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FATIGUE UNDER PULSATING HYDROSTATIC PRESSURE

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ABSTRACT

Polycrystalline copper, brass, and a single crystal of copper were subjected to pulsating hydrostatic pressure of 40,000 psi. Ultrasonic attenuation measurements showed no change for fine grain brass but showed increasing attenuation with pressure cycles in polycrystalline copper and decreasing attenuation in a single crystal of copper. Explanations advanced for the changes were: that cyclic hydrostatic pressure reduced the density of effective dislocations causing reduced attenuation; and local deformation at grain boundaries resulting from inhomogeneity there and possibly anisotropy caused increased attenuation.

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Introduction

Available evidence suggests that fatigue is caused by cyclic shearing stresses acting on some critical plane and that the normal stress acting on the critical shear plane affects the ability of the material to resist fatigue--a tensile normal stress weakens and a compressive normal stress strengthens[1]*.

With this concept of fatigue failure it is natural to think of limiting conditions, such as what precisely happens when a metal is subjected to cyclic hydrostatic (or volumetric) stress. In this instance no shearing stress is generated in a perfect single crystal of an anisotropic material. Shearing stresses would not be generated even at the micro scale for a homogeneous polycrystalline metal whose crystals had cubic structure and were either isotropic or anisotropic regarding elastic constants. This may be determined from the nature of the stress tensor for a cubic metal[2]. However, shearing stresses would be generated by volumetric stresses (such as hydrostatic pressure) in polycrystalline metals having inhomogeneity such as may occur at grain boundaries or whose crystals had certain classes of anisotropic elastic constants found in some non-cubic crystals. The latter results from the fact that in a polycrystalline metal the neighbors of a given crystal would have different orientations. Thus, for crystals whose strain under hydrostatic compression is anisotropic hydrostatic compression would cause neighboring grains in general to resist compression with different compression stresses in a given direction. This difference in compression stress in neighboring grains would induce distortion and shearing stresses in other neighboring grains. Nonuniformity due to regions of disorder of grain boundaries could also introduce shearing stresses under hydrostatic compression in a somewhat similar manner.

Thus, cyclic volumetric stresses might be expected to induce changes (perhaps fatigue cracks under sufficiently intense stresses and sufficiently strong anisotropy or nonuniformity) in some polycrystalline metals under hydrostatic pressure; but no change could be expected in perfect single crystals.

To investigate these possibilities, specimens of polycrystalline copper and alpha brass and a single crystal of copper were subjected to cyclic hydrostatic pressure from zero to 40,000 psi to determine whether cyclic pressure would produce structural changes in metals that could be measured by ultrasonic attenuation.

While Bridgman[3] had reported hysteresis and a permanent change in linear strain in copper following hydrostatic pressure of 427,000 psi, information on the effect of cyclic pressure was limited to two or three cycles when the present investigation was begun. The results of two related studies have been reported since, however. Davidson and Homan[4] described widespread slip and cross slip (observed metallographically) on polycrystalline bismuth (a trigonal system having strong crystal anisotropy) resulting from noncyclic pressures from 73,000 to 294,000 psi. Seely, Baker and Gibbs[5] reported internal friction measurements by means of mechanical vibration at 50 kc. of 99.99% aluminum (cubic nearly isotropic metal) subjected to hydrostatic pressure of 29,000 psi. They found no observable residual pressure effect on an annealed single crystal, but cold worked and annealed (two hours at 572°F) polycrystal aluminum showed an increase in damping after increasing amounts of pressure. This pressure effect progressively decreased with repeated runs to nearly zero after a few cycles. However, Miles and Gibbs [6] later found that the pressure effect on polycrystalline aluminum could be eliminated by removing hydrogen from the sample.

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* Numbers in brackets identify the references at the end of the paper.

Materials and Specimens

The metals investigated to date were copper and cartridge brass. The attenuation of these metals was easy to determine with available apparatus. Some other metals in the hexagonal and trigonal system were of possibly greater interest from the anisotropy standpoint, but involved seemingly too great experimental difficulties for an exploratory study.

