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EVALUATION OF SOME CANDIDATE MATERIALS FOR AUTOMOBILE THERMAL REACTORS IN ENGINE-DYNAMOMETER SCREENING TESTS - INTERIM REPORT

by Robert E. Oldrieve
Lewis Research Center
Cleveland, Ohio
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This information is being published in preliminary form in order to expedite its early release.

Fourteen materials were evaluated in engine screening tests on full-size thermal reactors for automobile engine pollution control systems. Cyclic test-stand engine operation provided 2 hours at 1040° C (1900° F) and a 20-minute air-cool to 70° C (160° F) each test cycle. Each reactor material was exposed to 83 cycles in 200 hours of engine testing. On the basis of resistance to oxidation and distortion, the best materials included two ferritic iron alloys (GE 1541 and Armco 18S/R), several commercial oxidation-resistant coatings on AISI 651 (19-9 DL), and possibly uncoated AISI 310. The best commercial coatings were Cr-Al, Ni-Cr, and a glass ceramic.

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SUMMARY

Fourteen materials, including iron and nickel-base alloys and several commercially-applied coatings on AISI 651 (19-9 DL) stainless steel, were evaluated for use in automobile thermal reactors in 200-hour engine dynamometer screening tests. Full size reactors (cores, liners, and port tubes) of candidate materials were subjected to cyclic engine operation with air injection to the test reactors to promote oxidation of the unburned exhaust products. Each test cycle consisted of a 2-hour exposure at a peak measured reactor-core-metal temperature of approximately 1040° C (1900° F) and cooldown to less than 70° C (160° F) in approximately 20 minutes before return to maximum temperature. All candidate materials were subjected to 83 test cycles in 200 hours of engine testing. The reactors were evaluated on the basis of core weight change, dimensional stability of reactor components, and coating life. The best of these candidate reactor materials evaluated in the screening tests were:

<u>Iron Alloys</u> (uncoated)

1. GE 1541 (Fe-15Cr-4Al-1Y)
2. Armco 18S/R (Fe-18Cr-2Al-1Si-0.5Ni-0.4Ti)
3. AISI 310 (Fe-20Ni-25Cr)

<u>Coatings on AISI 651 Alloy</u> (19-9 DL stainless steel)

1. Cr-Al, pack cementation (HI-15)
2. NiCr, slurry applied (NC-9)
3. Glass ceramic, slurry applied (A-418A)

All of these materials exhibited a maximum of 3 percent weight loss and less than 9 percent core distortion in the 200-hour screening test. The associated reactor components of these test materials performed comparably as the reactor cores of the same material.

By comparison, a test core of the nickel-base superalloy Hastelloy X had approximately 1 percent or less change in weight or dimensions. The liner of Hastelloy X, however, was cracked and penetrated in the areas of impingement by the hot gases leaving the core. Thus, Hastelloy X appears

to be less satisfactory than the best of the candidate materials in the screening test.

Materials which did not perform as well as those above included:

Cr-Al coated AISI 310 (which grew 10 percent in length and 25 percent at maximum cross-section); S-6100M (coating destroyed), Sermetel J (hole in core), and NC-630 (excessive weight loss and locallized spall) coatings on AISI 651 alloy; and uncoated AISI 651 (which lost 6 percent by weight in 110-hours test exposure).

INTRODUCTION

Thermal reactors have been shown to be an effective means of reducing exhaust gas pollutants from automobile engines (refs. 1 to 4). A thermal reactor is essentially a close-coupled afterburner (installed in place of the cast-iron exhaust manifold) into which air is injected to oxidize unburned hydrocarbons and carbon monoxide. From the oxidation process, the core of the reactor reaches temperatures from about 870° to 1040° C (1600° to 1900° F) under normal driving conditions. This high temperature oxidizing environment in combination with high-velocity corrosive constituents in the exhaust gas presents a severe environment for reactor materials. Of particular concern has been the inability of low-cost, abundantly-available, materials to survive the reactor environment (ref. 3).

The program reported herein is a part of an overall evaluation of potential materials which may be suitable for thermal reactors. The main purpose of this work is to determine which materials can best endure the severe operating conditions encountered in thermal reactors. This report covers preliminary screening test results on full-size thermal reactors fabricated from 14 different materials. The materials evaluated included ferritic and austenitic iron-base alloys, nickel-base superalloys, and metallic and ceramic coatings on iron-base alloys. The reactors were subjected to 200 hours of cyclic automobile engine operation on an engine dynomometer, motoring-engine test stand. Comparison was made of the effects of short-cycle testing (10 min at peak temperature) and long-cycle testing (2 hr at peak temperature). The bulk of the reactor tests were conducted with the long cycle which consisted of 2 hours at a reactor-core-metal temperature of approximately 1040° C (1900° F) with a cooldown to less than 70° C (160° F) for a total time of 20 minutes before return to peak temperature each cycle. This test procedure provided 83 cycles in a total test time of 200 hours of engine testing per reactor. The reactor test material components were evaluated on the basis of core weight change, dimensional stability, and coating life. Materials with the best performance are currently being endurance tested under simulated driving conditions (to be reported at a later date).

This program is being conducted in cooperation with the office of Air Programs of the Environmental Protection Agency. The reactors were fabricated and tested under a NASA contract with the Teledyne-Continental Motors Division of Teledyne Industries, Incorporated, Muskegon, Michigan, under the direction of Mr. L. D. Carufel, Project Manager, and Mr. J. Sykes, Designer and Test Engineer.

MATERIALS AND PROCEDURE

Materials Evaluated

The fourteen materials selected for this screening phase of the program were as follows:

1. Armco 18S/R
2. GE 1541
3. AISI 651
4. AISI 310
5. Nimonic 75
6. Hastelloy X
7. AISI 651 + NC-9 coating
8. AISI 651 + NC-630 coating
9. AISI 651 + S-6100 M coating
10. AISI 651 + 418A coating
11. Incoloy 800 + Al coating
12. AISI 651 + Cr-Al coating
13. AISI 310 + Cr-Al coating
14. AISI 651 + Sermetel J coating

Compositions of the base alloys by alloy type and nominal values of major constituents are given in table 1. The coatings are proprietary and are listed in table II by nominal composition, application method, amount of coating deposited (based on core weight and thickness before and after coating), coating vendor, and trade name. The key designation assigned to each coating in table II is referenced throughout this report for coating identification.

Selection of the alloys to be evaluated in this program was made to include austenitic stainless steels, ferritic alloys, and nickel-base superalloys. The material thicknesses (table I) were about 1.6 mm (0.063 in.) for the ferritic iron alloys and the austenitic iron alloy selected for coating (AISI 651) and were about 1.25 mm (0.049 in.) for the other austenitic iron and the nickel-base alloys. The AISI 310 alloy, Hastelloy X, one of the ferritic alloys (Armco 18 S/R), and Al-coated Incoloy 800 had previously been evaluated for automobile thermal reactors (ref. 3) and were known to have potential for the application. Nimonic 75 was included as representative of a conventional high-nickel superalloy with a relatively simple composition. As representative of a higher aluminum content, ferritic alloy GE 1541 was selected because of availability in large sheets required for reactor fabrication.

