https://ntrs.nasa.gov/search.jsp?R=19630004526 2020-03-24T06:11:56+00:00Z

N 53 14402

NASA TN D-1531



**NASA TN D-1531** 

# TECHNICAL NOTE D-1531

CONTINUED INVESTIGATION OF AN ADVANCED-TEMPERATURE,

TANTALUM-MODIFIED, NICKEL-BASE ALLOY

By John C. Freche and William J. Waters

Lewis Research Center Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION WASHINGTON April 1963

#### NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

#### TECHNICAL NOTE D-1531

#### CONTINUED INVESTIGATION OF AN ADVANCED-TEMPERATURE,

#### TANTALUM-MODIFIED, NICKEL-BASE ALLOY

By John C. Freche and William J. Waters

#### SUMMARY

The investigation of an advanced-temperature, NASA nickel-base alloy having a nominal composition in weight percent of 8 tantalum, 6 aluminum, 6 chromium, 4 molybdenum, 4 tungsten, 2.5 vanadium, 1 zirconium, 0.125 carbon, and balance nickel was continued. This alloy appears to have considerable potential for various aerospace applications.

The average ultimate tensile strength of the as-forged alloy ranged from 158,000 psi at room temperature to 18,000 psi at  $2100^{\circ}$  F. This compares with 135,000 and 34,000 psi, respectively, for the as-cast alloy. Stress-rupture data obtained up to  $2100^{\circ}$  F at 15,000 psi indicate average rupture lives for the as-cast alloy of 1000, 100, and 10 hours at  $1817^{\circ}$ , 1915°, and 2015° F, respectively. Impact tests provided evidence of alloy ductility. Additional evidence of work-ability was also obtained. Room-temperature forging, accomplished by unidirectional forging techniques, readily flattened 1/2-inch diameter, as-cast bars, and thickness reductions of 50 percent were consistently obtained.

In oxidation tests at  $1900^{\circ}$  F, relatively low oxidation rates were obtained, although spalling of the oxide scale was observed upon cooling through the  $800^{\circ}$ to  $600^{\circ}$  F range. The weight gain per unit surface area for the NASA alloy was 2.9 and 6.5 milligrams per square centimeter after 50 and 200 hours exposure, respectively. These are not excessive oxidation rates, particularly for short-time applications; however, spalling may pose a problem in applications where frequent temperature cycling occurs.

#### INTRODUCTION

The demand for higher performance turbines in aircraft engines and in the turbopump components of rocket engines, as well as the continued need for improved high-temperature metals in many structural applications, has intensified research efforts devoted to alloy development. Research with nickel-base alloys affords considerable promise for achieving materials with improved, elevated-temperature properties for the applications cited. As a class, these alloys have excellent strength up to approximately 1800° F and have generally good oxidation and impact resistance.

A continuing program exists at the Lewis Research Center to provide alloys with improved high-temperature strength and ductility that also have satisfactory oxidation resistance. To date, a series of nickel-base alloys has been developed as a result of this program; this series is fully described in references 1 to 4. In summary, the nominal composition of the basic NASA alloy was (in weight percent) 8 molybdenum, 6 chromium, 6 aluminum, 1 zirconium, and the balance nickel. The basic alloy was essentially a cast material and was generally limited to a use temperature of 1800° F for reasonably long-time, high-stress applications.

Modifying the composition of the basic alloy resulted in substantial increases in use temperature, high-temperature strength, and ductility. One of the first significant improvements in high-temperature strength was obtained by means of a titanium and carbon modification of the basic alloy (ref. 1). This alloy also performed quite well as a turbine-bucket material (refs. 2 and 5) in a turbojet engine at a temperature of  $1650^{\circ}$  F. Additional increases in hightemperature strength and impact resistance were obtained with a tungsten, vanadium, and carbon modification of the basic alloy (ref. 3). The improved impact resistance over the earlier alloys in this series, as well as many commercial cast nickel-base alloys, was encouraging because it indicated increased ductility. Finally, making tantalum alloying additions to the tungsten, vanadium, carbon modification of the basic alloy, resulted in substantial increases in both high-temperature strength and ductility (ref. 4). The use temperature of this alloy for long-time, high-stress applications was  $1900^{\circ}$  F, and its ductility was such as to permit limited cold forging of 3/4-inch as-cast bars without cracking.

Other investigators have developed cast nickel-base alloys with good hightemperature stress-rupture properties. Among the strongest and most common of these are Nicrotung and Inconel 713C. More recent developments include IN 100 and TRW 1800 (ref. 6), which compare closely with the tantalum-modified NASA alloy in high-temperature strength. All these alloys have a considerable potential for aerospace applications, in which castings can be used, particularly spacevehicle structural members and turbojet-engine buckets. At the same time, nickel-base alloys with adequate workability are also much in demand for other aerospace applications, such as outer panels of reentry vehicles. For example, the Air Force has recently sponsored a program to produce a nickel-base alloy sheet with optimum high-temperature properties (refs. 7 to 10). The tantalum modified alloy of this report was included among the nickel-base alloys studied in the Air Force sponsored investigation. Thus far, cast sheets of this alloy approximately 0.100 inch thick have been rolled to thicknesses of 0.015 inch (ref. 9).

On the basis of earlier work done at Lewis (ref. 4) and by others (refs. 7 to 10), it appears that the tantalum modified NASA alloy has considerable potential for aerospace applications that require workability as well as high-temperature strength. The present investigation was conducted to determine more fully the properties of this alloy in order to provide an additional basis for evaluating its potential for various aerospace applications. The alloy was investigated to (1) extend as-cast short-time and long-time strength data to higher temperature levels, (2) obtain as-cast impact resistance data, (3) determine as-wrought tensile properties, and (4) obtain elevated-temperature oxidation-resistance data. Both tensile and stress-rupture data were obtained up to  $2100^{\circ}$  F. Oxidation tests were run at  $1900^{\circ}$  F. Wrought tensile test bars were

made by forging. As in the previous investigations, melts were made by highfrequency induction heating under an argon blanket, and investment casting techniques were employed.

#### MATERIAL, APPARATUS, AND PROCEDURE

#### Alloy Investigated

The alloy studied in this investigation had a nominal composition (in weight percent) of 8 tantalum, 6 chromium, 6 aluminum, 4 molybdenum, 4 tungsten, 2.5 vanadium, 1 zirconium, 0.125 carbon, and the balance nickel. In the nomenclature of reference 3, this alloy is designated herein as Mo-4-W + 8Ta + 2.5V + 0.125C.

Table I lists the nominal composition of this alloy, as well as chemical analyses of typical heats. The analyses were conducted by an independent laboratory. Considerable variation in zirconium content occurred. As in earlier investigations (refs. 1 to 4), great care was exercised in maintaining quality control of castings in order to obtain the desired 1 percent of zirconium. Unavoidable variations in melting time (average exposure time, 20 min), however, that would affect the amount of zirconium picked up from the crucible occurred occasionally. Stabilized zirconia crucibles, from the same supplier as those used in the previously reported investigations, were used.

