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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS3-7981

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Fourth Quarterly Progress Report

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ABSTRACT

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Molybdenum and rhenium wires have been cycled and tested ultrasonically up to 4000[°]R. After annealing, the wires exhibited sound velocities which were in reasonable agreement with velocities calculated from independent elastic moduli data.

Ultrasonic instrumentation for automatically measuring temperature has been constructed. Preliminary bench tests of the unit have been conducted using a two inch sensor at room and elevated temperatures.

L SUMMARY

Molybdenum and rhenium wires have been thermally cycled and tested ultrasonically up to 4000° R. After cycling, the wires exhibited sound velocities which were in reasonable agreement with velocities calculated from independent elastic moduli data.

Ultrasonic instrumentation for automatically measuring temperature has been constructed. Preliminary bench tests of the unit have been conducted using a two inch sensor at room and elevated temperatures.

IL. PROGRESS

A. High Temperature Ultrasonic Tests

Several molybdenum and rhenium wires of 0.040 in. dia. x 2 in. sensor length have been thermally cycled, including two hour soaks at 4000°R, at a pressure of ~10⁻⁵ torr. The oven used was an Abar Model C3T2-Lab. This oven utilizes a folded tungsten strip, 1-1/4 in. wide x 0.010 in. thick, as the heater element. Figure 1 schematically shows the oven hot zone, specimen and thermocouple position, sight port and radiation shields. Preliminary runs to ~2300°R were required to bake out the oven, to reduce outgassing sufficiently. Saturable reactor temperature control over the range from room temperature to 5000° R was accomplished by modifying the available 115 kva Miller dc power supply, Model 30*4C, to operate on only one of its three phases. With 220 volts applied to the primary, temperatures to 3800° R are obtained, and with 440 volts, temperatures to at least 5000° R are obtained.

The maximum heater element temperature was measured with an optical pyrometer. Maximum specimen temperature was measured with W-5% Re/W-26% Re thermocouples, whose junctions were at the same relative position as the ends of the ultrasonic sensors. Discrepancies between thermocouple and pyrometer readings indicate thermal gradients in the hot zone, as much as 180° R in some cases.

The thin wire technique (see First Quarterly Progress Report, NASA CR-54780, October, 1965) was used for the measurements. Extensional wave echoes were obtained from the kink and end of 2 in. straight samples (and also a 3 in. coiled Re sample) using 300 kHz video pulses. The 0.040 in. dia wire specimens were electrically

1

welded to 10 to 15 ft lengths of 0.040 in. dia Mo lead-in wire, which in turn was welded to a 0.050 in. dia magnetostrictive wire.

Extensional wave sound velocity was measured during heating. soaking and cooling. Typical results are plotted in Figures 2, 3 and 4. Figure 2 shows the initial molybdenum heating and cooling run after a 2 hour soak at 4500 R. As can be seen, the initial heating values are on a different curve than the annealed values. This is probably due to the effects of recovery and recrystallization. The annealed points are within + 3% of all measurements. By repeatedly cycling an annealed sample (not shown) it was found that each run generates a curve slightly different from the previous runs. The spread in measured values is to be expected since Mo is, to some extent, anisotropic. As the grains reorient, the sound velocity along the wire's axis represents a slightly different averaging process of the individual grains. It has been estimated that the velocity-temperature curve would stop changing when the grains occupy the full diameter, and are about 1 in. long. It appears that recrystallization effects could also be eliminated by using single crystal sensors.

The question of how much annealing is required to fully anneal the specimen must be answered experimentally. Fifty years ago, Langmuir^{*} showed that a drawn tungsten wire undergoes a 15 to 20% decrease in cold resistance when first heated to 2700° R for 1 minute, and a further decrease of 2 to 4% upon aging for 24 hours at 4300° R. Much later, Langmuir and Taylor^{**} pointed out that Kannuluik^{***} measured resistivities which were high by 13% due to annealing tungsten only at 2800° R.

With respect to sound velocity measurement, it appears that annealing should be done at a temperature above the sensor's maximum use temperature, or 5000° R in the present work.

- *L Langmuir, Phys. Rev. 7 302 (1916), see p. 313.
- **Langmuir and Taylor, Phys. Rev. <u>50</u> 68 (1936).
- ***W. G. Kannuluik, Proc. Roy. Soc. (London) <u>A131</u> 320 (1931); <u>A141</u> 159 (1933).