The selection of materials was based in part on a preliminary survey of materials properties and performance in nonautomobile high-temperature applications and in part on test exposures of coupons exposed in thermal reactors (refs. 3, 5, 6, and 7). Consideration was given to alloys more readily available and/or potentially less-costly than superalloys and to include alloys with greater hot strength than ferritic iron alloys. Several commercially-available coatings generally used only on nickel-base superalloys were evaluated on austenitic iron-base alloys for their ability to provide improved oxidation protection of these alloys.

Engine Test Procedures

All aspects of the experimental program were directed toward the goal of obtaining reproducible test conditions for the screening test purposes. For operation of identical engines using identical loads and test cycles, insofar as practical, the principal parameter of concern was the reactor core temperature. Accordingly, the test reactors were designed and operated by injection of air to provide control of the peak-measured material temperature for exposure conditions provided by engines on test stands which were operated using predetermined test cycles. The peak controlled material temperature was established at 1040° C (1900° F) for each of two test cycles. A description of the parameters of interest in each of several task areas is provided in the following subsections.

Test reactor design and operation. - The test reactors designed and fabricated for this study are illustrated in figure 1. The reactor core, liner, and port tubes were fabricated from the materials to be evaluated. The metallic cores were all fabricated by the same vendor, but the coatings were applied by the particular coating developer, where possible. The outer housing is a casting of Meehanite type SF 60 alloy. Each exhaust port is ducted to flowing air supplied by the air pump furnished with the test engine as well as supplemental air to raise core temperature to the 1040° C (1900° F) specified for the test. At 3200 rpm the combined reactor injected air was about 16 percent of the engine total air flow with a 12:1 air/fuel ratio (the air pump alone provided 10 percent). A second air ducting system which would provide abundant cooling (quench) air to prevent overtemperature was seldom, if ever, used.

The regulation of the reactor air supplies was an automatic operation. Input to a temperature set-point air controller was provided by the highest of four spring-loaded thermocouples in contact with the reactor cores at a point opposite each exhaust port (fig. 1(b)). In a typical 2-hour uniform temperature exposure, it was noted that supplemental air was automatically introduced about 10 to 20 times an hour. With each introduction of supplemental air, the temperature climbed steadily until an overtemperature set point of about 1055° C (1925° F) interrupted supplemental air flow and the temperature declined to about 1025° C (1875° F) at the lower set point. The 30° C (50° F) temperature variation in the highest measured core-metal temperature was considered to be acceptable for this type of test. Also, the temperature variations along the length of the reactor core ranging from about 940° C (1720° F) to 1050° C (1920° F) were considered acceptable for this type of test (see fig. 2 for typical temperature distributions in each reactor core). Cyclic test operation (described in the Screening Test Conditions section) was accomplished by automatically controlling engine conditions and air flow to the reactors.

Engine-dynamometer test stands. - The automobile engine selected to provide the test conditions was a 1970 Ford 302 V-8 engine with a 210-horsepower rating at 4400 rpm. The engines were equipped with an air pump for injection of air into standard cast-iron manifolds. In place of each of the two standard manifolds, a test reactor was installed for the reactor materials evaluation as illustrated in figure 1(b). To provide simulation of operation under vehicle loading, an eddy-current dynamometer, a clutch, and a reverse-rotation slave engine (standard 210 horsepower, 302 Ford V-8 marine powerplant) were installed on each of three test stands as shown in figure 3.

To regulate engine operation for test cycling, a punched paper-tape controller provided throttle adjustment using pneumatic actuators and clutch engagement and throttle control for the motoring engine. Automatic recycling was accomplished by inputs provided by the time-controlled punched tape. For the purposes of the screening tests reported herein, the engines were operated using commercially-available leaded gasoline (about 2 to 3 grams tetraethyl lead per gallon and 0.2 theories of phosphorous).

Screening test conditions. - Two types of tests to be used in sequence were planned to provide screening of materials. Engine operating conditions were adjusted to provide the test conditions and peak cycle temperature for the two screening tests which are outlined as follows.

Accelerated creep and corrosion (AC/C) test (see fig. 4(a)). -

(1) Heat to 1040° C (1900° F) in 5 minutes
(2) Hold at 1040° C (1900° F) peak core temperature for two hours
(3) Idle engine 5 minutes with quench air to <200° C (<390° F)
(4) Continue air cooling with engines off for 15 minutes to <70° C (<160° F)
(5) Repeat cycle to attain at least 80 cycles in 200 hours of engine operation

Thermal cycle (TC) test (see fig. 4(b)). -

(1) Heat to 1040° C (1900° F) in 2 minutes or less
(2) Hold at 1040° C (1900° F) for 10 minutes
(3) Cut fuel flow
(4) Cool for 3 minutes (with air injection and engine motoring) to <370° C (<700° F)
(5) Continue cooling at least 5 more minutes to <150° C (<300° F)
(6) Repeat cycle for up to 200 cycles

The purpose of the accelerated creep and corrosion (AC/C) tests, as the name implies, was to provide sufficient time at temperature each cycle to allow a significant amount of corrosion and creep to occur. It was planned that the AC/C test would allow screening of the oxidation resistance of coated materials with minimum effects from rapid cycling. The best materials which survived AC/C testing would then be subjected to the thermal cyclic (TC) testing to determine if the candidates were prone to failure in rapid cycling. In the course of the program, however, it was determined that the AC/C test resulted in greater core weight changes, core distortion, and visually-apparent damage than did equivalent test durations using the TC test. This is illustrated in figure 5 which compares the weight changes of two core materials under both test conditions. For these reasons, use of the TC test was discontinued. All other test reactors were evaluated using the AC/C test cycle.

The engine operating parameters which provided the 1040° C (1900° F) reactor core temperature for the AC/C test cycle are tabulated below:

Engine parameter	At peak cycle temperature	At idle
rpm	3200	800
Dynamometer load N-m (ft-lb)	135.58 (100)	40.67 (30)
Ignition timing	6° retard	6° retard
Fuel flow kg/hr (lb/hr)	20 - 23 (45-50)	----------------
Manifold vacuum kN/m^2 (in. Hg)	37.1 - 40.5 (11-12)	64.2 (19)
Reactor annulus pressure kN/m^2 (in. H_2O)	1.5 - 2.5 (6-10)	0.5 - 0.75 (2-3)

At no time during AC/C testing was spark outage observed. Presumably, even with the injection of excess air (for cooling) by the automatic quench air system, the reactor core temperatures would climb excessively if an unburned fuel/air mixture were injected from one or more exhaust ports.

