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ANN ARBOR, MICH.

EIGHTH PROGRESS REPORT

TO

MATERIALS CENTRAL

WRIGHT AIR DEVELOPMENT DIVISION

ON

STUDIES OF HEAT-RESISTANT ALLOYS

by

A. P. Coldren
J. W. Freeman

Project 02760

Air Force Contract
AF 33(616)-5466
Task No. 73512

March 31, 1960

SUMMARY

Progress is reported for research carried out under Air Force Contract No. AF 33(616)-5466 covering the period of December 31, 1959 to March 31, 1960.

It was previously demonstrated that if "A"Nickel is slowly strained at 1550° or 1600°F, a substructure was formed that could be outlined by etch pits and measured under the microscope. However, decarburization and intergranular cracking at the surface occurred under these conditions, making it impossible to produce etch pits in localized areas and introducing uncertainties into the stress calculations. Attempts to solve these problems by the use of a dynamic helium atmosphere were only partially successful. Finally, a specimen creep tested 8.53 percent at 1550°F in a vacuum (10^{-4} mm. of Hg) was found to be completely free of decarburization, internal oxidation, and surface cracking.

Rupture tests at 1100°F and 20,000 psi on "A"Nickel showed that prestraining as little as 5.37 percent at 1600°F at 30 percent per hour caused the rupture life to be doubled with only a minor decrease in ductility.

The double-electron-gun-melted pure niobium was received during this period. The adaptation of the rolling mill for rolling in an inert atmosphere was completed except for the carbon-resistor furnace.

INTRODUCTION

This report, the Eighth Progress Report issued under Air Force Contract AF 33(616)-5466, covers the period December 30, 1959 to March 30, 1960.

The present investigation is part of a research program being carried out under the sponsorship of the Materials Central, Wright Air Development Division. The overall objective of the program is to establish basic relationships between the structure and creep-rupture properties of high-temperature alloys. The current work concerns the effect of hot working on the structure and properties of single-phase type materials.

Substructure and substructure-impurity interactions--presumably the two main factors besides work hardening that determine the creep-rupture properties of hot-worked metals--are being investigated in nickel. Also, the influence of hot rolling on the structure and properties of pure niobium is being evaluated.

WORK DONE DURING PERIOD

The main work done during the period involved clarification of procedures to be used to show the role of substructures in the creep of nickel. The development of equipment for hot rolling and creep testing refractory metals in inert atmospheres was continued.

Substructures in "A"Nickel

The experimental difficulties associated with the problem of relating substructures to creep-rupture properties of nickel were discussed in the Seventh Progress Report. For the "A"Nickel used in the initial hot-rolling experiments, it has been fairly well demonstrated that measurable substructures can only be delineated when the straining rate is sufficiently slow and the temperature sufficiently high to allow polygonization to occur. It was established that this occurred during creep testing at 1550°F and during slow tensile straining at 1600°F. Deformation or exposure at temperatures in the range of 1200° to 1550°F apparently resulted in precipitation of a carbon-bearing phase and thereby prevention of the "decorator" effect of this element to outline substructures by etch pits formed during etching with H_3PO_4. Other experimenters apparently had less difficulty from this cause inadvertently through the use of lower-carbon material. The "A" Nickel being used contained 0.06-percent carbon. In using slow straining at temperatures of 1550°F or higher to develop measurable substructures, it was found that intergranular cracking and surface decarburization was causing difficulty.

Analysis of the problem indicated that the most informative procedure would be as follows:

1. Use creep straining at 1550°F to produce samples with variable initial substructures which can be measured.
2. Utilize the creep tests at 1550°F to extend and confirm the information relating substructure formation to creep.
3. Check the influence of these substructures on creep-rupture properties at lower temperatures and correlate the results with those produced by hot rolling.

