EXPERIMENT SS-6

Internal Friction in Tantalum Containing Oxygen

In this experiment, kinetic processes occurring on an atomic scale will be studied by mechanical means. Measurements of the temperature dependent internal friction and shear modulus will be used to observe the rearrangement of oxygen atoms dissolved in small concentration in crystalline tantalum, when the latter is periodically stressed. This is an example of the so-called Snoek Effect.

Specifically, this experiment involves three tasks:

1) Determine the activation energy for the rearrangement of the oxygen atoms
2) Determine the diffusion constant (units: cm^2/sec) of oxygen in tantalum, as a function of temperature, and from this estimate the temperature at which 10% of the oxygen would diffuse out of the tantalum wire used in this experiment in, say, 1000 hr (many students have worked on this wire!)
3) Prepare a critical review of earlier investigations of the first task. Make an estimate of the oxygen concentration in your setup. Identify open questions or contradictory statements. To simplify this task reprints of relevant investigations are appended.

The experiment involves measuring from 50^oC to 300^oC the resonance frequencies and the amplitude decay of a torsional oscillator in which the torsional member is a tantalum wire (diameter 0.76mm, length 25cm, see the appended description of the apparatus (1)). Use six different positions of the brass counter-weights to achieve resonance frequencies between ~ 0.4 and 1.0 H_z. In order to prevent further oxidation of the wire flush argon gas through the furnace housing the Ta wire. A very small rate (about 1cfh) is adequate (is this argon flow at all necessary? See Task 2 above). The experiment is straight-forward, but as every experiment involving temperature, thermal equilibration is crucial and can be tedious. It is suggested that the experiment is done in two steps: On the first day, go through the experiment quickly, for one frequency . Demonstrate at least once that the pendulum amplitude does indeed decay exponentially with time! Evaluate the data so that you learn what to watch out for. Then go back, with one full day set aside for this purpose, and do the entire experiment in one go. In your analysis, consider both the variation of the internal friction and of the shear modules.

For a review of damped harmonic motion and the definition of internal friction, see Nowick and Berry (2). A good and concise introduction to the topic of internal friction is contained in the article by Berry (3), which is addressing problems arising in the relaxation of thin films, a topic of current interest. Earlier investigations of oxygen in tantalum are contained in Refs. (4) to (9).

Brief introductions into diffusion and random walk can be found in many texts, e.g. in Ref (10). A discussion of diffusion into various geometrics is contained in Ref. (11). Although this reference deals with the diffusion of heat, the solutions are the same for any material diffusion characterized by a diffusion constant (units: cm^2/sec). Note that in Carslow and Jaeger, the diffusion constant is labeled κ and is called: diffusivity. See the attached pages taken from this text. Note that the 10% oxygen loss (Task 2) is intended only as an approximate value.

References

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VI A. Apparatus

The apparatus is described as follows: From a brass wall bracket a torsional pendulum was hung in a vertical position by means of a 5/16" stainless steel tube about 18" long. The tube is held in a vertical hole in the wall ~~clamp~~ bracket. At the bottom of the tube is a hollow pin vise which holds the top of the torsion member, a tantalum wire 0.030" diameter, approximately 12" long. (Courtesy of G.E. Co.). Fastened to the bottom of the tantalum wire, by means of another pin vise, is a brass sleeve on which is placed a 9/16" diameter x0.006" wall thickness stainless tube which encloses the tantalum wire. The purpose of this was to form a chamber around the tantalum specimen which would be filled with argon as it passed slowly thru the 5/16 stainless tube and thru the jaws of the pin vise into the chamber. This served not only to prevent oxidation but to prevent the specimen from absorbing more oxygen and nitrogen from the air.

From the bottom of the sleeve a 1/8" diameter x 7½" stainless tube connected to the pendulum. The inertial part of the pendulum consisted of a 8½" x 1/8 diameter brass rod held in the middle. On either side was a cylindrical brass weight free to slide on the rod and on each end of the rod, with a slight press fit, was a piece of armco iron.

The pendulum was accuated on two solenoids mounted to apply a brief torque to the pendulum when a micro switch was closed momentarily. The amplitude of oscillation was measured using a galvonometer light source, mirror, and scale. The mirror was fastened to the 1/8 stainless tube just above the pendulum by G.E. glyptol cement.

From the bottom of the pendulum suspension a 1/8" diameter x3" rod extended to be partially emmersed in 30 weight oil to damp out any lateral vibrations.

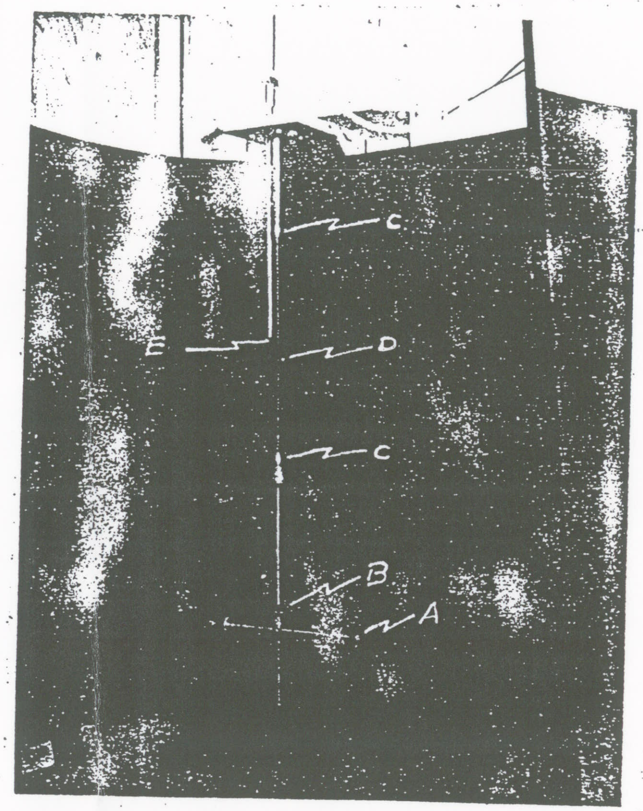

Figure 6-1. Torsional Pendulum showing (a) armco iron, (b) galvonometer mirror, (c) pin vise, (d) tantalum wire, (e) thermocouple.

