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FIFTH PROGRESS REPORT

TO

MATERIALS LABORATORY

WRIGHT AIR DEVELOPMENT DIVISION

ON

STUDIES OF HEAT-RESISTANT ALLOYS

by

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SUMMARY

This report covers the period of March 30, 1959, to June 30, 1959. Efforts during this period centered on preparing the Summary Report for work carried out during the fifteen-month period ended June 15, 1959. The remaining time was spent planning the research to be carried out during the twelve-month period beginning June 15, 1959.

The objectives of the research planned for the coming year are discussed, and the general experimental approach to be used is outlined for each of the subject materials.

INTRODUCTION

This report, the Fifth Progress Report issued under Air Force Contract AF 33(616)-5466, covers the period March 30, 1959, to June 30, 1959. Most of this period was spent in preparing the Summary Technical Report covering the fifteen-month period ended June 15, 1959. The present report gives an outline of the research objectives for the coming year, and the experimental approach planned for each material is discussed.

The present investigation is an extension of previous research initiated at The University of Michigan for the Wright Air Development Division on the relation of microstructure to creep-rupture properties in heat-resistant alloys. The objective of the present work is to study the variations in structure and properties associated with hot working. Specifically, it is sought to obtain an approximate evaluation of the relative effects of substructure and strain hardening, the two factors believed to be the major causes of strengthening when a single-phase material is hot worked at temperatures too low for simultaneous recrystallization.

The creep-rupture properties of a commercial grade of pure nickel ("A" Nickel) were previously determined (Ref. 1) as a function of hot rolling conditions, but extensive efforts to identify the substructure in the rolled bars were only partially successful (Ref. 2). The study of "A" Nickel is being continued in the belief that a slower type of hot deformation will produce substructures that can be delineated by etching, making it possible to measure the subgrain size and study its effect on creep resistance. Also, it is planned to evaluate the influence of impurities by measuring the strengthening effect of substructures in high-purity (99.95+ percent) nickel and comparing it with the effect of similar substructures in "A" Nickel (99.4 percent pure).

In view of the increasing interest in the metals having very high melting points, it was decided to include refractory metals in the program. The importance of studying controlled hot working as a means for strengthening both pure and alloyed refractory metals is emphasized by the general practice of working such metals to avoid brittleness. Furthermore, from an academic point of view it will be helpful to compare the hot working response of the body-centered-cubic refractory metals to that of the face-centered-cubic nickel previously investigated. The program also included the purchase of a suitable furnace and adaptation of a rolling mill to enable the hot rolling of the reactive refractory metals.

WORK DONE DURING PERIOD

The major effort of the period was devoted to analysis of previously obtained data (Refs. 1 and 2). The most pressing problem was to determine ways in which the role of substructures and impurities could be determined. This was done by analysis of both the previous experimental results for "A" Nickel from this program and the data in the literature. The plans for the adaptation of the rolling mill to inert-atmosphere hot rolling were made, and the types of niobium available were investigated in preparation for ordering stock.

Substructure and Impurity Effects in Nickel

In the previous research on "A" Nickel, both substructures and impurities could be influencing results. Accordingly, a thorough analysis of available data was carried out with correlation to published research in the literature on substructures in an effort to establish the most promising procedure to delineate the two effects.

Delineation and Measurement of Substructures

The earlier attempts to delineate substructures by the etch pits produced by electrolytic etching in a 40 percent H_3PO_4 solution (Ref. 2) were only partially successful. The literature search indicated that this technique could only be expected to be successful if the structures were highly polygonized so that the sub-boundaries would be sharp. From this viewpoint then, the cases where the substructures were delineated involved rolling under conditions which would allow polygonization to take place. Those structures rolled at the highest temperature (1800°F) with small reductions would be most likely to polygonize. Thus the sample reduced 5.8 percent at 1800°F responded to the etch.

Samples of several bars rolled under slightly less favorable conditions for polygonization exhibited substructures after creep testing for several hundred hours at 1100°F. Polygonization could be expected to occur slowly at 1100°F and in a few favorable conditions resulted in structures which responded to the etch. Large reductions by rolling at 1800°F and reductions at lower temperatures resulted in such disturbed structures that sufficient polygonization could not take place even during several hundred hours at 1100°F.

