NASA TECHNICAL NOTE

NASA TN D-7966

NASA TN D-7966

MODIFIED FERRITIC IRON ALLOYS WITH IMPROVED HIGH-TEMPERATURE MECHANICAL PROPERTIES AND OXIDATION RESISTANCE

Robert E. Oldrieve

Lewis Research Center
Cleveland, Ohio 44135

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JUNE 1975

1. Report No. NASA TN D-7966	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle MODIFIED FERRITIC IRON ALLOYS WITH IMPROVED HIGH-TEMPERATURE MECHANICAL PROPERTIES AND OXIDATION RESISTANCE		5. Report Date June 1975 6. Performing Organization Code
7. Author(s) Robert E. Oldrieve		8. Performing Organization Report No. E-8185
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		10. Work Unit No. 505-03 11. Contract or Grant No.
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		13. Type of Report and Period Covered Technical Note 14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

An alloy modification program was conducted in which the compositions of two existing Fe-Cr-Al alloys (Armco 18SR and GE-1541) were changed to achieve either improved high-temperature strength or improved fabricability. Only modifications of Armco 18SR were successful in achieving increased strength without loss of fabricability or oxidation resistance. The best modified alloy, designated NASA-18T, had twice the rupture strength of Armco 18SR at 800° and 1000° C. The NASA-18T alloy also had better oxidation resistance than Armco 18SR and comparable fabricability. The nominal composition of NASA-18T is Fe-18Cr-2Al-1Si-1.25Ta. All attempted modifications of the GE-1541 alloy were unsuccessful in terms of achieving better fabricability without sacrificing high-temperature strength and oxidation resistance.

17. Key Words (Suggested by Author(s)) Ferritic-iron alloys; Stainless steels; Oxidation; Stress-to-rupture data; Materials for emission control devices	18. Distribution Statement Unclassified - unlimited STAR Category 26 (rev.)		
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 34	22. Price* $3.75

* For sale by the National Technical Information Service, Springfield, Virginia 22151

CONTENTS

MODIFIED FERRITIC IRON ALLOYS WITH IMPROVED HIGH-TEMPERATURE MECHANICAL PROPERTIES AND OXIDATION RESISTANCE

by Robert E. Oldrieve

Lewis Research Center

SUMMARY

An alloy modification program was conducted in which two existing iron-chromium-aluminum (Fe-Cr-Al) alloys, Armco 18SR and GE-1541, were changed in composition in an attempt to achieve better high-temperature strength or better fabricability without sacrificing oxidation resistance. Armco 18SR was modified by the addition of refractory metal to increase high-temperature strength. Yttrium was deleted from the GE-1541 alloy in an attempt to improve fabricability, and refractory metal was added to maintain high-temperature strength. The alloys were evaluated in sheet form in stress-to-rupture tests at 800° and 1000° C and in cyclic furnace air-oxidation tests at 1150° C.

The best of the modified alloys were those with tantalum added to the Armco 18SR base alloy (nominally Fe-18Cr-2Al-1Si). They were designated NASA-18T and NASA-18T-A and contained 1.25- and 0.45-wt. % tantalum, respectively. The fabricability of both these alloys was comparable to that of Armco 18SR. Compared to the latter alloy, the NASA-18T alloy had about 1.5 times the tensile strength and twice the stress-to-rupture strength at 800° and 1000° C. The NASA-18T-A alloy had about 1.5 times the stress-to-rupture strength of Armco 18SR at 800° and 1000° C. In 1150° C cyclic oxidation tests, both modified alloys exhibited improved oxidation resistance, forming a spall-resistant oxide scale composed mainly of Al_2O_3. In 1040° C automotive exhaust-gas exposure, also, the NASA-18T alloy exhibited improved corrosion resistance.

Additions of molybdenum plus niobium to Armco 18SR were not as effective as tantalum additions in improving strength and oxidation resistance.

All attempted modifications of the GE-1541 alloy were unsuccessful in terms of achieving better fabricability without sacrificing high-temperature strength and oxidation resistance.

INTRODUCTION

This study was conducted to improve the high-temperature strength and/or fabricability of ferritic, Fe-Cr-Al alloys. It is part of an automobile thermal reactor technology program that included development and evaluation of both metallic and ceramic materials (refs. 1 to 3). In this ferritic alloy study, the approach used to achieve the objective was to modify two existing Fe-Cr-Al alloys, Armco 18SR (nominally Fe-18Cr-2Al-1Si) and GE-1541 (nominally Fe-15Cr-4Al-1Y). For the Armco 18SR modifications, emphasis was placed on improving high-temperature strength while retaining good fabricability and oxidation resistance. Modifications were made to the stronger, more oxidation-resistant GE-1541 alloy primarily to improve fabricability so that warm forming would no longer be required to fabricate parts from this alloy (ref. 2). Alloys that can be formed cold are preferred for use in automobile thermal reactors for economy.

The refractory metals tantalum (Ta) or molybdenum (Mo) plus niobium (Nb) were added to both alloys. Tantalum was used because prior studies indicated improved fabricability, ductility, and oxidation resistance when about 1-wt. % Ta was added to an Fe-25Cr-4Al base alloy (ref. 4). The Mo + Nb addition was evaluated as an alternative to Ta. Total refractory metal content was kept below 3 wt. % in an attempt to retain fabricability (ref. 5). Yttrium, which is known to decrease fabricability and promote embrittlement upon long-term exposure of Fe-Cr-Al-Y alloys (refs. 5 and 6), was deleted from all modifications of the GE-1541 alloy. The GE-1541 was selected instead of GE-2541 (nominally Fe-25Cr-4Al-1Y) because the latter alloy is more susceptible to embrittlement upon long-term exposure due to its higher Cr content.

The base alloys and their modifications were prepared in sheets about 1.6 mm thick. Mechanical property evaluations of selected alloys included tensile tests from ambient temperature to 1025° C and stress-to-rupture tests of all alloys at 800° and 1000° C. Cyclic oxidation resistance of the alloys was evaluated at 1150° C in air. Formability was assessed in terms of ease of sheet manufacture and component parts fabrication and in some cases was based on cup drawability and bend testing.

The various alloy modifications that were evaluated and their properties are described in this report. Also included is a discussion of the performance of the best modified alloys in automobile thermal reactor tests, as reported in reference 2.

MATERIALS AND PROCEDURE

Alloys and Compositions Evaluated

The alloys selected as base compositions for this modification program were

Armco 18SR and GE-1541. A total of six modified alloys were made to NASA specifications by the vendors listed in table I. For convenience in discussion, the modified Armco 18SR alloys are referred to as Fe-18Cr-2Al compositions and the modified GE-1541 alloys as Fe-15Cr-4Al compositions, with specific alloys in each series identified by the designations used in table I. The Armco 18SR alloy was modified by two levels of Ta (0.45 and 1.25 wt. %) and by the addition (in wt. %) of 2.0Mo + 0.5Nb to one alloy heat. The GE-1541 alloy was modified by the deletion of yttrium (Y), by the addition of two levels of Ta, and by the addition of 2.0Mo + 0.5Nb + 0.04C to one heat. Carbon (C) was added to the NASA-15M heat to evaluate carbide strengthening in these otherwise low-C type alloys. Additional aluminum (Al) was added to the NASA-15T2 heat to improve oxidation resistance. The alloy designated GE-1540 is a version of GE-1541 which contains no Y (ref. 5).

Melting and Rolling Practice

All modified alloys used in this investigation required only slight modification of existing melting and rolling practices normally used for the respective Fe-Cr-Al base compositions. The GE-1541 alloy and the Ta modifications of GE-1541 were vacuum cast and processed to sheet as described in reference 5. The Armco 18SR alloy and all its modifications, as well as the NASA-15M alloy, were melted and poured under argon by using commercial AISI 400 series stainless-steel-type melting stock. Ingots of all modified alloys were conditioned by surface machining prior to a 50 percent reduction to slab. The slabs were heated and cross rolled to sheet at about 1010° C in 10 percent reductions. After hot rolling, the sheet was descaled, pickled, and cold rolled to approximately 1.6 mm thick and annealed at about 1000° C. Portions of the as-received Armco 18SR alloy and the Ta-modified Armco 18SR alloy were cold rolled to permit determination of mechanical properties of 0.5-mm-thick stock and to investigate alternative annealing treatments (see appendix).

Mechanical Property Testing

Test specimens for rupture and tensile testing were pin-loaded, sheet-metal type with a uniform reduced cross-sectional gage length of 31.8 mm and a width of 9.5 mm. Test specimens were punched from each alloy both parallel and perpendicular to the rolling direction.

Stress-to-rupture testing of all alloys was conducted at 800° and 1000° C in air in conventional lever-arm stress rupture machines. An indication of creep behavior was made by dial-gage measurement of beam deflection, which was verified by interrupting long-term tests (>2000 hr) of selected alloys for measurement of the actual extension of the test specimen.

Tensile tests were made on die-punched specimens taken parallel to the rolling direction for the Armco 18SR and NASA-18T alloys and are compared with available data (refs. 5 and 7). Tensile data were obtained by using a load-cell machine with mechanical crosshead drive at crosshead speeds of 0.0125 cm/min to yield and 0.125 cm/min to failure.

Cyclic Oxidation Tests

Cyclic oxidation testing was conducted on all alloys for as long as 600 hours at 1150° C. Die-punched and ball-milled oxidation coupons (nominally 45 mm by 11 mm in size) were exposed to as many as 600 test cycles. Each test cycle consisted of 1 hour at 1150° C followed by a minimum of 40 minutes static-air cooling to room temperature. Cyclic oxidation was selected as being more representative of service conditions than isothermal testing. Oxidation products that spalled from the specimen during cooling were collected on a tray. Test specimen weight was determined after 10, 20, 40, 60, 80, and 100 cycles and each 50 cycles thereafter. The spall was weighed at completion of test exposure for each test specimen. Further details of the cycles and the test apparatus are given in reference 8. Test specimen failure is defined as excessive weight loss.

Exposed test specimen surfaces and spalled oxides were analyzed by X-ray diffraction, electron dispersive analysis, and emission spectrographic techniques.

Metallurgical Studies

Microstructures of each sheet alloy after fabrication and after oxidation test exposure were evaluated by conventional metallurgical techniques. Sections of exposed alloys were also examined with the scanning electron microscope (SEM) to aid in identifying oxide scales and to observe the oxide-substrate morphology. An electrolytic etchant consisting of 10 parts H_2SO_4 and 90 parts H_2O plus 2 grams CrO_3 was used for all alloys in the unexposed condition. The same etchant was employed after 1150° C cyclic oxidation exposure for all alloys except NASA-15T, GE-1541, NASA-18M, and GE-1540, which required an immersion etchant (33 parts HNO_3, 33 parts CH_3COOH, 1 part HF, and 33 parts H_2O).

RESULTS AND DISCUSSION

Stress-to-Rupture Properties

The rupture strengths of the program alloys, for 400-hour life at 800° and 1000° C, are summarized as bar graphs in figure 1. Stress-to-rupture data are summarized in table II and are presented in figure 2. From these graphs and plots, the alloys are compared and the effectiveness of the alloy additions is evaluated.

Fe-18Cr-2Al alloys. - The strongest Fe-18Cr-2Al alloy was NASA-18T with about twice the rupture strength of Armco 18SR at both 800° and 1000° C (figs. 1(a) and 2(a)). The NASA-18T-A alloy had about 1.5 times the rupture strength of Armco 18SR at both temperatures (figs. 1(a) and 2(b)). In tests of both 0.5- and 1.6-mm-thick sheet, gage thickness had little effect on rupture life (table II). However, the thicker gage material had greater percentage of elongation for equivalent rupture stress or rupture life, as would be expected by consideration of volumetric distortion. That is, a greater thickness and the same gage length provide greater volume to allow greater total elongation of the test section.

The addition of 2Mo + 0.5Nb to Armco 18SR to form NASA-18M provided the least rupture strength improvement of any modified Fe-18Cr-2Al alloy (figs. 1(a) and 2(c)). Thus, Ta is a more effective addition than Mo plus Nb to the Fe-18Cr-2Al alloys.

Fe-15Cr-4Al alloys. - At 1000° C, GE-1541 had the highest rupture strength of all Fe-15Cr-4Al alloys, as well as all Fe-18Cr-2Al alloys, for 400-hour life (fig. 1). The NASA-15T2 alloy was the only modification that closely approached the strength of GE-1541 at 1000° C (figs. 2(e) to (g)). At 800° C, the NASA-15M and NASA-15T2 modifications showed greater rupture strength than GE-1541.

The NASA-15T2 alloy had about twice the rupture strength of NASA-15T at 1000° C. The NASA-15M alloy, with 2Mo + 0.5Nb, had a rupture strength at 1000° C which was between those of the Ta-containing alloys. At 800° C, the NASA-15M alloy had a rupture strength advantage over the other Fe-15Cr-4Al alloys, possibly because of the higher carbon content.