The copper specimens were from commercial electrolytic tough pitch (ETP) and oxygen free high conductivity (OFHC) polycrystalline rods and from a random orientation single crystal of good quality but unknown specification. As received the copper was hard drawn with a tensile strength of 40,000 psi. Both polycrystalline specimens were annealed at 1000°F for 1 hr. after machining. The single crystal of copper was annealed at 1800°F for 1 hr. in a helium atmosphere. The polycrystalline specimens had a grain size of 0.025 mm. and 0.050 mm., after annealing.

The cartridge brass specimens were from half hard commercial unleaded 70-30 alloy rod having a tensile strength of 75,000 pst. Two different grain sizes were used, 0.030 mm. and 0.090 mm. These were obtained by annealing at 750°F for 1 hr. and 1200°F for 1 hr., respectively. The larger grain size was the largest that could be employed and still measure the attenuation accurately in the frequency range available.

The anisotropy of elastic constants for these metals is expressed by the parameters[7],

$$A = C_{11}/C_{33} \quad , \quad B = 2C_{44}/(C_{11}-C_{12} \quad ,$$

where C_{11} , C_{33} , C_{44} , and C_{12} are the elastic constants and isotropy is indicated by $A = B = 1$. For the cubic metals copper and α-brass, $A = 1$ and B has the values [7] 3.2 and 4.0 for copper and alpha (cartridge) brass, respectively.

The polycrystalline samples were annealed in an electric furnace with automatic temperature control and a reducing atmosphere.

All specimens were machined (except the single crystal) to 3/4 inch in diameter and 1-3/4 inches long. Two flats were machined on the sides and polished to reveal the microstructure.

The ends of the cylinders were polished parallel to within 0.0002 in. as required for the pulse echo technique of ultrasonic attenuation measurement.

Experimental Procedure and Apparatus

These samples were subjected to repeated applications of hydraulic pressure of 40,000 psi, and inspected for changes in the attenuation of pulses of 3 or 30 megacycle ultrasonic dilatation waves. Three megacycles was used for the polycrystal specimens and 30 megacycles for the single crystal of copper. The measurements were carried out using an ultrasonic attenuation unit which measured ultrasonic energy losses in solid materials by means of the pulse echo method[8].

The ultrasonic waves were generated by a quartz crystal transducer of about 1/2 in. diameter driven by an untrasonic attenuation measuring instrument designed by Chick, Anderson and Truell[9]. In order to insure pure hydrostatic compression, it was desired that no shearing stresses be introduced during compression because of mismatch of elastic constants between the quartz and the test sample. This was achieved by removing the quartz crystal during application of fluid pressure and making measurements before and after each sequence of pressure applications. This precaution required developing the skill required for applying and reapplying the transducer so as to obtain the same result.

The transducer was wrung on to the sample as follows: (1) A thin coating of Nonaq Stopcock grease was applied to one end of the test specimen. (2) The transducer was spun onto the grease with a shear motion. If the bond was satisfactory, an exponential echo pattern was produced on the scope of the attenuation measuring unit. (3) Successive readings were taken with increasing pressure on the transducer as it was applied. The attenuation reading decreased as the film of grease thinned out. (4) Stabilization of the readings, together with a consistent exponential echo pattern, indicated that the correct attenuation had been reached. The values recorded were the average of the stabilized readings. To verify that the technique did not change, a control specimen, which was not subjected to hydraulic pressure, was employed to check on the technique. Consistent readings reproducible within ±.001 db/μ sec. were obtained at all times.

Hydrostatic pressure cycles from approximately zero to 40,000 psi were applied to two or three samples simultaneously by compressing Shell Tellus hydraulic oil in a one-inch bore pressure vessel. Specimens, A Fig. 1, were supported in a steel cage mounted in the pressure vessel; see Fig. 1. The cage was provided with dowel pins, B Fig. 1, passing through transverse grooves at midlength in the polycrystalline specimens to prevent the specimens from being battered about by the hydraulic fluid. Coiled lead wire was employed at the ends of the single crystal specimen as protection against damage. Spiral grooves and soft wires C and holes D in the steel cage permitted flow of the hydraulic fluid.

