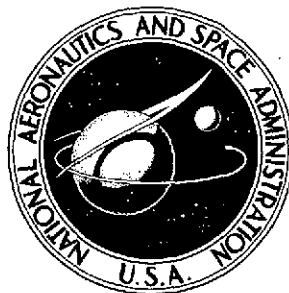


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SURFACE RECRYSTALLIZATION THEORY OF THE WEAR OF COPPER IN LIQUID METHANE

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16. Abstract The recently proposed surface recrystallization theory of wear has been found to accurately predict the wear rate of copper sliding against 440C steel in liquid methane. The wear rate was proportional to the sliding velocity squared and to the normal load for sliding speeds from 3.1 to 24 m/sec, and for loads from 0.25 to 2 kg. Transmission electron microscopy revealed that the microstructure near the wear scar surface was characterized by a fine cell recrystallized zone in which individual dislocations could be distinguished in the cell walls. The diameter of the cells was about 0.5 μ m, and the cell interiors were nearly dislocation free. The depth of this fine-cell zone was roughly proportional to the sliding velocity. A first-order analysis suggested that this depth was also proportional to the shear yield stress of the copper and inversely proportional to the recrystallization temperature and specific heat of the copper.			
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SUMMARY

Copper was subjected to sliding against 440C in liquid methane. The normal load range was from 1/4 to 2 kilograms, and the sliding velocity range was from 3.1 to 25 meters per second. Over this range of experimental parameters, the wear rate of the copper rider was found to be proportional to the sliding velocity squared and to the normal load. Transmission electron microscopy was used to study the dislocation structure in the copper very near the wear scar surface. It was found that near the wear scar surface, the microstructure was characterized by a fine-cell recrystallized zone in which individual dislocations could be distinguished in the cell walls. The interiors of the cells, about 0.5 micrometer in diameter, were nearly dislocation free. Below the recrystallized layer was a zone that was intensely cold worked by the friction process. With increasing depth, this intensely cold worked zone gradually became indistinguishable from the partially cold worked bulk of the copper, representative of the initial condition of the material. Analysis showed that the fine-celled recrystallized layer thickness was proportional to the sliding velocity and the shear yield stress of the copper, and inversely proportional to the recrystallization temperature (estimated), and the specific heat of the copper.

INTRODUCTION

It has been demonstrated that under certain circumstances the wear of metals in sliding contact occurs largely by the mechanism of intense plastic deformation of the surface and near subsurface regions. For example, Antler (refs. 1 to 3) and Cocks (refs. 4 and 5) developed a description of a "wedge flow" mechanism applicable to sliding combinations of soft metal pairs.

In a recent work on the investigation of experimental parameters affecting adhesive metal transfer (ref. 6), Landheer and Zaat pointed out that the wedge flow mechanism might be suppressed when a soft metal is slid against a hard, flat metal surface. This is in agreement with the observations made by the authors of this report in the case of relatively soft copper in sliding contact with a 440C steel disk, reported in reference 7. In this case the wear occurred by the incremental shearing of copper in a thin recrystallized layer comprising the wear scar surface. The copper gradually migrated toward the exit of the wear scar in a series of plastic "waves" forming a neatly layered mass of wear material adhering to the wear scar exit. The model proposed to describe this mechanism included the effects of load and velocity on the observed wear rates.

An article by Suh (ref. 8) describes a wear model based on dislocation dynamics in which wear is envisioned to occur by the coalescence of voids or cracks. The cracks form at a critical distance below the sliding surface. This is the distance over which dislocation image forces due to the proximity of a free surface can overcome the internal friction forces. Thus the wear occurs by the "delamination" of sheets of metal from the nearly dislocation-free surface layer.

The objectives of this report are threefold: (1) to refine the model proposed by the authors in reference 7 (the model to be called the recrystallization theory of wear); (2) to expand the range of loads and speeds over which the recrystallization model is observed to be valid (The ranges of sliding speeds and loads were 3.1 to 25 m/sec and 0.25 to 2 kg, respectively.); (3) to examine the microstructure present in the copper nearest the surface of the wear scar by transmission electron microscopy and optical microscopy; and (4) to further develop the wear model proposed in reference 7 showing how the recrystallized layer thickness relates to the material properties of the copper.

SYMBOLS

A	real contact area
A'	apparent contact area (wear scar area)
b	dislocation Burgers vector
C _P	specific heat
D	average diameter of discrete contact event
d	average spacing between discrete areas of real contact
G	shear modulus
H	dislocation-free layer thickness proposed in ref. 8

h	recrystallized layer thickness
h_0	reference value of h
k	shear yield stress
L	normal applied load
N	rate at which discrete contact areas move across wear scar
\dot{Q}	rate of frictional heat generation
S	cell size
T	temperature
T_R	recrystallization temperature
V	sliding velocity
V_0	reference value of V
v	volume
\dot{W}	wear rate (volume)
μ	coefficient of friction
ρ	density

APPARATUS AND PROCEDURE

The apparatus used in this study is shown in figure 1 and is more completely described in reference 9. The basic elements consisted of a hemispherically tipped 4.76-millimeter-radius rider specimen, composed of 99.95 percent purity copper ($R_E = 85$), held in sliding contact with the lower flat surface of a 63.5-millimeter-diameter rotating disk composed of 440C steel ($R_C = 58$). The experiments were conducted with specimens completely submerged in liquified methane. The sliding speed was varied from 3.1 to 25 meters per second (1000 to 8500 rpm). The rider specimen was loaded against the disk by a helium-pressurized bellows assembly. The frictional force and the normal load were measured by strain-gage dynamometer rings. The wear was continuously measured by a linear voltage differential transformer (LVDT). The load, wear, and friction coefficient were continuously recorded during a sliding experiment.