Reactor Evaluation Methods

The relative performance of the materials evaluated in this screening test was based on reactor core weight change, dimensional stability of the core, and visual appearance of coatings for the different coatings evaluated. A complete metallurgical analysis of the reactors was not included in this part of the study but is planned for later work. Test reactor cores were weighed on a beam balance before test exposure and after approximately 12, 25, 50, 100, 150, and 200 hours of accelerated corrosion and creep testing. The nominal weight of the cores prior to testing was 950 to 1300 grams. For reporting purposes, core weights are given to tenths of grams but should be considered to be significant to about ±2 grams. The surface area of all reactor cores was about 1650 cm^2. The comparison of alloys and coatings was based on mg/cm^2 weight change. Coating systems were considered to have failed with a loss of 20 mg/cm^2 which exceeds the deposited weight of all except the NC-9 and NC-630 nickel-chromium systems (see table II). Uncoated metals were considered to have failed with greater than 36 mg/cm^2 loss in the 200-hour test (approximately 5 percent core weight loss in perhaps 15 percent of the expected reactor life).

Reactor cores were dimensionally measured prior to test and after approximately 15, 75, 100, 150, and 200 hours of exposure. The core diameter dimension was obtained at right angles at both ends of each core during test and at the point of maximum deformation at the completion of the test.

RESULTS AND DISCUSSION

Uncoated Reactors

Reactor core weight change data obtained in the AC/C tests for the uncoated reactors are shown as a function of test time in figure 6. Dimensional changes of the test reactor cores after 100 and 200 hours of testing are presented in table III along with corresponding weight changes for these test intervals. Cores of GE 1541, Armco 18 S/R, AISI 310, and Hastelloy X exhibited adequate oxidation resistance for the 200-hour test. The AISI 651 core showed excessive oxidation in 100 hours and was removed from the test. The Nimonic 75 alloy reactor also was removed from test at the completion of 100 hours, since less expensive alloys were shown to be as adequate.

In most cases the weight and dimensional changes of the reactor cores appear to represent the performance of the material being tested. But for Hastelloy X, the lower half of the reactor liner (which surrounds the core) was found to be locally cracked at three of four sites where the exhaust gases impinge on the liner as they leave the core. The largest of these sites is locally bulged and penetrated, as is evident in figure 7. Part of the reactor liner support strip cracked and became detached at the tack welds. (The support strip failure was caused by poor weld penetration for this particular assembly and is not considered to be a weld problem associated with the material.) Although the reactor core is not generally bulged or distorted (approximately 1.3 percent maximum after 100 hours with no subsequent change in the second 100-hour exposure, as indicated in table III), the Hastelloy X material is apparently not as satisfactory for the reactor application as some of the other materials with unaffected liners.

Both ferritic alloy cores were found to have excellent oxidation resistance, but both were somewhat bulged after 200 hours of test exposure (see fig. 8). The GE 1541 ferritic alloy exhibited the least distortion with about half as much as Armco 18 S/R in the major diameter (table III). Indentation of the Armco 18 S/R core by the spring-loaded thermocouple (fig. 8(b)) and the missing "foot" on the GE 1541 core (fig. 8(a)) are minor indicators of possible strength and weldability deficiencies associated with these ferritic alloys. The 4.7 percent diametral growth of the GE 1541 core is considerably less than might be expected, however, and should be fully acceptable since the end-supported core design used for this program allows for some growth and sag, as well as loss of "support" feet, without resulting in exhaust blockage.

The 3 percent weight loss of the AISI 310 austenitic iron alloy core after the 200 hours of exposure (table III) is not excessive if it is considered that 160 hours of the 200-hour test exposure is at speeds equivalent to 113 km/hr (70 mph) which might convert to about 10 percent of the 160 000 km (100 000 mile) lifetime of a typical automobile.

From these screening test results, the GE 1541 alloy appears to be the best candidate for further evaluation as a reactor material in engine tests of longer duration. Armco 18 S/R and AISI 310 have potential for use in reactors and should be considered for further evaluation using heavier gage sheet. This would tend to minimize distortion of the Armco 18 S/R and to allow for the greater oxidation rate of the AISI 310.

Coated Reactors

The test results for the coated reactors are discussed by coating types below and are summarized in figure 9. Core distortion is given in table IV and is plotted in figure 10. Photographs of the exposed cores are given in figures 11 to 13. All data are for cores exposed for 200 hours using 2-hour AC/C cycles at 1040° C (1900° F). Reactor liners and port tubes of each of the coated materials evaluated were comparable to the cores in distortion and visual changes, except as noted in the discussion following.

Aluminide coatings. - The chromium-aluminum coatings on AISI 651 and AISI 310 stainless steel and the aluminum coating on Incoloy 800 either gained or lost less than 1 percent of the core weight in the 200-hour exposure. The weight change of these reactor cores is less than 20 percent of the weight of the deposited coating. Therefore, it is considered to be probable that none of the aluminide coatings have suffered either general or localized oxidation failure.

Of the three aluminide coating systems evaluated, it was noted that the Al/Incoloy 800 reactor showed blistering (wrinkling) on the exterior of the core at 150 hours. The blistering did not progress further, however, in the subsequent 50 hours of test exposure. A photograph of the reactor core is shown in figure 11(a). The Cr-Al/AISI 310 coated reactor core grew 4 percent in length and 17.5 percent in diameter after 100 hours of exposure. After 200 hours of exposure, the length increased 7 percent and the reactor distorted elliptically with the core diameter increasing as much as 25 percent (see table IV and figs. 10 and 11). This gross distortion was not exhibited by either of the two other aluminide-coated systems, which were on thicker material. In 200 hours, the Cr-Al/AISI 651 coated core grew 3 percent in length and 5 percent in diameter as compared with 2.7 percent in length and 7 percent in diameter for the Al/Incoloy 800 system. The Cr-Al/AISI 651 core also had the least general distortion and had the least net bow (warp of core) as the result of the exposure. Thickness measurements made at the impingement areas opposite the exhaust ports show no locallized loss of thickness to indicate coating failure.

It is concluded that the aluminide coatings generally appear to provide excellent oxidation resistance for the 200-hour test duration and that they were perhaps 20 percent consumed overall in these tests. A selection was made of Cr-Al/AISI 651 stainless steel for future endurance testing because it was equally oxidation resistant and had the least core distortion of the three aluminide systems evaluated.

Glass-ceramic coatings. - The weight changes of the two glass-ceramic coatings are plotted against test cycles and time in figure 9. From table II, the nominal weight of S-6100M coating applied to AISI 651 was about 7 to 12 mg/cm^2. The weight loss equivalent to the amount of coating deposited was exceeded between the 100- and 150-hour inspection after which the reactor core failed at approximately the same rate as uncoated AISI 651 alloy. The entire coating appears to be gone except for residual sheen which can be seen in an area to the right-hand side of figure 12(a). The S-6100M coated reactor core has distorted into an oval shape in the direction perpendicular to that observed with the bare metals.

