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SEVENTH PROGRESS REPORT

TO

MATERIALS LABORATORY

WRIGHT AIR DEVELOPMENT DIVISION

ON

STUDIES OF HEAT-RESISTANT ALLOYS

by

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SUMMARY

Progress is reported for research carried out under Air Force Contract No. AF 33(616)-5466 covering the period September 30, 1959 to December 30, 1959.

It was established during this period that substructures (in "A" Nickel water quenched from 1800°F) resulting from creep alone can be completely delineated by the H_3PO_4 etch-pitting technique only if the creep temperature is above 1500°F. Metallographic examinations indicated that creep tests at lower temperatures resulted in early, general precipitation that removed carbon from solid solution and thereby made it unavailable to decorate dislocations. Partial substructure delineation was observed in longitudinal banded regions in specimens crept at 1350°F. It was suggested that there was a smaller tendency for precipitation in these bands because of deviations from the mean chemical composition. Differences in chemical composition were also proposed to explain some apparent conflicts between the present work and other published data on the use of H_3PO_4 etch pitting to reveal sub-boundaries in "A"Nickel.

During the period, an order was placed for double-electron-gun-melted pure niobium. A study will be made of the effect of hot working on the structure and properties of this refractory metal. Progress is reported on the adaptation of the rolling mill for hot working in an inert atmosphere.

INTRODUCTION

This report, the Seventh Progress Report issued under Air Force Contract AF 33(616)-5466, covers the period September 30, 1959 to December 0, 1959.

The present research is a continuation of work previously initiated at the University of Michigan for the Materials Laboratory, Wright Air Development Division, on the relationship between microstructure and creep-rupture properties in heat-resistant alloys. Specifically, the current studies deal with the influence of hot working on the structure and creep-rupture properties of materials having essentially single-phase structures.

Special emphasis is being placed upon the evaluation of substructure and impurity effects in nickel. The creep-rupture properties of "A"Nickel were previously (Ref. 1) determined as a function of rolling conditions. Those data suggested that the improvement in strength at 1100°F from rolling was due primarily to substructures plus any interaction there may have been between the substructures and impurity atoms. The present work is designed to test the accuracy of this hypothesis by (1) introducing measurable substructures into "A"Nickel and high-purity nickel under conditions that do not cause strain hardening of the type that broadens X-ray diffraction lines at high Bragg angles, and (2) determining the strength of these structures at 1100°F to see whether or not they approach the strength of the rolled bars. Information on the relative importance of impurities will be obtained by comparing the data from the "A"Nickel and the high-purity nickel. It was demonstrated in the Sixth Progress Report (Ref. 2) that by slow tensile straining at 1600°F, measurable substructures can be introduced into "A"Nickel. In the present report, it is demonstrated by an X-ray diffraction experiment that internal lattice strains are not introduced when substructures are formed by slow tensile straining at 1600°F.

In the area of refractory metals research, it is planned to study the influence of hot working on the structure and properties of pure niobium. During this work period, an order was placed for double-electron-gun-melted niobium to be delivered in the form of 1-inch diameter round bars. Also, progress is reported on adaptation of the rolling mill to permit hot rolling in an inert atmosphere.

WORK DONE DURING PERIOD

During the period, further studies were carried out to develop suitable tests of the role of substructures in hot-worked "A"Nickel. The procurement of high-purity nickel and niobium was advanced. Construction of the atmosphere chamber for the rolling mill was also carried along.

Substructure and Impurity Effects in Nickel

Substructures in "A"Nickel

In the Fifth Progress Report (Ref. 3), it was reported that there appeared to be sufficient precipitation of carbon during air cooling of "A" Nickel from 1800°F to prevent it from suitably decorating the dislocations so that etch pits could develop and outline the sub-boundaries. At least this appeared to be the reason why substructures could not be revealed after creep at 1100°F in air-cooled specimens or in specimens rolled below 1500°F. Microstructural evidence indicated that the precipitation could be suppressed by water quenching. Accordingly, a creep test was conducted on a sample water quenched from 1800°F using a test temperature of 1100°F and a stress of 11,000 psi. When examined after 4.1 percent creep, there was no visible substructure and a considerable amount of general precipitation as shown in Figure 1a. Presumably, sufficient precipitation took place before the substructure was formed to prevent decoration. This is suggested by the fact that specimens prestrained by slow tensile deformation at 1600°F to develop a well-defined substructure were shown in the Sixth Progress Report (Ref. 2) to retain delineation of the substructure during creep testing.

This result suggested that the lowest temperature for creep testing which would result in a measurable substructure ought to be established. A test at 1350°F under 5,250 psi with 1.42 percent creep resulted in banded areas of precipitation and visible sub-boundaries (Fig. 1b). A test at 1500°F under 3,325 psi with 3.48 percent deformation gave a well-delineated substructure, although there was still some precipitation (Fig. 1c shows area of complete delineation).

