THE UNIVERSITY OF MICHIGAN RESEARCH INSTITUTE
ANN ARBOR, MICH.

FOURTH PROGRESS REPORT

TO

MATERIALS LABORATORY

WRIGHT AIR DEVELOPMENT CENTER

ON

STUDIES OF HEAT-RESISTANT ALLOYS

Phase A

Influence of Hot Working on Structure and Creep-Rupture Properties

Phase B

Relationship Between Strain Aging Phenomena and High-Temperature Strength

by

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Project 2760

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

March 31, 1959

SUMMARY

Progress is reported for research carried out under Air Force Contract No. AF 33(616)-5466 covering the period of December 31, 1958 to March 31, 1959.

The overall objective of the present research is to study the relationship between microstructures and creep-rupture properties in heat-resistant alloys. The research is handled in two parts: (a) the influence of hot working on the structure and creep-rupture properties of "A" Nickel, A-286 alloy, and "17-22-A"V steel, and (b) the relationship between strain aging phenomena and high-temperature strength in two 1020 carbon steels and in the A-286 alloy.

The problem of revealing and measuring substructures in "A" Nickel is discussed in this report. Also included is a description of an attempt to use transmission electron microscopy to reveal substructures in rolled nickel samples.

In the structural study of A-286 alloy it was found that the problem of excessive attack of a grain boundary phase during electropolishing could be avoided by using a combination of mechanical polishing plus ion bombardment instead of electropolishing.

Results of the latest rupture tests on "17-22-A"V steel at 1100°F have established a definite beneficial effect of plastically deforming the austenite just prior to the bainite transformation which in this steel is 100-percent complete on air cooling 1/2- to 1-inch rounds to room temperature. A brief survey of the "17-22-A"V structures with the electron microscope

showed no obvious differences in the matrix structures of rolled and non-rolled specimens at a magnification of 22,000 diameters. At 2,200 diameters there appeared to be a difference in the distribution of the excess phases (probably mostly carbides). In the cases where the bainite was formed from strain-free austenite, the carbides precipitated preferentially at the former austenite grain boundaries; whereas in specimens rolled below the simultaneous recrystallization temperature the carbides were distributed more randomly throughout the structure.

In the strain aging study, preliminary electron micrographs of the aluminum-deoxidized and the silicon-deoxidized carbon steels indicate a difference in the structure of the two steels. A study of the influence of plastic strain on precipitation in the carbon steels is nearly complete. Constant strain rate tests on A-286 are being carried out in the new automatic machine.

INTRODUCTION

This report, the fourth quarterly progress report issued under Air Force Contract No. AF 33(616)-5466, covers work done from December 31, 1958 to March 31, 1959.

The present research follows earlier work carried out for the Wright Air Development Center at the University of Michigan on the relationship between microstructure and creep-rupture properties of heat-resistant alloys. The earlier work was done on low-alloy steels, and the microstructures were varied by heat treatment alone. The present research has been extended to include materials of a more refractory nature and to include hot working as a processing variable which affects microstructure and properties. For convenience, the work is handled under two general phases:

Phase A - Influence of Hot Working on Structure and Creep-Rupture Properties

Phase B - Relationship between Strain-Aging Phenomena and High-Temperature Strength.

The hot working studies are being carried out on three materials: (a) a commercially pure metal ("A" Nickel), (b) a precipitation-strengthened, austenitic alloy (A-286), and (c) a ferritic alloy of low strategic element content (Timken "17-22-A"V steel). The strain aging studies are being done on (a) a silicon-deoxidized 1020 steel (Steel C) which is susceptible to strain aging in all conditions, (b) an aluminum-deoxidized 1020 steel (Steel F) which may or may not be susceptible to strain aging, depending on the heat treatment, and (c) a high-strength, austenitic alloy (A-286).

TEST MATERIALS

The materials for the hot-working study were supplied gratis by the following organizations: "17-22-A"V steel from the Timken Roller Bearing Company, A-286 alloy (Heat 21030) from the Allegheny-Ludlum Steel Corporation, and "A" Nickel from the International Nickel Company.

The plain carbon steels (Steel C and Steel F) used in the strain aging study were obtained from the Chemical and Petroleum Panel of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals. The A-286 alloy (Heat 82073) used in the strain aging experiments was supplied by the Materials Laboratory, Wright Air Development Center from the stock used by Captain Domian to study strain aging by hot-hardness tests.