The glass-ceramic developed by the National Bureau of Standards and designated A418A, was applied to AISI 651 in about the same thickness as the S-6100M coating. Although this coated reactor lost weight nearly equivalent to the original coating weight, the core visually appears to have little or no coating depletion outside and a general change of coating appearance of the core interior (fig. 12(b)). The coating, which was originally a glossy bright green, as-fired, (as is the S-6100M coating) had dulled and turned yellow-green on the reactor core interior except at the raised surface of the longitudinal weld at the side of the core which is still bright green (but cannot be seen in fig. 2). Although the coating had probably partially devitrified; no failed areas were evident. The interior of the liner surrounding the core provided further encouragement in that the exposed surface, which was scoured on the S-6100M coated reactor, was still bright green on the A418A coated components. Since both coatings are of glass-ceramic composition, local abrasion and loss of coating are evident at sites where contact of supporting or chattering parts has occurred (at port tubes, etc.). Because the glass-type coating systems are potentially the least expensive of the coating-types evaluated here and the A418A coating evidenced excellent protection of the substrate and was free from distortion, it was concluded that an A418A glass-coated reactor should be considered for further evaluation.

Slurry-metal coatings. - Other coatings which are relatively inexpensively applied are the sprayed or dipped slurry metallic coatings. Two of the selected slurry-metal coatings are of nickel-chromium composition while the third is an aluminum coating containing approximately 10 weight percent silicon. The performance of these coating systems is given in table IV and figures 9 and 10 along with the other coatings data. Photographs after test exposure are given in figure 13.

The Sermetel J coating on AISI 651 (which might be expected to perform as well as the aluminum diffusion coatings because of the aluminum content) lost weight equal to the coating failure criteria in less than 100 hours of exposure. Between 100 hours and 200 hours exposure, the weight loss rate was equivalent to bare AISI 651 (slope of curve, fig. 9). The coated reactor core at the 100-hour inspection was observed to have a hole approximately 2.5 cm (1 in.) by 1.9 cm (3/4 in.) which enlarged (see fig. 13(a)) in 200 hours. A second hole along the weld seam, 0.32 cm (1/8 in.) wide and 2.54 cm (1 in.) long, also was observed after 200 hours of exposure.

Because the holes and the loss of a 10 to 15 gram core support foot cannot account for the nearly 10 percent weight loss after 200 hours of exposure, it is tentatively concluded that the Sermetel J coating has suffered general failure in addition to the failure at local areas.

The NC-630 coating on AISI 651 lost weight after the 200-hour exposure equivalent to about one-third of the estimated amount of coating deposited. Such a loss must be attributed to general coating depletion in addition to losses accountable to small areas of localized spall and subsequent oxidation of the exposed substrate (fig. 13(b)). By comparison to NC-630, the NC-9 coated material performed with weight loss equivalent to only 20 percent of the coating deposited. For the NC-9 system, also, general depletion outside as well as inside of the core appears to be the probable cause of the measured weight loss (only one spall area is observable, fig. 13(c)). It should be noted that both the NC-9 and NC-630 coating systems were heavily deposited with at least 10 percent of the final core weight consisting of coating. Therefore, either of these coated reactors might survive considerably longer exposure with probable progression to local failure only in those areas where spalling is evident. Such a progression to total penetration of 1.7 mm (0.067 in.) material was noted at one of the core support feet of the NC-630 coated reactor (see inset, fig. 13(b)). The "spot failure" was only 0.32 cm (1/8 in.) by 0.64 cm (1/4 in.). But this is of some significance to a reactor designer as well as to the general consideration of using coated materials in a reactor, not because of the failure of the foot (even uncoated reactors had feet missing), but because the failure was in a high-temperature stagnation zone, downstream of the exhaust flow. In such a zone, the coating may spall and the base metal will fail not only on the foot but on the core beneath the foot (as is evidenced by the crater in the core material and the penetration of the foot from the underside outward). Thus, a substitution of support materials would probably not have prevented this highly localized initiation of failure of the coated metal core. Sharp edges, heat concentration, and areas of coating buildup are all to be avoided in design applications where coated materials are to be used. The oxidation of the NC-9 coating on the core interior of the thermocouple well is noted in figure 13(c), as an example of heat concentration and deterioration which has not yet progressed to probable coating spall.

For the conditions of this program, the NC-9 system is the best of the three metallic slurry coatings evaluated, and it is to be included in further testing.

FUTURE WORK

Further evaluation of the best materials from the screening tests is now being started using an endurance test cycle that simulates driving conditions. Full-size reactors are being used in the engine-dynamometer tests with the peak reactor temperature controlled at 1040° C (1900° F). In these endurance tests, each reactor can be exposed up to 1000 hours with about 45 percent of the exposure to the peak temperature. Both ferritic iron alloys, GE 1541 and Armco 18 S/R, are being tested to obtain additional performance data on these potentially low-cost materials. AISI 310 will be included in these tests. Also, the best coatings from the screening tests (Cr-Al, Ni-Cr, and a glass ceramic) will be further evaluated. And a nickel-base alloy, probably Inconel 601, will be included to give a further basis for comparative performance. Inconel 601 is lower in cost than the other nickel-base alloys used in this program and appears to have excellent corrosion resistance for the thermal reactor application based on a related coupon evaluation study (ref. 7).

SUMMARY OF RESULTS

Fourteen materials including iron- and nickel-base alloys and several coatings were evaluated in full-size thermal reactor screening tests. The reactors were subjected to 200-hour, cyclic heating on an engine test stand. Each test cycle consisted of a 2-hour exposure at a peak measured metal temperature of approximately 1040° C (1900° F) and a cooldown to less than 70° C (160° F) before return to peak temperature. Although the number of cycles is low, the reactor exposure results indicate that the test cycle is severe. The reactor materials were evaluated on the basis of weight change, dimensional distortion, and coating life. The results are summarized as follows:

1. The best of these candidate reactor materials evaluated in the screening tests were:

Iron alloys (uncoated)

Type	Nominal composition
GE 1541	Fe-15Cr-4Al-1Y
Armco 18 S/R	Fe-18Cr-2Al-1Si-0.5Ni-0.4Ti
AISI 310	Fe-25Cr-20Ni-0.25C

Coatings on AISI 651 alloy (19-9DL stainless steel)

Type	Designation	Nominal composition
Cr-Al (pack)	Cr-Al	M(Al+Cr)[a]
Ni-Cr (slurry)	NC-9	Ni-18.5Cr-10Si
Glass ceramic (slurry)	A418A	70(FRIT)-3Cr_2O_3-6 Clay

[a] M = substrate composition

All of these materials exhibited a maximum of 3 percent weight loss and less than 9 percent core distortion in the 200-hour screening test.

2. The performance of two ferritic iron alloys, GE 1541 and Armco 18S/R, was comparable in their oxidation-erosion resistance (both less than 1 percent weight loss) but notably different in core distortion. The GE 1541 alloy survived the full-term (200-hr) screening test with less than 5 percent swelling of the core and no elliptical distortion. Armco 18S/R, however, became elliptical in cross section with a -5 percent and +9 percent distortion. of the major axes.

3. AISI 310 was the better of two austenitic-iron alloys evaluated with 3 percent weight loss in 200 hours; whereas, the core of AISI 651 (19-9DL) alloy lost about 6 percent of its weight in only 100 hours of testing. Neither alloy had excessive distortion, although the AISI 310 core was 30 percent thinner than the core of AISI 651.