Further tests were then undertaken at 1550°F. No evidence of precipitation was found. Three tests were conducted at a stress of 3,000 psi to creep strains of 0.56, 1.91, and 3.53 percent (in 1, 5.1, and 22.5 hours, respectively). The substructure was rather well developed after 1.91 and 3.53 percent deformation. The number of small subgrains appeared to increase up to 3.53 percent deformation without much change in size.

The specimens tested at the higher temperatures and larger deformations were subject to extensive intergranular cracking at the surface. In those tested at 1550°F, there was very little cracking when the creep was limited to 0.56 percent. The two larger deformations exhibited about 40 cracks per inch, averaging 0.0018 inch in depth with cracks as deep as 0.005 inch. The surface layers were also depleted in decorator atoms so that the substructure would not etch in these areas.

These results indicate that measurable substructures can be produced either by slow tensile straining (Ref. 2) or by creep above 1500°F. The use of creep for this purpose would require that the surface by protected against reaction with air; or that large specimens be used which could be remachined to remove cracks and the decarburized surface metal.

The conditions under which measurable substructures can be produced have been surveyed. Pre-induced substructures require slow straining at temperatures above 1500°F to permit a very high degree of polygonization. In so doing, however, there are problems arising from surface cracking and surface decarburization. The cracking introduces an uncertainty into the stress calculation, and the decarburization prevents the formation of etch pits to delineate the substructure. The possibilities of deriving a series of samples produced by hot straining which can be used to determine the role of hot working in increasing strength are being evaluated.

One of the questions concerned with the evaluation of pre-induced substructures under creep conditions is the role of internal strain which may be present from cold work. In the experiments, it is desirable to separate the two effects. The well-defined, nearly equiaxed substructures associated with slow tensile straining at 1600°F described in the Sixth Progress Report (Ref. 2) indicated that there might be very little cold work present. This was verified by comparing the width of the X-ray diffraction line from the 420 reflection for a sample deformed 19.7 percent at 1600°F at a rate of 30 percent per hour with that of an annealed specimen. The line widths were equal as shown by the microphotometer traces in Figure 2. This indicated that samples prepared in this manner would reflect the role of substructure (plus impurities) alone.

Considerable effort was expended in attempts to understand the results in view of possible discrepancies with published information in the literature. The limited delineation of substructures in the samples being studied in comparison to published data is particularly puzzling. The "A" Nickel being used is rather high in carbon (0.06 percent) and in other trace elements as well in comparison to other investigators' "A"Nickel. Guard for instance (Ref. 4) used "A"Nickel with 0.015 percent carbon. It has been noted (Ref. 3) that when precipitates form, it is not possible to obtain proper etch pitting with H_3PO_4. Apparently, the larger amounts of contaminants in the material used for this investigation has intensified this problem. Guard's photomicrographs, for instance, indicated a depleted zone along grain boundaries which would not etch when grain boundary precipitates did form. There is a good possibility that the fewer impurities in his material was responsible for the better delineation of substructures in Guard's work because of a smaller tendency for overt precipitation.

It should also be recognized that this investigation has the objective of studying substructures as established by hot deformation. Other investigators have been concerned with structures derived from cold work plus polygonization heat treatments or those developed by creep alone. The problems of delineating structures developed by hot deformation may be quite difficult.

Further work will consist of utilizing the known conditions of delineating substructures and utilizing these to define the role of substructures in creep-rupture as developed by hot working.

High-Purity Nickel

The initial attempts to deoxidize carbonyl nickel powder with magnesium to produce a relatively pure nickel ingot were unsuccessful due to the violent vaporization of magnesium. The procedures used were reviewed with the individuals who had recommended the method. No obvious differences in procedure were determined. Several suggested modifications did not solve the problem. At present, the procedure is being studied for a way to avoid the difficulty.

REFRACTORY METAL (NIOBIUM)

An order was placed with the Mallory-Sharon Metals Corporation for 25 pounds of double-electron-gun-melted niobium. The stock is to be supplied as 1-inch diameter annealed bar stock.

The material is to be produced to a Brinell hardness less than 85. Typical chemical composition was stated to be: 170-270 ppm of oxygen, 35-100 ppm of nitrogen, and 100-155 ppm of carbon.

Fabrication is to consist of machining the surface of a 3-inch diameter ingot, forging from argon at 2000°F to 1.25-inch diameter, and rolling to 1-inch round. The round bar is to be conditioned by sand blasting and pickling. It will be cut into 10-inch lengths and vacuum annealed.

The order was placed with Mallory-Sharon on the basis that they could provide the size bar needed in a reasonable time. Other sources required the acceptance of a large-diameter ingot or a prolonged delivery time for making the bar stock. The cost between the different sources did not vary appreciably.