The test chamber was cleaned with 90 percent ethanol before each run. After cleaning and the installation of the specimens, the test chamber was closed, purged for 15 minutes with helium gas, and then filled with liquified methane. After the test

chamber was full and the liquid boiling has stabilized, the rider specimen was loaded against the disk and the disk was rotated at the desired speed. The duration of the runs was 1/2 hour.

The disk specimen preparation was as follows: The surfaces were (1) finish ground and lapped to 5×10^{-2} micrometer rms, (2) scrubbed with moist levigated alumina, (3) washed in tap water, (4) washed in distilled water and air dried.

Transmission electron microscopy specimens were prepared from a 99.95 percent copper rider in the following manner. After being subjected to sliding wear, a circular cut circumscribing the wear scar was electron discharge machined to about a 6-millimeter depth below the wear scar surface. The nose of the rider was cut off 6 millimeters below the wear surface, and the cylinder generated by the circular electron discharge machine (EDM) cut was extracted. Slices about 200 micrometers thick were then cut, by EDM, from the cylinder. The first slice thus cut had the wear scar as one of its surfaces. The disk-shaped slices were then electrolytically thinned to about 500 Å and prepared for transmission electron microscopy studies. Comparison of the micrographs from different depths revealed no obvious artifacts due to the EDM process. In the case of the first slice, with the wear scar as one of its surfaces, thinning was carried out from the inner surface, and the oxide film was removed from the wear scar surface. In this manner, a transmission electron microscopy specimen was obtained that was as close as practicable to the wear surface itself.

The rate of wear to the copper rider was calculated from the continuously monitored LVDT measurements. The wear volume at any instant is given by

$$v = \frac{1}{6} \pi l \left(l^2 + \frac{3}{4} a^2 \right)$$

where l and a are defined in figure 2. Since $l^2 \ll a^2$,

$$v = \frac{\pi}{8} l a^2$$

From figure 2,

$$a^2 = 8rl - 4l^2 = 38.2 l - 4l^2$$

since r is a constant 4.76 millimeters. Now,

$$v = \frac{\pi}{8} l (38.2 l - 4l^2)$$

The wear rate is obtained by

$$\dot{W} = \frac{dV}{dt} = \frac{\partial V}{\partial l} \frac{\partial l}{\partial t} = (30.6 l - 4.8 l^2) \frac{dl}{dt}$$

The instantaneous wear rate is calculated by measuring the slope of the l -curve and the value of l at the instant in question.

RESULTS AND DISCUSSION

Wear Rates and Microscopic Observations

The wear rate measurements are shown graphically in figures 3 and 4. In figure 3, the wear rate is plotted as a function of sliding velocity for the four values of normal load. For all normal loads, the wear rate is roughly proportional to the sliding velocity square. Figure 4 shows the wear rate as a function of normal load for the four values of sliding velocity. The wear rate is seen to be approximately proportional to the normal load. These observations agree with the theoretical relation proposed in reference 7.

The essence of this wear model (ref. 7) was that wear to the copper rider progressed by the extensive plastic deformation, in a series of discrete steps or increments, of a very fine grained recrystallized surface layer. In this way the wear material was transported in an incremental manner to the exit region of the wear scar where it formed an adhering tail observed to have a lamellar microstructure.

Measurements of the recrystallized layer thickness on specimens subjected to sliding under various load and sliding velocity conditions were made after 1/2 hour of sliding. Cross-sectional micrographs from two of the specimens are shown in figure 5 and the recrystallized layer thicknesses h are summarized in table I. Comparison of these data suggest that the thickness of the layer is roughly proportional to the sliding velocity and that the load has a rather secondary effect. This is reasonable if one envisions the recrystallization as occurring under discrete regions of instantaneous real contact between the rider and the disk. The normal load would determine the proportion of the wear scar over which simultaneous recrystallization occurs. The amount of heat generation under a contacting region on the rider surface is determined by the shear yield strength of the layer, a material property, and the shear rate related to the sliding velocity. It is the amount of heat generated in the discrete contacting regions that governs the extent and depth to which recrystallization occurs. Therefore, the recrystallized layer thickness is a function of sliding velocity and not so much a function of load. This theory is further developed in the following paragraphs.

The very fine microstructure of the recrystallized regions nearest the wear surface is revealed in the electron micrographs of figure 6. The metal nearest the wear surface consists of a very fine cell structure, the average cell diameter being 0.3 to 0.4 micrometer. The cell walls are sharply defined with the individual dislocations forming the walls being clearly discernible. The interiors of these cells are nearly dislocation free. Such a microstructure is indicative of the very early stages of recrystallization in which new grains are nucleated from a cold worked microstructure (ref. 10). The recrystallized layers are not exposed to the frictional heating for a sufficient length of time to allow the development of large grain sizes to occur before being transported out of the contact region.