The alloying constituents were commercially pure materials with the following compositions: nickel, 99.95<sup>+</sup>; electrolytic chromium, 99.5<sup>+</sup>; molybdenum, 99.0<sup>+</sup>; llOO aluminum, 99.0<sup>+</sup>; vanadium, 99.8<sup>+</sup>; tungsten, 99.9<sup>+</sup>; tantalum, 99.8<sup>+</sup>; and carbon, 99.5<sup>+</sup>.

#### Casting Techniques

The casting techniques employed were similar to those described in detail in references 1 and 3. A brief description of the major aspects of the casting procedure is given in the following paragraphs. A 50-kilowatt, 10,000-cycle per-second, water-cooled induction unit was used for melting. All melts were made in stabilized zirconia crucibles under an inert gas (commercially pure argon) blanket. The average exposure time between melt and crucible was 20 minutes. This time was determined from a series of experimental melts and is a function of a variety of factors that include melt size, melting constituents, crucible material, and induction unit characteristics. Vanadium and carbon additives were made in the form of powders or granules that previously had been compressed inside aluminum-foil containers. Other element additives were in the form of roundelles, rods, platelets, or chips. The order of addition of alloying metals was similar to that described in reference 3 and is as follows: carbon, vanadium, and nickel were first melted together; then tungsten, tantalum, chromium, and molybdenum were added, followed by aluminum just prior to pouring. Pouring temperature, determined by optical pyrometer measurements on all heats and checked by an immersion thermocouple in randomly chosen heats, was 3150°±50° F. All melts were top poured into 1600° F investment molds without the inert-gas-blanket coverage maintained during melting and were permitted to cool slowly (overnight) to room temperature before the investment was removed.

The lost-wax process was employed to make molds of the test specimens. Stress-rupture bars (also used for tensile tests) had conical, unthreaded ends to avoid stress concentrations and had a cylindrical test section  $1\frac{5}{16}$  inches long and 1/4 inch in diameter. Charpy impact bars were unnotched and were 0.395 by 0.395 by 2.25 inches. Precision dies were used to make wax patterns of the test specimens. The impact bar wax patterns were made slightly oversize to permit finish machining of the cast product to exact dimensions. Wax patterns of cylindrical rods nominally 1/2 inch in diameter and 4 to 6 inches long were used to provide forging samples. A silica slurry with a commercial binder was used as the investment material.

#### Forging Procedure

The forge bars were as-cast rounds 0.55 inch in diameter by  $3\frac{1}{2}$  inches in length. Forging was done at room temperature in a 4500-pound drop forge. Hardened steel dies were attached to the base and to the movable head of the forge. The forge stroke distance was maintained at approximately  $2\frac{1}{2}$  inches, and the applied force was increased progressively during the forging process. The bars were heat-treated during the reduction process after every 0.060-inch reduction in thickness except after the final reduction. Heat treatment consisted of heating the bars to  $2150^{\circ}$  F in a resistance-wound tube furnace, air-cooling to  $1650^{\circ}$  F, and then quenching in water. Several heat treatments were tried during preliminary forging attempts, and this one proved to be most satisfactory for minimizing cracks. After each heat treatment, the bars were visually inspected. If cracks occurred, they were removed by grinding before continuing the forging process.

#### Inspection of Specimens

All cast-test specimens including bars that were used for forging were vapor-blasted prior to inspection. Stress-rupture and tensile specimens, as well as forge bars, were subjected both to radiographic and zyglo inspection. The impact bar blanks were radiographed, machined, and zyglo inspected after machining.

#### Alloy Property Determinations

Tensile and stress-rupture tests. - Tensile tests were made in both the ascast and as-forged (50-percent reduction in bar thickness) conditions over a range of temperatures up to 2100° F. Table II summarizes the tensile-test data. At least two tests were made for each alloy condition (as-cast or as-forged) at each test temperature to determine tensile properties. Drawings of the test specimens used to obtain tensile data with the as-cast and as-forged alloys are shown in figure 1. In conducting the tensile tests, the loading rate for both types of specimen was maintained at approximately 24,000 psi per minute in the

elastic portion of the test. This rate corresponds approximately to a load application of 1200 pounds per minute for the as-cast specimens and 500 pounds per minute for the as-forged specimens.

Stress-rupture tests were made with the as-cast alloy at 15,000 psi up to 2100° F. Table III summarizes all the stress-rupture test data. A minimum of three and a maximum of five runs were made at each test temperature to determine stress-rupture properties.

Impact tests. - Impact resistance data were obtained with the cast 8-percenttantalum-modified alloy (Mo-4-W + 8Ta + 2.5V + 0.125C), an earlier cast NASA alloy (Mo-4-W + 2.5V + 0.125C), and two representative commercial nickel-base alloys, Nicrotung (cast) and René 41 (wrought). Both Izod and Charpy impact tests were made. Unnotched impact bars were used in all tests. Izod tests were made with a Bell Telephone Laboratory Impact Tester, which is fully described in reference 11. Test specimens that measured 3/16 by 3/16 by  $1\frac{1}{2}$  inches were mounted in a vertical position and gripped at one end by a vise. The test bars were inserted in the vise to a depth of 1/2 inch, and the point of impact of the striking pendulum was 1/8 inch from the free end of the bar. Total capacity of the unit was 62.5 inch-pounds. Charpy impact tests were made on a standard Charpy impact tester. Impact specimens, which measured 0.395 by 0.395 by 2.25 inches, were inserted in the tester in a horizontal position and were supported at both ends. The point of impact of the striking pendulum was the center of the bar. The tester had a total capacity of 220 foot-pounds.

Hardness determinations. - Hardness data were obtained with a Rockwell hardness tester for the 8-percent-tantalum-modified alloy in the as-cast and asforged conditions. Samples used for hardness tests were 1/4-inch slabs cut from as-cast and as-forged forging bars.

Oxidation tests. - A large (approximately 20 cu ft) resistance-wound hearthtype furnace was used for the oxidation tests. Oxidation specimens, which were ground cylinders 0.225 inch in diameter by 0.875 inch in length, were suspended from aluminum oxide rods inserted in an Inconel support placed in the furnace. The specimens were suspended from the ceramic rods by means of platinum wires spot-welded to the specimen ends. Furnace air temperature was maintained at 1900° F. A recording potentiometer was used to monitor furnace temperature continuously for the duration of the test. Alloys Mo-4-W + 8Ta + 2.5V + 0.125C, Nicrotung, and René 41 were tested simultaneously. The Nicrotung and René 41 were obtained from commercial suppliers. Specimens were weighed prior to insertion into the furnace both with and without the platinum wire attached. One specimen of each alloy was removed from the furnace after 50, 100, and 200 hours. In order to eliminate the possibility of loss of spalled material, the specimens were immediately put into separate glass containers upon removal from the furnace. After cooling, each specimen was weighed together with any spalled material to determine total weight gain.