Pressure was measured by a strain gage bridge cemented to the outside of a 1/2-inch bore, thick cylinder used as a pressure gage, which was attached to one end of the pressure vessel. This gage was calibrated by a dead weight tester.

The first 100 cycles of pressure were applied by hand, using a pressure intensifier and hand valves at the rate of one cycle every four minutes. Larger numbers of cycles of pressure were applied by attaching the pressure vessel to a Morrison high pressure fatigue machine[10]. This machine was designed to produce a constant pulsating pressure cycle as high as 45,000 psi. The pressure resulted from reciprocating a ram in a closed cylinder filled with oil, thus compressing the oil. In the present tests the specimens were cycled at about 400 cycles per minute. A 15°F increase or decrease occurred in the temperature of the oil in the pressure vessel when the pressure was applied or released, respectively, during hand loading. A 60°F gradual rise in temperature of the outside of the pressure vessel occurred on the long test runs. Ultrasonic attenuation measurements were made before and after each cycling on all test samples.

Results

Photomicrographs made periodically during the tests showed no slip lines or any other structural change. No change was found in typical grains spotted before and after cycling. The grain boundary deformation found by Davidson and Homan[4] in bismuth at pressures above 75,000 psi was not observed.

The attenuation for each specimen is shown as a function of number of cycles in Fig. 2. All measurements shown were made by removing the sample from the pressure vessel and reapplying the transducer. As shown in Fig. 2, both polycrystalline copper specimens showed a nearly continuous increase in attenuation with cycles of hydrostatic pressure. The oxygen free copper (OFHC) showed a greater increase than the electrolytic tough pitch (ETP) in the first 100 cycles, but the total change over 50,000 cycles was about the same, 0.043 db/μ sec.

However, the single crystal of copper showed an unexpected decrease in attenuation with cycles of pressure. The decrease for the total number of cycles was the same or greater than the increase observed in the polycrystalline copper. As shown in Fig. 2, however, the range of values for the single crystal was greater than for the polycrystals since the single crystal showed a rapid decrease up to 20 cycles and then an increase. After 100 cycles there was a gradual increase in attenuation for the single crystal as measured from the original end of the specimen. When the transducer was placed on the opposite end, no significant change was observed from 10^2 to 5×10^5.

Between 10 and 20 cycles for the OFHC and the single crystal of copper a blockage caused the pressure to be released slowly during some of the cycles. This is the only irregularity which occurred during this period, but it is not clear how it could have caused the decrease in attenuation found in these two tests.

Experimental difficulties encountered after 20 cycles on the single crystal admit of the possibility that a nonhydrostatic stress component may have been introduced in some cycles. If present this could have caused the increase in attenuation observed after 20 cycles.

It was also observed that the attenuation was different for different positions of the transducer on the end of the single crystal specimen. The same location, however, consistently gave the lowest readings, which are the values shown in Fig. 2.

The test results for the fine grain cartridge brass showed no signiicant change in attenuation. A small increase occurred between 10^2 and 6×10^3 cycles. This may have resulted from a scratch on the end of the specimen. These results, as shown in Fig. 2, indicate that the attenuation readings were probably accurate within ±0.001 db/μ sec. The change in the attenuation measured on the control specimen before and after the test run on the companion specimen differed by -0.001, -0.001, and +0.004 db/μ sec. for copper, brass, and copper single crystal runs, respectively. The control specimen used with the single crystal was a specimen previously subjected to cyclic compression.

The specimen of coarse grain brass was observed only before and after 4.5×10^5 cycles of pressure. It showed a decrease in attenuation of 0.050 db/μ sec. This single observation is so difficult to explain that it will not be considered seriously pending confirmation of the result.

Rockwell hardness measurements of the test and control specimens after completing the testing showed no significant differences.

Discussion

The results of the experiments indicate that no gross deformation occurs, but progressive changes occur on a submicroscopic scale resulting in an increasing attenuation for polycrystalline copper, decreasing attenuation in a single crystal of copper, and no change in fine grain α-brass. The fact that no change was observed in the α-brass may result from the fact that in brass the dislocation loops are more firmly pinned by impurity atoms so that motion was not produced at the pressure available in these experiments.