In comparison to the recrystallized microstructure described previously, figure 7 reveals an extremely cold worked microstructure typical of the substrate metal about 200 micrometers below the wear surface. The significant features are extremely dense dislocation entanglements and some evidence of ill-defined cell walls. Even the barely distinguishable cell wall network was not seen everywhere.

For comparison, micrographs of specimens made from regions 3 millimeters below the wear surface, representing the initial microstructure of the copper rider, are shown in figure 8. An intermittent, elongated cell structure is seen with some dislocation entanglements in the cell interiors. The cells that do exist are roughly 2 to 3 micrometers long and 0.5 micrometer wide. This sort of microstructure indicates that the starting material was cold worked to some degree.

Thus, with increasing depth below the wear surface of the copper, regions showing three rather distinct microstructures are identifiable. First, there is the recrystallized layer nearest the wear surface produced by the combination of plastic deformation and frictional heating. Just below this layer is a region of intense plastic deformation resulting from the frictional shear forces. Finally, toward the interior of the copper specimens there is the partially cold worked microstructure that represents the initial condition of the metal.

Theoretical Development of Proposed Wear Theory

The observation that the interiors of the cells nearest the wear surface are almost dislocation free is in agreement with the mechanism proposed in reference 8. In reference 8, however, it was hypothesized that the dislocations in the uppermost layers were drawn out of the metal by the surface image forces experienced by the dislocations. In the appendix it is shown that the image forces would be insufficient to move a dislocation out of the interior of the cell structure found in the copper specimens. The nearly dislocation-free cell interiors must therefore have resulted from the movement of dislocations from entanglements, such as those seen in figures 7 and 8, to

more stable positions in the cell walls during the recrystallization process.

The extreme ductility of the wear scar surface may now be understood on the basis of the observed microstructure. The recrystallization process is a dynamic one occurring simultaneously with the plastic deformation that takes place in the thin surface layer during wear. Strain hardening and crack initiation, both of which normally accompany such extensive plastic deformation, are prevented from occurring by this recrystallization.

The wear model proposed in reference 7 is given by

$$\dot{W} \propto \frac{h_0}{V_0} \frac{1}{DA'} \frac{V^2 L \sqrt{1 + 4\mu^2}}{2k} \quad (1)$$

The term h_0/V_0 can now be further developed to show the relation between the recrystallized layer thickness to sliding velocity ratio and various properties of the copper.

The development of a relation between h_0/V_0 and the properties of the copper is based on the assumption that shear heat is generated in a small volume of metal under a discrete area of real contact. It is further assumed that this heat generation occurs over a very short period of time so that heat-transfer effects may be neglected. The combination of shear heating and plastic deformation results in the recrystallization of the small volume of copper under consideration. Since the recrystallization process is a somewhat exothermic reaction, once triggered by the temperature rise due to frictional shearing, it will perpetuate itself for a short period of time after the frictional shearing event.

The volume of material being sheared during a contact event is approximately D^2h . The rate of heat generation in this volume is given by

$$\dot{Q} \approx D^2h \frac{V}{h} k$$

Now, neglecting heat-transfer effects, the temperature of this volume will increase with time according to

$$D^2h\rho C_P \frac{dT}{dt} = D^2h \frac{V}{h} k$$

which may be approximated by

$$\rho C_P \frac{T - T_0}{t} = \frac{V}{h} k$$

where T_0 is the initial temperature of the volume, and t is the duration of the event. The problem now is to find the layer thickness h which undergoes recrystallization. The recrystallization temperature, a function of prior cold work, purity, and grain size, is taken to be about 288°C (550°F) (ref. 11). The time duration of an event is approximated as being 10^{-4} second, the time required for 1 millimeter of motion at 10 meters per second. The effective bulk temperature T_0 is assumed to be about 0°C . Thus, a 288°C (550°F) temperature rise is considered to occur in a layer of thickness h in 10^{-4} second. Then, for copper

$$\frac{h}{V} = \frac{h_0}{V_0} = \frac{kt}{\rho C_P (T_R - T_0)} = \frac{1.38 \times 10^7 \text{ N/m}^2 \times 10^{-4} \text{ sec}}{8.94 \times 10^6 \text{ g/m}^3 \times 0.1 \text{ cal/(g)}(^{\circ}\text{C}) \times 4.18 \text{ N-m/cal} \times 288^{\circ}\text{C}}$$

$$\approx 10^{-6} \text{ sec}$$

or at a 10-meter-per-second sliding speed, $h \approx 10$ micrometers, on the order of the observed recrystallized layer thickness.

Referring now to table II, the wear results are not inversely proportional to A' (the wear scar area), contrary to the behavior predicted in equation (1). The inverse proportionality to A' entered the wear model as a result of the original assumption that the wear rate should decrease as the average spacing between discrete areas of real contact increased. The spacing was expressed as being proportional to the apparent contact area (wear scar surface area).