<u>Metallographic studies</u>. - Metallographic studies were made of the alloy in both the as-cast and as-forged conditions and after oxidation tests. Macrophotographs (magnification, 3) as well as photomicrographs at magnifications of 250 and 750 are presented.

#### RESULTS AND DISCUSSION

#### Tensile Data

Tensile-test data are summarized in table II. Ultimate tensile strength and percent elongation were obtained for alloy Mo-4-W + 8Ta + 2.5V + 0.125C at room temperature, 1800°, 1900°, 2000°, and 2100° F in both the as-cast and the asforged (50-percent reduction in thickness) conditions. Figure 2 shows curves of the average ultimate tensile strength and percent elongation as a function of temperature for the as-cast alloy and the as-forged alloy. The trend pattern for the as-forged-alloy curves is almost the same as that for the as-cast-alloy curves. Room-temperature ultimate tensile strength was increased from 135,000 to 158,000 psi by forging. At the higher temperatures, the as-forged alloy demonstrated somewhat lower ultimate strength values than the as-cast alloy. The asforged alloy strengths were still quite high at these temperatures, ranging from approximately 59,000 psi at 1800° to 18,000 psi at 2100° F, as compared with 80,000 and 34,000 psi for the as-cast alloy. A general trend of increasing percentage elongation with increasing temperature was obtained. Average elongations ranging from approximately 5 percent at room temperature to 17 percent at 2100° F, were obtained with the as-cast alloy. Values ranging from 2 to 8 percent were obtained with the as-forged alloy at these same temperatures. Some necking of the test specimens was noted; however, this was not pronounced and did not account for the major part of the elongations observed.

The investigators of references 7 to 10 report lower ultimate tensile strengths in the as-cast condition for the NASA 8-percent-tantalum-modified alloy than those reported herein at room temperature, 2000° and 2100° F. In general, lower elongations accompanied these lower ultimate strength values. Their data and that reported herein, however, coincide quite well at 1900° F. At this temperature, an average ultimate tensile strength of 54,900 psi was reported. Because of specific requirements, a different specimen configuration and a modified melting practice were employed by these investigators (refs. 7 to 10). These differences may account for the differences in properties. In this regard, it is also interesting to note that a vacuum melt of this alloy has been made by another vendor. Unpublished data obtained from limited tensile and stress-rupture tests with samples from this melt agreed well with data reported herein.

Figure 3 presents bar chart comparisons of the average ultimate tensile strength of alloy Mo-4-W + 8Ta + 2.5V + 0.125C with an earlier NASA alloy in this series (ref. 3) and with several of the strongest, most recent high-temperature nickel-base superalloys. Comparisons are made at both room temperature and 1800° F (figs. 3(a) and (b)). Commercial alloy data were obtained from reference 12 and commercial data folders. At room temperature, the ultimate tensile strength of the 8-percent-tantalum-modified alloy in the forged condition is approximately 25 percent less than that of René 41 and Udimet 700. In the as-cast condition, its ultimate tensile strength is slightly less than that of IN 100 and TRW 1800 (135,000 psi compared with 143,000 and 140,000 psi, respectively). At 1800° F, however, higher ultimate strengths were obtained with the cast 8percent-tantalum-modified alloy than with all the other cast alloys. These differences were not too pronounced, 80,000 compared with 77,500 and 74,000 psi for IN 100 and TRW 1800, respectively. In the as-forged condition, greater differences in ultimate strength were observed between the 8-percent-tantalum-modified

alloy and the fully wrought commercial alloys. The comparative values were 58,500 psi for the NASA alloy compared with 50,000 and 40,000 psi for Udimet 700 and René 41, respectively.

#### Stress-Rupture Data

The results of all stress-rupture tests are given in table III. The 15,000-psi rupture properties are shown in figure 4 for a temperature range from  $1800^{\circ}$  to  $2100^{\circ}$  F. A least-square line was drawn through the data. The plot indicates that 1000-, 100-, and 10-hour rupture lives can be obtained at  $1817^{\circ}$ ,  $1915^{\circ}$ , and  $2015^{\circ}$  F, respectively. It should be noted that all the data shown were obtained with as-cast samples. Additional increases in rupture life may possibly be obtained by suitable heat treatments. Further investigation to determine a beneficial heat treatment for a specified test condition would appear warranted for those interested in pursuing the development of these alloys, particularly in view of the favorable results obtained with certain heat treatments employed with the strongest titanium and carbon modification of the basic alloy (ref. 1).

Figure 5 indicates the maximum temperature for 100-hour life at 15,000 psi with alloy Mo-4-W + 8Ta + 2.5V + 0.125C and the same representative nickel-base alloys considered in the earlier tensile-strength comparison. Commercial alloy data were obtained from references 12 and 13. A 1915<sup>O</sup> F use temperature is indicated for alloy Mo-4-W + 8Ta + 2.5V + 0.125C in the as-cast condition as compared with 1900<sup>O</sup> for an earlier NASA alloy Mo-4-W + 2.5V + 0.125C and for IN 100. Maximum use temperatures for the other alloys under these stress conditions range from approximately 1880<sup>O</sup> F for TRW 1800 and Nicrotung to 1740<sup>O</sup> F for René 41. The limited number of specimens available prevented obtaining data in the wrought condition with the tantalum-modified NASA alloy.

Maximum use temperature for long-time service under high-stress load is extremely important in many applications. This is particularly true in certain aerospace applications such as structural members of reentry vehicles and turbine buckets in turbine aircraft engines. In such applications, each permissible increase in operating temperature can be reflected in increased vehicle performance. The NASA alloy, with its high use temperature of approximately 1915° F for 100-hour life at 15,000 psi stress, affords a particularly interesting potential for such applications.

Figure 5 is based on rather limited data for the NASA alloys, as compared with what may be a large amount of data for the commercial alloys. Also, the NASA alloys were cast in small experimental heats; if they are to be used in production, a casting procedure suitable for production practice should be evolved. There is some indication that satisfactory production melting practices can be evolved since, as previously indicated, larger heats of the alloy have been cast satisfactorily in commercial practice.

#### Impact Resistance

Unnotched, room-temperature, impact-resistance data are summarized in ta-

ble IV. The 8-percent-tantalum-modified alloy in the as-cast condition is compared with an earlier NASA alloy in this series as well as with Nicrotung and René 41 (annealed condition). In the Izod tests, impact resistances above 62.5 inch-pounds were obtained with all the alloys tested. None of the specimens was broken; all had an impact resistance above the tester capacity. In the Charpy tests with alloy Mo-4-W + 8Ta + 2.5V + 0.125C, an average impact resistance of 33 foot-pounds was obtained. This value compares quite favorably with that obtained for the cast alloy Nicrotung and is considerably lower than the impact resistance of René 41. René 41 could not be broken in the Charpy tester, and its excellent impact resistance might be expected from a completely wrought product in the annealed condition.