Using published data^{*} for Young's modulus E. sound velocity v was calculated from $v = \sqrt{E/\rho}$. Figure 3 compares these calculated values with our values measured in Mo after several annealing cycles. The graph shows that from room temperature to \sim 3500[°]R our results agree with the generally accepted Armstrong and Brown data to + 1%. Above that temperature the calculated Armstrong and Brown values are lower than our measured ones, for example, about 4, 7% low in Mo at 4000°R. Future runs will determine the degree of reproducibility obtainable in these polycrystalline wires after prolonged soaking and repeated cycling. Discrepancies are apparently attributable to a thermal gradient in the hot zone, such that the average sensor temperature is less than the thermocouple temperature. The effects of thermal gradients may be eliminated by testing various length samples and extrapolating the results to the condition of l = 0, or a point of the material. Use of small coiled sensors may also obviate this thermal gradient problem.

Thermal gradients, however, pose no serious problem with respect to the radiation experiment. Different samples of the same materials, when installed in the same oven location, yield reproducible sound velocity vs. temperature curves. Thus, ultrasonic tests before and after radiation will determine the extent of radiation effects on sound velocity.

Final calibration up to 5000° R will utilize a uniform hot zone, such that there is no thermal gradient over the sensor length. The present tests were purposely restricted to 4000° R to prolong heater life, and to avoid interruption of runs where as many as five sensors were heated simultaneously.

Rhenium

The results in Re (Figure 4) show that Re is much more isotropic with respect to extensional wave sound velocity than Mo since successive cycles repeat previous cycles to $\sim 0.3\%$. The present work is in excellent agreement with the previously published data of Sims and Jaffe^{**}

^{*}P. E. Armstrong and H. L. Brown, Trans. Met. Soc. AIME 230, 960-966 (Aug. 1964); B. A. Kalugin and L. G. Mikhailov, Akusticheskii Zhurnal 12 (1) 114-116 (1966).

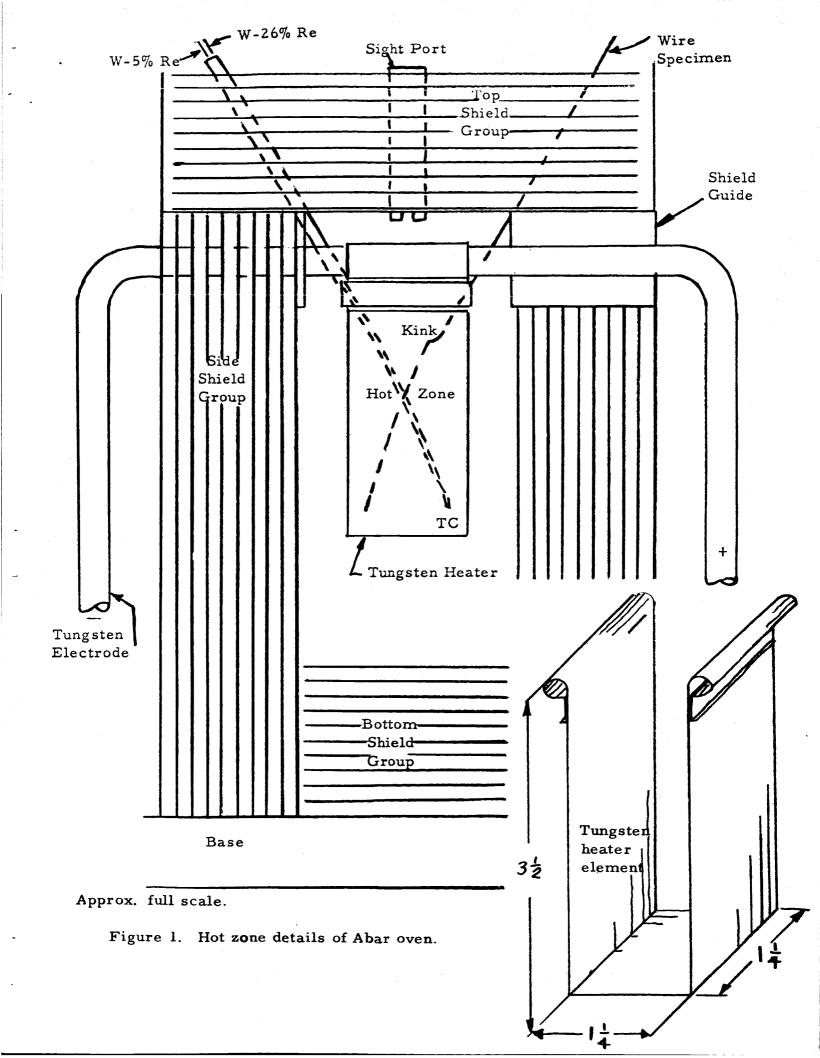
C. T. Sims and R. L. Jaffe, "Further Studies of the Properties of Rhenium Metal," AIME Trans., 206 (8), pp. 913-917 (1956); C. T. Sims, "Properties of Rhenium," pp. 23-35 (see p. 26, Fig. 3) in B. W. Gonser (Ed.), Rhenium, Elsevier Publishing Co., Amsterdam-New York (1962).