On the other hand, if the discrete areas of real contact are generated by a process such as the thermoelastic instability model proposed by Burton (ref. 12), the spacing between these areas should not increase strongly as the wear scar area grows. The average rate at which these discrete contact events move across the wear scar may be expressed as

$$\dot{N} \propto \frac{VA}{D^2 d} = C \frac{V}{D^2 d} \frac{L \sqrt{1 + 4\mu^2}}{2k} \text{ (see ref. 7)}$$

where A is the total real contact area, and d is the average spacing between the discrete contact events comprising the total real contact area. The wear volume per event v is given by

$$v \approx hD^2 = \frac{h_0}{V_0} VD^2$$

as described in reference 7. The wear rate may now be expressed as

$$\dot{W} \propto \frac{VL \sqrt{1 + 4\mu^2} h_0 VD^2}{D^2 d 2k} = C \frac{h_0 V^2 L \sqrt{1 + 4\mu^2}}{V_0 2kd}$$

which implies that

$$\dot{W} = \frac{C}{d} \frac{h_0}{V_0} \frac{V^2 L \sqrt{1 + 4\mu^2}}{2k} \quad (2)$$

The wear scar area A' does not appear in equation (2). The only undefined quantities are C and d , the spacing between microscopic areas of real contact. Lacking sufficient information to describe d , the ratio C/d may be treated as a single constant applicable to the data in figures 3 and 4. The wear rate data combined with the estimated value of h_0/V_0 gives a value of 10^{-5} centimeter $^{-1}$ for C/d . Equation (2) could form a basis for predicting the wear of sliding combinations of soft metals in continuous contact against a hard material in liquid methane if the ratio C/d is not found to vary significantly for different material combinations.

CONCLUSIONS

High purity copper was subjected to sliding against 440C steel at various sliding speeds and normal loads in a liquid methane environment. Measurements of wear rate of the copper in conjunction with microstructural studies led to the following conclusions:

1. The wear rate was proportional to the velocity squared and to the load, as proposed in reference 7, over the load and speed ranges of 0.25 to 2 kilograms, and 3.1 to 25 meters per second, respectively.

2. With increasing depth below the wear scar surface, three zones consisting of different microstructures were identified:

- a. The region immediately below the wear scar surface consisted of a very fine cell ($\sim 0.3\text{-}\mu\text{m}$ cell diam.) recrystallized structure. The cell interiors were nearly dislocation free, and individual dislocations comprising the cell walls were visible.

- b. Below the recrystallized region was a heavily cold worked layer. The cold work resulted from the friction process.

c. Finally, with increasing depth, the microstructure typical of the bulk copper was encountered. This was characterized by a moderate amount of cold work.

3. The thickness of the fine-cell recrystallized region was found to be roughly proportional to the sliding velocity. Further analytic approximation showed that, at a given sliding speed, the thickness of the recrystallized layer may be expressed in terms of the shear yield stress, specific heat, and recrystallization temperature of the copper.

Lewis Research Center,
National Aeronautics and Space Administration,
and
U.S. Army Air Mobility R&D Laboratory,
Cleveland, Ohio, August 22, 1974,
501-24.

APPENDIX - DISLOCATION IMAGE FORCE CALCULATION

The segment of dislocation shown in figure 9 is attracted to the nearby free surface according to the following equation (ref. 13):

$$F = \frac{Gb^2}{4\pi\left(\frac{S}{2} - y\right)} dx, \text{ where } y = y(x)$$

where F is the image force experienced by the dislocation line segment. This image force is opposed by a dislocation line tension force T given by

$$T = Gb^2$$

Summation of the forces on a differential segment of dislocation line gives

$$-T \frac{dy}{dx} + T \frac{dy}{dx} + T \frac{d^2y}{dx^2} + F = 0$$

which implies that

$$\frac{d^2y}{dx^2} = - \frac{1}{4\pi\left(\frac{S}{2} - y\right)}$$

The boundary conditions on y are

$$y(0) = 0; \quad \left. \frac{dy}{dx} \right|_{x=S/2} = 0$$

There is no known analytic solution for the aforementioned equation, so an approximate technique is followed. This technique is a negative definite test as to whether the dislocation line will be drawn out of the crystal by the free surface image force.

The test proceeds as a series of iterative steps. A constant value is assumed for y on the right-hand side of the equation, in effect making the equation linear. As a first step, y is assumed to be zero:

$$\frac{d^2y}{dx^2} = -\frac{1}{2\pi S}$$

which implies that

$$y = -\frac{x^2}{4\pi S} + Ax + B$$

From the boundary conditions

$$y = -\frac{x^2}{4\pi S} + \frac{x}{4\pi}$$

The constant value for y chosen for the second iteration is y at $x = S/2$ from the first trial:

$$y|_{x=S/2} = 0.0199 S$$

Putting this value of y into the right-hand side of the equation results in

$$\frac{d^2y}{dx^2} = -\frac{1}{4\pi S(0.4801)}$$

The second iteration gives

$$y|_{x=S/2} = 0.0207 S$$

Using this value for the third trial results in

$$y|_{x=S/2} = 0.0208 S$$

Thus the iterative process is rapidly converging, and clearly the dislocation line will not approach the free surface. The test is considered to be negative definite because at each iterative step the image force is maximized over the entire length of dislocation line.

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TABLE I. - SUMMARY OF
RECRYSTALLIZED LAYER
THICKNESSES

Normal load, L, kg	Sliding speed, V, m/sec	Thickness of layer, μm
2	12.4	12
1	12.4	^a 10
1/2	6.2	3 to 4

^aFrom ref. 7.