Although it is impossible to make a broad generalization regarding the adequacy of the 8-percent-tantalum-modified NASA alloy for various aerospace applications. the results of these impact tests indicate that its impact resistance is probably adequate for applications where impact resistance is a critical factor, such as turbine-bucket applications. Earlier Lewis investigations with turbine buckets of various superalloys (refs. 14 and 15) indicated that satisfactory engine operation was possible with buckets made of alloys having a much lower impact resistance than the 8-percent-tantalum-modified alloy. In reference 14 it is shown that Guy alloy buckets demonstrated satisfactory impact resistance in engine tests, and, in reference 15, S-816 + B showed similar results. The average Izod impact resistances of both these alloys at room temperature (as determined in the manner described herein) were only 11.6 and 6.6 inch-pounds for Guy alloy and S-816 + B, respectively (ref. 15). Of course, in order to evaluate this or any other alloy fully with respect to impact resistance for specific. long-time, high-temperature applications, impact tests should also be made after long-time exposure to high temperature. In this way the embrittling effect (if any) of phases that may be precipitated during aging can be determined.

#### Hardness

Hardness data are summarized in table V. Rockwell C hardness values were converted to the nearest whole number from the experimentally obtained Rockwell A values by using a standard conversion table. Listed hardness values represent the average of approximately five readings for each alloy specimen. A considerably higher as-cast hardness (Rockwell C, 41) was obtained with the 8-percenttantalum-modified alloy than with alloy Mo-4-W + 2.5V + 0.125C. An average Rockwell C hardness of 35 was obtained with this alloy. A hardness increase might be expected because of the higher strengths obtained with the tantalum-modified alloy. After 1/2-inch rounds of the tantalum-modified alloy were so forged as to achieve 50-percent-thickness reductions, average hardness was increased to C-46.

#### Workability

Additional evidence of workability was obtained with the 8-percent-tantalummodified alloy by means of unidirectional forging techniques. In initial attempts at forging this alloy (ref. 4), techniques similar to those described herein were utilized, and the maximum thickness reduction obtained with as-cast rounds was 29 percent. In the present investigation, 1/2-inch as-cast rounds were consistently flattened to achieve thickness reductions of 50 percent without significant cracking. Figure 6 illustrates a typical bar before and after forging. The microconstituents were elongated by the forging process. This is evident from microphotographs that are presented in the Metallographic Studies section of this report. Added strength was imparted to the alloy by forging, as indicated by the substantial increase in room-temperature tensile strength. Although the elevated temperature tensile strengths of the as-forged alloy were lower than those of the as-cast alloy, choice of working temperature and/or subsequent heat treatments could conceivably reduce this difference.

Others have investigated the workability of this alloy (refs. 7 to 10) by utilizing rolling techniques. Substantial reductions in cast sheet thickness were obtained by rolling. Reference 9 indicates that cast sheets 0.100 inch thick have been rolled to thicknesses of 0.015 inch. It would appear from the results described herein, as well as those obtained by others, that this alloy has considerable workability potential. As a consequence, its potential usefulness for aerospace applications that require formed shapes is considerably enhanced.

#### Oxidation Resistance

The results of oxidation tests at 1900° F with alloy Mo-4-W + 8Ta + 2.5V + 0.125C are shown in figure 7. Total weight gain per unit surface area is plotted as a function of time. Data for Nichrome, obtained from reference 16, are included to provide a reference basis. Shown also for comparison are data that were obtained simultaneously with two representative high-temperature commercial nickel-base alloys, Nicrotung and René 41. The latter alloy is generally used at temperatures lower than 1900° F. This is a desirable use temperature for Nicrotung and the NASA alloy, however, because of their high strength at 1900° F. Oxidation specimens of the commercial alloys were obtained from material supplied by vendors. As shown in figure 7, the NASA alloy compares rather closely in oxidation rate with that of Nicrotung and René 41 up to 50 hours of exposure. After 50 hours, a weight gain of 2.9 milligrams per square centimeter was obtained with the NASA alloy as compared with 2.7 and 2.4 milligrams per square centimeter for Nicrotung and René 41, respectively. As exposure time was increased beyond 50 hours, the difference in weight gain became more marked. After 200 hours, weight gains of 6.5, 12.6, and 3.5 milligrams per square centimeter were obtained with the NASA alloy, Nicrotung, and René 41, respectively. It should be noted that the Nicrotung oxidation specimens exhibited some porosity, which was not present in the other alloys. This may have adversely influenced the results obtained with Nicrotung because of the potentially greater surface area exposed.

It is interesting to note that, although the NASA alloy contains a relatively low percent of chromium as compared with other nickel-base alloys (table I), its oxidation rate based on weight-gain tests was not particularly excessive. It was assumed in the design of this alloy that the relatively high percent of aluminum used in conjunction with the chromium present would contribute substantially toward providing adequate oxidation resistance as well as superior strength. The effectiveness of aluminum in achieving improved oxidation resistance with iron-base alloys is shown in the literature (ref. 17). Also,

although the NASA alloy contains alloying constituents, such as vanadium, that oxidize catastrophically at temperatures near 2000° F, the oxidation rate for this alloy was far from catastrophic. In fact, its relatively low oxidation rate up to 50 hours is significant when considered in the light of aerospace applications involving short exposure times. For such uses as surface panels or structural members of missiles or reentry vehicles in which high-temperature atmospheric exposure is of short duration, the oxidation resistance of this alloy, based upon these data, would appear to be adequate. Additional investigations are required to determine the effect of possible volatile oxides and the adequacy of the alloy in applications where temperature cycling occurs. These studies are indicated since spalling of the oxide scale occurred in the 800° to 600° F range upon cooling from the oxidation test temperature. In a turbojet engine, turbineinlet temperature is varied frequently during the course of engine operation. The degree of deterioration that turbine buckets of this alloy might experience as a result of spalling under cyclic operation cannot be conclusively determined, however, without conducting cyclic temperature oxidation tests or actual engine tests.

#### Metallographic Studies

Macrophotographs of alloy Mo-4-W + 8Ta + 2.5V + 0.125C in the as-cast and the as-forged (50 percent reduced) conditions are shown in figure 8. The dendritic structure in a typical as-cast bar is evident in figure 8(a). The extent to which this structure has been affected by forging is shown in figure 8(b). Deformation of the dendritic pattern was achieved although its presence is still readily apparent. Grain boundaries are also evident in the as-cast and as-forged samples.

