

Growth and Crystallography of Deformation of β -Phase Uranium Single Crystals

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Single crystals of the tetragonal β -phase of uranium were grown in material containing small amounts of chromium by a $\gamma \rightarrow \beta$ phase-transformation method. These crystals could be retained in a metastable condition for long periods following their quenching from normal β -phase temperatures into water at room temperature. The crystals were found to be ductile, with slip occurring on the $\{110\}$ type plane in the $\langle 001 \rangle$ direction, a slip system analogous to that of β (tetragonal) tin.

Introduction

Over the past two years, several β -phase uranium single crystals have been grown and retained in a metastable condition at room temperature. The first few crystals were used by Tucker (1951) to determine the structure of the β -phase. In the early attempts to produce these crystals, a good crystal, even of the size required for X-ray study, was rarely grown. Only recently has it been possible to grow excellent β -phase crystals at every attempt and the present method will be described.

With the availability of these large β -phase single crystals, it has been possible to negate a long-standing conclusion that the β -phase was completely brittle; the single crystals of β -phase are usually ductile and they deform by slip. The slip plane has been identified as the $\{110\}$ type plane in the β crystal and the direction of slip as the $\langle 001 \rangle$ direction. Only when they are unfavorably oriented, do β crystals behave in a brittle fashion.

Growth of β -phase crystals

It was found possible to grow β -phase single crystals in uranium containing 0.5%–1.5% (atomic percentages) chromium. The technique used was as follows:

Wires of the material were sealed off in evacuated quartz tubes. The tube containing the wire was then suspended by a thin nichrome filament in a vertical tube furnace of a special design. This furnace had three separate temperature zones, each of which could be controlled individually. It was thus possible, for example, to have one zone in the temperature range where the α phase is stable, one in the β range, and one in the γ range, with temperature gradients between zones.

For our purpose, however, the lower two zones in the furnace were maintained at about 850° C., well in the γ range, and the upper zone was maintained at 700° C., in the β range. The tube containing the

uranium wire was positioned initially in the γ zone and raised slowly up into the β zone at a rate of about 3 in./hr. The rate of movement was not critical, however. When the quartz tube was entirely in the β zone, the supporting nichrome filament was cut and the tube dropped out of the bottom of the furnace into a beaker of cold water. This technique produced β crystals several centimeters in length.

Several attempts have been made to grow β -phase crystals using the transformation from α to β and then quenching. None of these attempts has produced a single crystal, but rather only an oriented growth of many crystals with rather irregular boundaries. The general appearance is not unlike the appearance of veining in iron that has been transformed from γ to α .

A possible reason for failure when using the $\alpha \rightarrow \beta$ transformation might be that the $\alpha \rightarrow \beta$ transformation is of a Widmanstätten type, wherein a plane in one phase contains the proper array of atoms to nucleate some plane in the other phase. Upon transforming a polycrystalline α phase to β it would then be difficult to cause growth from only one of the many available nuclei.

While such an explanation must not be discounted completely, there is one other matter that must be considered, namely, the relative strengths of the growing and parent phases. The volume changes involved in the transformation may well introduce sufficient stresses to distort the transformation interface. The question, therefore, of whether the growing or parent phase is the softer, and consequently receives most of the deformation, is important. If, for example, the parent phase were extremely soft at the transformation temperature, the deformation would be confined to it, and the nucleus of the growing phase would remain undistorted. If the converse situation in regard to relative strengths existed, one might expect lineage in the growing crystal, probably caused by the migration of dislocations into boundaries to relieve the distortion in the region of the interface.

β uranium is known to be much stronger than γ

at the $\beta \rightarrow \gamma$ transformation temperature. β , however, is comparable in strength to α at the $\beta \rightarrow \alpha$ transformation temperature. One would then expect that any distortion during the $\gamma \rightarrow \beta$ transformation would be confined to the parent γ phase, and that good undeformed single crystals of β could be produced. On the other hand, the β crystal grown from α is distorted because of the small difference in strength of the two phases.

