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NINTH PROGRESS REPORT

TO

MATERIALS CENTRAL

WRIGHT AIR DEVELOPMENT DIVISION

ON

STUDIES OF HEAT-RESISTANT ALLOYS

by

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SUMMARY

Progress is reported for research conducted under Air Force Contract No. AF 33(616)-5466 covering the period of March 31, 1960 to June 30, 1960.

Rupture data at 1100°F and 20,000 psi for 0.06-percent carbon "A"Nickel are presented for the case where measurable substructures were pre-induced by tensile straining at 1600°F. The maximum subboundary density was limited due to recrystallization during prestraining at 1600°F. The rupture life was increased almost three fold as the material was prestrained up to 5.37-percent elongation. For prestrains of 10.8 and 23.2 percent, the rupture life decreased to a value only slightly higher than that of non-prestrained material.

Creep rate versus substructure density data are given for low-carbon (0.01 percent) "A"Nickel creep tested in vacuo at 1550°F and 3,360 psi. The same type of measurements are planned for vacuum-melted carbonyl nickel to check the generality of the present results and to determine the influence of impurities on this substructure phenomenon.

A procedure has been developed for rolling in an argon atmosphere which results in carbon steel remaining bright during heating and rolling at 2100°F. It is expected that the niobium bars can be rolled without contamination. Vacuum creep-rupture testing units are being assembled and proven out for testing the Nb.

INTRODUCTION

This report, the Ninth Progress Report issued under Air Force Contract AF 33(616)-5466, covers the period March 31, 1960 to June 30, 1960.

The research described in this report is part of an investigation being carried out at the University of Michigan for the Materials Central, Wright Air Development Division. The purpose of the investigation is to establish basic relationships between the structure and creep-rupture properties of heat-resistant alloys. The present phase of the work concerns the influence of hot working on the structure and properties of materials having essentially simple, single-phase microstructures.

Data are presented showing the effect of pre-induced substructure by tensile prestraining at 1600°F on the rupture life of 0.06-percent carbon "A"Nickel at 1100°F and 20,000 psi. The sub-boundary density was measured by a lineal intercept technique following delineation by etch pitting. Substructure data are also presented for 0.01-percent carbon "A"Nickel creep tested in vacuo at 1550°F and 3,360 psi.

Equipment for hot-rolling electron-beam-melted niobium in an argon atmosphere has been developed. Vacuum creep-rupture testing will be carried out.

WORK DONE DURING PERIOD

Substructures in Nickel

Substructures Produced by Tensile Straining at 1600°F

As discussed in Reference 1, the use of tensile straining at 1600°F to develop substructures by "hot working" was adopted. The main reason was to allow time for polygonization to occur so that the substructures could be delineated by etch pitting. The limitations on the conditions under which carbon would permit etch pit delineation of substructures in the "A"Nickel being used severely limited the conditions for even this type of working. The following results have been obtained:

Prestrain at 1600°F (%)*	Substructure Density (sub-boundaries per inch)**	Rupture Data at 1100°F and 20,000 psi: Rupture Life (hrs)	Elongation (% in 4D)	Red. of Area (%)
0	--	12.0	35.5	61.5
0	--	7.4	33.9	56.6
1.67	830	27.4	41.8	58.0
1.69	840	19.8	39.1	53.9
5.37	2,240	26.0	33.8	51.3
10.80	2,700	15.3	39.8	70.8
23.21	2.230	15.1	46.4	72.9

* At 30 percent per hour.

** Densities obtained by lineal intercept technique. Approximately 350 grains were sampled.

To check on where the maximum substructure density occurred as a function of prestrain, a specimen was prestrained 11.56 percent. The substructure density appeared to reach a maximum at about this point or a little earlier. (Refer to Figure 2 in Reference 2.) Because partial recrystallization occurred in the samples strained 10.80 and 11.56 percent, it is evident that substructure density in unrecrystallized grains increases sufficiently to cause an over-all increase while recrystallization occurs. The sample strained 23.21 percent recrystallized to a greater extent and, therefore, had a reduced mean substructure density. There was an increase in rupture strength with prestrain in the absence of recrystallization. The increase, however, was less than expected. For prestrains greater than about 5 percent recrystallization that occurred during prestraining limited the strengthening. Also, the fact that rupture life did not increase as the prestrain was increased from 1.69 percent to 5.37 percent suggests that further recrystallization may have occurred during rupture testing. This will be checked by metallographic examination.

Comparison of these results for tensile straining with those for rolling in Figure 3 of Reference 3 indicates that the strength level and increase in strength from rolling were much greater than when they were tensile strained.

