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Renewal Proposal to the

U. S. ATOMIC ENERGY COMMISSION

For a Research Program on

CREEP MECHANISMS IN B.C.C. ALLOY CRYSTALS

by the

Engineering Experiment Station
and the
Department of Minerals and Metals Engineering
College of Engineering
The University of Wisconsin
Madison, Wisconsin 53706

January, 1968

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Renewal Proposal to the

U. S. ATOMIC ENERGY COMMISSION

For a Research Program on

CREEP MECHANISMS IN B. C. C. ALLOY CRYSTALS

NAME AND ADDRESS OF INSTITUTION

This proposal is submitted by the Department of Minerals and Metals Engineering and the Engineering Experiment Station of the College of Engineering, The University of Wisconsin.

Address: 1509 University Avenue
Madison, Wisconsin 53706

TITLE OF PROPOSED RESEARCH

The title of the proposed research is, "Creep Mechanisms in B. C. C. Alloy Crystals."

DESIRED STARTING DATE

It is desired that support of the research commence on May 1, 1968, so as to permit an uninterrupted research program.

TIME PERIOD

Support is being requested for a period of one year.

PROGRESS REPORT NO. 3

For the Period January, 1967 to January, 1968

Apparatus and Equipment

1. Two Heath Model EUW 20-A Servo Recorders have been purchased. These permit more convenient and accurate recording of creep strains.
2. Five diamond cut-off wheels have been purchased for sectioning ingots into single crystal and polycrystal blanks, using the Do-All machine. A motor and gear train have been installed on the Do-All as an automatic feed system.
3. The electrolytic lathe has been further improved by incorporating a reversible drive to the horizontal motion of the planing wheel. This permits precisely flat surfaces to be electrolytically planed.
4. A 30° tilting stage has been ordered for the electron microscope, using U. W. Engineering Experiment Station funds. This will permit the easier determination of Burgers vectors of dislocations.

Data and Discussion: Stoichiometric NiAl

Much of the research on stoichiometric NiAl is discussed in the three papers listed below which were recently submitted for publication, the first two to Acta Metallurgica and the last to Trans. Met. Soc. AIME.

1. Creep of Stoichiometric NiAl, I. The Thermally Activated Cross Slip Range
2. Creep of Stoichiometric NiAl, II. Diffusional Creep
3. Glide Systems in NiAl at Elevated Temperatures

Since these papers are appended to the present proposal, only the essential conclusions will be summarized at this time. Below $\sim 750^{\circ}\text{C}$ the steady-state creep rate of stoichiometric NiAl single crystals is governed by the rate of thermally-activated cross-slip. Abundant evidence for cross-slip has been obtained through transmission electron microscopy of foils prepared from creep specimens. Slip trace analysis suggested a (103) slip plane and [010] slip direction. Contrast experiments

confirmed a $[0\bar{1}0]$ Burgers vector, while pencil glide on (001) and (101) accounts for the observed macroscopic (103) plane.

At temperatures $> 750^{\circ}\text{C}$ the creep rate has been found to be very orientation dependent (this is probably true below 750°C but has not been studied). Certain orientations cross slip readily and give much faster creep rates than orientations which result in thin films showing little evidence of cross slip. The behavior is accountable in terms of the resolved shear stress on the cross slip plane. The effect is such that single crystals of some orientations have considerably greater creep resistance than polycrystals. The activation energy for steady-state creep of polycrystalline NiAl has been found to be equal to that for diffusion of Co^{60} in NiAl, and further indication of the role of diffusion is given by well-formed sub-boundaries due to dislocation climb.

At high temperatures $\{110\} \langle 110 \rangle$ slip has been identified, although there is reason to believe that this type of slip is intrinsically more difficult than cube slip, i. e., the operative system is not determined solely by the respective Schmid factors. These high temperature glide systems are the subject of the third paper listed.

Non-Stoichiometric NiAl

No difficulty has been experienced in growing Ni-rich NiAl, using the same techniques as for the stoichiometric alloy. Three crystals containing 54 at% Ni have so far been creep tested, indicating an increase in creep strength as compared with stoichiometric NiAl. However, the relative magnitudes of orientation-dependence and alloying-dependence of strength have not yet been studied.

On the other hand, serious difficulty has been encountered in preparing Al-rich, i. e., vacancy-defective, NiAl single crystals. Growth from the melt is impossible, the large freezing range resulting in a very high degree of porosity. However, stoichiometric NiAl single crystals in principle can be converted into Al-rich crystals either by removal of nickel or addition of aluminum. The removal of nickel as nickel carbonyl has been attempted but was unsuccessful, probably because only "active" nickel is easily converted into the carbonyl. The addition of aluminum to stoichiometric NiAl crystals is now being attempted by a diffusional technique. The NiAl crystals are suspended in liquid aluminum, a chemical melting flux being used to ensure that diffusion is not impeded by oxide films. The degree of success of this technique is not yet known.

Stoichiometric CoAl

A coarse-grained ingot was prepared by the method described for NiAl. This material appears to be easily fragmented and it has not been possible to slice it into single crystal blanks suitable for tensile creep

specimens. Instead, small compression samples have been obtained and will be tested. This alloy may accidentally be aluminum-rich which would account for its behavior. Chemical analysis will determine this point, and in any event additional material will be prepared.

Beta Brass, CuZn

Hot-rolled rod, 3/8 in. diameter, has been grown into single crystals in graphite molds by a Bridgman method. Single crystals up to 10 in. long have been prepared and are presently being oriented and electroshaped into creep specimens. Since these are ~2 in. long, it is apparent that four or five crystals of each orientation will be available; this is important in isolating the effect of crystal orientation from other variables.

In most respects this program will parallel the research on NiAl.

Etch Pit Techniques and X-Ray Topography

Efforts have been made to develop additional techniques for studying dislocation arrangements in the alloys, not to supplant electron microscopy, but for use in those instances where electron microscopy is not entirely suitable. These include determinations of low dislocation densities and subgrain dimensions.

Figure 1 shows etch-pit structures developed in NiAl using chemical etching. The reproducibility of the pits and the similarity of the etch-pit pattern on both sides of a thin slice suggest that the pits are dislocation etch pits. A correlation with X-ray topographs will be attempted; in this connection a two-circle goniometer has been converted into a Lang camera, and the topographical technique is being developed.