EFFECT OF POINT DEFECTS ON MECHANICAL
PROPERTIES OF METALS

Technical
Progress Report
for the Period January 1, 1976 - December 31, 1976

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TECHNICAL PROGRESS REPORT FOR THE PERIOD
FROM JANUARY 1, 1976 TO DECEMBER 31, 1976

ABSTRACT

This Technical Progress Report consists of five sections:

(1) Surface Film Softening, (2) Electron Irradiation Softening of Iron, (3) Comparative Study of Two Softening Phenomena, (4) Preparation and Plastic Deformation of Niobium Sheet Single Crystals, and (5) Dislocation Motion in Copper. Of the publications listed at the end of this Progress Report, COO-1367-74, -76, -77, and -79 correspond with section (1), COO-1367-78 corresponds to section (2) and COO-1367-75 corresponds to section 5. The research activities described in section 4 are still preliminary investigations and those in section 5 are analyses of and numerical calculations for completed experiments.

1. Surface Film Softening

It was reported in the last annual report (COO-1367-73) that the coating of iron single crystals with thin nickel film reduced the yield stress at low temperature. This finding was rather surprising since surface coating was generally expected to cause a hardening effect. We have studied this phenomenon during the past year and have come to grips with the scope of this phenomenon and its cause at least in iron.

The mechanical characteristics of the surface film softening are summarized first:

(a) Serration. The surface film softening is nearly always accompanied by visible serrations on the stress-strain curve. The amplitude of the

serrations varies with the experimental conditions. Metallographic examination, using both light and electron microscopes, rejects twinning as the cause of the serrations.

(b) Reversibility of surface film softening. The surface film softening can be removed from or reintroduced to a specimen by electropolishing the surface film away or by reelectroplating a film even after some deformation. Simultaneously, the serration disappears or reappears along with the softening.

(c) Strain rate effect. The effect was examined by both strain rate cycling tests and comparison of the stress-strain curves obtained at different strain rates in the range of $10^{-4} \sim 10^{-2}$ /sec. Three significant points were noted: 1) The strain rate cycling tests indicated that the surface film softening reduced the strain rate sensitivity drastically. 2) The strain rate sensitivity also appears to be dependent on the strain rate. 3) The strain rate dependence of the yield stress gave the stress exponent, $m^* = \partial \ln \dot{\gamma} / \partial \ln \sigma$, similar to the value obtained from the motion of edge dislocations in iron. This value was smaller than the value obtained in a control specimen (iron single crystal without surface film). The latter value is usually considered as that for screw dislocations.

(d) Temperature dependence. The temperature dependence of the yield stress, $d\tau/dT$, decreases with the surface film softening. Although an iron specimen of initial purity of 99.95 wt% (before the ZrH_2 treatment) fractures before plastic deformation takes place at 4.2° K, the nickel-coated iron showed a substantial plastic deformation. Obtaining the yield stress of control specimens at 4.2° K by extrapolating the yield stress-temperature curve, the magnitude of the surface film softening is greatest at 4.2° K.

(e) Orientation dependence. The yield stress (in tensile stress) appears to be nearly independent of the angle, χ , except near $\chi = -30^\circ$ where the yield stress appears to be slightly higher. χ is the angle between [110] and the normal to the maximum resolved shear plane, as defined in an earlier report (cf. C00-1367-58, Scripta Met. 8, 851 (1974)). Consequently the greatest surface film softening effect was observed at $\chi = 30^\circ$, the hard (112) slip orientation.

The surface morphology of deformed specimens shows characteristics of surface film softening. Coarse striation becomes visible under a low magnification light microscope. Two types of striation are observed; they are cracks nearly perpendicular to the tensile axis on the edge-faces and slip-like lines on the screw-faces. The details of these observations are described in C00-1367-77, and analysis of these observations led to the mechanism responsible for the observed surface film softening. Although the details of the mechanism are still to be worked out with a number of calculations, the mechanical behaviors discussed in the preceding paragraph are in good agreement with this mechanism.

The essential part of the mechanism is as follows: When a surface coated specimen is extended, the nickel film and the iron single crystal substrate deform elastically at the low stress level. As the stress increases, the microplastic strain (due to the motion of preexisting edge dislocations) begins to occur in the iron, while the nickel film still deforms elastically. In consequence, the tensile stress is considerably greater in the film than in the substrate. Upon reaching a certain critical stress the film begins to crack, generating numerous dislocation loops at the root of the cracks. If the stress in the iron substrate is still below the stress level for the

screw dislocation motion, only the edge component of the dislocation loops moves. The observed geometry of the surface striations clearly substantiates that such is the case. The dependence of the nature of the striation on the surface character (edge or screw) indicates the importance of the interaction between the crack initiation and the deformation in the iron substrate.