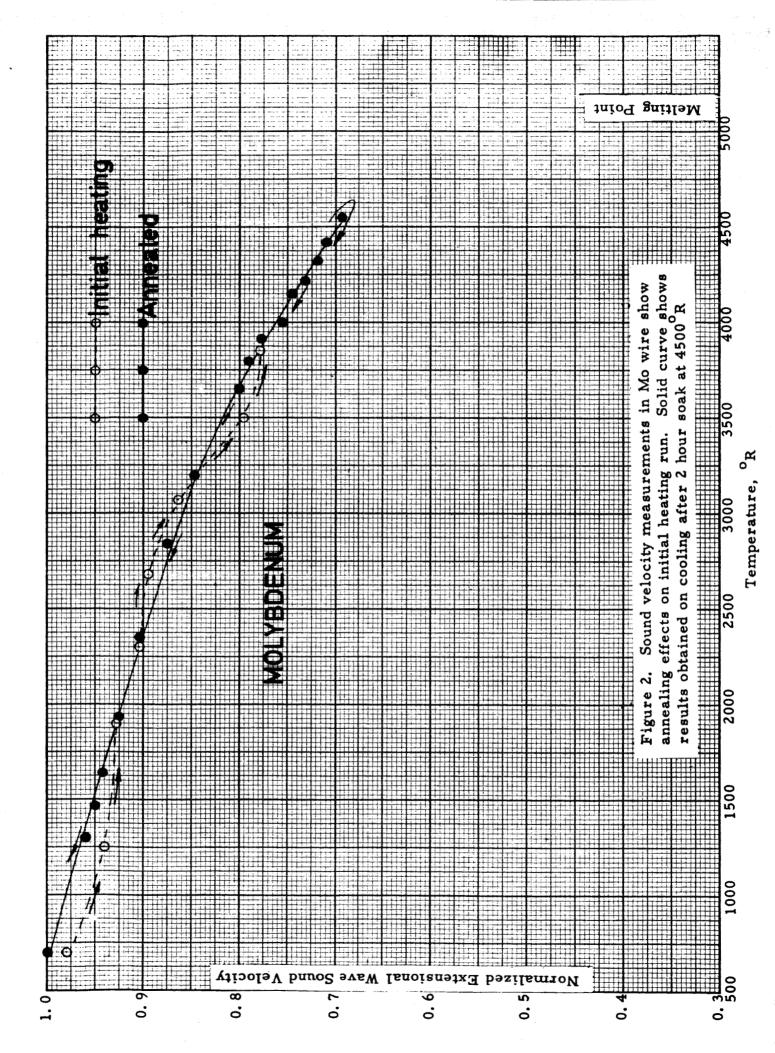
up to 2100° R, the highest temperature at which published Re modulus data are available.

In the future, rhenium will be tested in intimate contact with graphite, at successively elevated temperatures up to 5000[°]R, to determine whether graphite significantly changes the sound velocity in rhenium.

B. Radiation Test

Radiation effects will be determined by testing the same wire material before and after irradiation. Wires of Mo, Re, W, Cb, Ta and W-Re alloys will be shipped to B & W in the first half of July. These wires will be irradiated starting in the second half of the month. The wires will subsequently be tested at room and elevated temperatures, in a vacuum environment, and then in a graphite environment.