Photomicrographs of the alloy at ×250 and ×750 in the as-cast and as-forged conditions are shown in figure 9. The as-cast alloy contains minor constituents randomly dispersed throughout the matrix that are probably of the gamma prime intermetallic compound type. Electron micrograph studies indicate that the larger massive particles (white phase) were also of this type. What may be a eutectic formation occurs adjacent to some of these large white phases. A few carbides are scattered throughout the matrix. These were evidenced by luster and by microhardness indentations. Electron-diffraction and electron-probe studies have indicated the presence of tantalum carbides, as well as the gamma-prime-type intermetallic compound phase, which, in this alloy, consists primarily of nickel, aluminum, and tantalum. Figure 9(b) shows the changes in microstructure brought about by forging. The microconstituents have evidently been elongated by the working process. The large white phase particles appear to have agglomerated and become realined. There is some evidence of cracking of the brittle carbides.

The microstructures of alloy Mo-4-W + 8Ta + 2.5V + 0.125C, Nicrotung, and René 41, after a 200-hour oxidation test at 1900° F, are shown in figure 10. Each alloy displays an external oxide scale, a depletion zone, and a relatively unaffected zone. The oxidized samples of the NASA alloy and Nicrotung are quite similar. The NASA alloy and Nicrotung (figs. 10(a) and (c)) do not display as continuous and tightly adherent an external scale as René 41 (fig. 10(b)). In the NASA alloy this is probably due to the spalling, which was observed during cooling of the oxidation specimens from the test temperature through the  $800^{\circ}$  to  $600^{\circ}$  F temperature range. The depletion zones, adjacent to the exposed surface of the NASA alloy and Nicrotung, are somewhat narrower than that of René 41. For applications in which thin sections are required, depletion of alloying constituents from a material in the region adjacent to the exposed surface can be detrimental insofar as structural integrity of the part is concerned. The relatively narrow depletion zone observed in the NASA alloy after 200 hours exposure at  $1900^{\circ}$  F indicates that depletion of alloying constituents may not be a severe problem with thin sections of this alloy operated up to a  $1900^{\circ}$  F temperature. In a relatively thick section, such as the 1/4-inch-diameter test section of the stress-rupture specimens used in this investigation, structural integrity would probably not be markedly affected by such depletion. The excellent elevated-temperature stress-rupture properties obtained with this alloy would tend to bear out such an assumption.

#### CONCLUDING REMARKS

On the basis of the evaluations described, the 8-percent-tantalum-modified alloy appears to have considerable potential for various aerospace applications, particularly where good impact resistance, high-temperature strength, at least limited workability, and oxidation resistance upon short-time exposure to a reasonably constant, high-temperature environment are required.

#### SUMMARY OF RESULTS

The following results were obtained from the continued study of an advanced temperature NASA nickel-base alloy having the following nominal composition in weight percents: 8 tantalum, 6 chromium, 6 aluminum, 4 molybdenum, 4 tungsten, 2.5 vanadium, 1 zirconium, 0.125 carbon, and balance nickel.

1. Ultimate tensile strengths of the as-forged alloy, ranging from 158,000 psi at room temperature to 18,000 psi at  $2100^{\circ}$  F, were obtained. These compared to ultimate strength values ranging from 135,000 to 34,000 psi at room temperature and  $2100^{\circ}$  F, respectively, for the as-cast condition.

2. Stress-rupture data for the as-cast alloy indicated that average rupture lives of 1000, 100, and 10 hours can be obtained at 1817°, 1915°, and 2015° F, respectively, under a stress of 15,000 psi.

3. Impact tests provided evidence that this alloy has a considerable degree of ductility. In Izod impact tests with as-cast, unnotched specimens, impact resistances greater than the tester capacity, 62.5 inch-pounds, were obtained. In Charpy impact tests at room temperature, an average impact resistance of 33 foot-pounds was obtained with standard unnotched specimens. This was considerably higher than that of Nicrotung, a typical, strong, as-cast nickel-base alloy, but much less than that of René 41, a completely wrought nickel-base alloy.

4. Additional evidence of workability was obtained with this alloy. Unidirectional forging techniques at room temperature were used to obtain consistent thickness reductions of 50 percent with 1/2-inch-diameter as-cast bars.

5. In oxidation tests at 1900° F, relatively low oxidation rates were ob-

tained, although spalling of the oxide scale was observed upon cooling through the 800° to 600° F range. The weight gain per unit surface area was 2.9 and 6.5 milligrams per square centimeter for the NASA alloy after 50 and 200 hours, respectively. These are not excessive oxidation rates, particularly for shorttime applications; however, spalling may pose a problem in applications where frequent temperature cycling occurs.

Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio, January 11, 1963

#### REFERENCES

- 1. Freche, John C., and Waters, William J.: Exploratory Investigation of Advanced-Temperature Nickel-Base Alloys. NASA MEMO 4-13-59E, 1959.
- Freche, John C., Waters, William J., and Riley, Thomas J.: A New Series of Nickel-Base Alloys for Advanced-Temperature Applications. ASM Trans., vol. 63, 1961, pp. 523-537.
- 3. Freche, John C., Riley, Thomas J., and Waters, William J.: Continued Study of Advanced-Temperature Nickel-Base Alloys to Investigate Vanadium Additives. NASA TN D-260, 1960.
- 4. Freche, John C., Waters, William J., and Riley, Thomas J.: A New Series of Advanced-Temperature Nickel-Base Alloys. Paper presented at High Temperature Materials Conf., AIME, Cleveland (Ohio), Apr. 25-27, 1961.
- 5. Johnston, James R., Gyorgak, Charles A., and Sinclair, John H.: Performance of Several Cast Nickel-Base Alloys as Turbojet-Engine Bucket Materials at 1650° F. NASA TN D-1318, 1962.
- 6. Lund, C. H.: The Use of Nickel-Base Alloys in the Rotating Parts of Gas Turbines for Aerospace Applications. DMIC Memo. 145, Defense Metals Info. Center, Jan. 11, 1962.
- 7. Greenewald, H., Jr., and Biretta, V. J.: First Quarter Report for Development of a Nickel Base Alloy Sheet for High Temperature Application. CVC Rep. AER-ELR-13527, Chance Vought Corp., July 13, 1961.
- 8. Greenewald, H., Jr.: Second Quarter Report for Development of a Nickel Base Alloy Sheet for High Temperature Application. CVC Rep. AER-ElR-13626, Chance Vought Corp., Sept. 15, 1961.
- 9. Greenewald, H., Jr., and Riley, T. J.: Third Quarter Report for Development of a Nickel Base Alloy Sheet for High Temperature Application. CVC Rep. AER-E2R-50012, Chance Vought Corp., Dec. 15, 1961.