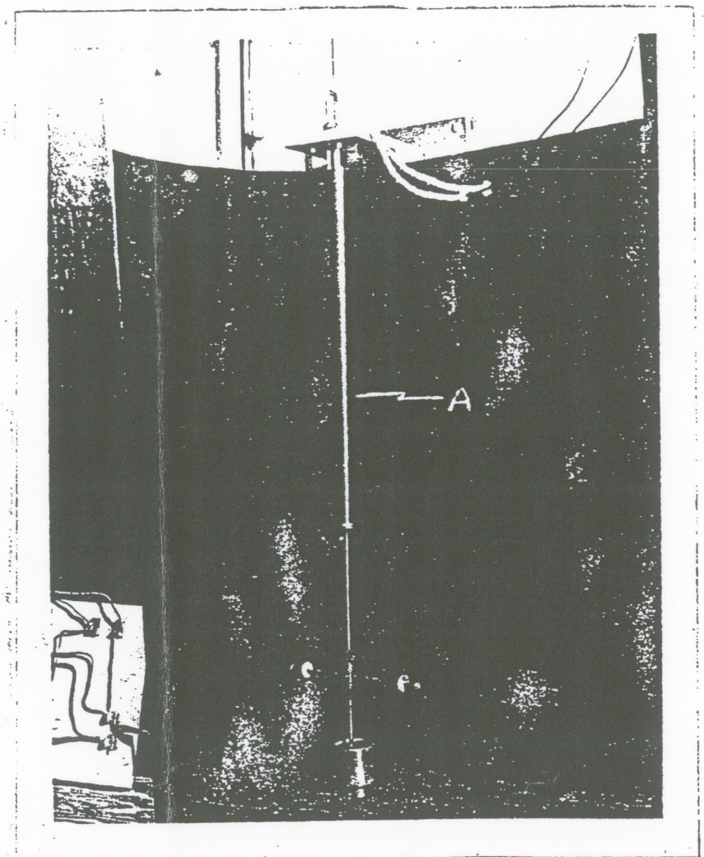

Figure 6-2. Torsional Pendulum showing (a) argon shield.

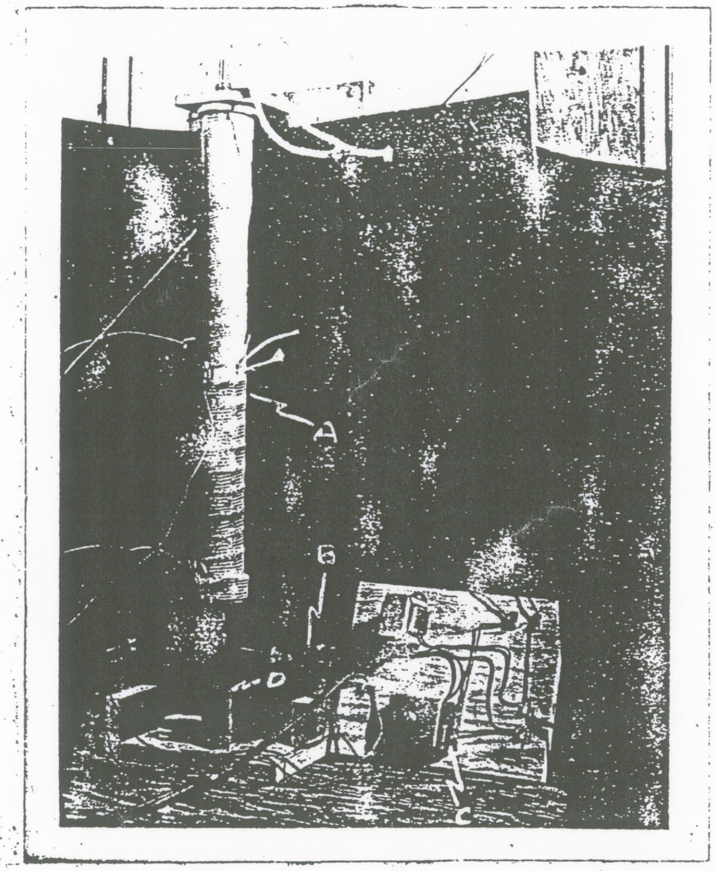

Figure 6-3. Torsional pendulum showing (a) electric furnace, (b) solenoids, (c) micro-switch, (d) damping oil.

The whole suspension system down to within about an inch above the galvonometer mirror was enclosed in an electric furnace. The furnace consisted of a stainless steel tube, 1 3/4" diameter x 22½" long which was wrapped with "Briskeat" flexible heating tape. Two six ft. lengths of 1" wide tape were required to cover the furnace with a little overlap at the ends. The two tapes were connected in parallel to a General Radio type 10-V variac. The furnace was also suspended from the brass wall bracket.

The temperature inside the furnace was measured with a chromel-p-alumel thermocouple (with a reference junction in ice) and a Leeds and Northrup No. 8657-C, double range potentiometer indicator.