Substructures Produced by Creep at 1550°F

At the time Reference 1 was written, it was indicated that there was reason to believe that a lower carbon "A"Nickel would respond to etch pit delineation of substructures over a wider range of creep temperatures than the 0.06-percent carbon "A"Nickel which had been used. The reported composition of a new heat was:

Ni	-----	99.78 percent
C	-----	0.01 percent
Mn	-----	0.03 percent
Fe	-----	0.032 percent
S	-----	0.005 percent
Si	-----	0.10 percent

The complete delineation of the substructure required for quantitative measurements was obtained by creep testing at 1550°F. This was similar to the higher-carbon material except that there were noticeably fewer areas in the low-carbon nickel where overt precipitation produced carbon-depleted areas where substructures could not be outlined by etch pitting with H_3PO_4. As the creep temperature was lowered, the delineation of substructures became less and less complete. The tendency for precipitation was greater in the high-carbon nickel. The lowest temperature for substructure delineation was not determined precisely for either material but the indications were that it was lower for the low-carbon nickel.

It was planned earlier to study the influence of impurities on substructure strengthening at 1100°F by comparing the slopes of rupture life vs substructure density curves for nickel at various impurity levels. Unfortunately, in the 0.06-percent carbon material, complications from recrystallization prevented the establishment of a well-defined rupture-life vs substructure curve over a reasonable range of prestrain values.

Also, the degree of substructure strengthening at 1100°F and 20,000 psi was smaller than expected on the basis of data for rolled bars.

For an alternate approach, a review of the literature suggested that a study of the effect of substructure on creep rate at constant stress might be feasible and profitable. As discussed earlier, a creep temperature of 1550°F was found suitable on the basis of the clarity of delineation of substructures produced. The selection of stress was based on (1) the obvious requirement that to determine the influence of an intragranular structure such as substructure on creep rate, deformation by grain boundary sliding and boundary migration must be very small relative to deformation from slip and lattice rotation within the grains and (2) the fact that excessive deformation on loading might complicate the study by introducing additional uncontrolled variables. The first requirement dictated the use of a high stress because grain boundary sliding is known to be most prominent at low stresses. The second requirement placed an upper limit on the stress. In the absence of specific precedents as to how much loading deformation could be tolerated, the stress was arbitrarily selected to give about 0.5-percent elongation on loading. For the low-carbon "A"Nickel, a stress of 3,360 psi at 1550°F was found to meet these requirements.

The procedure followed was to obtain substructure vs creep rate measurements on specimens vacuum creep tested at 1550°F and 3,360 psi with and without pre-induced substructures. The pre-induced substructures were obtained by creep to about 3.8-percent elongation at 3,860, 4,360, and 5,360 psi at 1550°F. The reason for including tests on specimens with substructures pre-induced at higher stresses was two fold: (1) to extend the range of substructure densities to higher values than might result from creep alone at 3,360 psi; and (2) to determine whether a true correlation exists between substructure density and creep rate by reaching the final substructures through various routes. Pre-creep at stresses below 3,360 psi was not planned because the sudden strain that would occur when the stress was raised to 3, 360 psi would obviously complicate matters by introducing a large supply of new dislocations. It was hoped that if undesirable effects were introduced by the higher loading strains at these stresses, these effects would be eliminated during creep before the stress was reduced to the correlation stress, 3, 360 psi.

It was decided to carry out the pre-creep at 1550°F so that the stress could be reduced to 3, 360 psi and an instantaneous value of creep rate obtained without cooling and reheating the specimen. This procedure would presumably allow one to detect transient variations of creep rate that might occur if significant changes in the equilibrium number of non-boundary dislocations occurred when the stress was lowered. The data obtained thus far are presented without comment in Figure 1.

Refractory Metals

Due to the delay in delivery of the high-temperature heating furnace for the rolling mill, a furnace with Hoskins 875 resistor wire was made. Investigation of the response of niobium to rolling indicates that this furnace should be adequate for at least most of the rolling temperatures which would be required. The equipment is being checked out rolling plain carbon steel. Indications have been obtained to prove that it will be possible to roll the niobium without contamination. A long and difficult process of elimination of sources of contamination of the argon has been carried out. Modifications of the unit to allow vacuum de-gassing while warming up have just been completed. Carbon steel bars rolled from 2100°F under this condition are bright with no indication of oxidation. Early completion of the rolling of niobium for the investigation is planned. Vacuum creep units have been constructed and operational difficulties are being eliminated.

REFERENCES

1. A. P. Coldren and J. W. Freeman, "Studies of Heat-Resistant Alloys", Eighth Progress Report to Wright Air Development Division under Contract No. AF 33(616)-5466 (March 31, 1960).

2. A. P. Coldren and J. W. Freeman, "Studies of Heat-Resistant Alloys", Sixth Progress Report to Wright Air Development Division under Contract No. AF 33(616)-5466 (September 30, 1959).

3. A. P. Coldren, J. E. White, R. K. Bowen, and J. W. Freeman, "Studies of Heat-Resistant Alloys", WADC Technical Report 59-606 (February, 1960).