4. Of the several coating systems evaluated on the AISI 651 substrate, the Cr-Al pack coating caused the greatest reactor core distortion. The exposed core (with 1.6 mm thick substrate) grew 4.8 percent in diameter and 3 percent in length. And the same coating on thinner gage (1.2 mm) AISI 310 alloy distorted unacceptably: it grew 25 percent in diameter and stretched 7 percent in length. By comparison, an aluminum-coated Incoloy 800 alloy reactor of 1.3 mm thickness grew 7 percent in diameter and 3 percent in length.

5. Cores of both Nimonic 75 and Hastelloy X had less than 1/2 percent weight change after 100 and 200 hours of test exposure, respectively. However, the liner of Hastelloy X was found to be cracked and penetrated in areas of exhaust gas impingement.

6. None of the cores or liners of the best systems or liners of any materials other than Hastelloy X were penetrated in 200 hours exposure although hot spots were noted at the impingement zones on all liners tested.

7. For comparable test hours, the primary test cycle used in this study (2 hr at 1040° C (1900° F)) was found to be more severe in terms of weight loss than a short cycle with only 10 minutes at 1040° C (1900° F).

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TABLE I. NOMINAL COMPOSITIONS OF ALLOYS EVALUATED IN THERMAL REACTOR SCREENING TESTS.

Alloy Designation	Material thickness		Alloying Elements, Weight %											Alloy Vendor
	mm	inches	Fe	Cr	Ni	Mo	W	Ti	Al	C	Mn	Si	Other	
Armco 18 S/R	1.75	0.069	Bal.	18.0	0.50	—	—	0.40	2.0	0.05	0.50	1.0	—	Armco Steel Co.
GE 1541	1.60	0.063	Bal.	14.5	0.03	—	—	—	4.5	0.002	—	—	1.0 Yttrium	General Electric Co.
AISI 651	1.65	0.065	Bal	18.5	9.0	1.4	1.35	0.25	—	0.32	1.15	0.6	0.4 (Cb+Ta)	several
AISI 310	1.22	0.048	Bal	25.0	20.0	—	—	—	—	0.25	2.0	1.5	—	several
Incoloy 800	1.37	0.054	46	20.5	32	—	—	0.4	0.4	0.04	—	—	—	International Nickel Co.
Nimonic 75	1.35	0.053	5.0	19.5	Bal.	—	—	0.4	—	0.10	1.0	1.0	0.5 Cu	International Nickel Co. Ltd.
Hastelloy X	1.25	0.049	18.5	21.8	Bal.	9.0	0.6	—	—	0.10	1.0	0.5	1.5 Co	Cabot Corp.

TABLE II. - COATING SYSTEMS EVALUATED IN REACTOR SCREENING TESTS.

Coating Systems		Composition (nominal)	Application Method		Amount Deposited on Reactor Core			Coating Vendor (Coating Trade name)
Key	Substrate		liner	core	μm	mg/cm^2 (1)	mg/cm^2 (2)	
Cr-Al	AISI 651	M(Cr-Al)(3)	pack	pack	76.2	25.6	9.7	Alloy Surfaces (HI-15)
Cr-Al	AISI 310	M(Cr-Al)(3)	pack	pack	111.8	37.6	17.0	Alloy Surfaces (HI-15)
Al	Incoloy 800	M(Al)(3)	pack	pack	50.8	17.1	19.7	Alloy Surfaces (HI-15-12)
S-6100M	AISI 651	Mix A(4) + Frit A(5)	spray	dip	58.4	12.2	6.7	Wall Colmonoy (Solaramic S-6100M)
A418A	AISI 651	Mix B(6) + Frit B(6)	spray	dip	40.6	8.5	2.9	Lycoming Div. Avco (NBS A-418A)
NC-9	AISI 651	Ni-19Cr-10Si+B	spray	spray	63.5	45.5	28	Wall Colmonoy (Nicrocoat 9)
NC-630	AISI 651	Ni-17Cr-5Si-7$TiSi_2$-7TiN	spray	spray	228.6	164.0	66.4	Wall Colmonoy (Nicrocoat 630)
Sermetel J	AISI 651	Al-5Si	spray	dip	25.4	8.6	1.8	Teleflex, Inc. (Sermetel J)

Notes:

(1) based on thickness

(2) based on apparent weight gain

(3) M = substrate constituents

(4) Mix A

100 parts Frit A
10 parts TiO_2
5 parts Cr_2O_3
1/8 part KNO_3
60 parts H_2O
6 parts Clay

(5) Frit A

Proprietary.
Contains:
SiO_2, MgO, CaO,
ZrO_2, BaO, ZnO
+ other

(6) MIX B: 30 parts Cr_2O_3 + 6 parts clay + Frit B

Frit B (by weight %)
44 BaO - 38 SiO_2 -
1.5 ZrO_2 - 10 B_2O_3 -
6 ZnO - 1 Al_2O_3

TABLE III - DIMENSIONAL AND WEIGHT CHANGE OF UNCOATED THERMAL REACTOR CORES SUBJECTED TO THE ACCELERATED CREEP AND CORROSION TEST CYCLE.(1)