- 10. Greenewald, H., Jr., and Riley, T. J.: Fourth Quarter Report for Development of a Nickel Base Alloy Sheet for High Temperature Application. CVC Rep. 2-53430/2R383, Chance Vought Corp., Mar. 15, 1962.
- 11. Probst, H. B., and McHenry, Howard T.: A Study of the "Toss Factor" in the Impact Testing of Cermets by the Izod Pendulum Test. NACA TN 3931, 1957.
- 12. Wagner, H. J.: Recent Developments in Superalloys. DMIC Memo. 64, Defense Metals Info. Center, Sept. 8, 1960.
- Simmons, Ward F., and Metzger, M. C.: Compilation of Chemical Compositions and Rupture Strengths of Super-Strength Alloys. Spec. Tech. Publ. 170-B, ASTM, 1961.
- 14. Signorelli, R. A., Johnston, J. R., and Weeton, J. W.: Preliminary Investigation of Guy Alloy as a Turbojet-Engine Bucket Material for Use at 1650° F. NACA RM E56I19, 1956.
- 15. Waters, William J., Signorelli, Robert A., and Johnston, James R.: Performance of Two Boron-Modified S-816 Alloys in a Turbojet Engine Operated at 1650° F. NASA MEMO 3-3-59E, 1959.
- 16. Barrett, C. A., Evans, E. B., and Baldwin, W. M., Jr.: Thermodynamics and Kinetics of Metals and Alloys - High Temperature Scaling of Ni-Cr, Fe-Cr, Cu-Cr and Cu-Mn Alloys. Final Tech. Rep. Mar. to Nov. 1955, Case Inst. Tech., Dec. 1955.
- Gulbransen, Earl A., and Andrew, Kenneth F.: Oxidation Studies on the Iron-Chromium-Aluminum Heater Alloys. Jour. Electrochem. Soc., vol. 106, no. 4, Apr. 1959, pp. 294-302.

#### TABLE I. - ALLOY COMPOSITIONS

Alloy Ta Cr Al Мо Ni W V Zr СЪ Ti Co В С Mo-4-W + 8Ta + 2.5V + 0.125C68+ 8 6 6 2.51 4 0.125 4 \_\_\_\_ ---\_\_\_\_ Inconel 713C Bal. -13 6 4.5 ----.10 2.25 0.6 -- 0.012 .12 IN 100 Bal. -10 5.5 3 .05 ---- 5 --- 1.0 15 .015 .18 TRW 1800 13 6.0 ---- 9.0 ----Bal. -.07 1.5 .6 --.07 .09 Nicrotung 61+ -12 4.0 ----8.0 ----. 05 ----4.0 10 .05 .10 René 41 Bal. -19 1.5 10 3.1 11 .01 .09 \_ \_\_\_\_

(a) Nominal composition

(b) Composition of typical heats of Mo-4-W + 8Ta + 2.5V + 0.125C as determined by chemical analysis

Ni	Ta	Cr	Al	Мо	W	V	Zr	C
69.06	8.10	5.74	5,18	4.02	3.90	2.42	1.00	0.14
Bal.	7.89	5.75	6.07	3.86	3.90	2.61	.54	.12
Bal.	7.92	5.82	5.92	3.88	3.89	2.48	. 49	.11

### TABLE II. - TENSILE-TEST DATA

Condition	Temper- ature, o <sub>F</sub>	Ultimate tensile strength, psi	Elonga- tion, percent
As cast	Room	133,700 126,400 143,500	1.2 9.5 4.8
	1800	84,500 75,700	7.2 4.8
	1900	54,100 54,400 58,500	11.9 7.2 7.6
	2000	50,900 47,500	7.2 7.2
	2100	29,700 39,000	26.8 6.9
As forged	Room	173,400 165,000 136,600	3.0 3.0 0
	1800	61,000 56,000	1.5 2.0
	1900	49,100 48,700	6.0 6.0
	2000	42,400 37,300	4.0 3.0
	2100	20,600 15,800	6.0 10.0

# TABLE III. - STRESS-RUPTURE DATA

Temper- ature, oF	Life, hr	Temper- ature, OF	Life, hr
1800	1493.8 637.5 1374.0 1338.4	1950	17.6 53.2 26.2 60
1850	540.5 274.2 861.5	2000	21.3 19.7 6.3
1900	237.5 229.5 123.0 137.3 186.1	2100	1.5 2.3 1.9

# AT 15,000 PSI

# TABLE IV. - ROOM TEMPERATURE IMPACT RESISTANCE DATA OF UNNOTCHED SPECIMENS

Alloy	Izod impact resistance, in1b	Charpy impact resistance, ft-1b
Mo-4-W + 2.5V + 0.125C	>62.5, >62.5	40.0, 77.0, 44.5; av. 53.8
Mo-4-W + 8Ta + 2.5V + 0.125C	>62.5, >62.5, >62.5, >62.5, >62.5, >62.5, >62.5	29.5, 35.5, 35.0; av. 33.3
Nicrotung	>62.5, >62.5	11.0, 8.0, 10.0; av. 9.7
aRené 41		>220, >220; av. >220

<sup>a</sup>Annealed.

Alloy	Condition	Average Rockwell hardness <sup>a</sup>	
		A	C,
Mo-4-W + 2.5V + 0.125C	As cast	67.9	35
Mo-4-W + 8Ta + 2.5V + 0.125C	As cast	71.0	41
Mo-4-W + 8Ta + 2.5V + 0.125C	As forged	73.7	46

TABLE V. - HARDNESS DATA

<sup>a</sup>Rockwell A results are an average of at least three tests. Rockwell C values are converted from Rockwell A values.