Many attempts also have been made to grow α crystals by using the $\beta \rightarrow \alpha$ transformation alone, or the $\gamma \rightarrow \beta \rightarrow \alpha$ transformations in that order. In these experiments directional growth occurred, but the specimens always contained a fine substructure with considerable orientation difference between sub-grains.

In spite of the fact that those specimens given a $\gamma \rightarrow \beta \rightarrow \alpha$ treatment were presumably good β crystals in the intermediate stage, α crystals were produced which were only slightly superior to those produced with only a $\beta \rightarrow \alpha$ treatment.

β -phase single crystals produced from 1 atomic% Cr-U alloy are not single phase. They contain the old γ grain network outlined with a precipitate, presumably of a chromium-rich phase.

β crystals grown in 0.5 atomic% Cr-U alloy are free of precipitate. However, whereas the alloy containing 1% or more chromium will retain the β -phase crystal for an indefinite time after quenching to room temperature, the 0.5% alloy β crystals transform to α within a few hours at room temperature.

Crystallography of deformation in the β -phase

There have been many references in the past to the brittleness of the β -phase in uranium. The assumption of complete brittleness has been largely based on some unfortunate forming experience in the β temperature range, but partly based on the fact that the structure was known to be complex. Furthermore, Tucker (private communication) has observed that quenched polycrystalline β -phase uranium (the 1 atomic% chromium alloy) is brittle. These β -phase single crystals, however, have been found to be ductile. They deform entirely by slip; no trace of twinning has been observed in any deformed β -phase crystal.

As a consequence of his structure studies on these crystals, Tucker has assigned a tetragonal lattice to the β -phase with

$$a_0 = b_0 = 10.52, \quad c_0 = 5.57 \text{ \AA},$$

and with 30 atoms per unit cell.

To determine the plane of slip in the β -phase, single-crystal wires were grown by the technique described in the first part of this report. These wires were square in cross section, 0.050 in. \times 0.050 in., and about 6 in. long. The orientation of the crystals was determined normal to one plane face by a back-reflection Laue method and the crystals were then

carefully electropolished. Slip was produced in the crystals by slight bending. The angle that the trace of the slip plane made with the axis of the wire was measured both in the face that had been normal to the X-ray beam in the Laue photograph and in one of the adjacent faces 90° from the first. These data were then plotted on a stereographic projection of the crystal.

Since the pole of the slip plane is contained in a zone normal to the slip trace, the intersection of the

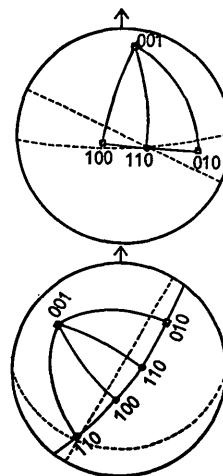


Fig. 1. Typical plots used to determine the slip plane. The pole of the slip plane occurs at the intersection of the dashed normals to slip traces.

zone that is normal to the slip trace in one face with the zone that is normal to the slip trace in the adjacent face will occur at the pole of the slip plane when these zones are plotted on a stereographic projection of the crystal. Two typical plots are shown in Fig. 1. It can be seen that the slip plane is the $\{110\}$ plane nearest the position of maximum shear stress.

The slip direction was determined using a crystal of cylindrical cross section 0.050 in. in diameter. The crystal was electropolished and then deformed in tension in a microtensile machine. The crystal was then mounted in a special horizontal goniometer head on the X-ray machine, arranged so that both a back-reflection Laue camera and a horizontal microscope could be focused on the crystal from opposing positions 180° around the axis of the crystal. With the aid of the microscope, the crystal was rotated about its axis until the slip traces faded out on its surface. Since it is known that slip traces fade out at 90° to the slip direction (Gough, 1928), the slip direction in the fade-out position was 90° to the axis of both the microscope and the X-ray beam. A back-reflection Laue photograph was taken in this position. The angle between the slip traces at the instant of fade-out and the crystal axis was measured with a protractor eyepiece on the microscope. From the known slip plane, the angle measured between the crystal axis and observable slip traces, and the fact that the slip direction must in

this setting occur at 90° to the X-ray beam, the direction of slip was easily determined to be the $\langle 001 \rangle$ direction.