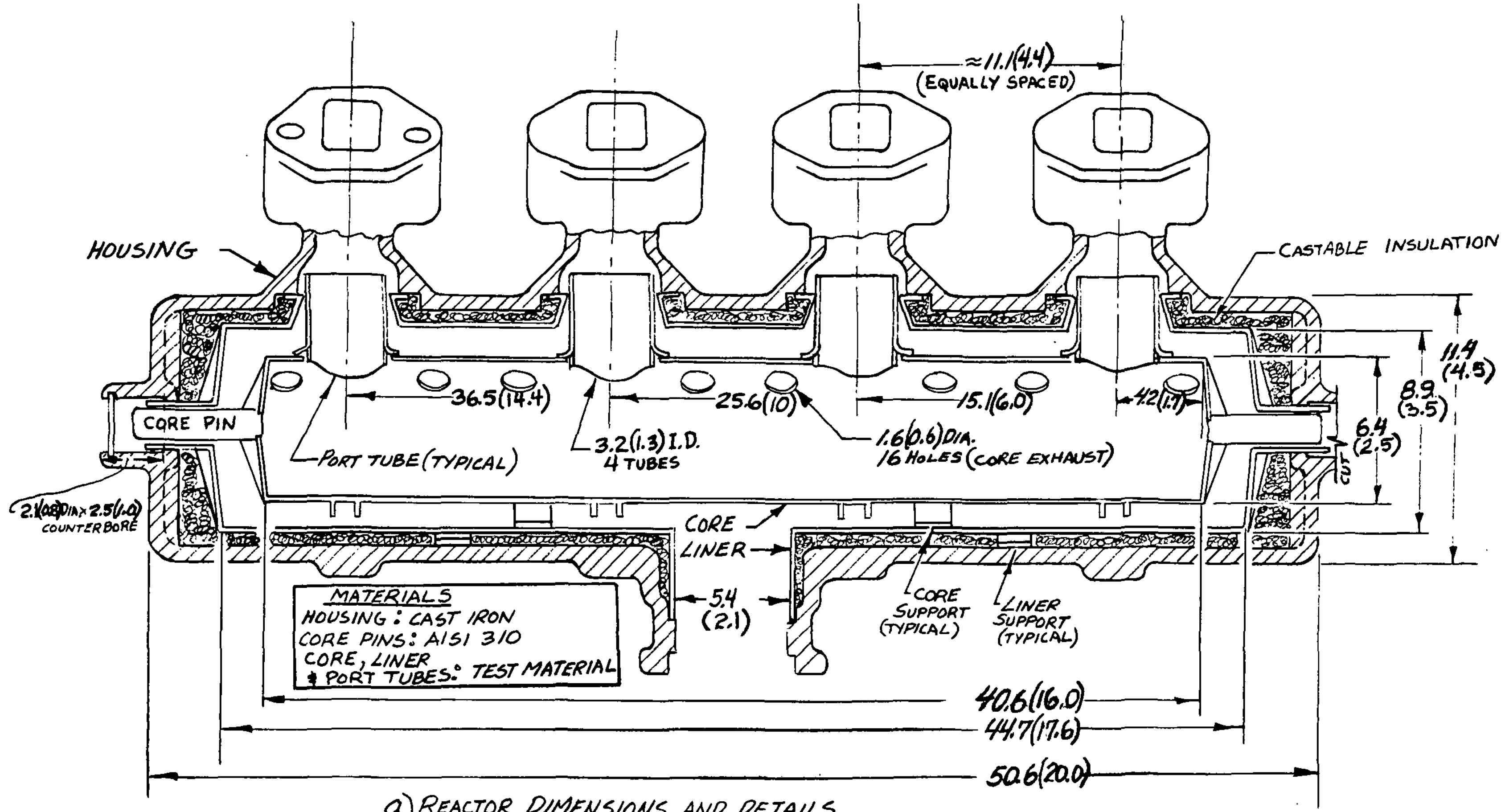
Thermal Reactor Core Material	Original Core			Reactor Cores after 100 and 200 hours of test							
	Length	Weight	End Dia.	Change in length, %		Change in weight, %		Change in end dia., %		Maximum/minimum dia.	Remarks
	cm.	gms	cm.	100	200	100	200	100	200	% in 200 hrs.	
Group 1. Ferritic Iron Alloys											
Armco 18S/R	42.261	1177.8	6.317 6.330	+0.30	+0.30	+0.44	+0.48	+1.5 -1.3	+1.7 -1.0	+9.0 -5.4	core distortion
GE 1541	42.344	1069.8	6.312 6.261	+0.07	+0.13	+0.06	-0.68	+0.40 -0.41	+0.5 -0.6	+4.7@port	
Group 2. Austenitic Iron Alloys											
AISI 651	42.431	1206.0	6.335 6.335	+0.22	—	-6.3	—	+0.52 -0.76	—	—	excessive weight loss
AISI 310	42.342	901.7	6.299 6.274	+0.10	+0.26	-1.5	-3.0	+0.44 +0.73	+0.8 +0.6	No bulge	
Group 3. Nickel-base Alloys											
Nimonic 75	42.278	1048.7	6.304 6.317	+0.14	—	+0.48	—	+1.01 -0.60	—	—	still good @100 hrs.
Hastelloy X	42.281	951.3	6.292 6.320	+0.13	-0.10	+0.74	+0.45	+1.25 -0.64	+1.30 -0.64	+1.01@ port	Reactor liner of Hastelloy X penetrated.

(1) Cyclic exposure: 2 hrs. at 1040°C followed by 20 min. forced cooling to <70°C and 5 min. return to 1040°C each cycle.

TABLE IV. - DIMENSIONAL AND WEIGHT CHANGE OF COATED THERMAL REACTOR CORES SUBJECTED TO THE ACCELERATED CREEP AND CORROSION TEST CYCLE.(1)

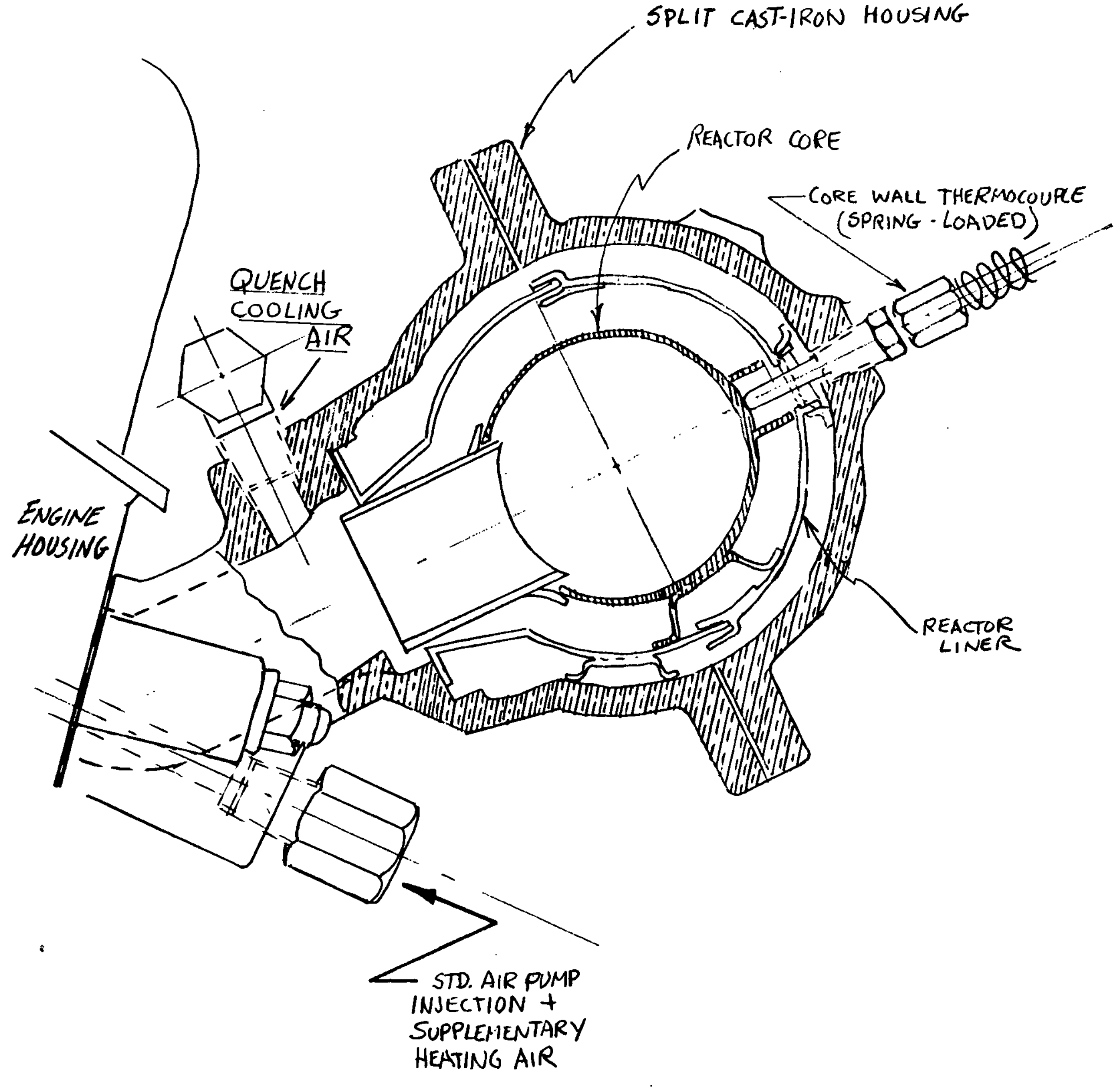
Coating/ Substrate	Original Core			Core after 200 test hours			
	Length cm.	Weight gms.	End diameter cm.	Change in Length %	Change in Weight %	Change in end dia. %	Maximum/ minimum dia. %, location
			Group I. Aluminide coatings				
CrAl/AISI 651	42.271	1222.8	6.299 6.299	+2.94	-0.26	+3.3 +0.65	+4.8, end port
CrAl/AISI 310	42.291	930.3	6.287 6.355	+7.25	+0.40	+11.9 +5.6	≈0 +24.8, between ports
Al/Incoloy 800	42.083	1050.5	6.299 6.299	+2.74	-0.24	+3.31 +1.21	+7.6 -2.9, 3" from end
			Group II Glass-ceramic coatings				
S-6100M/AISI 651	42.329	1217.6	6.284 6.335	+0.12	-8.3	+0.61 +0.36	-5.0 +3.1, between ports
A418A/AISI 651	42.268	1210.8	6.335 6.294	+0.31	-0.61	+0.68 -0.27	No bulge
			Group III Slurry-metal coatings				
NC-9/AISI 651	42.228	1249.0	6.327 6.309	+0.22	-0.64	-0.24 +0.16	+0.64, end port
NC-630/AISI 651	42.367	1315.0	6.421 6.350	+0.28	-2.36	+0.87 +0.84	No bulge
Sermetel J/AISI 651	42.329	1208.8	6.317 6.358	+0.82	-9.27	+1.33 -0.44	—, hole in core