TABLE II. - WEAR RATE AS A
FUNCTION OF WEAR SCAR AREA

Normal load, L, kg	Sliding speed, V, m/sec	Wear scar area, A', cm^2	Wear rate, W, cm^3/min
1/2	25	0.019	35×10^{-6}
		.049	35
		.094	40
2	12.4	0.019	78×10^{-6}
		.049	80
		.094	120
1	25	0.019	54×10^{-6}
		.094	46
1/2	12.4	0.019	15×10^{-6}
		.049	7
2	6.2	0.019	16×10^{-6}
		.049	16

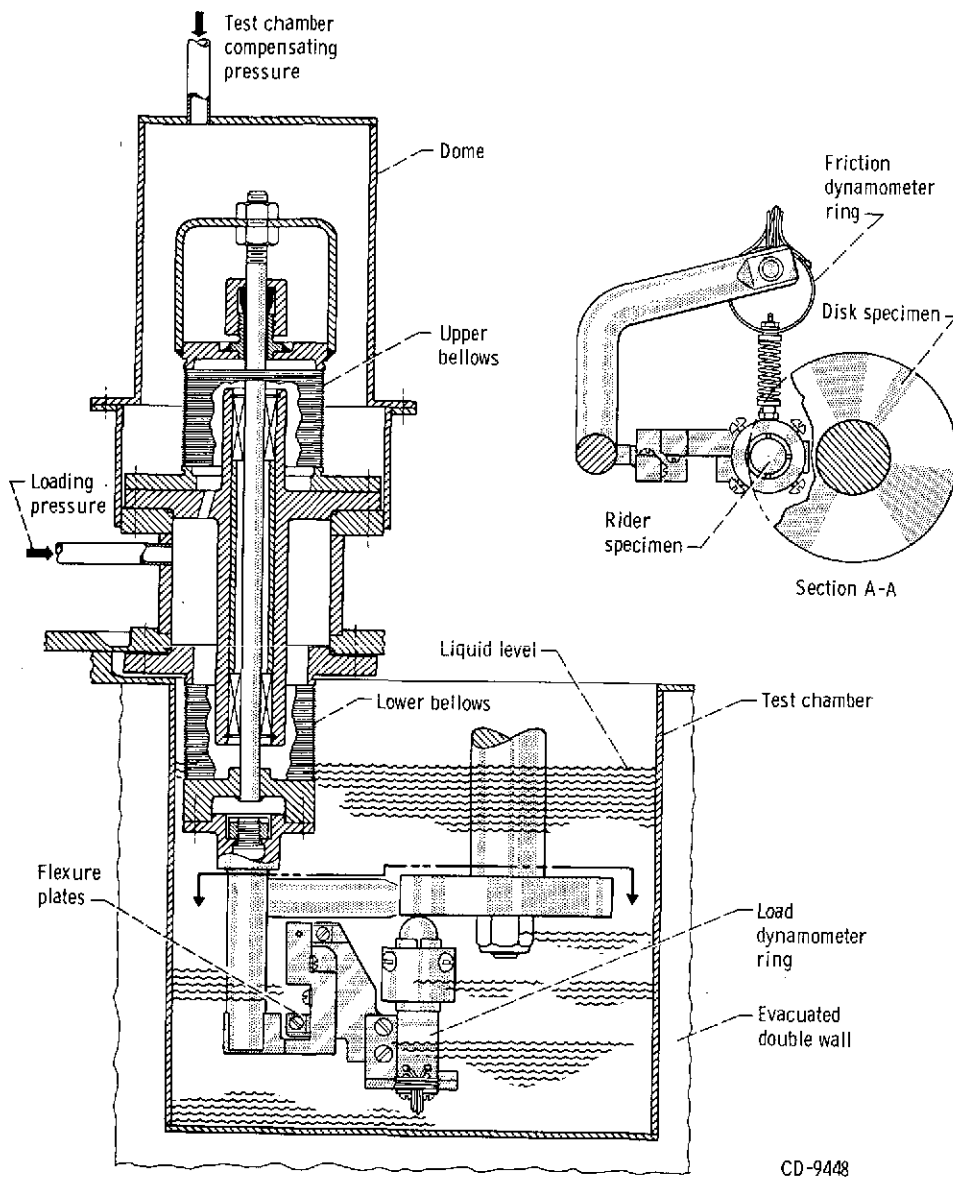


Figure 1. - Cryogenic fuel friction apparatus with specimen loading system.

