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DYNAMIC MODULUS OF ELASTICITY AND THE
INTERNAL FRICTION OF ZIRCONIUM

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THE VARIATION WITH TEMPERATURE OF THE DYNAMIC MODULUS OF ELASTICITY AND THE INTERNAL FRICTION OF ZIRCONIUM

By A. D. Schwope and G. T. Muehlenkamp

INTRODUCTION

The purpose of the investigation described in this report is the determination of the variation with temperature of the dynamic modulus of elasticity and of the internal friction of both the arc-melted Bureau of Mines sponge and crystal-bar zirconium.

The method adopted here is one in which the modulus is derived from the measurement of the natural frequency of a free-free beam in transverse vibrations. The internal friction is derived from the measurement of the width at half amplitude of the resonant frequency curve. In this method, the stress, and consequently the strain, to which the specimen is subjected is very small, and thus plastic deformation and its effect on the internal friction is minimized.

SUMMARY

Arc-melted Bureau of Mines sponge zirconium has a lower dynamic Young's modulus (12.8×10^6 p.s.i.) at room temperature than crystal-bar zirconium (13.9×10^6 p.s.i.). Both materials have approximately the same rate of decrease with temperature, with the crystal-bar modulus decreasing a little more rapidly, until at 1200°F. the moduli of both are approximately the same, being 7.8×10^6 p.s.i.

The internal friction (Q^{-1}) of the arc-melted Bureau of Mines sponge zirconium (2.4×10^{-4}) is much lower than crystal-bar zirconium (17×10^{-4}) at room temperature. Both decrease with rise in temperature, until at 500°F., the arc-melted sponge zirconium has Q^{-1} value of 1.4×10^{-4} and the crystal-bar zirconium has a Q^{-1} value of 6.2×10^{-4} . These values of Q^{-1} remain fairly constant until at approximately 900°F. there is a rapid increase in internal friction. At 1130°F., the arc-melted sponge zirconium has a Q^{-1} value of 14×10^{-4} and the crystal bar has a value of 100×10^{-4} .

EXPERIMENTAL PROCEDURE

The method used is similar to that of Roberts and Nortcliffe⁽¹⁾. The test specimen (3/16 inch diameter and 5 inches long) is hung from two slings of asbestos string, one connected to the exciting system, and the other to the receiver (Figure 1). The exciting sling is connected to a speaker coil, which is set in vibration by a variable-frequency oscillator. If the frequency is sufficiently distant from the natural period of the specimen, the latter remains essentially at rest. As the resonant frequency is approached, the bar is set in oscillation, which is a maximum when the exciting and natural frequencies coincide. This vibration of the test piece is, after damping, reconverted, by means of a crystal pickup supporting the receiving sling, into electrical oscillations which are measured on an oscilloscope. The slings supporting the test specimen must be displaced from the nodes of vibration or suspension at the nodes results in zero amplitude at resonant frequency. The nodes for the first harmonic are at points which are 0.224 of the specimen

1. M. H. Roberts and J. Nortcliffe, "Measurement of Young's Modulus at High Temperatures", Journal of Iron and Steel Institute, V. 157, 1947, p. 345.

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FIGURE 1. GENERAL ARRANGEMENT OF THE APPARATUS FOR DETERMINING DYNAMIC YOUNG'S MODULUS AND INTERNAL FRICTION

length (in this case 5 inches) from the ends of the specimen. The distance between the supporting slings was arbitrarily set at 3 inches. The setup was checked with bars of various materials and known moduli.

For the high-temperature determinations, the test specimen is supported in a Vycor tube (1-inch diameter) which has two smaller tubes (1/4 inch diameter and 2 inches long), perpendicular to the main tube, through which the slings pass. (See Figure 1) A steady stream of purified argon was passed through the main tube for all temperatures above room temperature.

The modulus of elasticity is determined from the resonant frequency using the relationship:

$$E = 9.18 \times 10^{-6} \times \frac{L^4}{d^4} \times \frac{m}{L} \times f^2$$

where

E = modulus of elasticity

L = length in inches

d = diameter in inches

m = weight in grams

f = resonant frequency in cycles/sec.

The above relationship is derived from the basic formula of lateral vibration of a prismatic bar:

$$f = \frac{a}{L^2} \sqrt{\frac{EIg}{AD}}$$

where

f = resonant frequency in cycles, sec.

a = constant depending on conditions at the ends of the specimen, = 3.58 for the free-free case.

E = modulus of elasticity

I = moment of inertia = $\frac{\pi d^4}{64}$

A = cross-sectional area

D = weight per unit volume

g = 32.2 ft./sec.²

L = length of specimen

The method of measuring the internal friction (Q^{-1}) is to plot the amplitude of vibration against the frequency of the exciting force in the region of the resonant frequency. The ratio of the width, Δf , of this curve at half maximum to the frequency, f , corresponding to the maximum is proportional to the internal friction⁽²⁾. $Q^{-1} = \Delta f / \sqrt{3} f$.