Relative Creep Resistance

A preliminary comparison of relative creep resistance was made on the NASA-18T, Armco 18SR, and GE-1541 alloys by constructing the relative creep extension curves depicted in figure 3. These alloys were subjected to interrupted stress-to-rupture testing in order to measure actual gage-length elongation. From these data and from extension as measured by dial gage, notably high creep resistance at low stresses was indicated for NASA-18T and GE-1541.

At 800° C and at stress levels of 10 MN/m^2 and higher, the NASA-18T alloy had considerably greater resistance to creep extension than GE-1541. The total elongation of NASA-18T was only 0.9 percent after 2480 hours, for an average creep rate of 0.0003 percent/hr.

At 1000° C, the total elongation of GE-1541 was 1.7 percent after 3000 hours at 3.5 MN/m^2, for an average creep rate of only 0.0006 percent/hr. For 500-hour life at these same test conditions, the NASA-18T alloy had a higher average creep rate of 0.08 percent/hr. For all test conditions, Armco 18SR exhibited lower resistance to creep extension. For the application intended, the NASA-18T and GE-1541 alloys appear to have adequate creep resistance.

Tensile Properties

The results of ambient- and elevated-temperature tensile testing are shown in figures 4(a) and (b) and are tabulated in tables III and IV. For 1.6-mm-thick sheet stock and at all temperatures from about 500° to 1000° C, the NASA-18T alloy had about 1.5 times the ultimate tensile strength (UTS) and about 1.5 times the yield strength at 0.2 percent offset (YS) of Armco 18SR. The Armco 18SR data obtained in this study correspond closely with those obtained by Armco and Chrysler research laboratories (ref. 7). The NASA-18T alloy also had 1.5 times the UTS and YS of GE-1541 to 850° C, the maximum temperature to which GE-1541 was tensile tested. At 1025° C, NASA-18T had 29.6 MN/m^2 UTS and 19.3 MN/m^2 YS.

The results of room-temperature tensile testing of as-received Armco 18SR, NASA-18T, NASA-18T-A, and GE-1541 alloy sheet are given in table IV. The addition of 0.45Ta (NASA-18T-A) provided no appreciable change in ultimate strength, yield strength, elongation, or hardness, as compared with Armco 18SR. For the 1.25Ta addition (NASA-18T alloy), the UTS was increased about 10 percent and the YS was unchanged as compared with Armco 18SR. The NASA-18T alloy had 30 percent greater UTS and 36 percent greater YS than GE-1541 at room temperature.

Cyclic Oxidation Resistance

Test coupon weight change of the program alloys in 1150° C cyclic oxidation is presented in figure 5 for Fe-18Cr-2Al alloys and in figure 6 for the Fe-15Cr-4Al alloys. Table V summarizes weight change and protective oxide composition after 200 and 600 hours of furnace test exposure. Photomicrographs of exposed alloys are presented in figures 7 to 9. From these data, it is seen that Ta improves oxidation resistance of these Fe-Cr-Al alloys as compared with the alloys which contained no Ta or Y.

Fe-18Cr-2Al alloys. - The best alloys overall were NASA-18T and NASA-18T-A. Both alloys had an Al_2O_3 protective oxide scale. From a electron dispersive analysis (EDAX), the oxide on the NASA-18T alloy was seen to contain primarily aluminum (Al) and oxides of Fe, Cr, silicon (Si), and titanium (Ti). A spot analysis (about 100 μm in diam) of the small light-grey particles within the oxide layer indicated that they contained primarily Ti with some Al (fig. 7(d)). The oxide scale on Armco 18SR was a mixed oxide containing primarily Al_2O_3 and oxides of Fe, Cr, Si, and Ti (fig. 7(c)).

Photomicrographs of specimens after 600 hours of cyclic furnace exposure reveal that oxide penetration into the substrate was greater for Armco 18SR than for NASA-18T (figs. 7(a) and (b)). The oxide penetration into Armco 18SR was about 50 μm; NASA-18T showed no evidence of oxide penetration into the substrate and no spallation.

The NASA-18T-A alloy was only slightly less oxidation resistant than NASA-18T (table V) and was reasonably free from oxidation damage (fig. 8(a)). The oxide on NASA-18T-A was identical in constituents to the oxide on NASA-18T and was protective.

The NASA-18M alloy test coupon, however, was heavily oxidized (30-mg/cm^2 weight loss, table V) after 450 hours at 1150° C. From figure 8(b), extensive internal oxidation and some void formation is evident. The surface oxide was nonprotective and consisted of Cr_2O_3 and spinel (table V).

It is concluded that all Fe-18Cr-2Al alloys evaluated, with the exception of NASA-18M, could be considered for oxidation service at temperatures to 1150° C. The NASA-18T and NASA-18T-A alloys had less than 2.5-mg/cm^2 weight change and were exceptionally oxidation resistant, with no evidence of spall or penetrating oxidation attack after 600 hours of cyclic exposure in air at 1150° C. These alloys retained a fully protective Al_2O_3 scale. It was indicated that Ta additions were at least less detrimental to oxidation resistance than the addition of 2Mo + 0.5Nb and that Ta was probably beneficial in improving oxidation resistance. The role of silicon in providing oxidation resistance was not evaluated.

Fe-15Cr-4Al alloys. - None of the alloy modifications in this series were better than GE-1541 in oxidation resistance. GE-1541 had the least oxidation weight change (table V) and, except for depletion of a second phase (FeY_9) for the depth of two grains (75 μm) below the surface, showed no evidence of internal damage (fig. 9(a)). The GE-1541 alloy had Al_2O_3 as its major protective oxide scale with some Cr_2O_3 evident as an oxide constituent in furnace testing after 200 hours of exposure. The microstructural constituents in GE-1541 are well known (ref. 6) and were confirmed as being a second phase of FeY_9, which formed as 20- to 30-μm-diameter precipitates throughout the matrix and as a grain boundary precipitate.

The NASA-15M alloy was the best of the modified Fe-15Cr-4Al compositions, with low weight change after 600 hours of cyclic exposure (table V) and a slight tendency for spallation. However, the NASA-15M alloy was internally oxidized to a depth of 90 to 300 μm (fig. 9(b)) in localized areas which covered about 20 percent of the perimeter of the metallographic sections evaluated.

The NASA-15T alloy was internally oxidized, with large hexagonally shaped oxides being formed after only 200 hours of exposure. These oxides tended to be on grain boundaries and were seen at least 200 μm into the substrate (fig. 9(c)).

The NASA-15T2 alloy exhibited poor furnace oxidation resistance in 480 hours (corners blooming, edges gone). The oxidation on unfailed surfaces and within the substrate (fig. 9(d)) was similar in type and extent to that of NASA-15T (fig. 9(c)). The additional 1-wt. % Al was apparently insufficient to provide oxidation protection in the absence of Y in these 15-wt. %-Cr alloys.

The GE-1540 alloy exhibited excessive spallation and weight loss in only 150 hours of 1150° C furnace oxidation testing. The edge attack clearly seen in figure 9(e) was typical of those alloys which were not resistant to oxidation attack. A companion test specimen of GE-1540 was two-thirds destroyed, with a 460-mg/cm^2 loss in less than 50 hours of testing. The oxide layer on GE-1540 after 100 hours was identified as Al_2O_3. After 150 hours, only Cr_2O_3 was evident.

The GE-1541 alloy was excellent in oxidation resistance, experiencing no internal oxidation and no spallation. The alloy had a tightly adherent Al_2O_3 protective oxide. The necessity for the inclusion of Y or another oxidation-resistance-promoting element in alloys such as NASA-15T and NASA-15T2 is evident. Although all additions of refractory metal benefited oxidation resistance as compared with GE-1540, none of the modified Fe-15Cr-4Al alloys provided oxidation resistance comparable to that of GE-1541.

Fabricability

Fabricability of the modified Fe-18Cr-2Al alloys was found to be comparable to that of Armco 18SR both in the manufacture of sheet by the alloy vendors and in the formability and weldability of the sheet to provide automobile thermal reactors for use in exhaust-gas-exposure tests, as reported in reference 2. All test reactor component parts shown in figure 10 were formed at room temperature from these alloys, which included Armco 18SR, NASA-18T, and NASA-18M. Limited drawability test data (table VI) indicated that the NASA-18T and NASA-18T-A modifications and Armco 18SR had similar drawability in the as-received condition. Armco 18SR showed an advantage over NASA-18T and NASA-18T-A in bend tests of as-received material and in drawability after welding.

In contrast, both the manufacture and the forming of sheet for the Y-containing GE-1541 alloys required special techniques. For sheet manufacture, the GE-1541 alloy had to be extruded to sheet bar prior to hot rolling to gage. The extrusion operation was required to break up the brittle FeY_9 phase, which solidifies in grain boundaries upon casting (ref. 5). Fabrication of parts from GE-1541 and from the NASA-15T2 and NASA-15T alloys by cold forming was not successful, probably because of the relatively high Al content in combination with Y and/or refractory metal additions. Reactor parts of these alloys had to be warm formed after being torch heated to about 300° C.

Automobile Thermal Reactor Tests

In tests of full-size automobile thermal reactors made of Armco 18SR, GE-1541, and selected alloy modifications, results similar to those obtained in the cyclic furnace oxidation tests were observed (ref. 2 and table VII). The NASA-18T alloy exhibited a marked improvement over Armco 18SR in cyclic exposure to exhaust gas at a peak temperature of 1040° C. After 600 hours of exposure, penetration of the 1.6-mm-thick Armco 18SR core was noted at exhaust port openings at either end and along the welded longitudinal seam. An identical NASA-18T test reactor survived 760 hours without penetration and exhibited less weight loss (table VII).

Photomicrographs of the Armco 18SR and NASA-18T alloys after exhaust-gas exposure are compared in figures 11 and 12. The relative depth of oxidation was somewhat less for the reactor exhaust-gas exposure than for cyclic furnace exposure at 110° C higher temperature and comparable duration. The oxide scale on exhaust-gas-exposed NASA-18T was continuous and protective and consisted of an outer layer of Al_2O_3 including some SiO_2 (table VII and fig. 12(b)). The inner layer contained principally Al_2O_3. Beneath the oxide were platelets which contained Ta, Fe, and Ti. The platelets may aid in keying the oxide to the substrate to minimize spalling. Except in the platelets, no evidence of Ta was found in the NASA-18T oxide scale. These results differ from the furnace oxidation test results, in which the oxide scale was a single layer containing primarily Al_2O_3 and some TiO_2. The differences in the exposure conditions (temperature and environment) probably caused these variations in the oxide scales. In either case, however, the NASA-18T alloy exhibited excellent resistance to oxidation. After test exposures to either air or exhaust gas, the NASA-18T alloy was free of substrate penetration beneath the oxide scale, which indicates that oxidation resistance was improved by the addition of Ta.

The Armco 18SR, exhaust-gas-exposed, reactor core exhibited oxide penetration into the substrate similar to that seen in the cyclic furnace oxidation test (figs. 7(c) and 12(a)). Also, the oxide scales were similar in both types of test exposure and contained mixed oxides of the substrate constituents. The amount of Al_2O_3 appeared to be greater and the oxide layer more uniform after the 600-hour furnace oxidation test (figs. 7(c) and 12(a)).

In similar thermal reactor tests, the GE-1541 alloy provided excellent performance (table VII), confirming the beneficial effects of Y in providing both good strength and oxidation resistance to the Fe-15Cr-4Al alloys. Neither NASA-15T nor NASA-15T2 survived exhaust-gas exposure beyond 470 hours, and neither alloy retained a protective Al_2O_3 oxide layer. Overall degradation of these two alloys in the thermal reactor tests was similar to that observed in the furnace oxidation tests.

CONCLUDING REMARKS

Of the many alloys evaluated, GE-1541, NASA-18T, and Armco 18SR were recommended as candidate materials for automobile thermal reactors in reference 2. Although the GE-1541 alloy exhibited the best performance in reactor tests, the poorer fabricability of this alloy is probably a major deterrent to its use. Both NASA-18T and Armco 18SR appear to have adequate fabricability. In terms of oxidation resistance and high-temperature strength, the NASA-18T alloy has an advantage over Armco 18SR, as shown in this study as well as in the thermal reactor tests (ref. 2). However, this advantage comes at a higher material cost because of the Ta addition. The cost difference could be as high as 40 percent. Thus, the technical advantages have to be considered in relation to the cost and to the final design requirements for thermal reactors as well as for other applications where Fe-Cr-Al alloys of this type have potential for use.