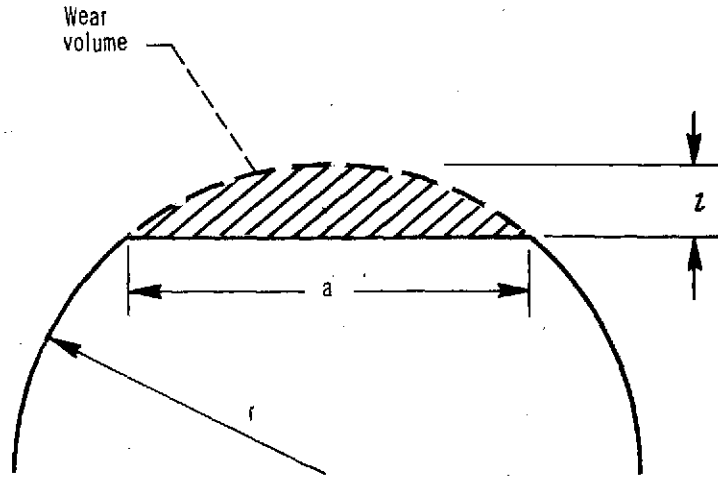
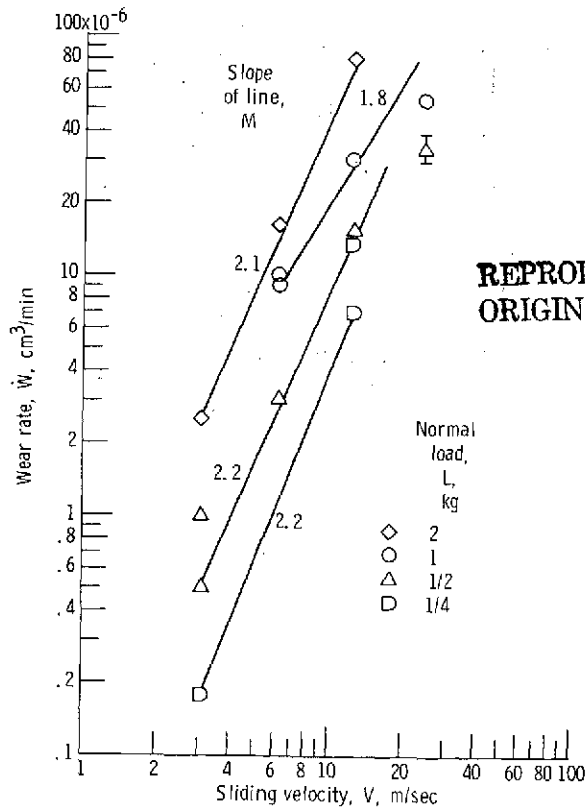


Figure 2. - Wear geometry and its relationship to continuously measured quantity Z .



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Figure 3. - Wear rate of copper as a function of sliding velocity.

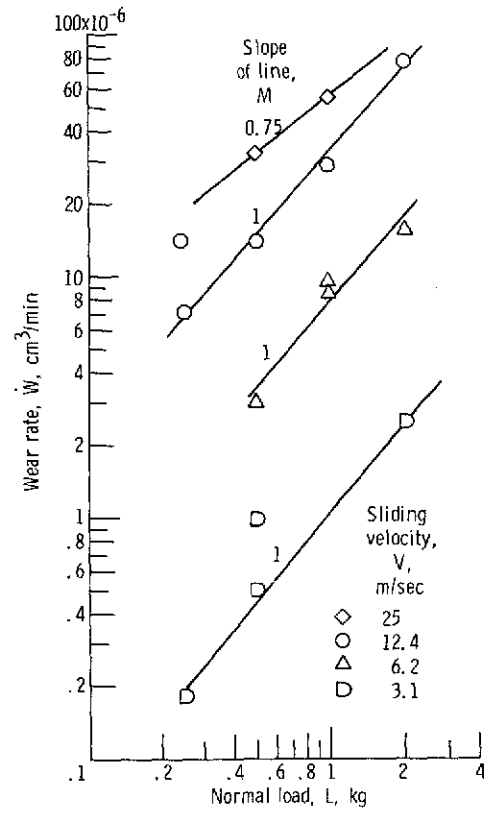
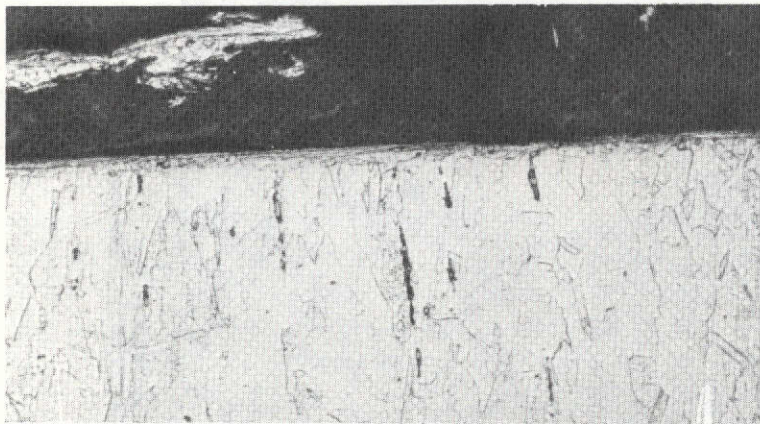
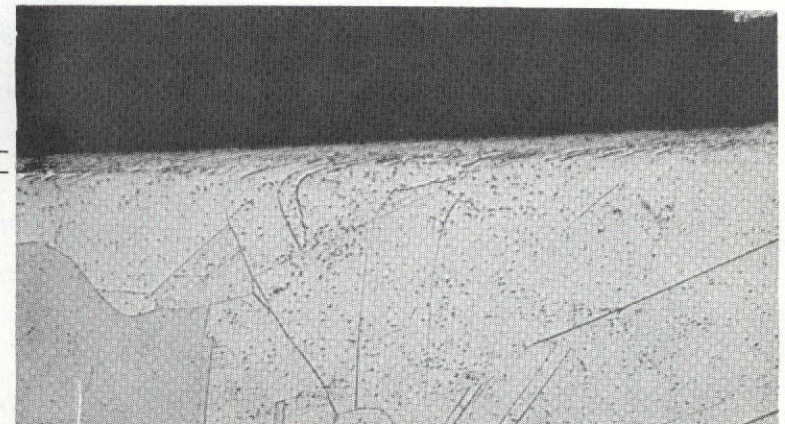
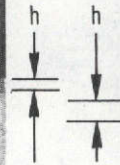


Figure 4. - Wear rate of copper as a function of normal load.