The review of the literature disclosed very few cases where etch pits delineated substructures unless impurity atoms were present in the boundaries as "decorator" atoms. Apparently, the H_3PO_4 etch requires such decorator atoms. Etch-pitting experiments by Guard (Ref. 4) on nickel indicated quite definitely that for etching with H_3PO_4, carbon is the decorator element in nickel. The samples from the hot-rolling program were again restudied under the microscope and it was verified that:

1. Precipitate particles formed when "A" Nickel in the form of a 7/8-inch square bar was air cooled from above 1500°F.

2. When the precipitate was present, etch pits would not form. Since all the bars were air colled from 1600°F before rolling, this precipitate had formed. It is presumed that this precipitate contains sufficient carbon to prevent carbon from decorating the substructures. Hence, only those samples rolled at 1600°F or higher had the carbon in a form suitable for decoration. Moreover, it appeared that the migration of the carbon to the sub-boundaries

prevented it from precipitating during cooling from rolling at 1600°F and 1800°F or during subsequent creep testing.

Extensive study of specimens under the microscope and analysis of the data and theory therefore led to the following conclusions regarding future work:

1. Polygonization rates are too slow for the delineation of substructures by the H_3PO_4 etch-pitting method in as-rolled samples unless the temperature of rolling is kept above 1600°F and the reduction limited to less than 10 percent. To be able to produce substructures which can be measured prior to testing, it would be necessary to use strain rates slower than those involved in hot-rolling so that polygonization would have time to occur. Information in the literature seems to indicate that this procedure is to be preferred to simply holding the material at temperature after rolling. Polygonization occurs more readily if the specimen is being deformed than if it is held at temperature after deformation. Perhaps it is more important that it avoids the possibility of recrystallization destroying substructures. This type of method of developing polygonization should also minimize or possibly even eliminate internal strains of the type evidenced by broadening of x-ray diffraction lines.

2. Any known technique for developing etch pits to outline the subboundaries in nickel requires the presence of decorator atoms. For the lot of "A" Nickel being used, deformations must be carried out above 1500°F to avoid precipitation of carbon and the loss of its effectiveness as a decorator.

3. Experiments will be necessary to determine the conditions of tensile

measured. Once this is done, the creep characteristics will be measured at 1100°F as a function of these substructures. Since it was indicated in Ref. 2 that substructures in the rolled samples was the controlling factor at 1100°F, the tests should prove or disprove this theory. Every effort will also be made to develop an etch which will produce etch pits outlining the subgrains of polygonized structures in which decorator elements are not present. This would remove many of the restrictions now imposed by the requirement that carbon being available for decoration. Moreover, it would enable similar studies with higher-purity nickel.

High-Purity Nickel

Because "A" Nickel contains substantial amounts of impurities, it is not certain to what degree the observed strengthening may be due to the presence of impurities at the sub-boundaries. The probability is that there is an interaction between the impurities and the substructure which can markedly increase creep resistance. Consideration of this problem lead to the following conclusions:

1. Hot rolling experiments similar to those conducted on "A" Nickel would be carried out on high-purity nickel. A comparison of the strength versus percent reduction curves would indicate what influence the impurities have.

2. Every effort would be made to compare the substructures in the two materials, including, if necessary, the introduction of decorator elements by diffusion after the substructures have been developed.

3. At least a limited amount of research should be done on "pure" nickel with slow deformation similar to that proposed for "A" Nickel to develop the

same structures.

Refractory Metals

The suitability of the suggested refractory metals for the research program (chromium, niobium, and molybdenum) are being investigated. When this is completed, it will be reviewed with representatives of the Materials Laboratory and a choice made. The several factors being weighed are as follows:

1. Availability of suitable experimental material.
2. Future usefulness of the metal.
3. Experimental difficulties in studying the metal.
4. Problem of reactivity with air.

The only metal which offers promise at the present time of resistance to reactivity with the atmosphere is chromium. This metal, however, is not available as a suitable experimental material due to brittleness. Moreover, no alloy has yet been devised which offers promise of high strength at the more elevated temperatures. It, therefore, begins to appear that the use of chromium would be premature.

Niobium would be the easiest to obtain as suitable experimental material. Moreover, the initial work on pure niobium would be at moderate temperatures where the experimental problems would not be as difficult as they would be for Mo, W or Ta. Data on niobium would have widespread practical use.

When the study is completed, the factors will be reviewed with the Materials Laboratory and a choice made.

Adaptation of Rolling Mill to Inert-Atmosphere Rolling

The factors involved to design a suitable method of hot-rolling in an inert atmosphere are being carefully reviewed. At the present time, it appears that the most practical procedure will be to build a chamber around the mill which can be filled with argon. Preliminary drawings are being prepared. The choice of a proper heating furnace is proving difficult and is being investigated with representatives of other laboratories who have had experience with extremely high temperature furnaces for heating refractory metals. It has been decided that the first furnace will be an ordinary resistance furnace. The experience gained with this furnace will be used to develop techniques.

The new rolls purchased from the Birdsboro Company by the University have been received and are being installed. These rolls were specifically designed to roll refractory metals.

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