The increase in attenuation for both forms of polycrystalline copper may result from local deformation along grain boundaries. This may be caused from shear stresses set up by inhomogeneity and anisotropy of the elastic constants associated with the collection of atoms in an irregular array at grain boundaries. Such deformation could generate dislocations and cause breakaway of dislocation loops with increased attenuation. The widespread slip observed metallographically by Davidson and Homan[4] in polycrystalline bismuth after a single pressure application is an extreme example of the mechanism which may be operating here. The progressive increase of attenuation at a decreasing rate with increasing cycles may result from further deformation occurring on unloading and reloading each cycle until all possible deformation at the given pressure range is exhausted. Again it is the inhomogeneity at grain boundaries and anisotropy which may cause shear stresses and local deformation to occur on loading, unloading, and reloading.

As reported[11], a slight deformation (such as would occur in polycrystals under hydrostatic pressure) tends to cause longer dislocation loops than observed in annealed material or heavily deformed material.

The greater increase in damping observed up to 100 cycles of pressure for OFHC compared to ETP copper may result from the larger grain size of the former.

Miles and Gibbs[6] observed an increase in damping in polycrystalline aluminum following application of about 58,000 psi hydrostatic pressure. The damping was measured by decay of free torsional vibrations at 50 kc. per sec., a much lower frequency than employed in the present experiments. These investigators concluded that the increase in damping resulted from quenched-in hydrogen present in cavities. No increase in damping with pressure was observed when the hydrogen was eliminated. No tests were performed in the present experiments at 50 kc. per sec., so it is not known whether the observed effects would appear also at this lower frequency. It seems unlikely that hydrogen was a dominant factor in the present experiments. However, the possible presence of hydrogen cannot be ruled out completely, since the specimens were annealed in a weak reducing atmosphere rather than vacuum. Hydrogen embrittlement was not seen at the surface of either specimen of polycrystalline copper under microscopic examination.

The progressive decrease in attenuation of the single crystal of copper was unexpected. It seems clear that no change should occur in a perfect single crystal under hydrostatic pressure, unless due to diffusion of the hydraulic fluid. Diffusion during the short time of one or even 20 pressure cycles would at best affect only a thin surface layer, whereas the attenuation measurements reflect changes throughout the volume. Diffusing foreign atoms may, however, lodge in dislocations causing pinning and in this way decrease the attenuation by immobilizing dislocation loops. Since this can be only a very small surface effect it cannot account for the magnitude of change observed.

Thus, the decrease in attenuation seems to result from imperfections in the crystal which are reduced in number or mobility under the action of hydraulic pressure. Two possibilities present themselves: dissipation of dislocations or pinning of dislocation loops present in the single crystal.

Since many dislocation loops are probably not excited by a given type, direction and frequency of ultrasonic wave, only those which are active contribute to the observed attenuation. Thus, in what follows references to dislocations or dislocation loops are to be considered as references to the active, or effective, dislocations.

The presence of dislocations increases the volume of a crystal slightly. Pressure tends to cause consolidation toward the least possible volume. Vacant lattice sites would thus tend to diffuse to the surface or migrate to dislocations producing climb, and dislocation pairs which were in a favorable situation to annihilate each other would have an increased tendency to do so. Some dislocations near a surface would tend to move to the surface and vanish. These tendencies would result in decreased attenuation.

A dislocation causes a local disturbance (or inhomogeneity) in the crystal constants. Thus, local shearing stresses might be introduced (in the plane of an edge dislocation, for example) by hydrostatic pressure. A single dislocation in an infinite volume under hydrostatic pressure would experience a symmetrical state of stress including shear stresses. Because of symmetry there would be no tendency for the dislocation to move. In general such symmetry would not exist, so that bias will be present which may permit some dislocations to move under repeated hydrostatic pressure.

If this is true, application of pressure may cause many dislocations to vanish, others to move to new equilibrium positions, and still others to remain unchanged. When the pressure is released the stresses around the remaining dislocations will change. New shearing stresses will be introduced on release of pressure which will cause further motion and possible vanishing of additional dislocations. Thus, the situation is changed so that on reapplication of pressure some dislocations whose motion was blocked on the first application of pressure are caused to move and vanish.