(a) Normal load, 1/2 kilogram; sliding speed, 6.2 meters per second.



(b) Normal load, 2 kilograms; sliding speed, 12.4 meters per second.

Figure 5. - Metallographic sections cut parallel to sliding direction, showing recrystallized layer thickness in a region about halfway across wear scar of copper slid on 440 C steel in liquid methane under indicated conditions. X250.



Figure 6. - Electron micrograph of copper subjected to sliding against 440C steel in liquid methane. Normal load, 1 kilogram; sliding speed, 6.2 meters per second; specimen made from metal adjacent to wear scar surface.

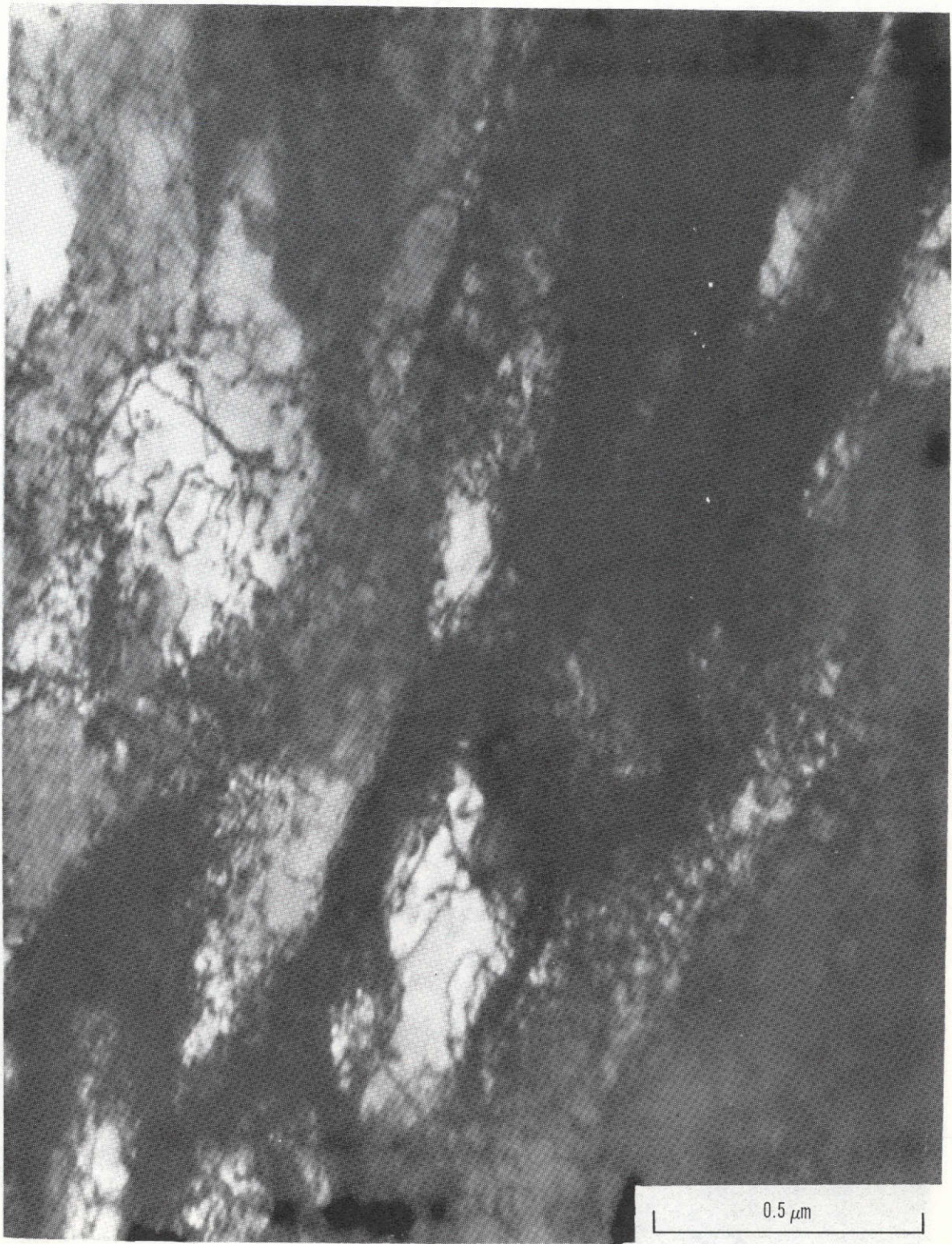


Figure 7. - Electron micrograph of copper subjected to sliding against 440C steel in liquid methane. Normal load, 1 kilogram; sliding speed, 6.2 meters per second; specimen taken from 200 micrometers below wear scar surface.