The chemical compositions were reported by the manufacturers as follows:

Alloy	C	Mn	Si	Cr	Ni	Mo	V	Fe	Other
"A"Nickel (Ht N9500A)	0.06	0.27	0.06	-----	99.46 (Ni+Co)	----	----	0.09	0.03Cu; 0.008S
A-286 (Ht 21030)	0.06	1.35	0.47	14.58	25.3	1.38	0.21	Base	2.00Ti; 0.17Al
A-286 (Ht. 82073)	0.03	1.27	0.62	14.58	25.44	----	0.59	Base	0.008N;0.12Al 0.37Co;Nom. Ti
"17-22-A"V (Ht. 11833)	0.29	0.70	0.71	1.43	0.31	0.51	0.81	Base	----
1020 Steel C	0.20	0.68	0.27	----	----	----	----	Base	0.015Al;0.0048N; 0.028P;0.034S
1020 Steel F	0.19	0.68	0.24	----	----	----	----	Base	0.053Al;0.0046N; 0.026P;0.036S

PHASE A - INFLUENCE OF HOT WORKING ON STRUCTURE AND CREEP-RUPTURE PROPERTIES

The purpose of this part of the investigation is to correlate creep-rupture properties with microstructure in the three subject alloys which were systematically hot rolled to produce structural differences. The "A" Nickel and A-286 alloy were processed and creep-rupture tested under a previous WADC contract (Ref. 1). The "17-22-A"V steel was hot worked and tested under the present contract. The last creep rupture data are presented in this report.

Commercially Pure Metal ("A" Nickel)

The choice of "A" Nickel for this study was based on the assumption that all of its important structural variables would be amenable to identification and subsequent correlation with the creep-rupture properties. However, as pointed out in the Third Progress Report (Ref. 2), efforts to identify and measure substructures have resulted in limited success.

Transmission Electron Microscopy

The failure of both X-ray diffraction and etching techniques to reveal the substructures in nickel rolled more than 6-percent reduction and/or at temperatures below 1600°F was discussed in the Third Progress Report (Ref. 2). It was assumed that the difficulty arose from the presence of diffuse regions of highly distorted lattice interspersed between regions of high crystalline perfection (subgrains). This model of the structure of a deformed polycrystalline metal was proposed by Gay, Hirsch and Kelly

(Ref. 3) who further described the distorted lattice regions as zones of high dislocation density. Theoretically, these regions ought to be visible by transmission electron microscopy since individual dislocations can be seen by this technique(Ref. 4).

Consequently, it was undertaken to develop a technique for preparing specimens suitable for transmission electron microscopy. A man was available who had been working for some time on transmission microscopy under the direction of Professor W. Bigelow. The problem was to prepare from a bulk piece of rolled nickel a specimen thin enough to allow an electron beam (accelerating voltage - 75 KV) to pass through it. To avoid deformation and alteration of the rolled structure a technique was developed whereby a 0.010-inch thick wafer cut from the bulk sample could be ground to a uniform thickness of 0.001-inch and then electro-polished to the final thickness. The grinding was done with diamond abrasive wheels under a stream of water. The grinding apparatus, which was specially designed and built for a University dental laboratory for the study of thin sections of teeth, was capable of removing exceedingly small amounts of material per pass over the specimen. The problem of achieving a uniform thickness during grinding was solved by mounting the wafer on a very flat metal plate using a special resin type of adhesive which could be used in extremely small amounts. The wafer was turned over midway through the grinding operation.

The 0.001-inch thick ground wafer was then electropolished in a perchloric acid-acetic acid solution until the specimen was sufficiently thin for viewing in an electron microscope.

Limited success was achieved in that only a few thin foils of the specimens yielded transmission micrographs showing recognizable structural features. The main difficulty in the technique of thinning the metal down appeared to arise from the many variables of electro-polishing. When this became evident the development was discontinued. Any further development of the technique would require the availability of a more promising electro-chemist than was available.

Precipitation-Strengthened, Austenitic Alloy (A-286)

Structural Studies

The A-286 alloy was hot rolled, heat treated, and creep-rupture tested under the previous contract (Ref. 1). The microstructural analysis of this material was hampered by two serious problems. The first problem was to find an etchant that would reveal the age hardening precipitate which in A-286 is very small and closely spaced. A modified form of glyceregia (5 Glycerine-4HCl-1HNO_3) was found to work, provided that the sample surface was free of disturbed metal. The glyceregia produced only etch pits on mechanically polished specimens.

The natural alternative was to electropolish the samples. Electropolishing was effective in producing the work-free surface, but it introduced a new problem. In many samples, a series of short, deep crevices were formed along grain boundaries during polishing, and it was not clear whether the crevices were microcracks produced during rolling or whether they were sites where a grain boundary phase had been attacked during electropolishing.