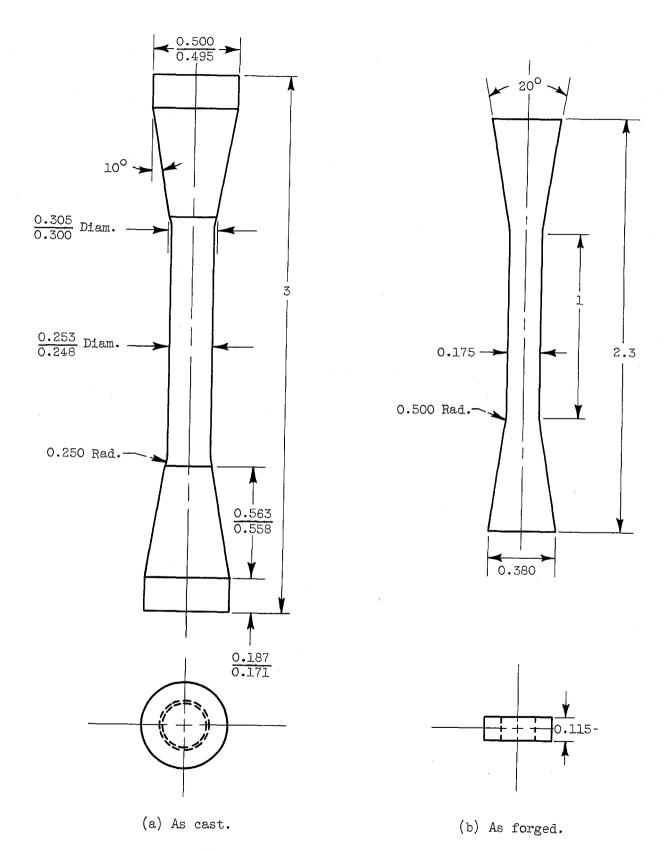
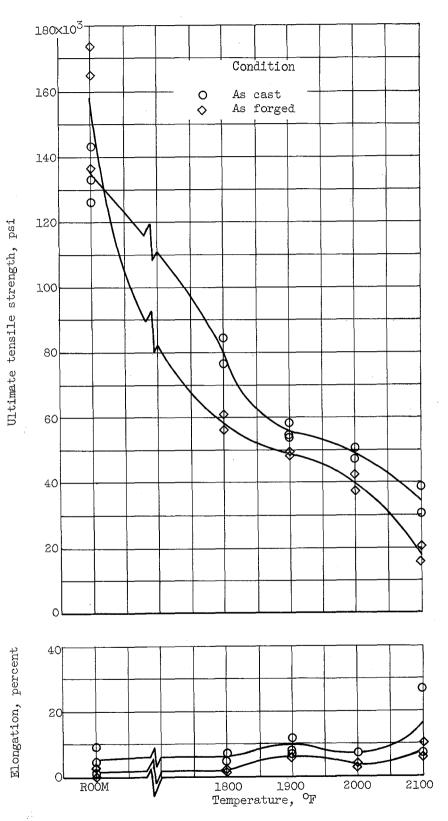
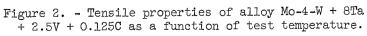
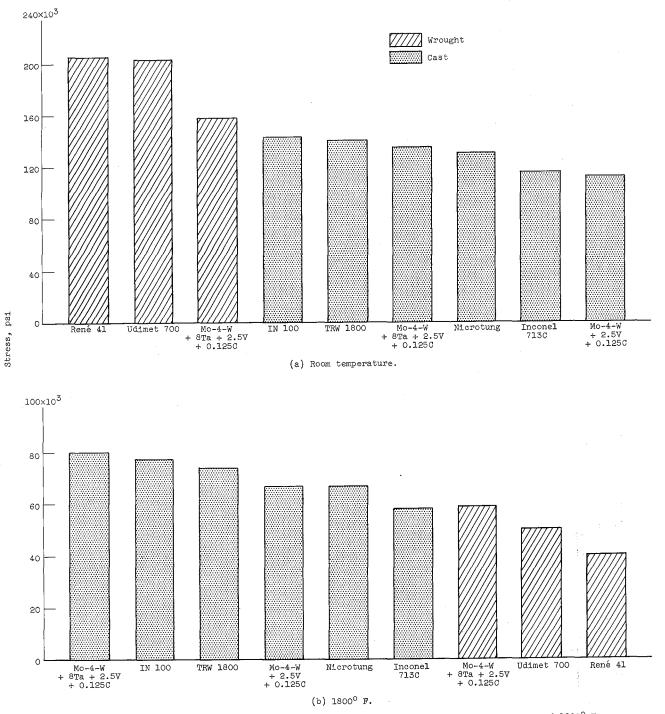


Figure 1. - Tensile-test specimens.







ļ

Figure 3. - Comparison of ultimate tensile strengths of superalloys at room temperature and  $1800^{\circ}$  F.

2100 г  $\mathbf{0}$ 2050 2000 ക o F Temperature, 1950 ο 0 1900 doo ന 1850 θ Θ

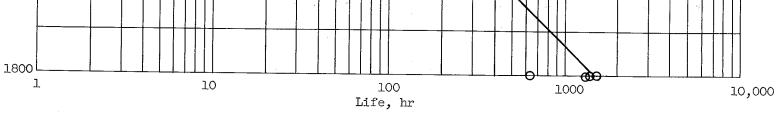
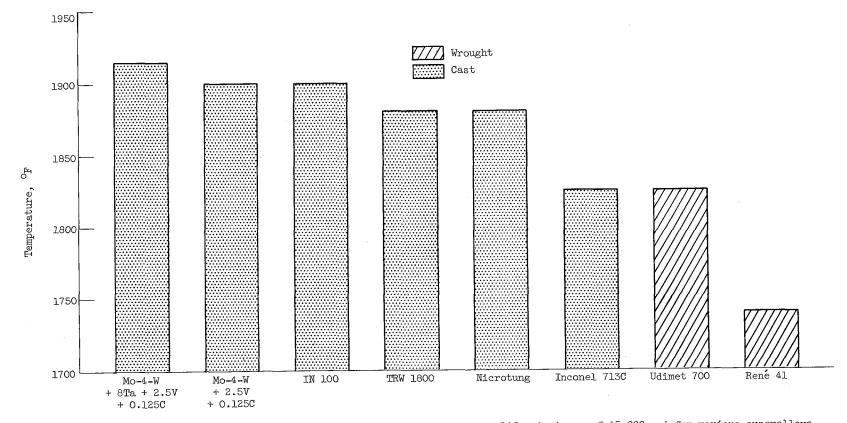
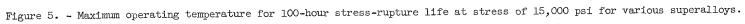
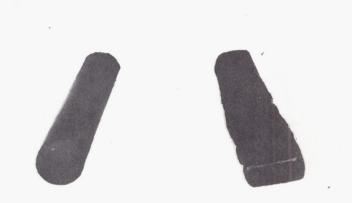


Figure 4. - As-cast stress-rupture life of alloy Mo-4-W + 8Ta + 2.5V + 0.125C at stress of 15,000 psi.



