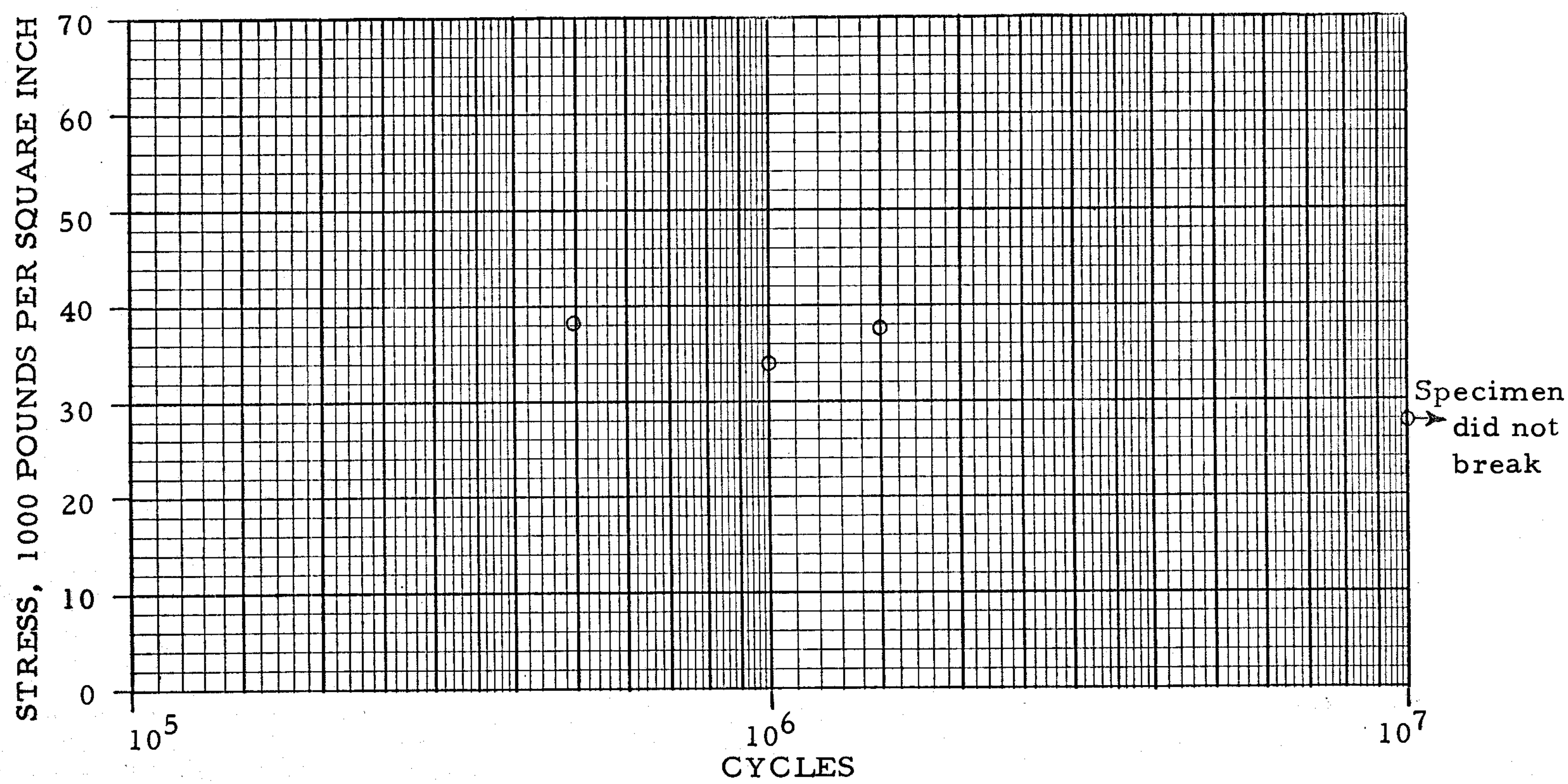


Figure 10. Reversed bending fatigue of recrystallized columbium - ten percent hafnium and one percent titanium columbium alloy A.