Modification of Rolling Mill for Inert-Atmosphere Rolling

The chamber sealing the rolls is nearly completed. It is expected that it will be possible to make the first trials with the mill early in January, 1960. First trials will utilize a Hoskins Alloy 875 resistance wound furnace capable of operating up to 2200°F.

After extensive checking of possible furnaces for very high temperatures, the choice of a graphite resistor tube furnace seemed most practical. Accordingly, as cleared with the WADD early in this reporting period, a tube furnace having an inside diameter of 4 inches and a total heated length of 24 inches was ordered from the Hevi-Duty Electric Company. This furnace is designed to have a relatively uniform hot zone over the center 12 inches of its length and will be capable of temperatures up to 4000°F with the 40KW saturable reactor control system to be provided. This will provide stepless control over the entire temperature range of the furnace. A combination water-jacketed cooling chamber and purge chamber will be provided at one end to provide a means for loading and removing bars from the furnac The other end of the furnace will be attached to the chamber around the rollin mill. Pieces up to about 2-1/2 inches in diameter and 12 inches long can be accommodated by the furnace. Delivery of the graphite furnace is anticipate in February, 1960.

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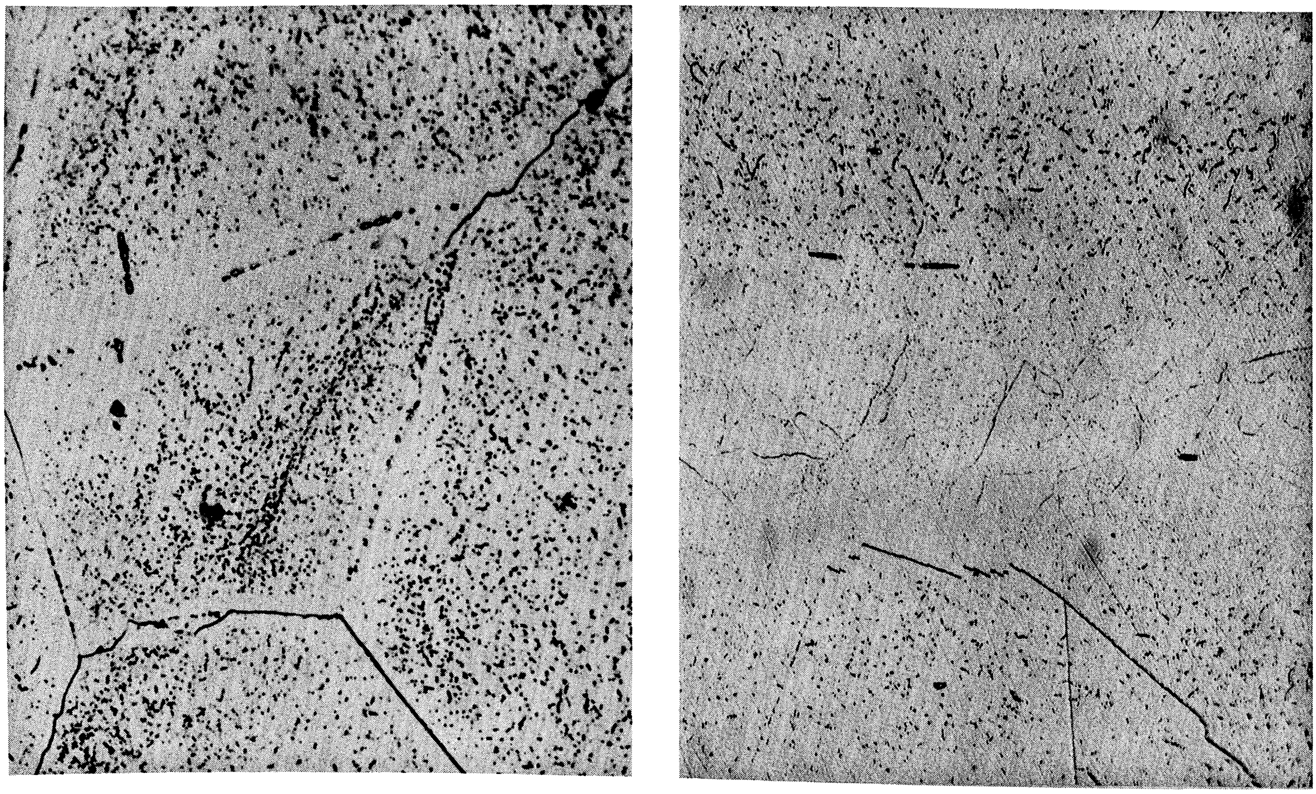
4. R. W. Guard, "The Role of Polygonization in Creep", Preprint Copy of WADC Technical Report Covering Work Done for Aeronautical Research Laboratory Under Contract No. AF 33(616)-3263, Appendix A (December, 1959).