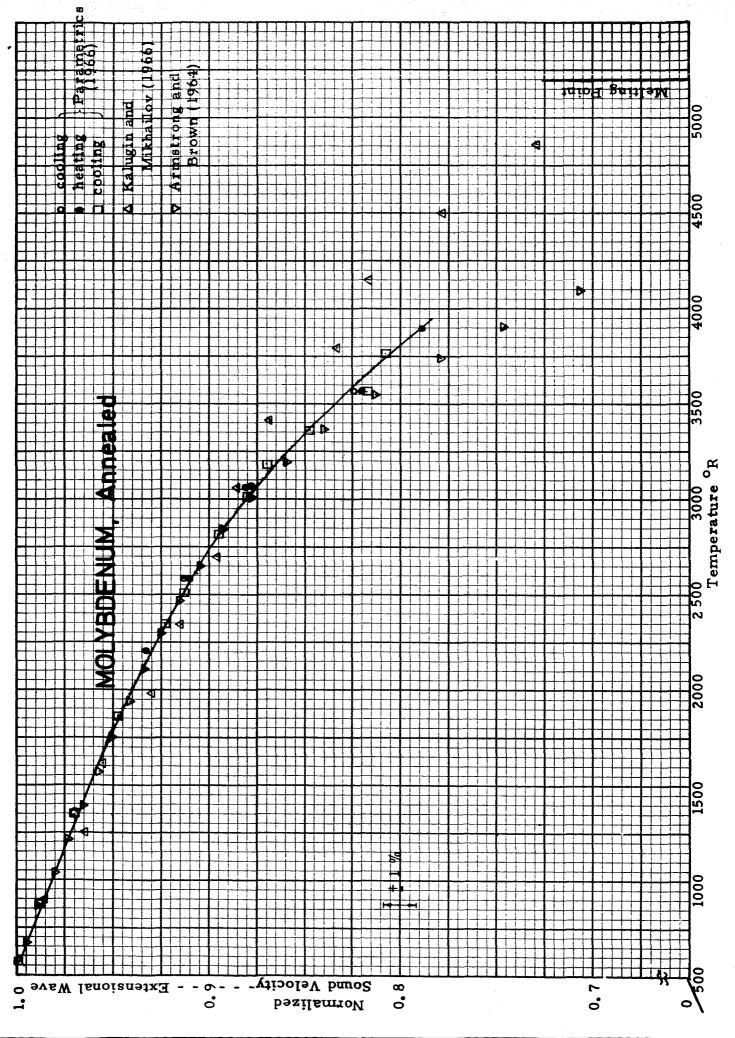


Figure 3. Temperature dependence of sound velocity in molybdenum, normalized to the room temperature annealed value

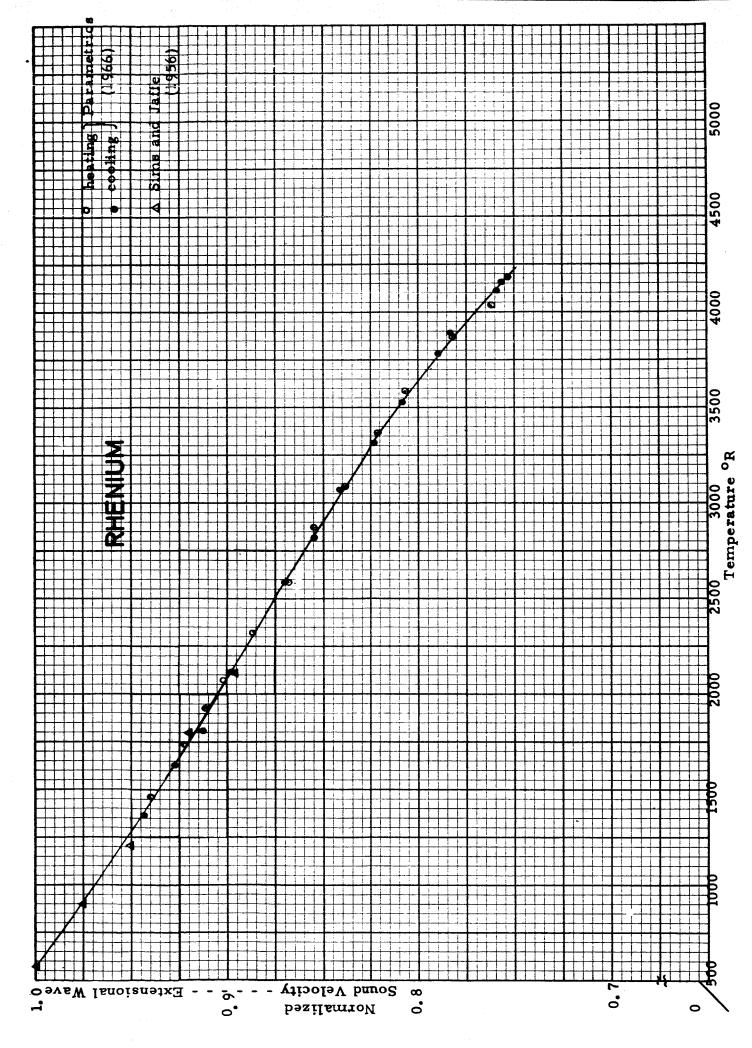


Figure 4. Temperature dependence of sound velocity in rhenium, normalized to the room temperature annealed value