(1) Cyclic exposure: 2 hrs. at 1040°C followed by 20 min. forced cooling to <70°C and 5 min. return to 1040°C each cycle.



a) Reactor dimensions and details.

Figure 1. Materials evaluation test thermal reactor. Dimensions are in centimeters (inches)



b) LOCATION OF REACTOR ON ENGINE AND LOCATION OF COOLING AND HEATING AIR.

Figure 1(cont.).-MATERIALS EVALUATION TEST THERMAL REACTOR

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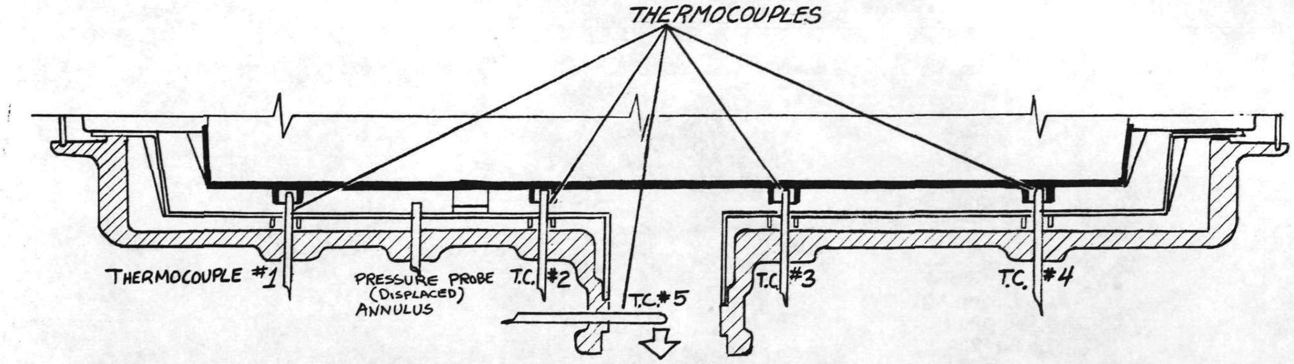


ENGINE R.P.M.	Reactor core temperature, °C (°F) (right and left bank reactors)								Exhaust temp. °C (°F)	
	#1 LEFT	#1 RIGHT	#2 LEFT	#2 RIGHT	#3 LEFT	#3 RIGHT	#4 LEFT	#4 RIGHT	#5 LEFT	#5 RIGHT
3200	965 (1770)	1040 (1900)	940 (1720)	1010 (1845)	1030 (1890)	1050 (1920)	1040 (1900)	1015 (1860)	990 (1810)	1015 (1860)
800	140 (290)	115 (240)	135 (275)	100 (215)	90 (190)	—	—	—	96 (205)	82 (180)

Figure 2:- Partial section of reactor showing temperature distribution for accelerated creep and corrosion (AC/C) test cycle.