The density of dislocations would thus decrease continuously but at a decreasing rate resulting in reduced attenuation, as observed during the first 100 cycles. The decreasing rate may be due to gradual exhaustion of mobile dislocations. Under this mechanism it is natural that the major change would occur on the first application of pressure as observed.

While pinning of dislocation loops in a way which would at least partially immobilize them after release of pressure would account for decreased attenuation, it is hard to imagine a mechanism which would cause pinning under these conditions in a single crystal. The rate of diffusion of foreign atoms into dislocations would be expected to be reduced under pressure. It might, however, be accelerated during release of pressure. The time available, however, was very small in most cycles. However, between the 10 and 20 cycle readings of the OFHC polycrystalline copper and the single crystal of copper there was a restriction of oil flow resulting in a slow release of the pressure. The results showed a decrease in attenuation in both samples. It is not clear that these observations were related.

The findings of Zumwalt, Skolnik and Ferron[12], while not corroborated by Hilliard, Lommel, Hudson, Stein and Livingston[13], support the hypothesis of pressure-induced dislocation motion. Zumwalt, Skolnik and Ferron presented X-ray evidence of motion of a few dislocations in a single crystal of aluminum toward the surface resulting in a decrease in density of dislocations at the center of the specimen and an increase at the surface. This effect was observed after release of a sustained hydrostatic pressure of 200,000 psi at room temperature. Since the ultrasonic measurements of the present paper reflect changes at the center of the specimen, the X-ray and ultrasonic observations agree.

Using an etch-pit counting technique, Hilliard, Lommel, Hudson, Stein, and Livingston[13] found no change, within an experimental uncertainty of 20 per cent, in the dislocation density of a copper single crystal following application of 58 x 10^4 psi hydrostatic pressure at 1112°F. Attenuation is considered to be a linear function of dislocation density[14] (other factors being equal). If this was the sole cause of changes in attenuation, the magnitude (47 per cent) of the pressure-induced reduction of effective dislocations in the copper single crystal reported in the present paper would be only about twice the estimated experimental error reported by Livingston[15]. Hence, it would be difficult to prove by etch-pit techniques that the reduction in attenuation was due to reduction in density of dislocations.

Other observations relating to the decrease in attenuation with cyclic pressure in a single crystal of copper include the observation of Hilliard et al[13] that sustained hydrostatic pressure at elevated temperature suppressed the migration of dislocations which occurred at elevated temperature and atmospheric pressure in LiF. This observation was made by a double etch technique on samples containing an extremely low dislocation density. It thus appears that while the pressure was on the pressure tended to pin dislocations, at least at the elevated temperature. Thus it would be expected that the mobility of dislocation loops would be reduced while under pressure, and hence damping would decrease. In the present experiments a permanent effect remained after release of pressure and at room temperature.

The observed difference in attenuation with position on the end of the sample was determined not to be a side wall effect. The cause is obscure but may result from a nonuniform distribution of dislocations and dislocation loop lengths within the crystal.

If, as suggested, pressure tends to reduce the dislocation density in a single crystal of copper, it should have a similar effect in a polycrystal except as inhibited by grain boundaries. Thus, the increase in attenuation of polycrystalline copper may be the net effect from two opposing tendencies: (a) reduction of dislocations within crystals causing reduced attenuation; and (b) breakaway of dislocation loops and possibly generation of dislocations due to small deformation at grain boundaries causing longer loop length and increased attenuation: the latter being dominant.

The fact that α-brass showed no change in attenuation at the pressures employed might result from a balance of the two tendencies discussed above or because the pressure employed was insufficient for these materials. This might also be true of the hydrogen free aluminum[6].

Conclusions

Cyclic hydrostatic pressure was found to cause ultrasonic attenuation to increase with cycles in polycrystalline copper, to decrease in a single crystal of copper and to remain unchanged in α-brass. The changes in copper have been attributed: (a) to pressure-induced reduction of dislocation density in crystals, causing reduced attenuation; and (b) to deformation at grain boundaries and breakaway of dislocation loops resulting from inhomogeneity and possibly anisotropy at the grain boundaries causing increased attenuation.

