EFFECT OF POINT DEFECTS ON MECHANICAL PROPERTIES OF METALS

Technical Progress Report
for Period December 1, 1979 - January 31, 1980

MASTER

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TECHNICAL PROGRESS REPORT FOR THE PERIOD
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ABSTRACT

This progress report consists of five sections. The importance of morphological study in conjunction with mechanical tests is emphasized and is explained in the first section. The succeeding sections are on the effects of oxygen in niobium, the surface effect on crystal plasticity of niobium, surface film softening in Ni-plated iron, and the hydrogen charging effect in iron. Twelve reports have resulted from the present project during this year. The first five (COO-1367-98 - COO-1367-102) are abstracts for talks and the next six (COO-1367-103 - COO-1367-108) are manuscripts, and the last (COO-1367-109) is the Technical Progress Report.

1. Introduction

It has been proposed, in reports resulting from this research project (COO-1367-97, -99, and -104), that consideration of the operating slip system is essential in the understanding of the solid solution effects on the yield stress of b.c.c. metals at low temperatures. We may have resolved the seemingly contradictory phenomenon, namely, solid solution hardening/softening. The confusion resulted from the complexity of deformation. For example, iron deforms by the primary slip system for any tensile axis orientation, while the anomalous slip operates prominently in niobium. Furthermore, the operating slip system depends on the tensile axis orientation and other experimental conditions. It has been shown that the solid solution softening can take place only when the primary slip system operates. When the deformation takes place primarily by the anomalous slip, only solid solution hardening is expected to

take place. The anomalous slip is identified as a coplanar motion of the primary and conjugate screw dislocations. This dislocation mechanism also explains why the anomalous slip occurs even when the Schmid's factor for the anomalous slip is considerably smaller than that for the primary and other slip systems. The information available on the operating slip systems during the low temperature deformation of b.c.c. metals is rather limited. It is felt that the determination of the operating slip systems for various experimental conditions and the understanding of why they operate for each condition are the keys to understand the low temperature deformation mechanism of b.c.c. metals. In the present project, this has been carried out with iron and niobium as specimen materials.

2. Solid Solution Hardening and Softening in the Nb-O System

The low temperature electron irradiation effects in niobium have demonstrated clearly that the effect due to dispersed interstitials can be either hardening or softening depending on whether the operating slip is on the anomalous system or the primary system. In this experiment, we examined the effect of oxygen atoms. The observation has been reported in COO-1367-99 and -108. The oxygen atoms were added to pure niobium by the oxidation and diffusion process. Again, two tensile axis orientations, C and H, were examined. The tensile axis of the C specimens was at the center of the reference triangle, while that of the H specimens was on the $[110]$ - $[111]$ line. The pure C specimens deformed exclusively by the anomalous slip. The results indicate that only the solid solution hardening can take place in the C specimens, while the softening effect was observed in the H specimens, in the concentration range up to 200 atomic ppm. Both types of specimens were purified and oxygen-doped in the same run to eliminate the possibility of

obtaining different chemical compositions.

The slip morphology indicated the following features: In the C specimens, the anomalous slip lines changed from fine to coarse as the concentration of oxygen was increased. They decreased in number and nearly disappeared at 400 at. ppm where a significant number of the primary and conjugate slip traces were observed. In the pure H specimens the primary slip dominates, and some unidentifiable slip lines appeared with increasing oxygen concentration. The observation suggests several interesting possibilities. Those coarse anomalous slip lines reported earlier in the literature may be the result of impure specimen materials. In pure specimens, nearly all deformation can take place on the anomalous slip system. Once the solid solution hardening effect becomes significant in the C specimens, single slips such as the primary and conjugate systems are favored over the anomalous system. The rotation of the tensile axis was also determined and substantiated the slip line observations.

3. Surface Orientation and Surface Film Effects in Niobium (C00-1367-102)

In view of the thinness of our specimens ($\sim 200 \mu\text{m}$) and a report indicating the surface orientation effect in molybdenum ($\sim 250 \mu\text{m}$),⁽¹⁾ we have examined the surface orientation effect in niobium. Again, C and H specimens were used and two surface orientations were prepared for each tensile axis orientation. The wider surface is chosen parallel to the primary Burgers vector (screw surface) or is rotated around the tensile axis 90° from this orientation (edge surface). In both the C and H specimens, no significant difference was observed in the stress-strain relation and the slip morphology between these two surface orientations although the tensile axis orientation effect was found to be significant. These experiments, therefore, assure us that our results are not biased by the specimen size and

(1) D. Vesely, Scripta Met. 6, 753 (1972).

should be comparable with the results expected from larger specimens. The role of the surface in the plastic deformation of niobium (such as providing dislocation sources, as speculated in a number of articles in the literature) is not major in the current experiments.