SUMMARY OF RESULTS

An alloy modification program was conducted in which two existing iron-chromium-aluminum (Fe-Cr-Al) alloys, Armco 18SR and GE-1541, were modified in an attempt to achieve either better high-temperature strength or better fabricability without degrading high-temperature oxidation resistance. In order to accomplish this, additions of tantalum (Ta) or molybdenum (Mo) plus niobium (Nb) were made to the base alloys in amounts less than 3 wt. %. The base alloys and their modifications were evaluated on the basis of stress-to-rupture properties at 800° and 1000° C, resistance to cyclic oxidation exposure at 1150° C, and fabricability. The results are as follows:

1. The best of the modified alloys in terms of oxidation resistance, strength, and fabricability was NASA-18T (Fe-18Cr-2Al-1Si-0.5Ni-0.5Ti-1.25Ta). The NASA-18T alloy had at least twice the rupture strength of Armco 18SR and 1.5 times the tensile

strength to about 1000° C. Its cyclic furnace oxidation resistance was also improved over that of Armco 18SR. There was negligible weight gain and no significant spall or internal damage. The protective oxide was Al_2O_3.

2. A second alloy, NASA-18T-A (Fe-18Cr-2Al-1Si-0.5Ni-0.5Ti-0.5Ta), had about the same oxidation resistance as NASA-18T with about 1.5 times the rupture strength of Armco 18SR to 1000° C. The protective oxide was Al_2O_3.

3. Fabricability of the NASA-18T and NASA-18T-A alloys was comparable to that of commercially available Armco 18SR.

4. The Mo + Nb additions to Armco 18SR were not as effective as Ta in providing improved strength and oxidation resistance.

5. All attempted modifications of the GE-1541 alloy were unsuccessful in terms of achieving better fabricability without sacrificing high-temperature strength and oxidation resistance.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 19, 1975,
505-03.

APPENDIX - ANNEALING BEHAVIOR OF Fe-18Cr-2Al ALLOYS

Portions of the sheet material produced in 1.6-mm thickness from Armco 18SR, NASA-18T, and NASA-18T-A were cold rolled to 0.5-mm thickness without intermediate anneals to study annealing effects on mechanical properties. The hardness and grain size of as-received material, as-cold-rolled material, and the 0.5-mm-thick material after annealing at several temperatures are given in table VIII. The as-received sheet stock differed in hardness only slightly for the three alloy compositions. Upon cold rolling, the NASA-18T alloy became 100 KHN harder than either NASA-18T-A or Armco 18SR. The hardness was not retained after annealing. And, as discussed in the section entitled Stress-to-Rupture Properties, the rupture strength of the 0.5-mm-thick material after annealing differed little from that of as-received 1.6-mm-thick sheet.

The NASA-18T alloy had uniform grain size for all annealing conditions investigated. A similar result with somewhat larger grain size was found for NASA-18T-A. In all cases the grain sizes of the modified alloys were smaller than the grain size of the Armco 18SR under similar annealing conditions. The selected annealing conditions for each of the reprocessed alloy sheet materials are designated in table VIII.

It is concluded that nominal variation in annealing time or temperature, as might be encountered in the processing of the NASA-18T and NASA-18T-A alloys, would have neither an adverse nor a beneficial effect. The as-received material which was used in this program is probably representative of the best properties of the alloy compositions investigated.

REFERENCES

1. Blankenship, Charles P.; and Hibbard, Robert R.: Status Report on the NASA-EPA Automotive Thermal Reactor Technology Programs. NASA TM X-68010, Jan. 1972.
2. Blankenship, Charles P.; and Oldrieve, Robert E.: An Evaluation of Alloys and Coatings for Use in Automobile Thermal Reactors. SAE Paper 740091, Feb. 1974.
3. Stone, Phillip L.; and Blankenship, Charles P.: Design and Evaluation of Experimental Ceramic Automotive Thermal Reactors. NASA TN D-7706, 1974.
4. Hill, V. L.; Misra, S. K.; and Wheaton, H. L.: Development of Ductile Claddings for Dispersion-Strengthened Alloys. (IITRI-B6083-13, IIT Research Inst.; NAS3-10494), NASA CR-72522, 1969.
5. Bartos, J.; Perkins, R.; and Robertshaw, F. C.: Mechanical Properties of Fe-Cr-Al-Y Alloys. GEMP-711, General Electric Co., 1969.
6. Wukusick, C. S.: The Physical Metallurgy and Oxidation Behavior of Fe-Cr-Al-Y Alloys. GEMP-414, General Electric Co., 1966.
7. Armco Product Data Sheet S47B. LA-13672, Armco Steel Corp., 1972.
8. Barrett, Charles A.; and Lowell, Carl E.: Comparison of Isothermal and Cyclic Oxidation Behavior of Twenty-five Commercial Sheet Alloys at 1150° C. NASA TN D-7615, 1974.

TABLE I. - COMPOSITIONS OF FERRITIC IRON ALLOYS EVALUATED

Alloy	Heat	Composition, wt. %													ASTM grain size	Vendor
		C	Mn	Si	Cr	Ni	Ta	Mo	Nb	Al	Ti	P	S	Other		
Fe-18Cr-2Al alloys																
Armco 18SR	490578	0.031	0.27	1.14	17.90	0.37	----	----	----	2.16	0.54	[a]0.015	[a]0.015	-----	2 - 5	Armco Steel Co.
NASA-18T-A	32	.038	.37	1.14	17.74	.17	0.45	----	----	2.10	.44	.008	.012	-----	4 - 5	Armco Steel Co.
NASA-18T	33	.040	.37	1.28	17.76	.20	1.25	----	----	2.10	.45	.007	.014	-----	6 - 8	Armco Steel Co.
NASA-18M	P-873	.041	[a].5	1.01	17.91	----	----	2.04	0.58	2.19	----	-----	-----	-----	3 - 4	Climax Molybdenum Co.
Fe-15Cr-4Al alloys																
GE-1541	M-287	0.012	0.001	0.07	15.20	----	----	----	----	4.95	----	0.003	0.001	0.7 Y	3	General Electric Co.
GE-1540	M-289	.007		.04	15.03	----	----	----	----	4.67	----	.002	.011	-----	1	
NASA-15T	M-290	.010		.05	14.72	----	0.91	----	----	4.27	----	.002	.003	-----	2	
NASA-15T2	M-292	.012		.05	15.23	----	1.97	----	----	5.35	----	.002	.004	-----	4 - 5	
NASA-15M	P-872	.041	[a].3	[a].1	14.37	----	----	2.02	0.53	4.72	----	-----	-----	-----	4	Climax Molybdenum Co.

[a]Expected or nominal.

TABLE II. - STRESS-TO-RUPTURE DATA FOR Fe-Cr-Al ALLOYS

[Nominal specimen thickness, 1.6 mm, except as noted.]

Temperature, °C	Stress, MN/m^2	Life, hr	Elongation, percent
Armo 18SR			
1000	3.3	105.7	65.7
	3.3	132.9	[a]48.6
	3.5	51.9	77.2
	5.2	3.6	113.0
	7.6	1.9	[a]57.2
	7.6	2.4	[a]88.5
900	5.0	489.4	[a]37.1
	8.0	59.5	[a]29.1
800	10.3	155.6	123.0
	13.8	40.4	126.0
	19.1	14.8	91.5
NASA-18T			
1000	3.5	483.4	[a]37.2
	3.5	749.5	100.0
	5.2	127.2	[a]22.2
	5.2	241.8	88.6
	6.9	23.6	[a]34.3
	8.3	340.9	[a]12.6
	8.6	7.9	111.5
900	6.9	449.4	[a]33.1
	8.0	270.1	[a]55.4
	10.0	138.2	68.6
800	10.3	2758.5	131.0
	14.5	2249.0	48.6
	17.2	586.0	57.0
	24.3	116.1	45.2
NASA-18T-A			
1000	3.5	429.6	31.4
	5.2	21.1	58.8
	7.3	7.1	85.1
	7.6	11.1	[a]31.4
	7.6	4.4	[a]44.6
900	6.0	600.3	[a]24.6
	8.0	185.2	[a]77.1
800	10.3	1570.9	74.3
	11.5	1076.2	65.7
	17.2	105.5	97.2
NASA-18M			
1000	3.5	245.8	120.0
	3.4	161.4	129.0
	5.2	48.7	110.0
	7.2	15.8	94.4
900	6.0	373.0	44.0
	15.0	4.9	53.1
800	11.6	299.5	62.8
	13.8	420.3	67.5
	17.2	191.7	71.4
	20.7	75.2	60.0
	30.1	16.2	93.8
NASA-15T			
1000	2.0	468.1	122.0
	3.5	61.4	99.0
	3.5	40.6	94.5
	3.6	54.2	83.0
	5.0	10.3	80.0
	6.9	6.5	99.0
900	5.0	447.3	54.3
	7.2	39.4	63.4
	12.0	4.7	93.1
	15.0	2.6	105.1
800	10.0	544.7	94.4
	17.2	49.9	74.4
	20.0	20.6	[a]61.1
NASA-15T2			
1000	3.4	978.9	101.1
	4.5	184.7	57.2
	5.2	77.2	65.6
	7.2	12.3	88.5
	7.6	41.1	60.0
900	6.9	475.4	71.4
	10.0	59.1	70.3
800	9.9	1429.0	54.3
	17.2	69.1	85.7
	17.2	64.9	65.7
	24.7	4.0	29.2
NASA-15M			
1000	3.5	125.1	104.5
	5.2	22.8	76.0
	7.2	8.8	111.0
900	6.0	178.0	81.7
	10.3	34.9	89.9
800	10.3	1715.8	54.3
	13.8	199.6	91.5
	15.5	282.7	60.0
	17.2	223.7	85.8
	20.7	92.0	94.4
GE-1540			
1000	2.0	215.8	67.8
	3.5	16.6	68.5
	4.9	6.1	71.4
	6.8	1.0	87.5
	7.2	1.2	82.9
900	4.0	194.3	98.9
	7.3	34.5	88.6
800	6.9	171.7	67.4
	9.0	67.6	91.4
	10.3	69.4	53.4
	17.2	5.2	82.8
GE-1541			
1000	3.5	3989.0	27.0
	5.2	1328.6	37.1
	6.9	44.5	85.7
	6.9	21.9	45.7
	8.6	6.7	100.0
	10.3	8.7	80.0
900	7.0	478.0	56.0
	8.0	116.8	58.9
	10.0	45.9	37.1
800	8.6	1078.2	48.6
	10.3	330.0	65.7
	17.2	16.7	85.8
	17.2	.1	64.0
	34.5	.8	74.3

[a]Specimen thickness, 0.5 mm.

TABLE III. - ELEVATED-TEMPERATURE TENSILE PROPERTIES OF NASA-18T, ARMCO 18SR, AND GE-1541 SHEET

[Nominal specimen thickness, 1.6 mm.]

Temperature, °C	NASA-18T			Armco 18SR			GE-1541[a]		
	Ultimate tensile strength, MN/m^2	Yield strength, MN/m^2	Elongation, percent	Ultimate tensile strength, MN/m^2	Yield strength, MN/m^2	Elongation, percent	Ultimate tensile strength, MN/m^2	Yield strength, MN/m^2	Elongation, percent
538	-----	-----	---	[b]358	-----	---	---	-----	-----
550	451	318	40	---	-----	---	243	192	29.6
650	319	251	25	206	161	69	140	101	68.9
	-----	-----	---	[b]193	-----	81	149	113	63.6
732	-----	-----	---	[b]83	-----	99	---	-----	-----
750	140	94	90	---	-----	---	68	61.5	18.8
800	92	62	102	---	-----	---	---	-----	-----
	95	63	86	---	-----	---	---	-----	-----
816	-----	-----	---	[b]42	-----	97	---	-----	-----
850	-----	-----	---	---	-----	---	39	30.5	128
900	55	37	106	---	-----	---	---	-----	-----
	61	37	83	---	-----	---	---	-----	-----
927	-----	-----	---	[b]28	-----	---	---	-----	-----
982	-----	-----	---	[b]14	-----	---	---	-----	-----
1025	[b]29	-----	---	[b]20	-----	---	---	-----	-----
	29.6	19.3	134	22	12.9	140	---	-----	-----

[a]From ref. 5.
[b]From ref. 7.

TABLE IV. - ROOM-TEMPERATURE TENSILE PROPERTIES OF NASA-18T, NASA-18T-A, ARMCO 18SR, AND GE-1541 SHEET

[Nominal specimen thickness, 1.6 mm; as received.]

Property	NASA-18T	NASA-18T-A	Armco-18SR	GE-1541[a]
Ultimate tensile strength, MN/m^2				
Longitudinal	661	596	552 - 620	503
Transverse	686	614	--------	----
0.2-Percent yield strength, MN/m^2				
Longitudinal	474	417	414 - 483	348
Transverse	495	436	--------	----
Elongation (50 mm), percent				
Longitudinal	25.5	27	25 - 30	25.2
Transverse	24	25	--------	----
Hardness, R_B				
Longitudinal	94	92	90	----
Transverse	95	91.5	--------	----

[a]From ref. 5.