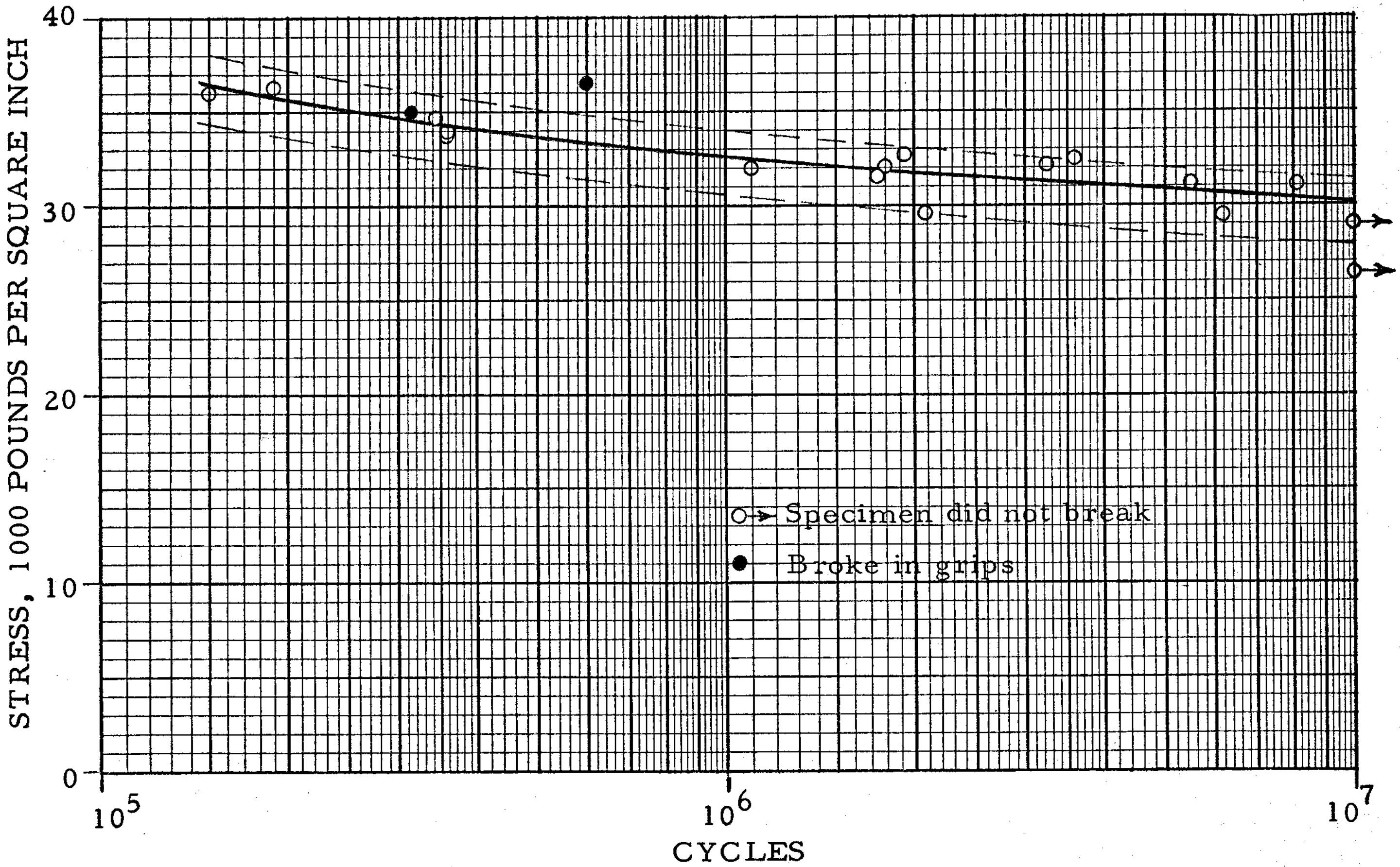


Figure 11. Reversed bending fatigue of recrystallized columbium - one percent zirconium columbium alloy B.

stress. The fatigue life in cycles is very nearly a power function of the maximum fiber stress.

