

A METHOD FOR DETERMINING THE MICROSTRUCTURAL CHANGES IN GRAPHITE THAT ACCOMPANY HIGH-TEMPERATURE DEFORMATION*

by

W. V. Green, L. S. Levinson, R. D. Reiswig and E. G. Zukas

Los Alamos Scientific Laboratory
The University of California
Los Alamos, New Mexico

ABSTRACT

Microstructural studies have contributed greatly to the understanding of the mechanical properties of metals in terms of structure. Without metallographic studies, it would have been virtually impossible to follow the structural changes which take place during creep deformation, such as slip band formation, wavy slip, subgrain formation, kink band formation, and fold formation. Such observations are a prerequisite for formulating a realistic theory for creep deformation.

Jenkins deformed graphite progressively while examining it on the stage of a microscope and thereby observed crack growth. Unfortunately, these studies were limited to deformation at room temperature. Other experimenters compared "typical" areas before deformation to other "typical" areas after deformation. Although such methods would undoubtedly show major changes in the structure, the more subtle structural changes probably would not be detected; but because of the complex structure of graphite, the subtle changes in structure could only be revealed by examining the same area before and after deformation.

The test temperature and strain rate exert a strong influence on the amount of deformation to fracture in graphite. This effect must be the result of some mechanism or mechanisms which come into play at high temperature. This paper describes a method which enabled sequential optical or electron microscope studies to be made on the same areas in its "as-received" condition and after deformation at high temperature. A special jig was used to polish flats on a tensile bar. Grinding and polishing were done to produce a metallographic surface. Then two selected areas in the gage length of each sample were cathodically etched. One area was etched for study by light microscopy, whereas the second area was etched for examination with the electron microscope.

Normally, the samples are heated by radiation from a graphite resistance tube heater in an inert atmosphere. Such conditions destroy the metallographic finish very rapidly at the high temperatures. Several modifications were made which enabled routine testing without excessive damage to the metallographic surfaces.

First, the metallographic surfaces were protected by wrapping several layers of pyrocarbon tape over the gage length. The samples were then loaded into the creep testing furnace and the furnace was evacuated. The samples were gradually heated to 2000°C while maintaining a vacuum of less than 5×10^{-5} torr. The test chamber was then filled to 2 psig with high-purity argon. The sample was then heated to the test temperature (typically 2500°C), and stretched the desired amount. The samples were then cooled in the argon atmosphere. The times at temperature ranged from one-half

* Work performed under the auspices of the United States Atomic Energy Commission.

to two hours.

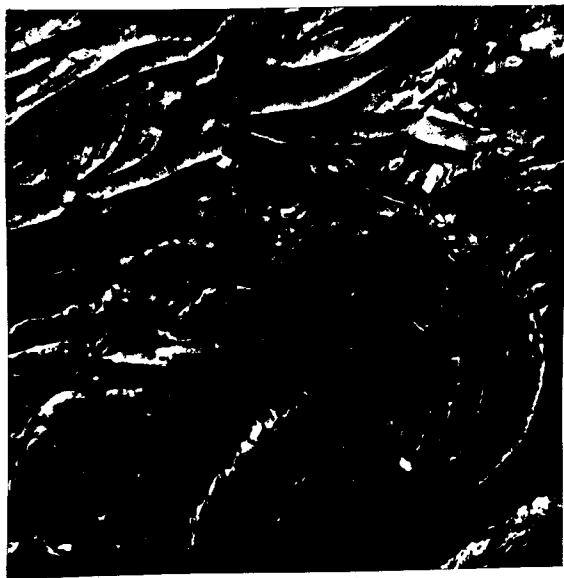
Deformation of a highly-oriented graphite (ZTA) was studied using the above technique. Several magnifications were tried, but 500X was the most satisfactory for optical microscopy while 5000X appeared best for electron microscopy.



Before strain, light microscopy, 500 X,



after 5% strain.



Before strain, electron microscopy, 5000 X,



after 5% strain.

Figure 1. Typical sequential photomicrographs.