TABLE V. - SUMMARY OF OXIDATION TEST CONDITIONS AND OXIDES ON EXPOSED ALLOYS

Alloy	Cyclic oxidation exposure at 1150° C				Remarks
	[a]200 Hours		[a]600 Hours		
	Weight change, mg/cm^2	Principal surface oxide	Weight change, mg/cm^2	Principal surface oxide	
NASA-18T	+1.7	Al_2O_3	+1.9	Al_2O_3	No spall
NASA-18T-A	+2.2	Al_2O_3	+2.4	(b)	No spall
Armco 18SR	+5.4	Al_2O_3	+7.8	Mixed oxides	Negligible spall
NASA-18M	-7.8	Cr_2O_3; spinel	-30.0 (450 hr)	Cr_2O_3; spinel	Failed
GE-1541	+1.0	Al_2O_3; Cr_2O_3	+1.1	(b)	No spall
NASA-15M	+4.0	Al_2O_3	+3.6	(b)	Light spall
NASA-15T	+8.4	Al_2O_3; spinel	+14.5	(b)	Light spall
NASA-15T2	+13.5	Al_2O_3; spinel	-30.0 (480 hr)	(b)	Failed
GE-1540	-30.0	Cr_2O_3 (150 hr)	(b)	(b)	Failed

[a]Exposure time, except as noted; each cycle consisted of 1 hr at temperature with a minimum 40 min static-air cooling to ambient temperature.
[b]Not determined.

TABLE VI. - FORMABILITY OF NASA-18T, NASA-18T-A, AND ARMCO 18SR AS-RECEIVED AND AS-WELDED SHEET

[Nominal specimen thickness, 1.6 mm; Armco-supplied data.]

Condition	Type of fabrication test	NASA-18T	NASA-18T-A	Armco 18SR
As received	Olsen cup drawability test[a]; height, cm	0.93	0.94	0.89
As received	180° bend test[b]; number of specimens without cracks	4 of 6	1 of 4	6 of 6
As welded[c]	Olsen cup drawability test[a]; height, cm	0.23	0.20	0.38

[a]2.2-cm ball; 8000-kg hold-down; mineral oil; polyethylene sheet.
[b]1 T-bend diameter.
[c]Gas tungsten-arc welded; no fillers; average of duplicate tests.

TABLE VII. - SUMMARY OF AUTOMOBILE THERMAL REACTOR ENDURANCE TESTS

[Fifty percent of exposure time at 1040° C; for a description of the test cycle, see ref. 2.]

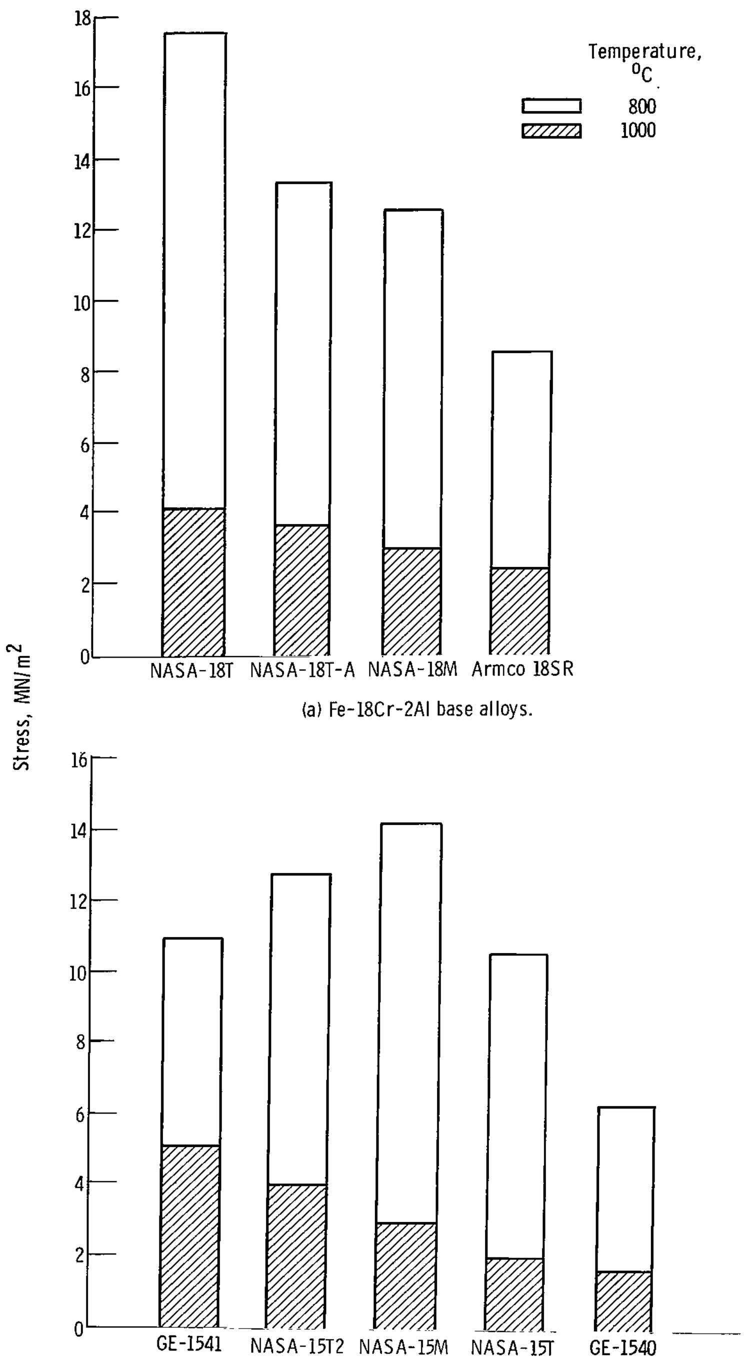
Reactor-core composition	Exposure time, hr	Weight change, percent	Principal surface oxide	Remarks
NASA-18T[a]	762	-0.3	Al_2O_3	Good performance; core distorted; did not fail
GE-1541[a]	684	+.3	Al_2O_3	Excellent performance; W-1 weld rod; did not fail
NASA-15T	404	+.6	Cr_2O_3	Localized oxidation failure
NASA-15T2	470	-.1	Spinel	Localized oxidation failure
Armco 18SR	616	-1.5	Mixed oxides	Localized oxidation failure

[a]Test terminated at end of program.

TABLE VIII. - EFFECT OF PROCESSING CONDITIONS ON GRAIN SIZE AND MICROHARDNESS OF NASA-18T, NASA-18T-A, AND ARMCO 18SR

Material condition	Annealing parameters[a]		NASA-18T		NASA-18T-A		Armco 18SR	
	Temperature, °C	Time, min	ASTM grain size	Hardness,[b] KHN	ASTM grain size	Hardness,[b] KHN	ASTM grain size	Hardness,[b] KHN
As received; 1.6 mm thick	----	--	7	228	4 - 5	218	4.5 - 5	207
As cold rolled; 70 percent reduced (0.5 mm thick)	----	--	(c)	435	(c)	340	(c)	335
Annealed; 70 percent reduced (0.5 mm thick)	926	5	--	---	-----	---	7	236
	1010	5	7	270	4 - 6	218	3 - 5	238
	1038	5		255	4 - 6	250	3 - 5	[d]207
	1065	5		250	4 - 6	[d]250	1 - 2	227
							5 - 6	
	1065	60		[d]250	-----	---	-----	---

[a]Air quenched.
[b]200-g load.
[c]Not determined.
[d]Selected for testing.



(a) Fe-18Cr-2Al base alloys.

(b) Fe-15Cr-4Al base alloys.

Figure 1. - Rupture strength of Fe-Cr-Al alloys for 400-hour life at 800° and 1000° C in air.

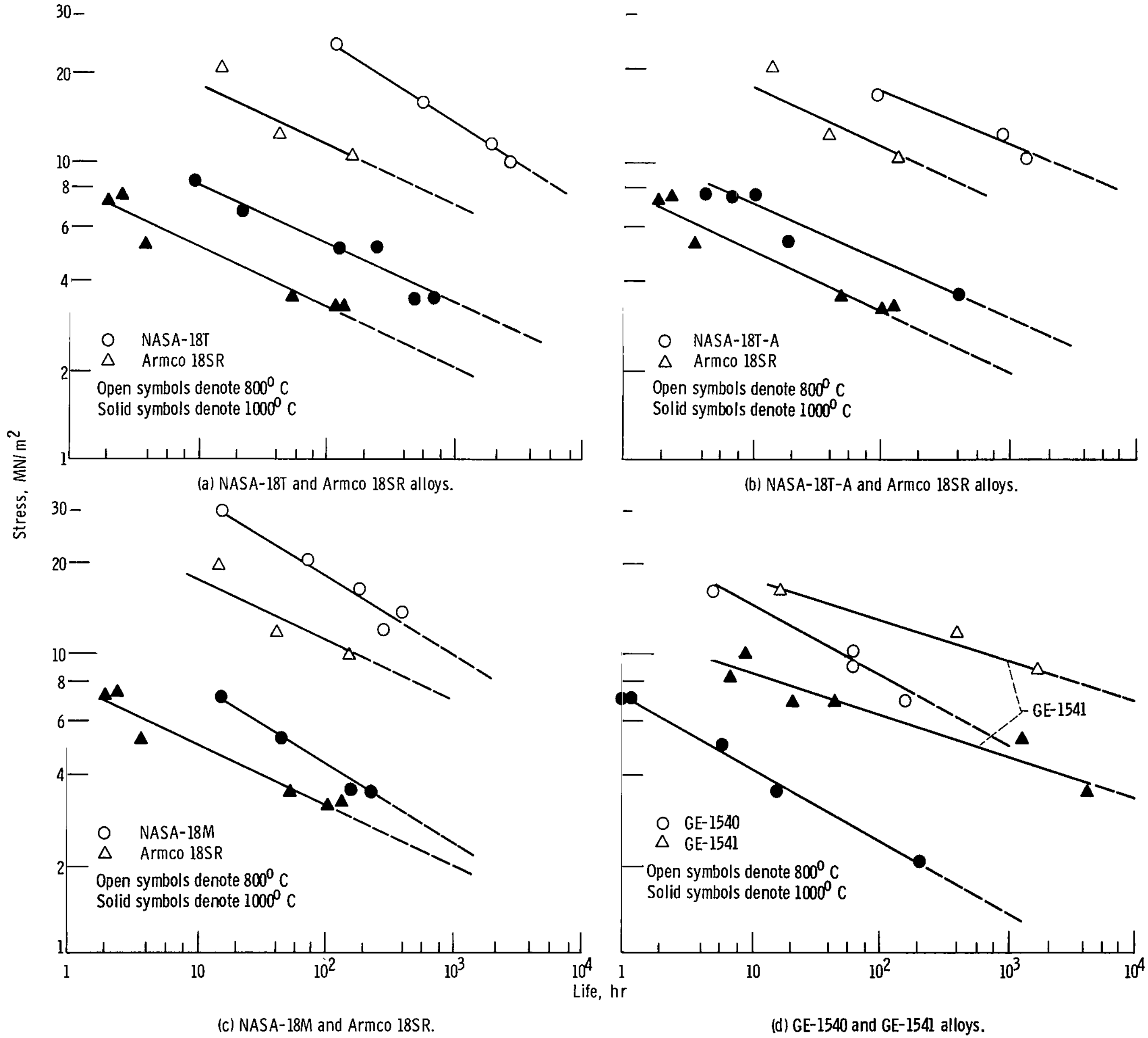
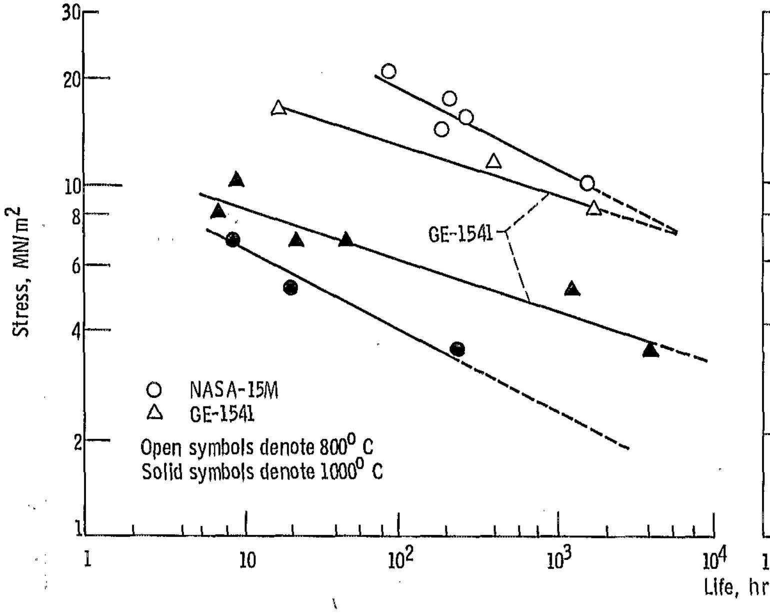
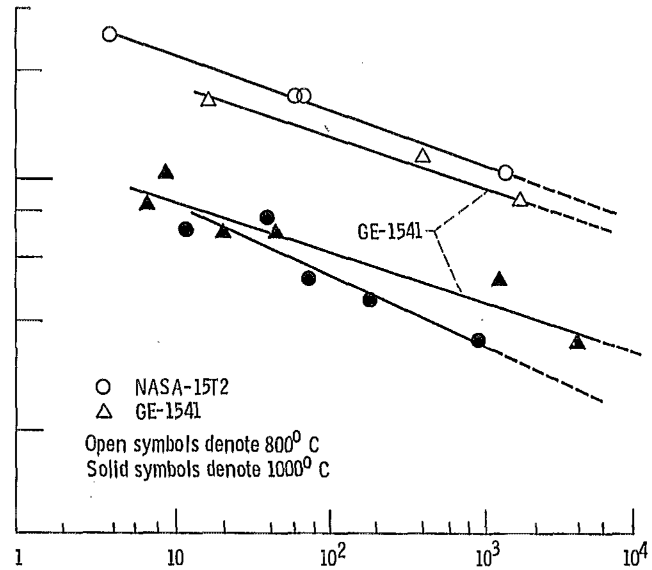


