the dislocations and prevent them from contributing to the dynamic elastic moduli are prestraining (Cu, Ag, Au),[7] and pinning with point defects produced by neutron irradiation or by impurity atom doping (Cu).[6]

Complete sets of TOEC have been determined for only those metals mentioned above, all fcc. Other materials for which all the TOEC have been reported are the semiconductors Ge,[8–11] Si,[8–11] GaAs,[12] InSb,[13] the alkali halides NaCl,[14,15] and KCl,[14] and the insulators MgO,[10] quartz,[16] and yttrium iron garnet.[17] Complete sets of TOEC for polycrystalline or amorphous materials that have been reported are for Cu,[1] Fe[1,18] fused silica,[10] pyrex glass and polystyrene,[18] a variety of steels, aluminum alloys, a magnesium alloy, and sintered Mo and W.[19]

Columbium is one of a class of materials, the bcc refractory metals, for which TOEC have not been reported, except for the sintered materials mentioned above. With the growing commercial use of these materials, there has been an increasing interest in their mechanical properties. The contribution of lattice anharmonicity in lattice-defect calculations is one area of theoretical study that is being pursued.[20]

Since the yield points of these metals are considerably higher than those of the fcc metals studied to date, it seems reasonable to expect less of a problem with dislocations when determining their TOEC using a uniaxial bias stress. This expectation was realized as will be seen in the following sections.

II. EXPERIMENTAL PROCEDURE

A. Apparatus

An ultrasonic phase comparison method was used to determine both the values of the pure-mode ultrasonic wave velocities and their dependence on an applied static bias stress, both hydrostatic and uniaxial. An Arenberg PG-650C pulsed oscillator was used as a gated amplifier for pulsing the sample with an rf signal derived from an external cw oscillator whose frequency was continuously monitored by a digital counter. The applied rf pulse length was adjusted to twice the round trip transit time of the ultrasonic pulse through the sample so that each echo overlapped one-half the following echo in the pulse-echo train. The frequency of the cw oscillator was then adjusted to obtain an out-of-phase condition between successive echoes so that the overlap regions presented a minimum or null signal level. The overlap region between the 1st and 2nd or 2nd and 3rd echoes was then gated out, amplified, and the unrectified signal displayed on a Hewlett–Packard 175A oscilloscope. The frequency at which the null condition occurs is simply related to the ultrasonic wave velocity in the sample. The null frequency can be determined to one part in 10^6 in the best cases encountered so far even with $\frac{3}{16}$ in. sample lengths. Typically, however, with this size sample, and with a low value of ultrasonic attenuation as experienced with a single crystal, the accuracy is 3–5 parts in 10^6 for shear waves and 3–5 parts in 10^5 for longitudinal waves because of their higher velocities. With larger samples and longer transit times the accuracies for the two types of waves should be the same. A complete description and analysis of the method is given elsewhere.[21]

Quartz crystals of $\frac{1}{8}$ in. diam were bonded to the nominally $\frac{1}{4}$ in. sq sample faces using Nonaq. Both *X*-cut and *AC*-cut crystals having 16 MHz fundamental frequencies were used for the single-crystal studies and 5 MHz for the polycrystals. Hydrostatic pressure runs were made with the sample enclosed in a high-pressure chamber pressurized with helium gas. A 16 in. 7500 psi Heise pressure gauge was used to monitor the helium gas pressure to $\pm 0.1\%$ of full scale. The uniaxial stress runs were made with the samples loaded in compression between optically flat stainless steel load platens in an isothermal enclosure on a table model Instron test machine. The load was monitored, by a load cell calibrated to $\pm 0.5\%$ of full scale. The sample temperature was recorded before and after each frequency reading to ± 0.025°C using a chromel-alumel thermocouple junction spring loaded to the side of the sample in both test arrangements.

B. Sample Description and History

Two single-crystal columbium samples were used in this study. They were both cut from the same $\frac{5}{16}$ in. diam 3-pass electron-beam zone refined single-crystal rod obtained from Materials Research Corp. A typical analysis for material prepared in this manner is 100 ppm Ta, <30 ppm P, 8 ppm C, 6 ppm W, 4 ppm N, and 23 ppm O, with traces of several other elements. After the initial cutting and lapping stages, special care was taken to alternately etch and polish each of the three pairs of faces of the samples until sharp Laue

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