In order to do this, it was necessary to avoid the intergranular cracking and decarburization associated with creep at 1550°F or higher. The first attempt to solve the problem involved creep testing in a dynamic atmosphere of dried and gettered helium. It was found that while the rate of scale formation was greatly retarded the surface cracking and decarburization rates were only moderately reduced. It was then decided to conduct creep tests in a vacuum creep unit that was recently developed using equipment purchased with University funds. A unit was adapted to vacuum testing of the nickel. A specimen tested at 1550°F and 3000 psi was crept to 8.5 percent elongation in 37.3 hours. Both cracking and decarburization were eliminated.

The creep curve for the vacuum test is compared with that for an air test in Figure 1. As is usually found at low stresses, the creep rate was higher in the inert atmosphere. This occurred even though surface cracking and decarburization was eliminated. These results demonstrate that it is feasable to proceed as outlined above.

Most creep theories are based on secondary or steady-state creep. It is, therefore, necessary to know when steady-state creep can be expected. A test at 1472°F and 2500 psi in helium required 2.3-percent creep before the creep rate became constant. This seems to be more than the amount of creep involved in tests reported in the literature for studies of substructures using nickel. Inspection of available curves suggests that true steady-state creep was not involved in those tests, even though there was a difference in the contaminants from those of the nickel used in the present investigation.

The use of precreep to produce known initial substructures will be a very slow process due to the limitations of available vacuum creep testing units. Secondly, this method is far removed from hot rolling. Thirdly, it would be desirable to produce known substructures by some other method than creep to check the results. The nearest approach to hot working in which it is possible to produce known initial substructures is by tensile straining. In hot working itself, there is insufficient time for complete polygonization and apparently precipitation of a carbon-containing phase during creep testing at 1100°F prevents etch pit delineation of the substructures which develop during testing at this temperature.

Accordingly, samples are being prepared by tensile straining at 1600°F and subjected to creep-rupture testing at 1100°F and 20,000 psi.

The following results have been obtained:

Condition	Rupture Life (hrs)	Elong. (% in 4 D)	Reduction of Area (%)
Base Condition (1 hr. 1800°F, Water Quench)	7.4	33.9	56.6
	12.0	35.5	61.5
Base Condition + 1.67% Prestrain at 1600°F at 30%/hr.	27.4	41.8	58.0
" + 1.69% "	19.8	39.1	53.9
Base Condition + 5.37% Prestrain at 1600°F at 30%/hr.	26.0	33.8	51.3
" + 11% "	15.3	39.8	70.8
" + 23.21% "	15.1	46.4	72.9

The expected increase in rupture life was obtained. However, the expected difference from the two degrees of prestrained was not obtained and is being checked. The substructure density indicated was estimated from Figure 2 of the Sixth Progress Report (Ref. 1).

In Figure 2 (Ref. 1), the curve was incomplete in that no information was available between 6 and 19 percent strain. The latter strain was accompanied by partial recrystallization. Another sample has been strained 11.59 percent and will be examined for substructure density and recrystallization.

A sample of lower carbon "A"Nickel has been obtained to determine whether substructures can be delineated in this nickel with fewer complications from precipitation than in the nickel currently being used. The composition in weight percent is as follows:

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

INFLUENCE OF HOT WORKING ON CREEP-RUPTURE PROPERTIES OF NIOBIUM

Prior research on the influence of hot working conditions on creep-rupture properties of alloys has been carried out on face-centered cubic alloys. Quite pure niobium and a niobium alloy will be used to determine how a body-centered cubic metal responds.

The stock ordered from Mallory-Sharon Metals Corporation as double-electron-gun-melted niobium has been received. The bars are one inch in diameter. The shipment consisted of two heats: 3.9 pounds of Heat No. 5138, and 18.6 pounds of Heat No. 6133. There was a difference in grain size between the heats as shown in Figure 2.

The rolling mill adaptation for rolling in argon is completed except for a furnace. A carbon-resistor furnace had been scheduled for delivery in March (after delays from the original schedule). At the time this is being written, the manufacturer has not been able to indicate delivery time and will only state that they have had trouble obtaining suitable resistors.