Further work would be desirable to verify the findings, to study materials having stronger anisotropy and to determine by other means, if possible, whether the dislocation density does decrease under the action of cyclic hydrostatic pressure.

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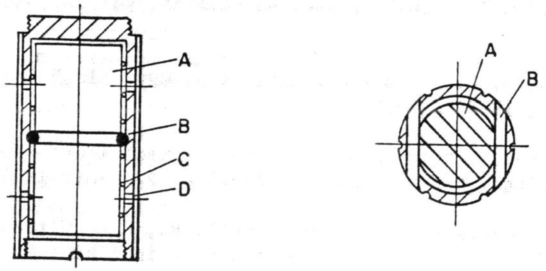

Fig. 1. Cage Used to Support a Specimen under Hydrostatic Pressure.

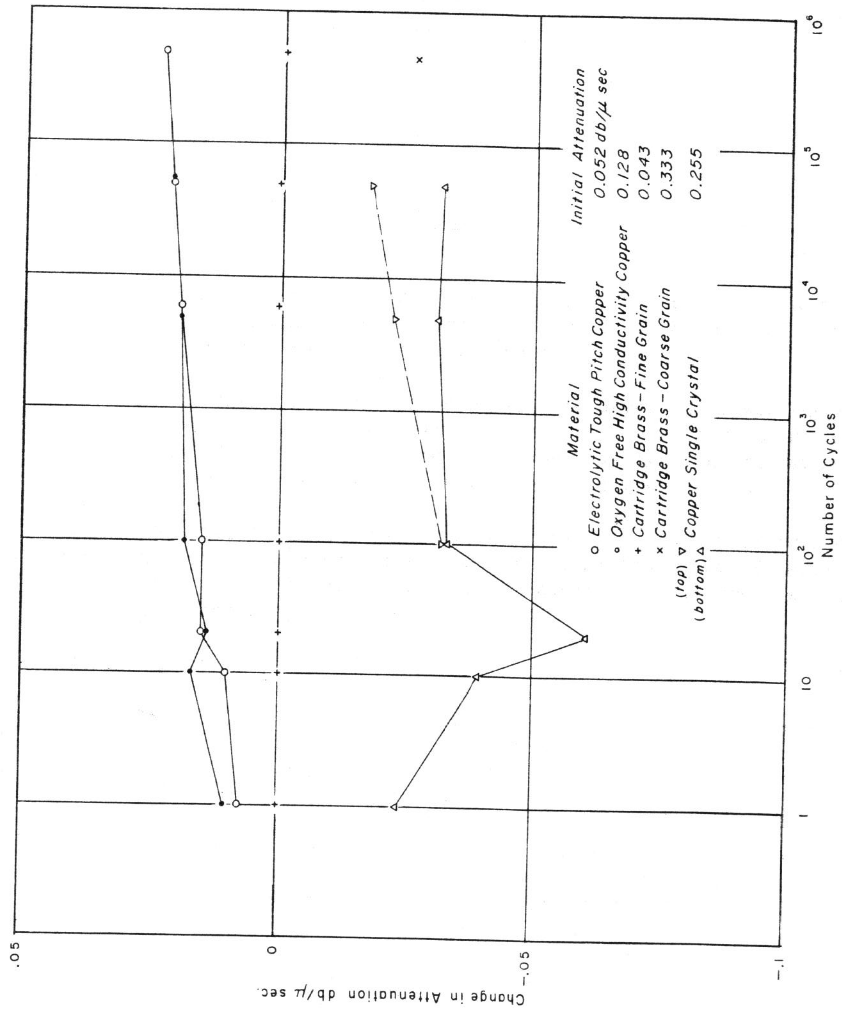

FIG. 2 EFFECT OF CYCLES OF HYDROSTATIC PRESSURE FROM ZERO TO 40,000 psi ON THE ATTENUATION OF ULTRASONIC PULSES