On the other hand, the surface orientation affects the yield stress and the stress-strain relation significantly when a specimen is coated with an oxide film (6×10^{-2} μm thick). The results are summarized as follows:

- 1) The surface film softening effect is largest in the case of edge surface.
- 2) The surface film completely eliminates the anomalous slip. The primary and conjugate slips become the only active slip systems.
- 3) The surface film virtually eliminates the trend to neck in H specimens.
- 4) The surface film softening eliminates the difference between C and H specimens.
- 5) In a partially coated C specimen, the primary slips terminate at the edge of the oxide film. At the ends of some of these slip lines, the anomalous slip was activated.

All of these observations indicate strongly the importance of the surface condition in the study of the low temperature crystal plasticity and further substantiate the massive edge dislocation motion as the mechanism of surface film softening. We can list three modes of dislocation motion with ease of motion, namely, edge dislocation motion, coplanar motion of two sets of screw dislocations, and motion of a set of screw dislocations. The details of this investigation will be written up shortly.

4. Surface Film Softening in Ni-Plated Iron Single Crystals

All of the experiments in this project have been completed in the last fiscal year (1978-79). The detailed analyses of the observations and of the

proposed mechanism have been carried out this year. Two reports have been prepared on them. The first (COO-1367-106) has been submitted to the Journal of Materials Science and Engineering, and the second is nearly completed and will be submitted to the same journal shortly.

5. Hydrogen Charging Effect on Plastic Deformation of Iron

It has been shown that the hydrogen charging has a drastic effect on the plastic deformation of pure iron (COO-1367-105 and -107). In a creep experiment, the charging increases the creep rate by 20 ~ 30 times. A significant decrease in flow stress also occurs in a tensile test. Two possible mechanisms can be suggested for the observed creep acceleration and the softening effect. Dissolved hydrogen atoms can act to promote dislocation motion. Although hydrogen atoms are mobile in this case, we shall term this mechanism as solid solution softening. Another possibility is that the precipitation of hydrogen atoms into small voids generates dislocation loops. Neither theory has been substantiated by direct evidence at this time. Further complications may come from the impurity effect on hydrogen trapping and precipitation.

In addition to acceleration of plastic deformation (or softening effect), hydrogen also embrittles iron. This was discovered by comparing the hydrogen charging effects in single crystals and polycrystals. Under certain conditions, polycrystalline iron specimens can be fractured along grain boundaries at a stress level at which little plastic deformation takes place. Therefore it appears completely brittle. This intercrystalline fracture is extremely sensitive to the strain rate. At the same time, it appears to be sensitive to the purity of the specimen and prior heat treatment. All the observations appear to point to a synergistic effect between hydrogen and some of the impurity elements segregated to grain boundaries. Further work is underway to clarify these points.

Reports resulting from the present research project:

- (1) - (97) COO-1367-1 through COO-1367-97. These reports have been included in previous Technical Progress Reports.
- (98) "Dose-Dependence and Recovery of Electron Irradiation in Niobium", abstract of talk presented at TMS-AIME Fall Meeting, Milwaukee, September 1979. COO-1367-98.
- (99) "Alloy Softening and Hardening in the Nb-O System", abstract of talk presented at TMS-AIME Fall Meeting, Milwaukee, September 1979. COO-1367-99.
- (100) "Effects of Hydrogen Charging on Creep Deformation of Pure Iron", abstract of talk presented at TMS-AIME Fall Meeting, Milwaukee, September 1979. COO-1367-100.
- (101) "Hydrogen Charging Effect on Creep of Pure Iron", abstract of talk presented at 2nd JIM International Symposium on Hydrogen in Metals, Minakami Spa, Japan, November 1979. COO-1367-101.
- (102) "Effects of Surface Orientation and Surface Film on Low Temperature Deformation of Thin Sheet Niobium Crystals", abstract of talk to be presented at TMS-AIME Annual Meeting, Las Vegas, February 1980. COO-1367-102.
- (103) "Surface Film Softening of Ni-Plated Iron Single Crystals", Proc. 5th Int. Conf. on Strength of Metals and Alloys, Aachen, Germany, (1979) 1, p. 151. COO-1367-103.
- (104) "Rationalization of Softening and Hardening Effects by Interstitial Atoms in B.C.C. Metals", Proc. 5th Int. Conf. on Strength of Metals and Alloys, Aachen, Germany (1979) 2, p. 989. COO-1367-104.
- (105) "Effect of Cathodic Charging on Creep and Tensile Deformation of Pure Iron", Scripta Metallurgica. In Press. COO-1367-105.
- (106) "Experimental Characterization of Surface Film Softening in Iron: Effects of Prestraining, Temperature and Orientation", submitted to Materials Science and Engineering. COO-1367-106.
- (107) "Hydrogen Charging Effect on Creep of Pure Iron", Proc. 2nd Int. Symposium on Hydrogen in Metals, The Japan Institute of Metals, Minakami, Japan (1979). COO-1367-107.
- (108) "Solid Solution Softening and Hardening in the Nb-O System", Scripta Metallurgica. In Press. COO-1367-108.
- (109) "Technical Progress Report for the Period from December 1, 1978 to December 31, 1979", submitted to the U. S. Department of Energy for renewal of Contract DE-AS02-76ER01367 (formerly EY-76-S-02-1367). COO-1367-109.