When it became evident that the carbon-resistor furnace was going to be unduly delayed, construction was started on a Hoskins Alloy 875 resistance furnace. It will be capable of heating bars to 2100°F for rolling. It will be used also to check technique and atmosphere control. It should enable initial rolling of the pure niobium late in April.

Ordering of the alloyed niobium has been held up pending the availability of the carbon-resistor furnace so that the choice can be based on the latest information consistent with availability of the rolling mill.

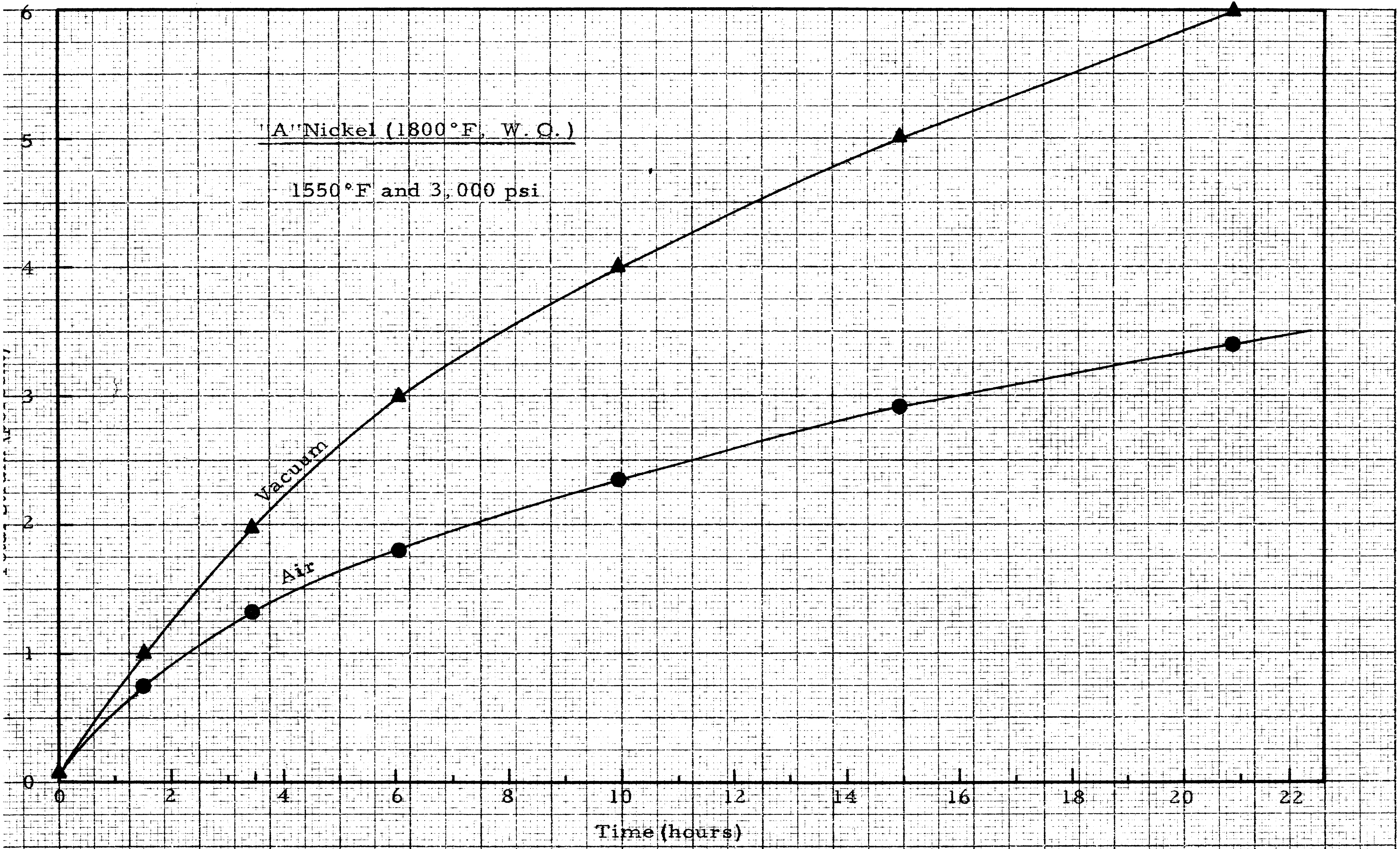
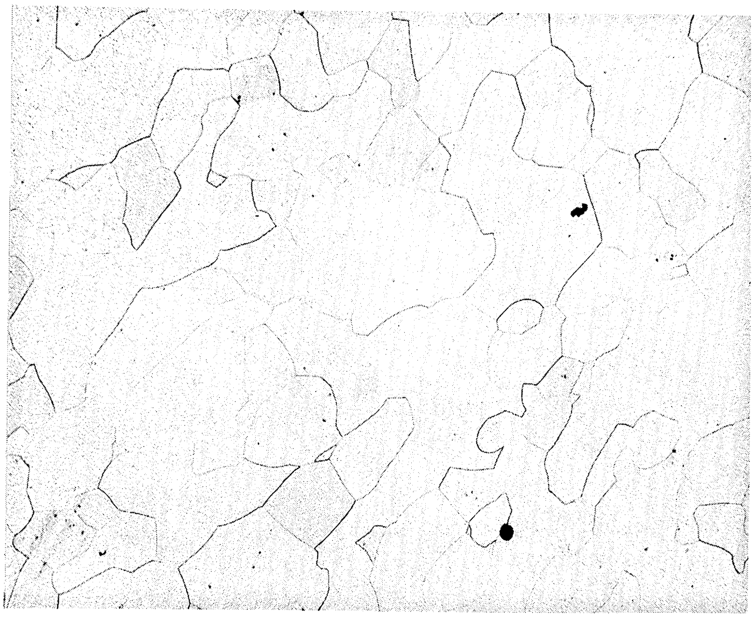