The S-N plot of the fatigue tests run on columbium alloy A (Figure 10) contains too few points to permit a curve to be drawn representing the probable fatigue strength. More tests with columbium alloy A could not be run because material was not available. All specimens of columbium alloy A were rectangular (Figure 2) and failure occurred at the grips. The effect of clamping stress, fretting corrosion and stress concentration due to constrained bending at the grips makes the true maximum fiber stress indeterminate. The stresses plotted on the ordinate are the calculated values of the maximum stress existing in the outer fibers. The isolated points on the S-N plot do seem to indicate that the fatigue strength at 10^7 cycles is probably greater than 27, 400 pounds per square inch and that the fatigue strength decreases with increasing cycles. Too few points are available to determine the exact shape of the S-N curve, so it is not possible to say whether an endurance limit exists in the range of testing. The results show a large scatter which is due in part to the statistical nature of fatigue failure and also to the indeterminant nature of the actual maximum stress. The actual maximum stress is probably higher than that calculated, so the actual fatigue strength is probably higher than would be apparent from the S-N plot.

The S-N curve of the fatigue tests run on columbium-one percent zirconium is shown in Figure 11. The points on the curve show the scatter that is characteristic of all fatigue tests. All the specimens of columbium-one percent zirconium were machined from the same piece of plate and in the same jig. All the specimens were polished using the same technique. The scatter can not therefore, be attributed to differences in heat treatment, chemical composition, machining, polishing or size effects. The scatter is due to the fact that the fatigue strength of a metal depends on the orientation and stresses in a few individual crystals which are nonisotropic in nature, producing a statistical variation in fatigue strength which depends on the statistical distribution of these crystal orientations and stresses.

The S-N curve shown in Figure 11 represents an average fatigue strength based on the resulting data. A scatter band is shown to indicate the upper and lower limits of the fatigue test scatter. The S-N curve does not indicate an endurance limit existing in the range of testing. The fatigue curve is similar in this respect to aluminum which has no endurance limit but shows a continual reduction in fatigue strength with increasing cycles. At 10^7 cycles the fatigue strength is greater than 26, 400 psi which is 5, 500 psi higher than the average yield tensile strength as given in Table 2. This would indicate that no endurance limit had been reached at 10^7 cycles since the endurance limit of most metals is lower than the yield strength. The fatigue strength curve shows a slight upward curvature which would indicate that an endurance limit would be reached at higher cycles. A rough extrapolation of the curve shows that the fatigue strength would not be lower than the yield strength until more than 10^8 stress cycles. An endurance limit on the order of 21, 000 psi might then be anticipated for recrystallized columbium-one percent zirconium between 10^8 and 10^9 cycles. Further testing, however, might show that no endurance limit exists.

The ratio of the fatigue strength at 10^7 cycles to the ultimate tensile strength is 0. 71 which is much higher than the usual 0. 50 endurance ratio for most metals. This is another indication that no endurance limit was reached at 10^7 cycles.

Further tests beyond 10^7 cycles were not conducted for several reasons. The tests in the 10^5 to 10^7 range required nearly a month of machine operation. To extend the testing range to 10^8 cycles would require machine operation for nearly a year which was not feasible because the air compressor had bad bearings. Lack of material also limited the amount of data and testing range. The lower test limit of 10^5 cycles was imposed by the maximum stress obtainable with the fatigue testing machine. The testing range of 10^5 to 10^7 cycles corresponds to the range used by Begley in his fatigue study of pure columbium.

Due to slight differences in specimen thicknesses and difficulty in setting and reading vibration amplitudes, it was not possible to test more than one specimen at exactly the same stress level. This made it impossible to produce the necessary requirements for a statistical analysis of the probable life at different stress levels. Normally five or more specimens would be run at the same stress and the probable life at that stress determined statistically. Restrictions on the amount of material and time available for testing limited the resulting data. The data available do give a rough statistical representation of the fatigue strength of recrystallized columbium-one percent zirconium.

A comparison of the fatigue strength curve of columbium-one percent zirconium with the fatigue characteristics of pure annealed powder-metallurgy columbium, as determined by Begley (9, p. A-32) (Figure 1), shows the shape of the alloy curve to be similar but with less scatter. The fatigue strength of columbium-one percent zirconium is slightly higher than that of pure powder-metallurgy columbium. This is probably due to the higher strength of the alloy and the improvement of metallurgical structure which arc melting produces over powder-metallurgy techniques. Since no tests have been run to isolate these two effects it is not possible to determine the individual contributions of each to the higher fatigue strength of columbium-one percent zirconium. However, powder-metallurgy techniques produce a porous metal matrix in which surface voids and defects should cause a reduction in fatigue strength. Also the low density of a powder-metallurgy metal causes the true stress to be higher than that calculated on the basis of measured area because of the presence of voids in the metal. This causes the fatigue strength to appear lower than it would be if based on the true stress in the metal particles.