The stereographic plot of a β crystal rotated so that its slip direction was at 90° to the X-ray beam is shown in Fig. 2. The slip trace and direction are indicated on the projection.

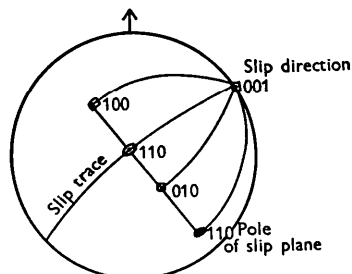


Fig. 2. Stereographic projection of a single β crystal obtained from a back-reflection Laue photograph taken normal to the slip direction.

The slip process at room temperature in β uranium is apparently limited to two slip systems since there are but two $\{110\}$ -type planes and a single $\langle 001 \rangle$ -type direction. This situation may be understood qualitatively by inspecting the β structure.

The $\{110\}$ planes are relatively close packed. Furthermore, of all the planes in β uranium, they are, according to Tucker, the only truly flat planes. They are logically slip planes. The arrangement of atoms on the $(\bar{1}10)$ and (110) planes is shown in Fig. 3. At first glance these two planes do not appear equivalent, and in the same unit cell the atom positions do differ. The periodic arrangement of atoms is the same in each plane, however, as one can see by merely shifting the unit-cell boundaries in the second plane in Fig. 3

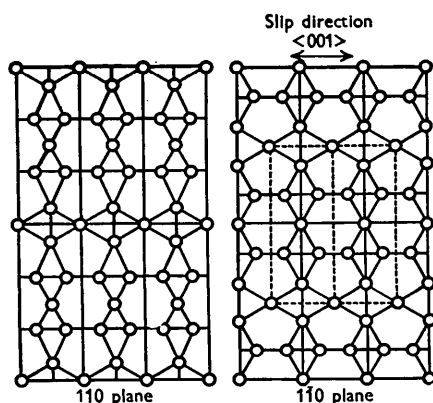


Fig. 3. Atom positions on $\{110\}$ slip planes in β uranium extended through six unit cells.

to the broken lines. Order could be restored to the lattice only by having a unit slip process extend over the entire unit-cell dimensions on the $\{110\}$ plane. The shortest and most logical path is then the $\langle 001 \rangle$ direction.

A few β crystals would not extend in tension, but instead fractured by cleavage. By compressing such a crystal normal to its axis, slip traces were produced at small angles to the axis. Apparently, these orientations were not proper for slip in tension. The cleavage plane has not been identified.

Several possible explanations exist for the observation that polycrystalline β material is quite brittle at room temperature. For example, a few of the crystals in a polycrystalline aggregate probably would be oriented for cleavage rather than slip, and cleavage once started would propagate through the remainder of the specimen. Even if some crystals did slip initially, stress concentrations at the end of the slip bands in crystals oriented for slip could start cleavage in crystals not favorably oriented for slip, and again the cleavage would propagate. This situation would be analogous to the brittleness of a coarse-grained cast zinc. Another possible explanation is that large cracks already exist in β quenched polycrystals, and such cracks have been observed. The cracks probably arise by cleavage of certain crystals due to stresses imposed by anisotropic thermal expansion of adjacent grains.

It is of some interest that the σ phase in stainless steels has been observed to have a structure like the β -phase in uranium (Tucker, 1951) and one might expect such a phase to deform similarly at room temperature.

White tin (β tin) has a tetragonal structure and deforms primarily on $\{110\}$ planes in $\langle 001 \rangle$ direction exactly as does β uranium (Schmid & Boas, 1935, p. 89). However, other slip systems have been observed in β tin both at room temperature and at higher temperatures, although these systems are not preferred. One may well expect other slip systems in β uranium to operate at higher temperatures, and twinning may occur as well. The high-temperature behavior of β has not been studied.

The author is indebted to Dr C. W. Tucker for the complete structure description which made possible a study of slip in β uranium.

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