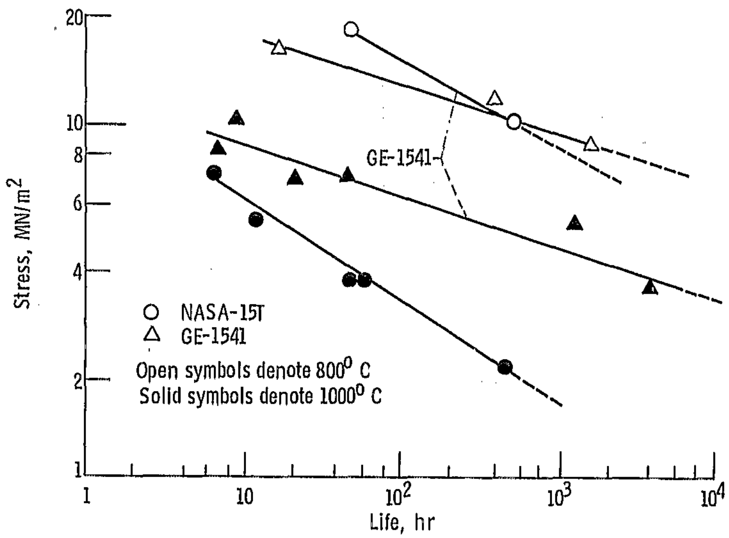
Figure 2. - Stress-to-rupture life of Fe-Cr-Al alloys at 800° and 1000° C in air.



(e) NASA-15M and GE-1541 alloys.

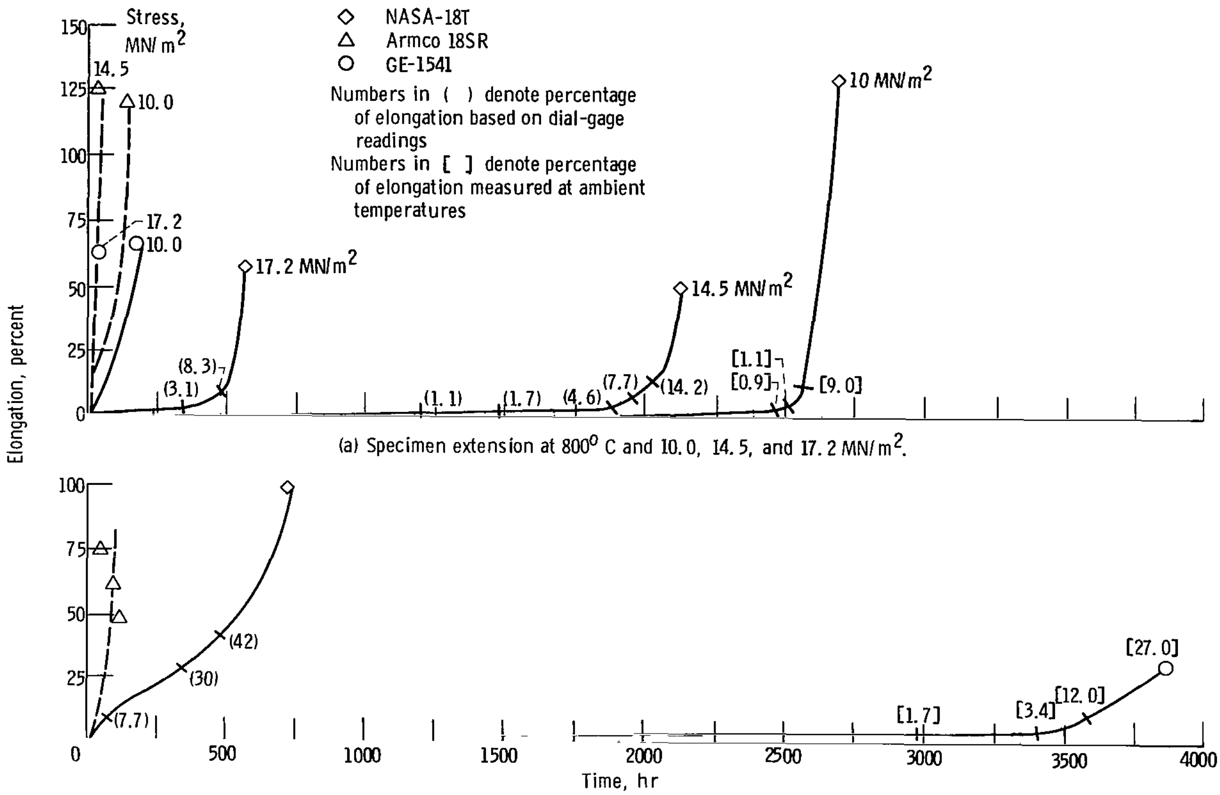


(f) NASA-15T2 and GE-1541 alloys.



(g) NASA-15T and GE-1541 alloys.

Figure 2. - Concluded.



(a) Specimen extension at 800° C and 10.0, 14.5, and 17.2 MN/m².

(b) Specimen extension at 1000° C and 3.5 MN/m².

Figure 3. - Relative creep extension of NASA-18T, Armco 18SR, and GE-1541 alloys in stress-to-rupture tests at 800° and 1000° C.

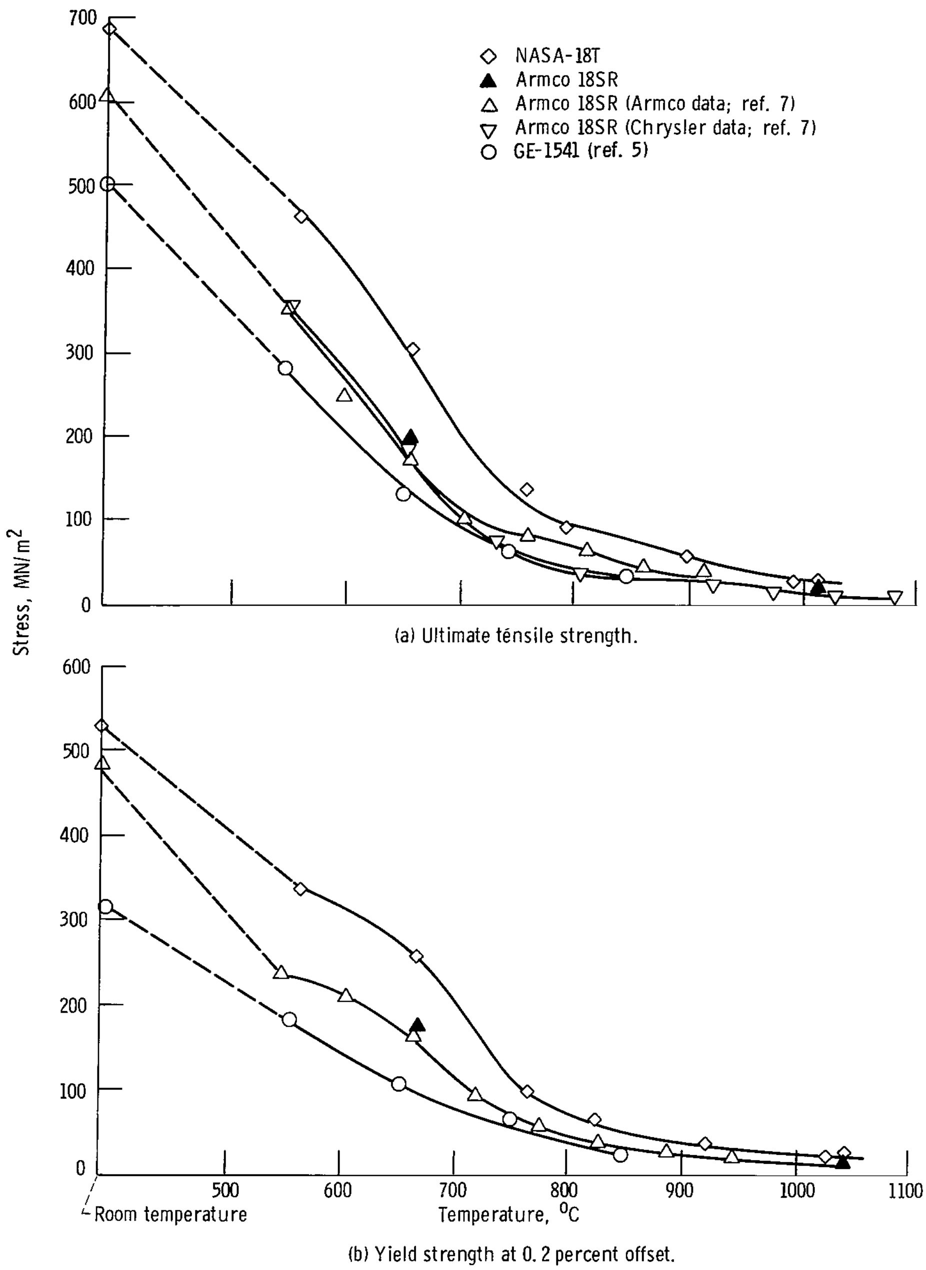


Figure 4. - Ambient- and elevated-temperature tensile data for sheet specimens of NASA-18T, Armco 18SR, and GE-1541 alloys.

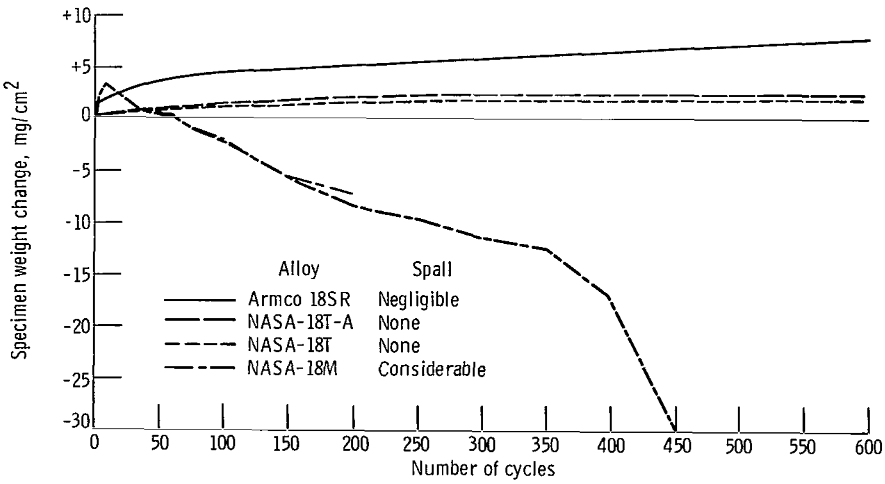


Figure 5. - Cyclic oxidation test results for Fe-18Cr-2Al alloys. Each cycle consisted of 1-hour furnace exposure at 1150° C followed by a minimum of 40 minutes static-air cooling to ambient temperature.