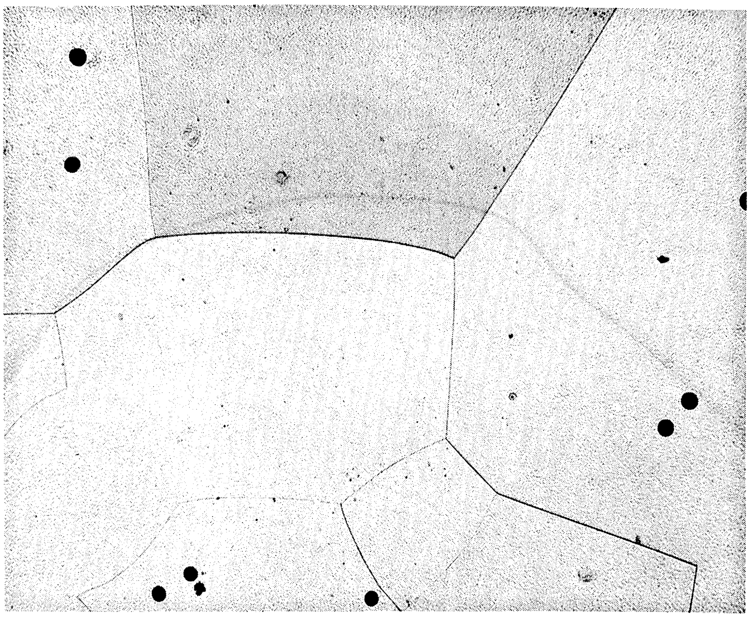


Figure 1. Influence of Atmosphere on Creep Curves of "A" Nickel at 1550°F and 3,000 psi Constant Stress. The vacuum was 0.1 micron of Hg or better.



(a) Heat No. 5138. Mag. 100X



(b) Heat No. 6133. Mag. 100X

Figure 2. Photomicrographs of Double-Electron-Gun-Melted Pure Niobium in the As-Received Condition. Electropolished in a solution containing 170 cc. HNO_3, 50 cc. HF, 510 cc. Methanol, and 5 g. citric acid. Chemically etched in same solution.

REFERENCES

1. 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, 1959).

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