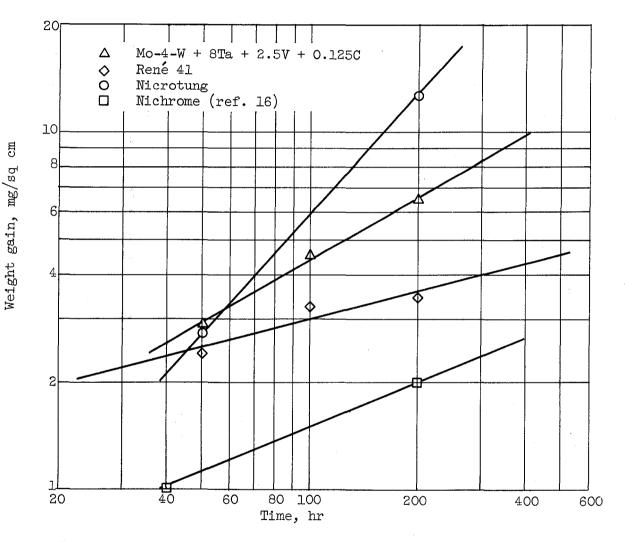


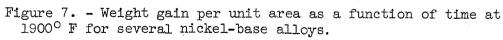


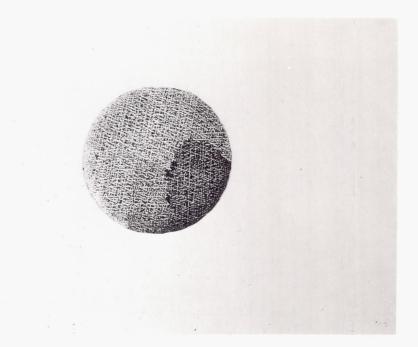
#### l inch

C-63157

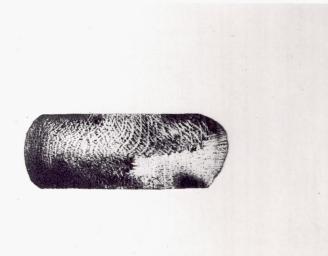
Figure 6. - Typical alloy Mo-4-W + 8Ta + 2.5V + 0.125C bars before and after forging at room temperature. Maximum reduction in diameter shown is 50 percent.







(a) As-cast.

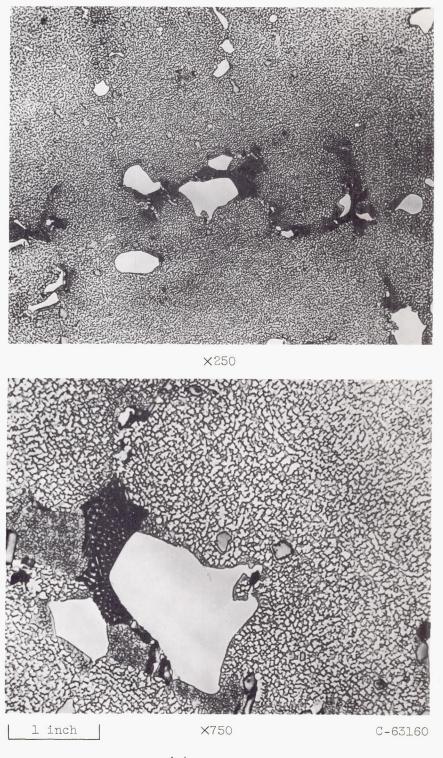


l inch

C-63158

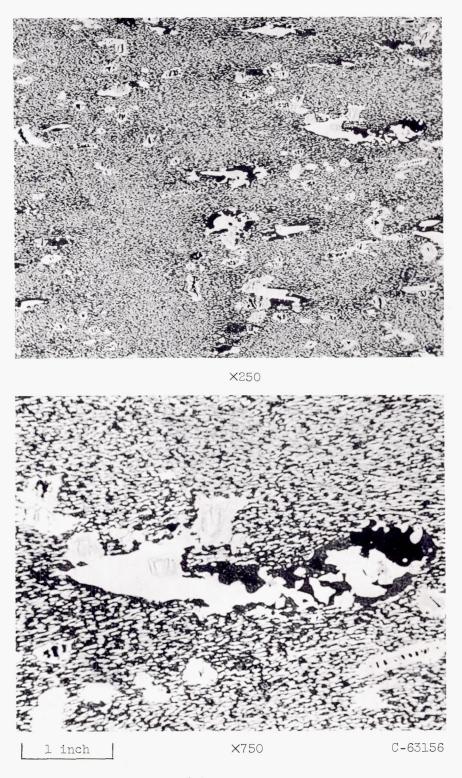
(b) As-forged (50 percent).

Figure 8. - Macrostructure of alloy Mo-4-W + 8Ta + 2.5V + 0.125C in the as-cast and as-forged conditions. X3.



(a) As-cast.

Figure 9. - As-cast and as-forged microstructures of alloy Mo-4-W + 8Ta + 2.5V + 0.125C.



(b) As-forged.

Figure 9. - Concluded. As-cast and as-forged microstructures of alloy Mo-4-W + 8Ta + 2.5V + 0.125C.

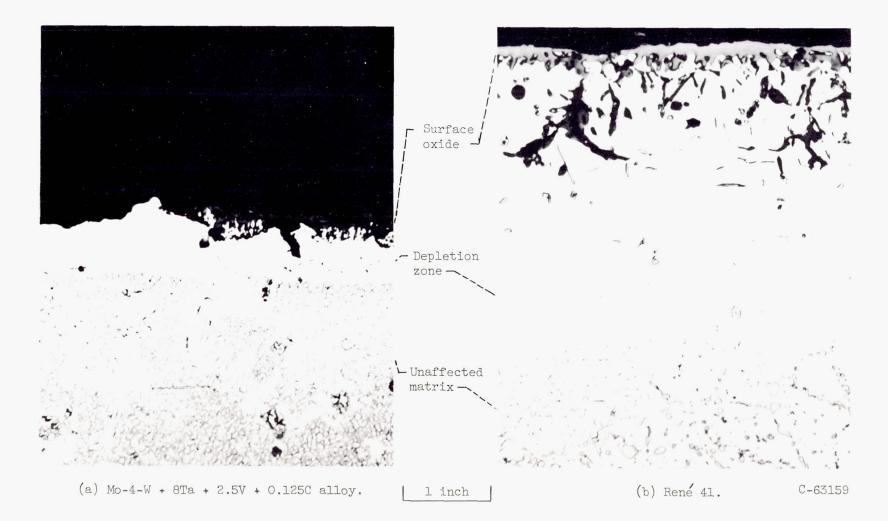


Figure 10. - Microstructure of oxidation test specimens in vicinity of exposed surface after 200-hour exposure at 1900° F. X750.

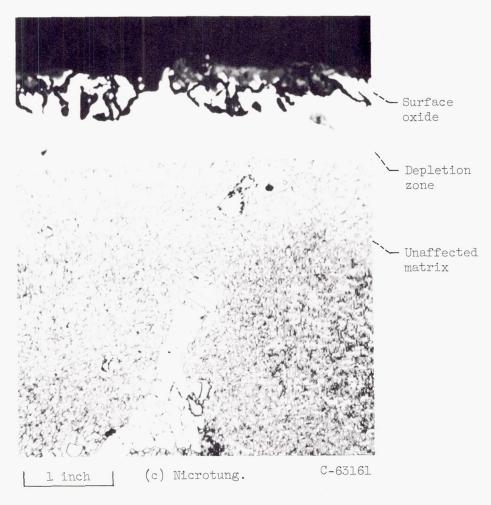


Figure 10. - Concluded. Microstructure of oxidation test specimens in vicinity of exposed surface after 200-hour exposure at 1900° F. X750.