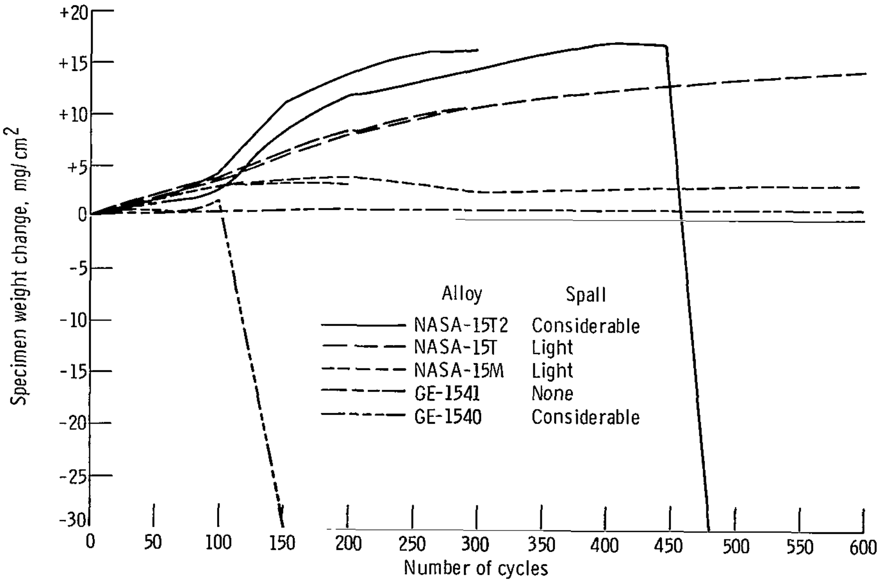
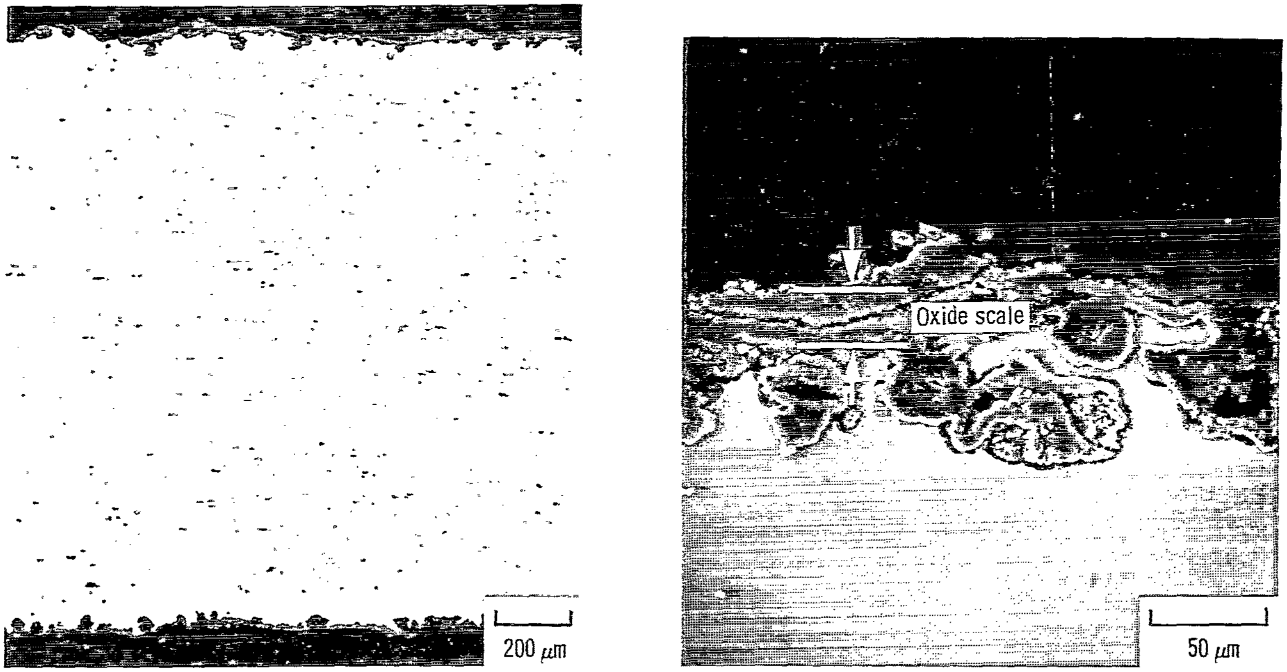
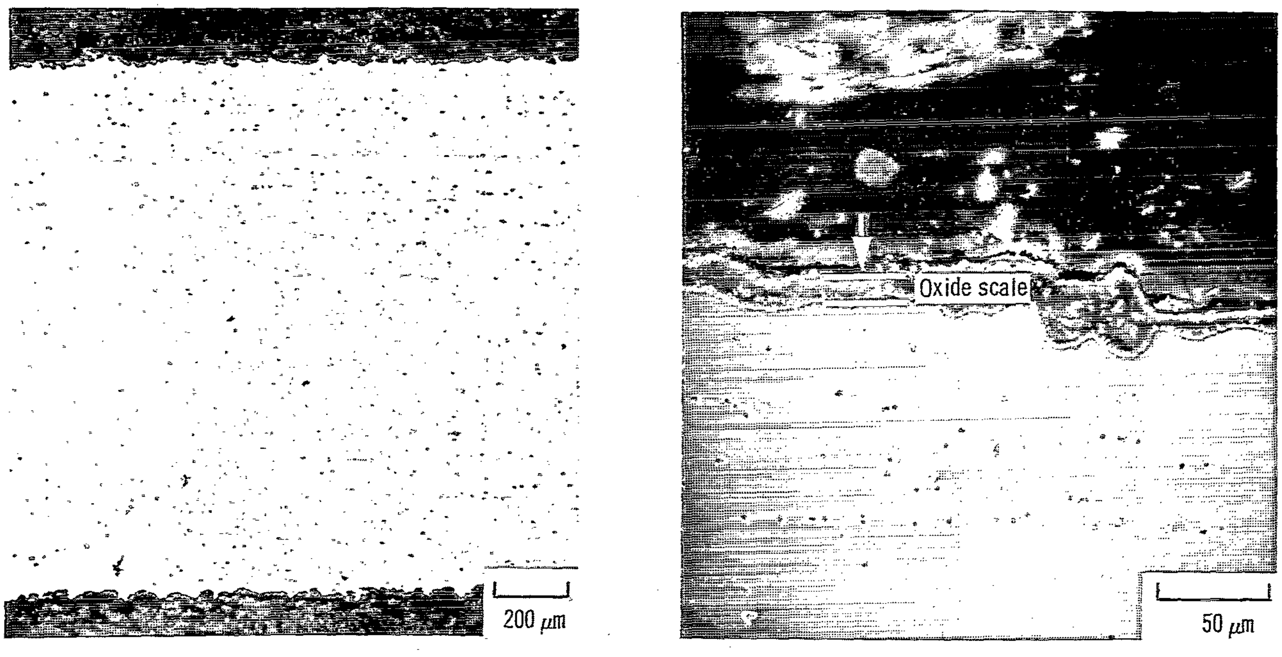


Figure 6. - Cyclic oxidation test results for Fe-15Cr-4Al alloys. Each cycle consisted of 1-hour furnace exposure at 1150° C followed by a minimum of 40 minutes static-air cooling to ambient temperature.

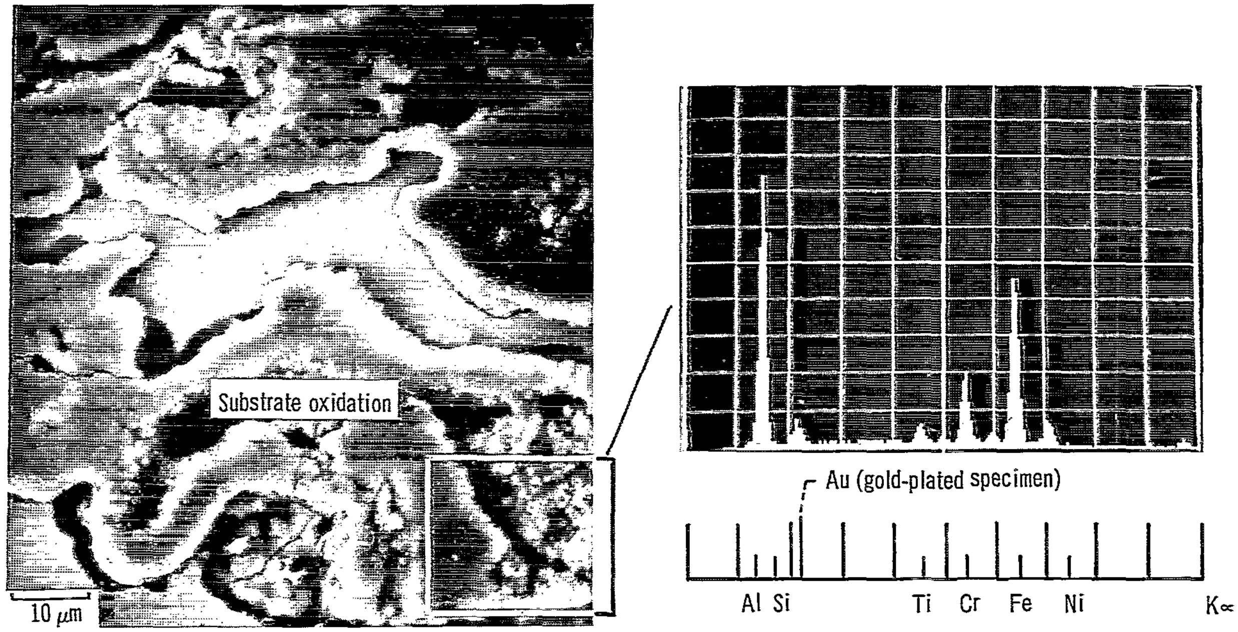


(a) Armco 18SR.

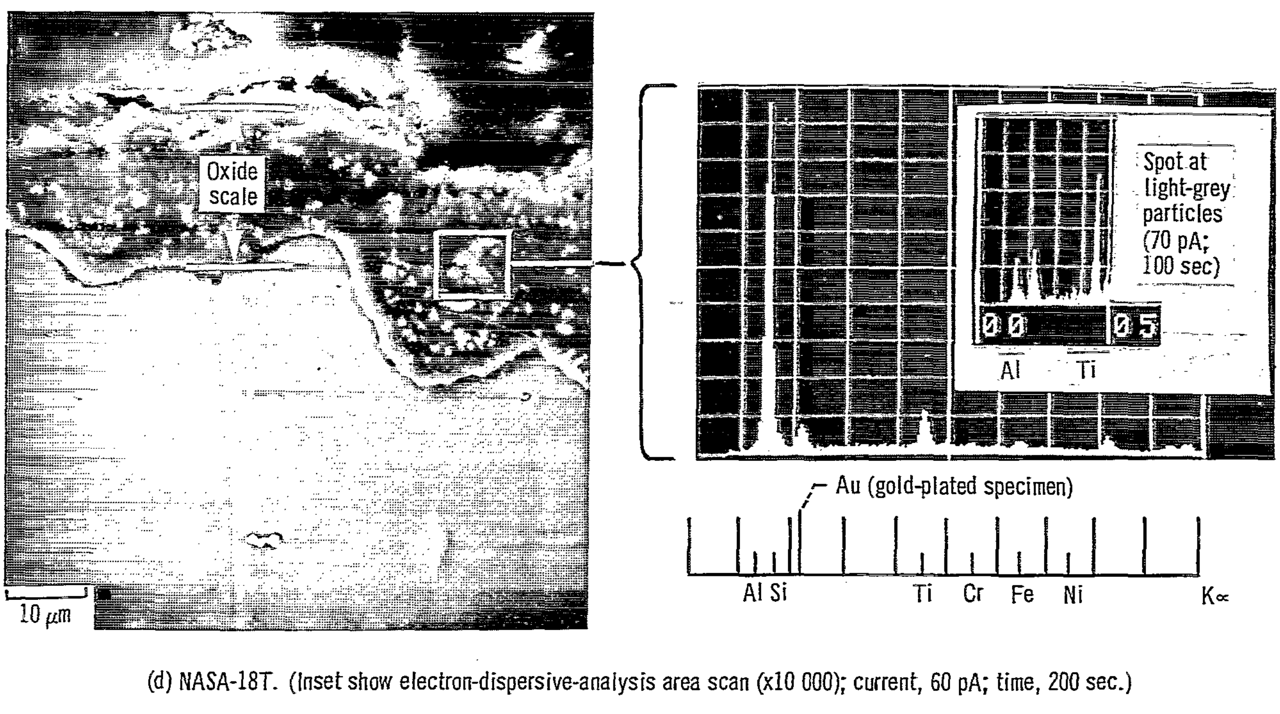


(b) NASA-18T.

Figure 7. - Photomicrographs of Fe-18Cr-2Al alloys after furnace oxidation testing at 1150° C for 600 cycles. Each cycle consists of 1 hour of heating and a minimum of 40 minutes static-air cooling to ambient temperature.