To determine whether the crevices were produced by the removal of a grain boundary phase, a process called ionic bombardment or vaccum cathodic etching was used to remove the cold-worked surface of mechanically polished samples. This method rules out the possibility of a chemical or electro-chemical attack of grain boundary phases because the surface metal atoms are removed mechanically by the impact of argon ions accelerated to the specimen in a vacuum chamber by a potential difference of about 5,000 volts. The freshly exposed surface was then swabbed with glyceregia. Replicas were made and examined in the electron microscope, and no crevices were found. It was concluded that the crevices in electropolished samples were produced by a rapid dissolution of a grain boundary phase.

The rate of metal removal under ionic bombardment is rather sensitive to crystallographic orientation, with the result that the surfaces of the individual grains are at slightly different levels. Also, the grain boundaries frequently appear as inclined planes connecting grains of different levels. This does not obscure boundary phases.

The sequence of grinding, mechanical polishing, ionic bombardment and swabbing with glyceregia is being used on all the A-286 specimens for the final correlation between structures and creep-rupture properties. This work is in progress.

Ferritic Alloy ("17-22-A"V Steel)

Hot Rolling Experiments

Hot rolling experiments on "17-22-A"V steel reported in the Third Progress Report (Ref. 2) showed a pronounced improvement in rupture strength at 1100°F when bainite was allowed to transform from plastically deformed austenite rather than from the usual strain-free austenite. Specifically, bars were heated to 2200°F for 1 hour, air cooled to a temperature too low for simultaneous recrystallization, 1900°F, rolled 50 percent reduction, and allow to transform to bainite during air cooling to room temperature. The bars were tempered for 6 hours at 1250°F prior to testing.

To test whether a still greater improvement in rupture strength could be produced by deforming the austenite at a considerably lower temperature, a bar was treated in exactly the same manner as described above, except that the rolling temperature and reduction were 1600°F and 60 percent, respectively. The rupture test results at 1100°F for the steel in this condition are compared with the data for steel rolled at 1900°F:

Rolling Temp. (°F)	Stress (psi)	Rupture Life (hrs)	Elongation (%)	R.A. (%)
1600	55,000	72.7	6.4	12.7
1600	40,000	493.8	1.8	6.0
1900	55,000	32.2	9.1	16.8
1900	40,000	324.6	1.5	4.8

Microstructural Study

Electron micrographs were taken on all of the "17-22-A"V samples to see whether structural differences could be found to explain the beneficial effect of working the austenite.

No appreciable differences could be detected in the structure of the matrix as viewed at a magnification of 22,000 diameters. In each sample there appeared areas of differing shade and texture. These differences are assumed to arise from differences in the composition of the austenite from which the bainite formed. Since the relative proportions of light and dark areas was the same in all samples it is assumed that the differences in properties are caused by some other structural features.

Micrographs at 2,200 diameters gave some indication regarding the size and distribution of excess phases which are assumed to be mostly carbides. In the bainite formed from strain free austenite there was a tendency for the carbides to precipitate preferentially at the boundaries--especially when the prior austenite grains were small. In the bainite formed from plastically deformed austenite, however, the carbides were more random throughout the structure, leaving the average concentration at prior austenite boundaries lower.

PHASE B - RELATIONSHIP BETWEEN STRAIN-AGING PHENOMENA AND HIGH-TEMPERATURE STRENGTH

During the previous periods carbon steels susceptible to strain aging were found to exhibit high strengths at higher temperatures for a strain rate of 0.004 percent per hour. In an attempt to follow up a precipitation strengthening hypothesis a program of microstructural studies using the electron microscope is in progress on the carbon steels.

A-286 alloy is concurrently being studied as to the effect of strain aging on its high temperature properties. In the Third Progress Report (Ref. 2) differences in strain-aging type characteristics were indicated between material solution treated at 1650°F and 1800°F when rapid strain rate tensile tests were conducted. However, no differences were found when constant strain rate tests at 0.004-percent per hour were conducted.

Carbon Steel

The study of the structures of the carbon steels by electron microscopic means has been continued. A series of samples of the aluminum-deoxidized and silicon-deoxidized steels are being heated at 1000°F for time periods up to 500 hours. These are being examined to determine if the precipitate particles previously observed in the Al-deoxidized steel would develop. This work is in progress. Preliminary results indicate a definite difference between the two steels. Specimens were also prepared in which both steels were strained at 10 percent per hour to 10 percent strain and to fracture. These are also being examined to study the effect of strain on the precipitate.

A-286 Alloy

Analysis of the results of constant strain rate tests suggested that the rate of 0.004 percent per hour used for previous tests was too slow to show the strain aging effect. For this reason, tests at 0.1 percent per hour are to be conducted. Tests at this rate require the use of the automatic machine for conducting constant strain rate tests. Difficulties in operation of this machine are being corrected and it is expected that the tests will be completed during the next period.

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