2. Electron Irradiation Softening of Iron

Earlier studies indicated that the electron irradiation softening might be extremely sensitive to the purity of the specimen. Since we found that the softening effect was drastically increased by using specimens of the hard (112) slip orientation, the impurity effect was reexamined using this orientation. The initial purity of the specimen material was 99.95 wt%. Single crystals were grown in the ZrH_2 purified hydrogen atmosphere and then annealed at 850°C for 48 hrs in the same atmosphere. The magnitude of the softening observed in these specimens was nearly comparable ($\sim 70\%$) to that observed in the Battelle-AISI iron (initial purity of 99.996 wt%). This large softening is rather unexpected in view of the earlier results. The present results suggest two possibilities:

- (a) The electron irradiation softening is insensitive to impurity content.
- (b) The electron irradiation softening is insensitive to substitutional impurities provided that the ZrH_2 treatment eliminates the difference in the interstitial impurity content. The interstitial doping experiment with the high purity iron should determine which is the correct answer.

The mechanistic aspect of the electron irradiation softening in iron has been reexamined. A review lecture was presented and its abstract is

enclosed with this report (see C00-1367-78). The most important point in the electron irradiation softening mechanism is that the interstitials distributed nearly randomly throughout the iron specimen are the cause for the softening effect. Furthermore, this softening is caused by the interaction of screw dislocations with these interstitials, resulting in enhanced mobility of the dislocations. The couple force on the screw dislocation due to the interaction has been shown to enhance the screw dislocation motion and to decrease the yield stress. In addition to the couple force, a modification in the core structure of screw dislocations may take place due to the interstitials, giving rise to the orientation dependence of the yield stress. It is believed that a similar interaction should operate between a screw dislocation and solute atoms (and possibly their clusters) and may be a cause for the solid solution softening. In the case of a solid solution, however, the substantial deviation of solute atoms from the randomness complicates the mechanism and a clear-cut interpretation of the observed phenomenon is difficult.

3. Comparative Study of Two Softening Phenomena in Iron: Electron Irradiation Softening and Surface Film Softening

It was suggested earlier by other studies that the electron irradiation softening might be a surface or a near surface effect; that is, spattering off the surface layer or the accumulation of radiation damage near the surface caused the observed softening. Although these possibilities were discounted at the time by the present investigator, it is important to clarify the relationship of the surface film softening, which has been recently discovered, with the electron irradiation softening. Indeed, both

phenomena showed some similarities. For example, in both cases, the greatest softening effect was observed when the tensile axis was oriented for the hard (112) slip. However, it is shown in the following comparative study that these two softening phenomena are distinctly different although work is still in progress:

- (a) Deformation morphology. The deformation morphology is characteristically different. In the case of electron irradiation softening, the slip lines are extremely fine and cannot be resolved at a small deformation. When the direction of the slip plane traces can be identified at a large deformation, it coincides with that of the primary slip plane. This morphology is also identical to that observed in a control specimen (annealed iron single crystal). On the other hand, the coarse striation appears from the onset of plastic deformation in specimens exhibiting the surface film softening. Cracks appear on the edge surfaces and slip-like lines appear on the screw surfaces. The cracks are always approximately perpendicular to the tensile axis while the slip-like lines deviate from the trace of the primary slip planes (see C00-1367-77 for the explanations).
- (b) Serration. The serrated stress-strain curve accompanies the surface film softening, while a smooth curve is always observed in the case of electron irradiation softening.
- (c) Recovery. The surface film softening can be eliminated by removing the surface film. The electron irradiation softening disappears after annealing near room temperature.
- (d) Strain rate effect. The strain rate effect is quite different between the two softening phenomena. When the strain rate cycling is applied, the strain rate sensitivity appears to be nearly zero in the

case of the surface film softening while the sensitivity increases with electron irradiation softening.

(e) Temperature dependence of yield stress. The electron irradiation softening increases the temperature dependence while the surface film softening decreases the dependence.

(f) Orientation dependence. The yield stress of the electron irradiated iron depends strongly on the orientation of tensile axis. On the other hand, the specimens exhibiting the surface film softening show hardly any orientation dependence.

The comparison of these two softening phenomena clearly shows that different mechanisms are involved in these two cases. It is suggested that the motion of screw dislocations controls the yield stress in electron irradiation softening and that the generation of edge dislocations determines the yield stress in the case of surface film softening.