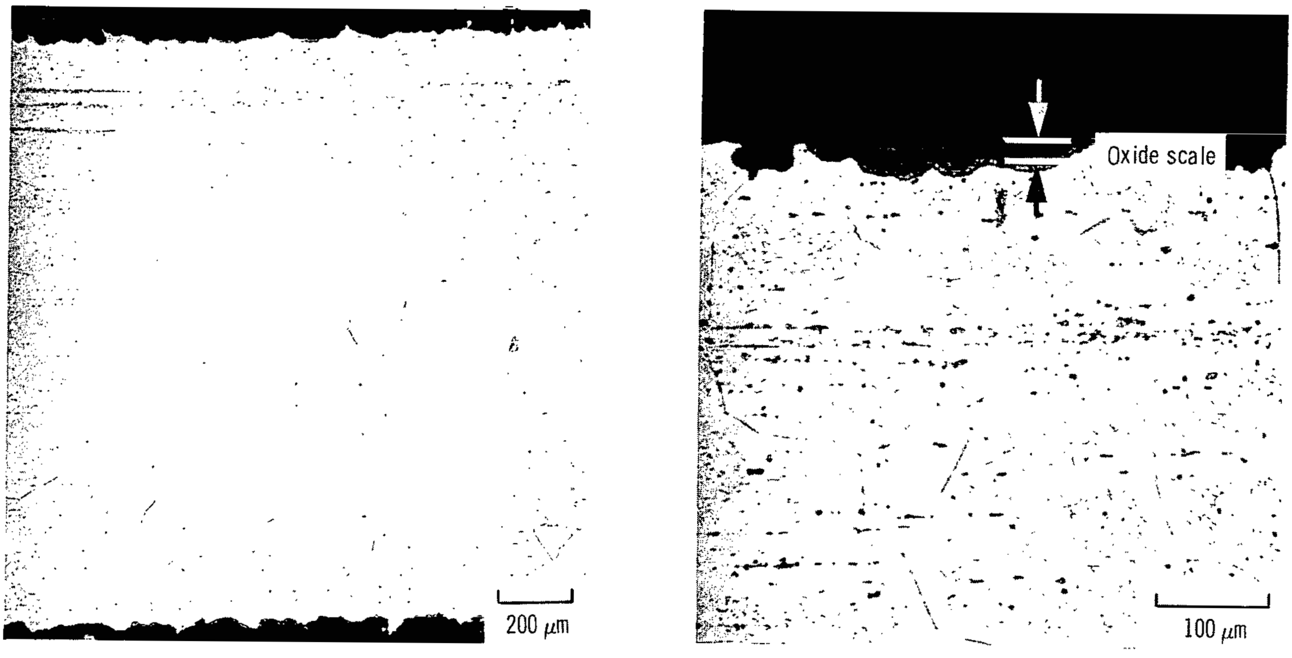


(c) Armco 18SR. (Inset shows electron-dispersive-analysis area scan (x3000); current, 30 pA; time, 100 sec.)

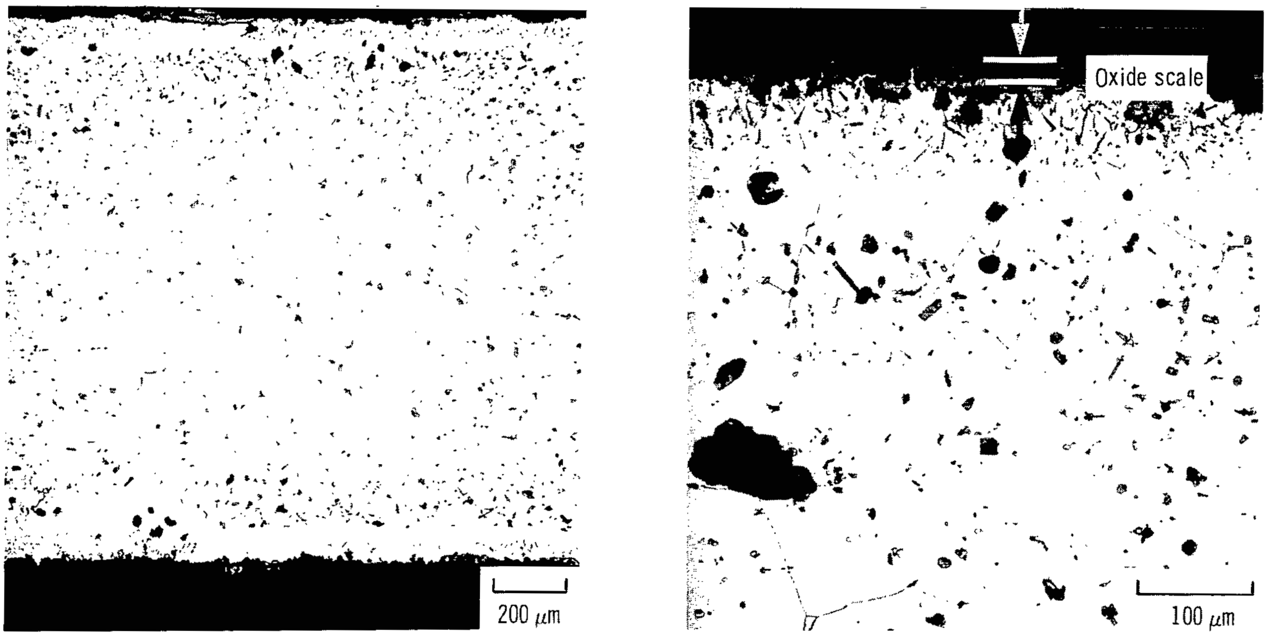


(d) NASA-18T. (Inset show electron-dispersive-analysis area scan (x10 000); current, 60 pA; time, 200 sec.)

Figure 7. - Concluded.

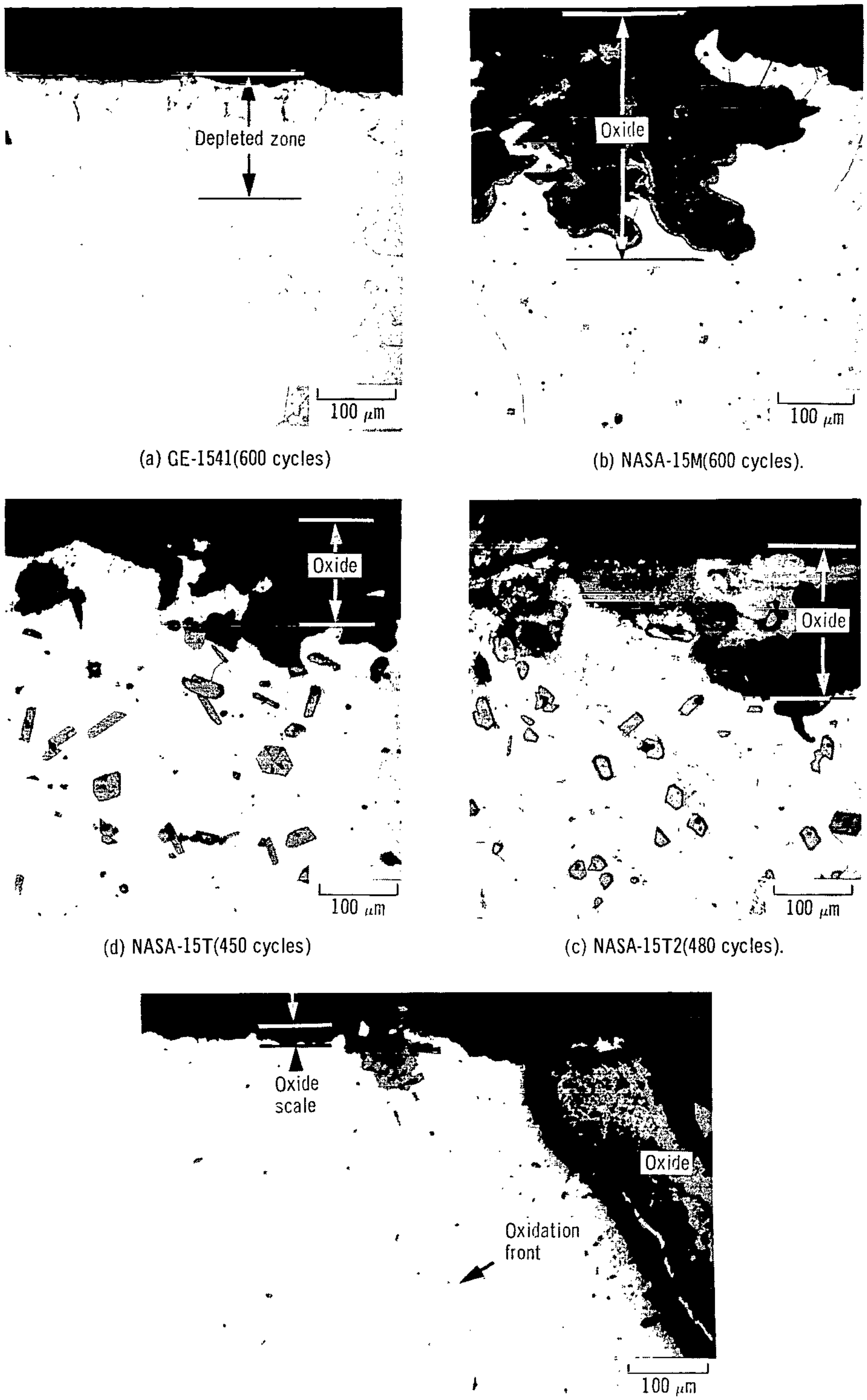


(a) NASA-18T-A (600 cycles).



(b) NASA-18M (450 cycles).

Figure 8. - Photomicrographs of Fe-18Cr-2Al alloys after cyclic furnace oxidation testing at 1150° C. Each cycle consists of 1 hour of heating and a minimum of 40 minutes static-air cooling to ambient temperature.



(a) GE-1541(600 cycles)

(b) NASA-15M(600 cycles).

(d) NASA-15T(450 cycles)

(c) NASA-15T2(480 cycles).

(e) GE-1540(150 cycles).

Figure 9. - Photomicrographs of Fe-15Cr-4Al alloys after cyclic furnace oxidation testing at 1150° C. Each cycle consists of 1 hour of heating and a minimum of 40 minutes static-air cooling to ambient temperature.