The arc-melted Bureau of Mines sponge-zirconium specimens were machined from rolled plate to a 3/16-inch-diameter bar. The crystal-bar zirconium specimens were machined from swaged crystal bar. All bars were then sealed in a Vycor tube under argon atmosphere and annealed at 1500°F. for 15 hours.

The approximate range of frequency encountered in these tests was between 1000 cycles per second (at room temperature) to 800 cycles per second (at 1100°F.). In all cases the heating and cooling curves coincide within the limits of experimental error. This indicates that there was very little effect of contamination of the specimens by foreign gases during the time (approximately 6 hours) it took to make the complete temperature cycle.

RESULTS

Curves showing the variation of the dynamic Young's modulus with temperature have been plotted in Figure 2 for both the arc-melted sponge zirconium and the crystal-bar zirconium. At room temperature, the modulus of crystal bar is 13.9×10^6 p.s.i., while, for the arc-melted sponge zirconium,

2. C. Zener, "Elasticity and Anelasticity of Metals", University of Chicago Press, 1948.

FIGURE 2. MODULUS OF ELASTICITY VS. TEMPERATURE FOR TWO TYPES OF ZIRCONIUM

it is 12.8×10^6 p.s.i. Both curves decrease linearly up to 1130°F. , the maximum temperature to which the crystal-bar zirconium was subjected. At 1200°F. , there is a more rapid decrease in the modulus of the arc-melted sponge zirconium.

The room-temperature modulus was checked for the crystal-bar zirconium using static tensile loading. The strain was measured using a Tuckerman extensometer with a sensitivity of two microinches. The resultant stress-strain curve is shown in Figure 3. The modulus of elasticity was 13.4×10^6 p.s.i., which agrees well with the dynamic value of 13.9×10^6 p.s.i. An interesting point to note is the low value of the proportional limit (4000 p.s.i.). The total elastic strain is approximately 300 microinches, which could account for the relatively low moduli values of 9 to 10×10^6 p.s.i. often reported in literature. If extensometers of the ordinary 50 to 100 microinch sensitivity are used, strains well in the plastic region would still be considered elastic and would result in a low modulus value. The ultimate strength of this specimen was 26,000 p.s.i., which is relatively low for zirconium.

The internal friction versus temperature curves for the two types of zirconium are given in Figure 4. At room temperature, the internal friction (Q^{-1}) of crystal bar is 17×10^{-4} , while the arc-melted sponge zirconium has an internal friction of 2.4×10^{-4} . Both show a decrease in internal friction as the temperature rises, until at approximately 900°F. there is a rapid increase in the Q^{-1} values. At 1130°F. , the internal friction values are 100×10^{-4} and 74×10^{-4} for crystal-bar and arc-melted sponge zirconium, respectively.

The rapid rise in internal friction at 900°F. can be associated with recrystallization and grain-growth characteristics of zirconium. If internal

FIGURE 3. ROOM-TEMPERATURE STRESS VS. STRAIN CURVE
FOR CRYSTAL-BAR ZIRCONIUM

FIGURE 4. INTERNAL FRICTION VS. TEMPERATURE CURVES FOR TWO TYPES OF ZIRCONIUM

friction is a measure of the relaxation time (ϵ) of viscous components, then it is expected that as the temperature is approached, at which the grain boundaries become relatively mobile, the internal friction would increase. The effect of impurities on the internal friction is indicated in Figure 2. The low internal-friction values for the impure Bureau of Mines zirconium can be associated with the precipitates present in this metal. These precipitates effectively act as blocks to relaxation occurring from the so called "stress relaxation centers"⁽²⁾. A further effect of the impurities is to shift the internal-friction peak to a higher temperature. It is quite probable that the shift of the peak is largely due to dissolved impurities, since these are much more effective than precipitates in raising the recrystallization and grain-growth temperatures. It should be remembered that the term recrystallization does not indicate that macroscopic grain formation is occurring during the measurements. Rather, that in an anisotropic metal, it is improbable that a completely stress-free structure can be obtained by annealing, and that the increase in internal friction is affected by the relatively small stress centers set up during the cooling process.

The decrease in internal friction from room temperature to 400°F. might be associated with the recovery process, which normally precedes recrystallization, or with a precipitation phenomenon. However, the fact that the internal friction followed the same change during the cooling cycle renders this explanation improbable. Further work is indicated to explain this unusual effect.

The break in the modulus curve of Figure 2 at 1200°F. is associated with the internal-friction peak of Figure 4. This is best explained in terms of relaxation times. As the relaxation time becomes shorter and the internal friction rises, more strain can occur per cycle of vibration. This is evidenced by a reduction in the frequency, and subsequently the calculated modulus.

It is noted that the modulus does not decrease immediately as the internal friction begins to increase, rather that the modulus follows the linear relationship until the relaxation time has been reduced to a period comparable to the frequency of vibration. It is, therefore, expected that as the resonant frequency becomes lower, due to shape considerations, the modulus would be affected sooner by temperature. The ultimate, a static test, would be expected to show a sharp drop around 900°F., where the recrystallization process is initiated.