(a) 4.1% Creep Strain in 166 Hours at 1100°F and 11,000 psi. X1000.

(b) 6.3% Creep Strain in 45 Hours at 1350°F and 5,250 psi. X500.

(c) 3.5% Creep Strain in 20 Hours at 1500°F and 3,325 psi. X250.

Figure 1. "A"Nickel Water Quenched from 1800°F Followed by Creep at (a) 1100°F, (b) 1350°F, and (c) 1500°F. Etched electrolytically in 40% H_3PO_4.

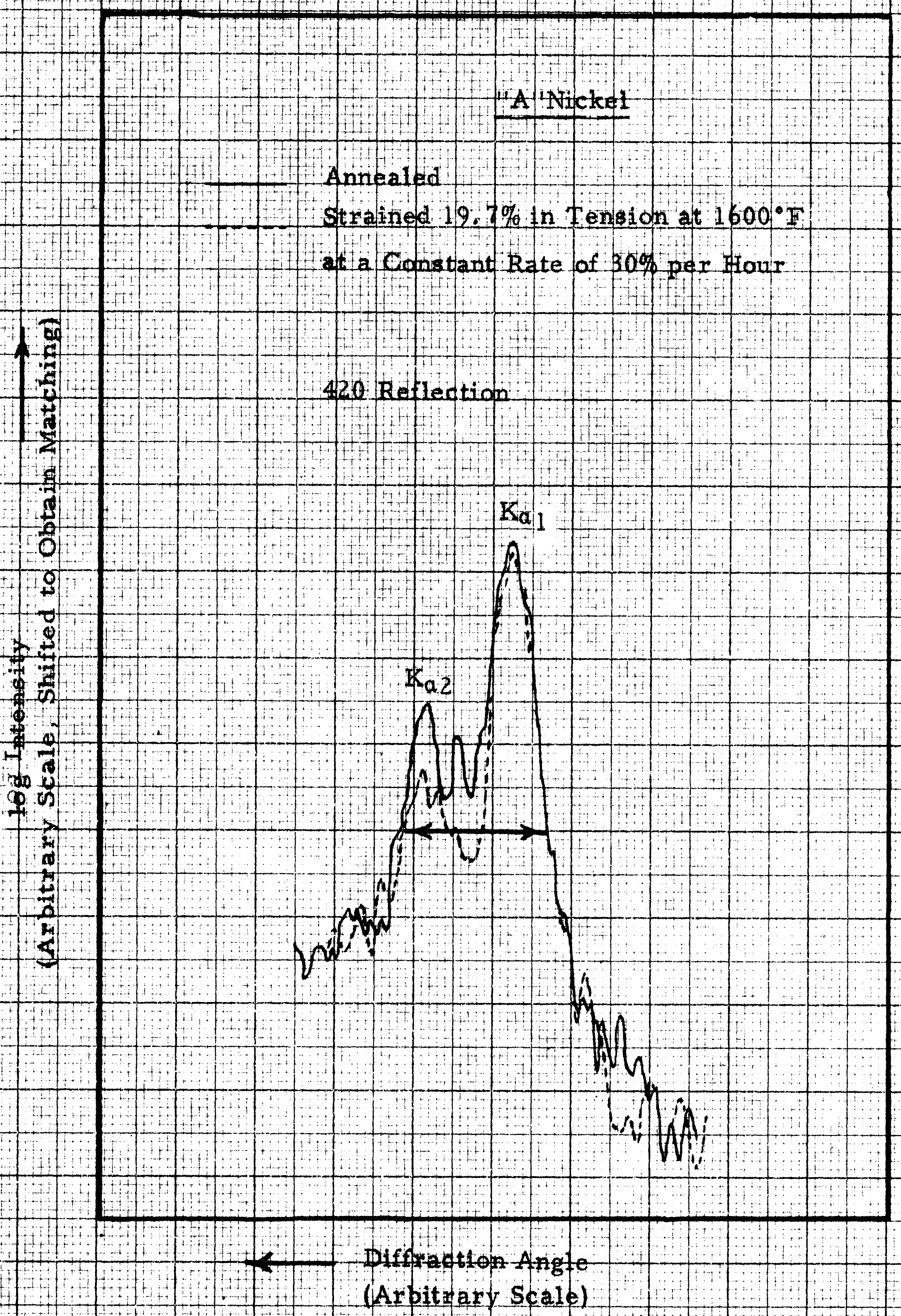


Figure 2. Profiles of X-ray Diffraction Lines from (420) Planes in Annealed and Hot-Strained "A" Nickel. Note that the widths of the lines are equal, indicating an absence of lattice stresses of the type found in cold-worked metals. The two photograms were taken on the same sheet of film so that photographic processing would be identical.

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