CONCLUSIONS

1. The fatigue strength curve of Figure 11 represents the room temperature fatigue properties of completely recrystallized columbium-one percent zirconium in reverse bending. The fatigue strengths shown are for carefully prepared test specimens and suitable safety factors must be used when applying these results to actual machine parts.
2. No endurance limit was observed for columbium-one percent zirconium within the range of 10^5 cycles to 10^7 cycles. However, an endurance limit of the order of 21, 000 psi might be anticipated beyond 10^8 cycles on the basis of the fatigue behavior of most metals.
3. No conclusions can be made for the columbium-ten percent hafnium-one percent titanium (alloy A) because all specimens broke in the machine grips and too few specimens were tested.
4. The specimen shown in Figure 4 can be used with the General Electric Pneumatic Fatigue Tester and will produce fatigue failures in the constant stress taper section of the specimen.
5. This specimen configuration should reduce the normal statistical variation in fatigue testing because of the large area of the specimen exposed to the maximum fiber stress.

RECOMMENDATIONS

1. Many more specimens should be tested in order to provide the necessary data for a statistical analysis which would indicate each fatigue strength curve as a probability curve.
2. In order to test a large number of samples, and increase the statistical certainty of the results without unduly increasing the duration of testing, the Probit method could be used (10, p. 138). This would require many identical testing machines, however.
3. Another type of machine, which does not require as much operator attention as the General Electric machine, should be used for the fatigue testing.
4. Fatigue tests of powder-metallurgy columbium and arc melted columbium should be conducted to isolate the effect of powder-metallurgy techniques on the results of Begley's fatigue tests of powder-metallurgy columbium.
5. The fatigue testing range should be expanded to 10^9 cycles, if feasible, to determine the endurance limit, if one exists.

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APPENDIX

APPENDIX

Table 3. Test data of reverse bending fatigue tests of columbium-ten percent hafnium-one percent titanium C-103 (Alloy A).

Specimen No.	Specimen Configuration	Stress in psi	Cycles for Failure	Notes
A-1	Figure 1	38, 200	495, 000*	Rolled
A-2	Figure 1	37, 400	1, 500, 000*	Polished
A-3	Figure 1	27, 400	20, 000, 000**	Polished
A-4	Figure 1	33, 900	1, 010, 000*	Polished

*Broke in grips
**Specimen did not break

Table 4. Test data of reverse bending fatigue tests of columbium-one percent zirconium (Alloy B).

Specimen No.	Specimen Configuration	Stress in psi	Cycles for Failure	Notes
B-1	Figure 2	36,500	590,000*	
B-2	Figure 2	35,000	314,000*	
B-3	Figure 3	34,700	345,000	
B-4	Figure 3	29,100	10,100,000**	
B-5	Figure 3	32,500	3,600,000	
B-6	Figure 3	33,900	360,000	
B-7	Figure 3	32,700	1,935,000	
B-8	Figure 3	UNKNOWN	UNKNOWN	Loss of air pressure during test
B-9	Figure 3	31,200	5,502,000	
B-10	Figure 3	32,100	1,100,000	
B-11	Figure 3	32,200	1,800,000	
B-12	Figure 3	29,500	6,160,000	
B-13	Figure 3	33,800	360,000	
B-14	Figure 3	32,200	3,240,000	
B-15	Figure 3	31,200	8,100,000	
B-16	Figure 3	29,600	2,070,000	
B-17	Figure 3	36,100	148,000	
B-18	Figure 3	36,300	180,000	
B-19	Figure 3	31,600	1,750,000	
B-20	Figure 3	26,400	10,100,000**	

*Broke in grips
**Specimen did not break

Table 5. Tensile properties of recrystallized columbium-one percent zirconium (transverse to rolling direction).

Specimen No.	Ultimate Tensile Strength, psi	Yield Tensile Strength, 0.2 Percent Off-set, psi	Modulus of Elasticity psi	Elongation in One Inch Percent	Reduction in Area Percent
B-1	36,500	22,500		49	95.4
B-2	37,800	19,300	14.7×10^6	50	95.5
Average	37,150	20,900	14.7×10^6	49.5	95.45