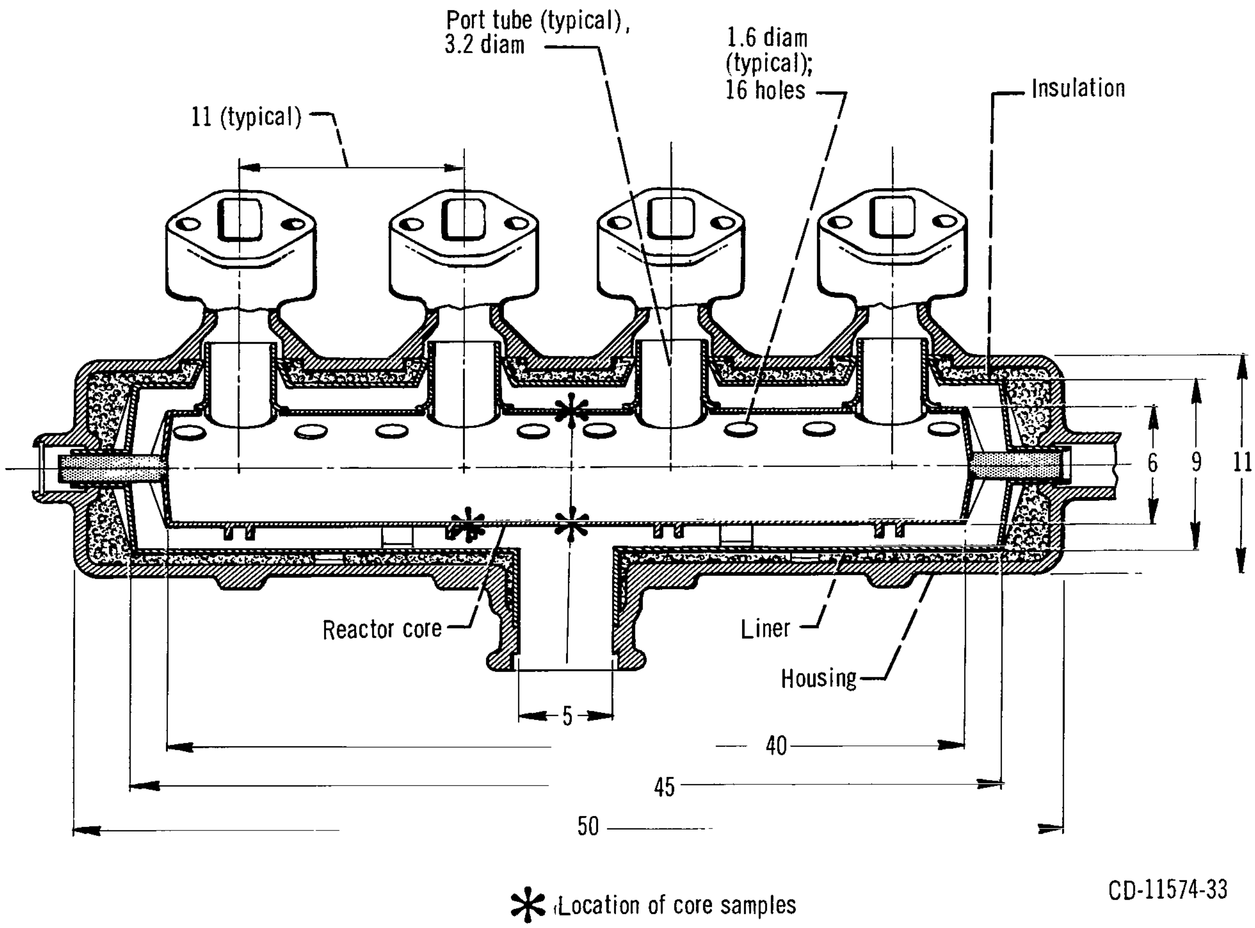
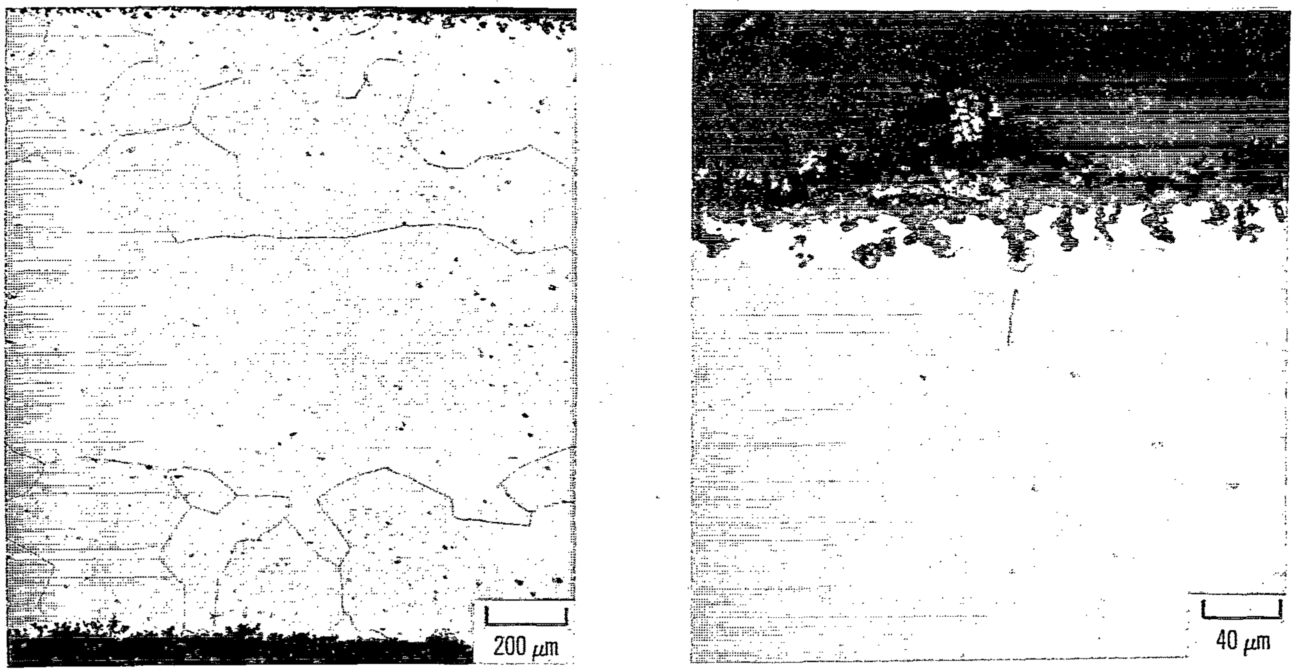
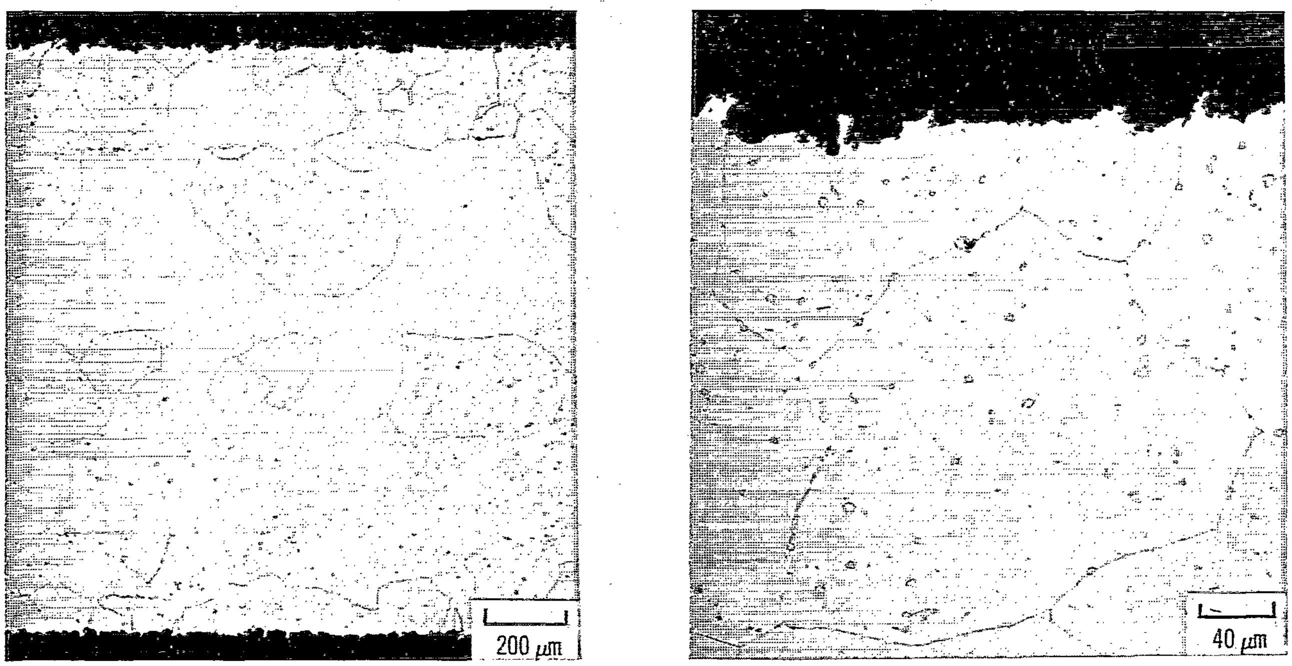


Figure 10. - Automobile thermal reactor design used for materials evaluation; core, liner, and port tubes fabricated of candidate Fe-Cr-Al alloys. (Dimensions in cm.)

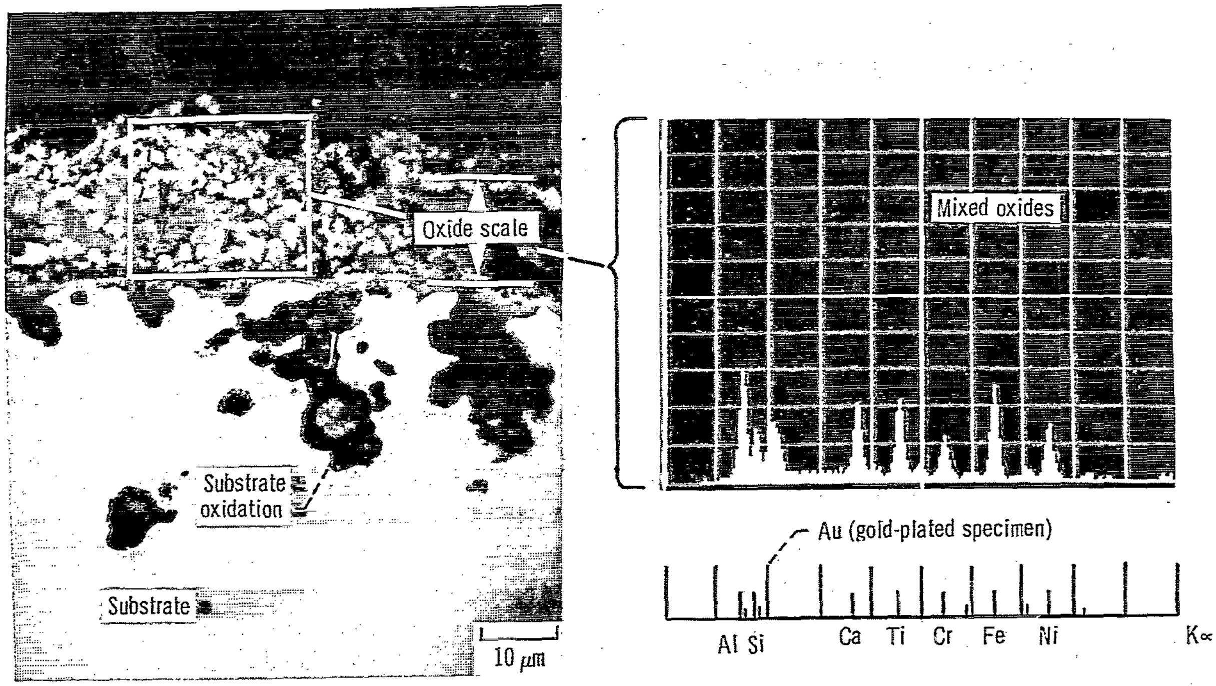


(a) Armco 18SR after 616 hours.

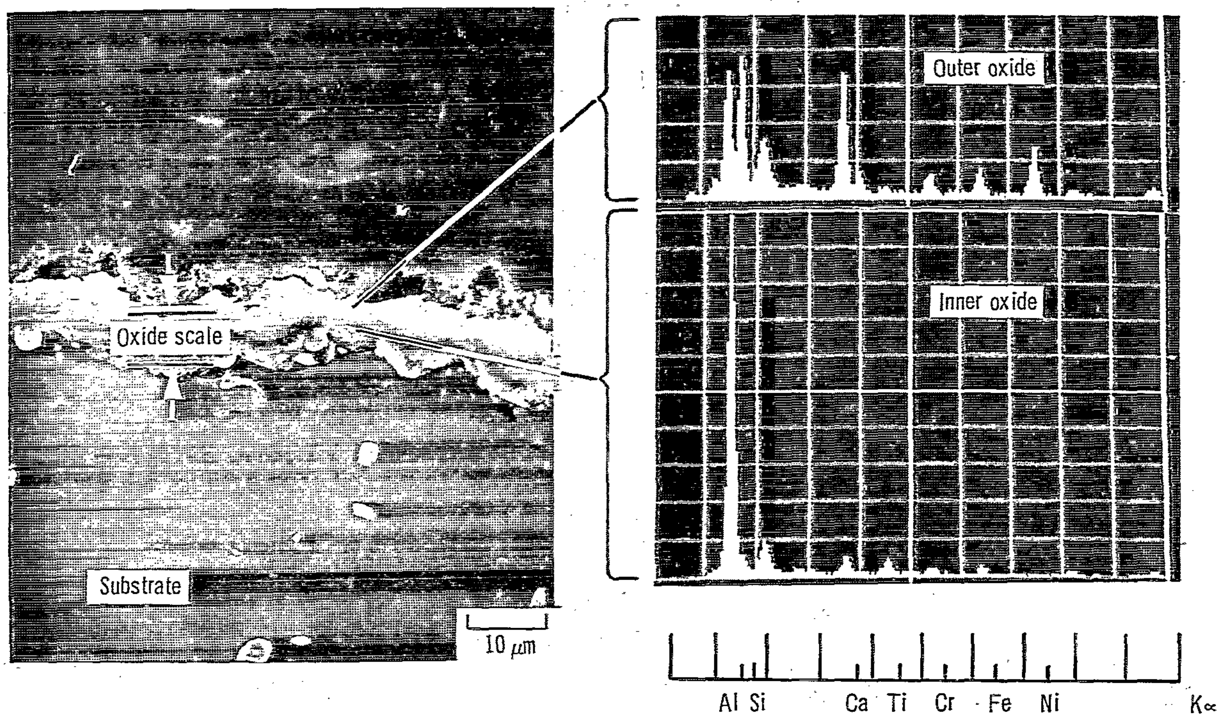


(b) NASA-18T after 762 hours.

Figure 11. - Photomicrographs of Armco 18SR and NASA-18T reactor core sections after automobile thermal reactor testing (ref. 2).



(a) Armco 18SR after 616 hours. (Inset shows electron-dispersive-analysis area scan (x3000); current, 70 pA; time, 100 sec.)



(b) NASA-18T after 762 hours. (Inset shows electron-dispersive-analysis area scan; current, 30 pA; time, 100 sec.)

Figure 12. - Scanning electron photomicrographs of oxide scales formed on Armco 18SR and NASA-18T after automobile thermal reactor testing (ref. 2).

NASA-Langley, 1975 E-8185

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C. 20546

OFFICIAL BUSINESS
PENALTY FOR PRIVATE USE $300

SPECIAL FOURTH-CLASS RATE
BOOK

POSTAGE AND FEES PAID
NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION
451

U.S.MAIL

569 001 C1 U C 750523 S00903DS
DEPT OF THE AIR FORCE
AF WEAPONS LABORATORY
ATTN: TECHNICAL LIBRARY (SUL)
KIRTLAND AFB NM 87117

POSTMASTER: If Undeliverable (Section 158 Postal Manual) Do Not Return

"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS: Information receiving limited distribution because of preliminary data, security classification, or other reasons. Also includes conference proceedings with either limited or unlimited distribution.

CONTRACTOR REPORTS: Scientific and technical information generated under a NASA contract or grant and considered an important contribution to existing knowledge.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities. Publications include final reports of major projects, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

TECHNOLOGY UTILIZATION PUBLICATIONS: Information on technology used by NASA that may be of particular interest in commercial and other non-aerospace applications. Publications include Tech Briefs, Technology Utilization Reports and Technology Surveys.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION OFFICE

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546