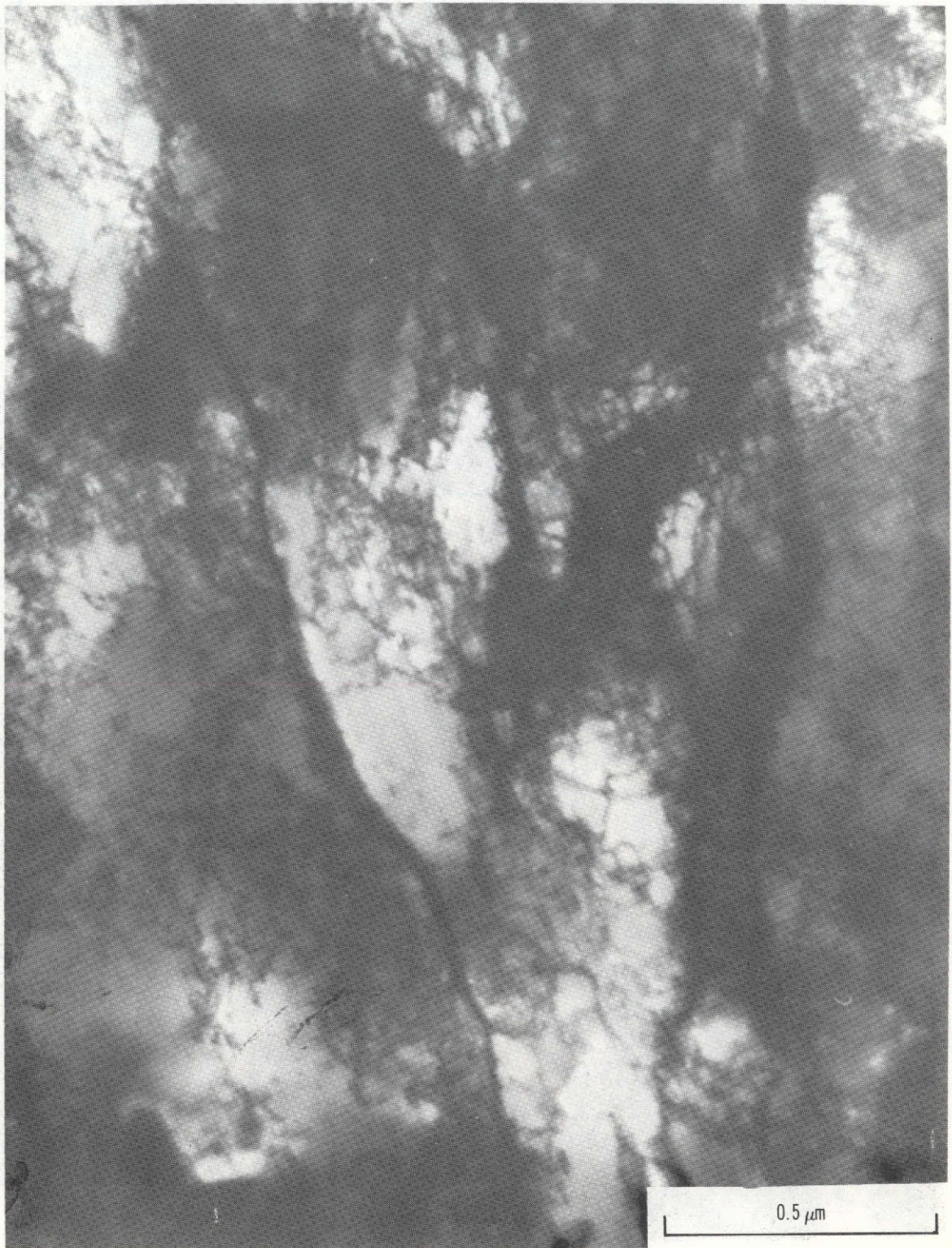


Figure 8. - Electron micrograph of bulk microstructure of copper rider.

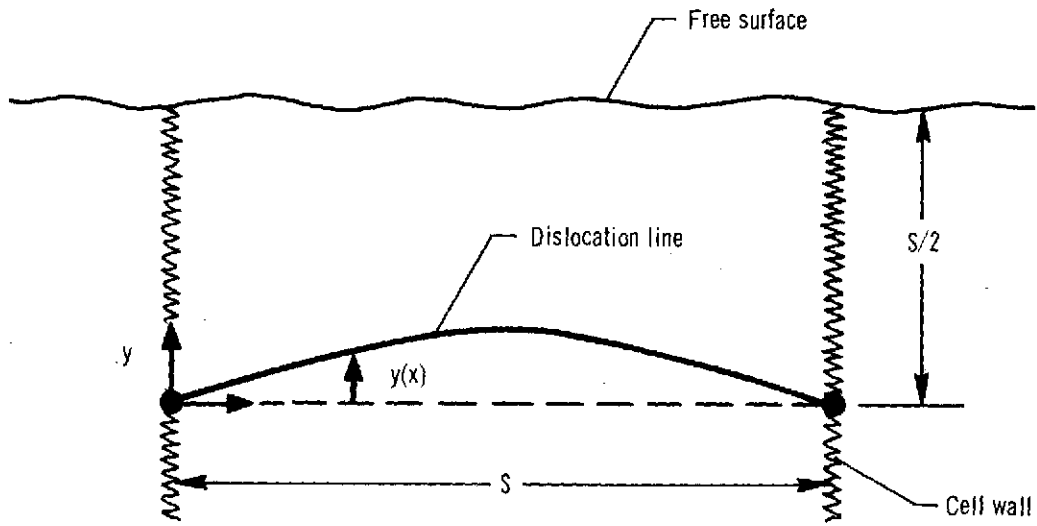


Figure 9. - Interaction between dislocation line pinned at cell walls and free surface.