4. Preparation and Plastic Deformation of Niobium Sheet Single Crystals

Specimens must be thin in order to produce a uniform distribution of point defects with electron irradiation. Accordingly, it is decided that thin sheet niobium single crystals will be used for the experiment.

Nearly all of the recent experiments on the low temperature deformation of refractory metals have been conducted using rod-shaped specimens except Vesely's experiment on Mo, in which the surface orientation effect was reported. Therefore, before investigating the electron irradiation effect, the plastic deformation of niobium sheet single crystals must be characterized. During the past twelve months, such sheet specimens were prepared and were plastically deformed at 77° K.

Niobium single crystal rods of $\frac{1}{4}$ inch diameter were grown by an electron beam zone melting apparatus at Argonne National Laboratory (Materials Science Division). Several sheet specimens with the tensile axis orientation near $[123]$ were sliced from one of the rods, using high speed mechanical slicing with a 15 mil thick carborundum wheel. After final polishing, the gauge sizes of $5.5 \times 1.8 \times 0.25$ mm and $7.8 \times 2.9 \times 0.3$ mm were obtained. The final annealing was carried out at $\sim 1960^\circ\text{C}$ and at $\sim 10^{-9}$ torr.

The preliminary results indicate the following: The yield stresses obtained were comparable to those obtained in rod specimens. The surface observation clearly revealed that the primary slip system $(a/2)[111](\bar{2}11)$ is in operation along with $(a/2)[111](\bar{1}01)$. The faint slip traces, which can be regarded as $(0\bar{1}1)$ anomalous slip planes, were also recognized. It appears that the surface orientation effect in Nb is not as remarkable as in Mo. Some specimens were anodized. The subsequent deformation revealed that the surface oxide film increased the yield stress slightly at room temperature and decreased the flow stress significantly at 77°K . The surface morphology was also altered drastically by the surface oxide film.

5. Dislocation Motion in Electron Irradiated Copper

The results of this experiment were summarized in C00-1367-75 and were presented at the Fourth International Conference on Strength of Metals and Alloys, August 1976, at Nancy, France.

The excellent correspondence between the dislocation behavior obtained directly from the etch pit measurements and those deduced from

macroscopic deformation was well accepted. However, a question raised by Professor P. Haasen of the University at Göttingen may be worthwhile to discuss. According to his study of dislocation motion by an etch pit technique in dilute copper alloys, the distance the dislocations moved did not depend on the loading time. He felt that our case should also be regarded as dilute solid solution; therefore, the dislocation motion should be more time-independent in nature. This is contrary to our observation. We believe that there is a difference between the case of a solid solution and the case of an electron-irradiated pure metal which can be regarded as a solid solution (electron-induced point defects being solutes). In a solid solution, there is a considerably greater degree of solute segregation to dislocations and other regions of a specimen than in an electron-irradiated specimen. In a sense, the electron irradiation produces a solid solution which is much more ideal for studying the random solute effect.

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Reports Resulting from the Present Research Project

- (1) - (73) COO-1367-1 through COO-1367-73. These reports have been included in previous Technical Progress Reports.
- (74) "Surface Film and Plasticity of Iron Single Crystals at 77° K", Proceedings of Second International Conference on Mechanical Behavior of Materials, Boston (1976) p. 78. COO-1367-74.
- (75) "Investigation of Dislocation Motion by Etch Pit and Yield Stress in Electron Irradiated Copper", Proceedings of Fourth International Conference on Strength of Metals and Alloys, Nancy France (1976), Vol. 2, p. 819. COO-1367-75.
- (76) "Surface Film Softening of Iron Single Crystals Oriented for Single Slip", physica status solidi (in press). COO-1367-76.
- (77) "Surface Observation of Nickel-Plated Iron Single Crystals Deformed at 77° K", submitted for publication to Acta Metallurgica. COO-1367-77.
- (78) "Interstitials and Low Temperature Strength of BCC Metals", Abstract of talk at Fall meeting of TMS/AIME, Niagara Falls (1976). COO-1367-78.
- (79) "Surface Film Softening in Nickel-Plated Iron Single Crystals", Abstract of talk to be presented at Annual Meeting of AIME, Atlanta (1977). COO-1367-79.
- (80) "Technical Progress Report for the Period from January 1, 1976 to December 31, 1976" submitted to the U. S. Energy Research and Development Administration for renewal of Contract EY-76-S-02-1367.*000. COO-1367-80.