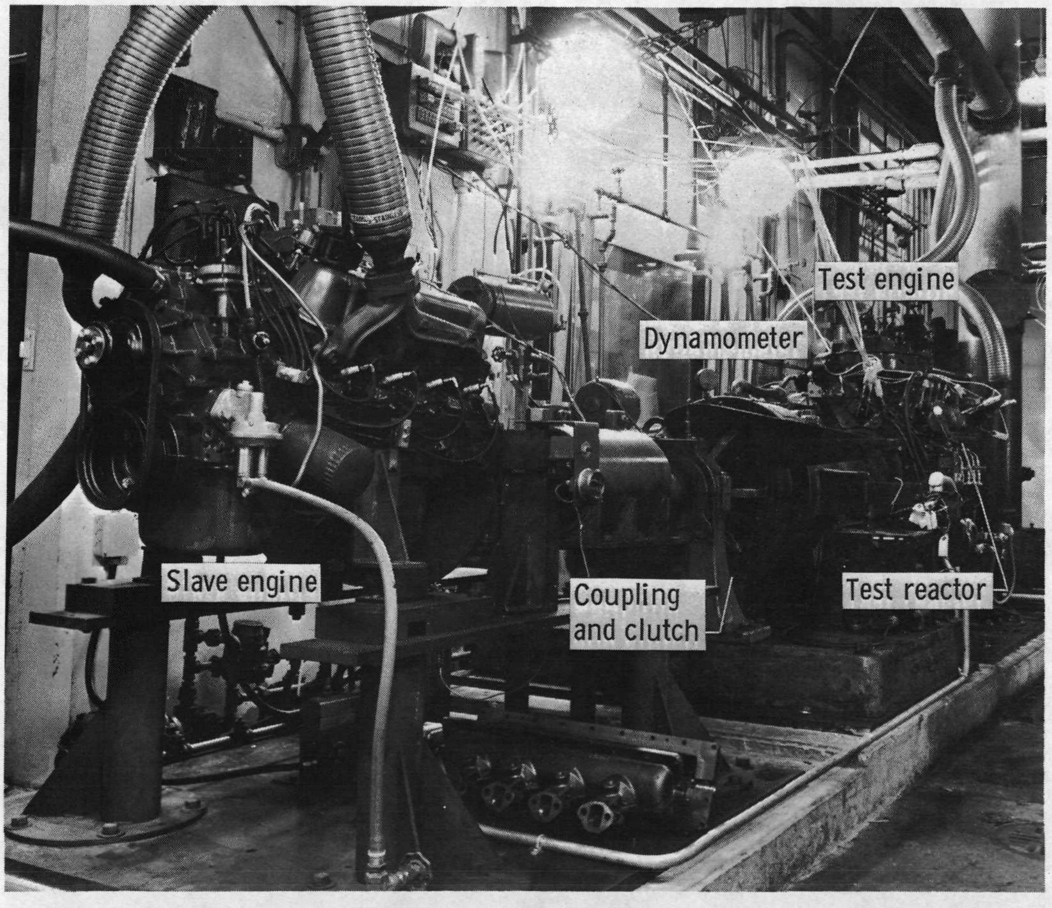
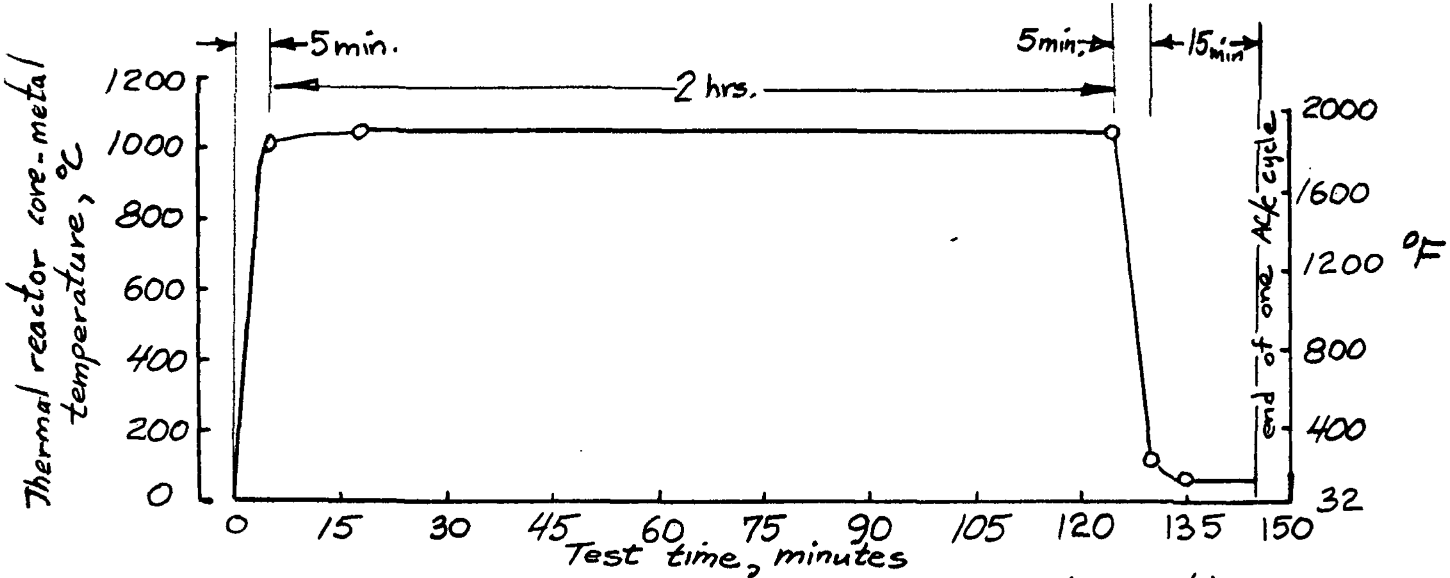
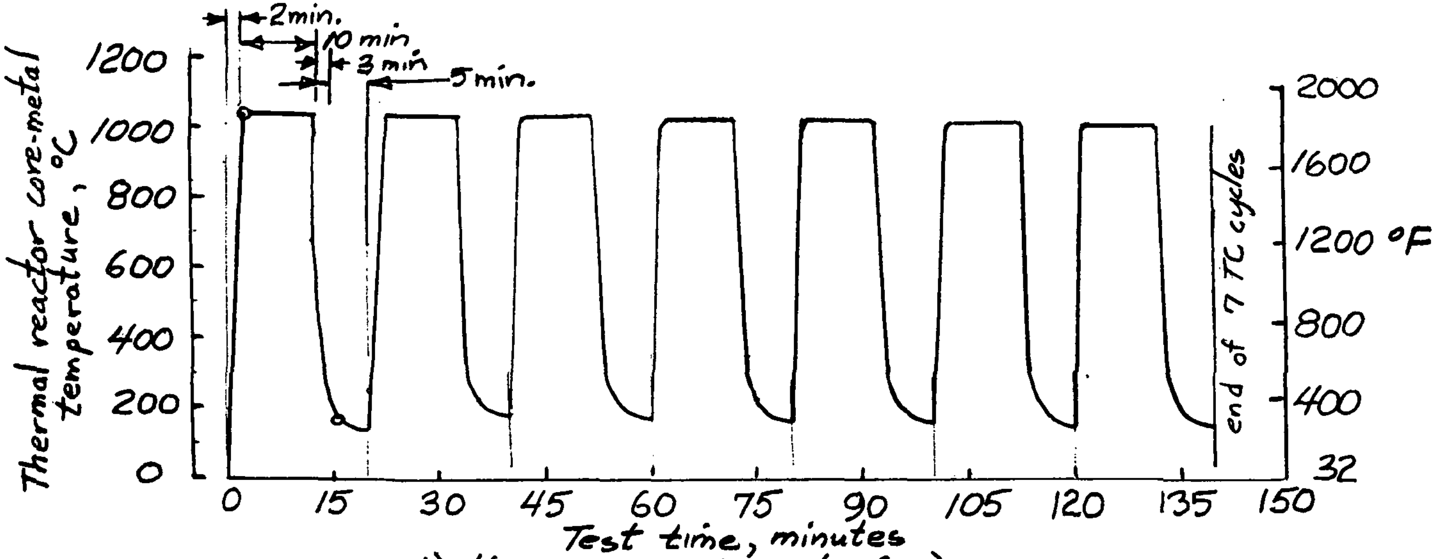


Figure 3. - Typical engine-dynamometer test stand installation showing slave-engine, eddy-current dynamometer, and test engine with reactors installed.



a.) accelerated creep and corrosion cycle (AC/C).



b) thermal test cycle (TC).

Figure 4.—Diagrams of screening test cycles.

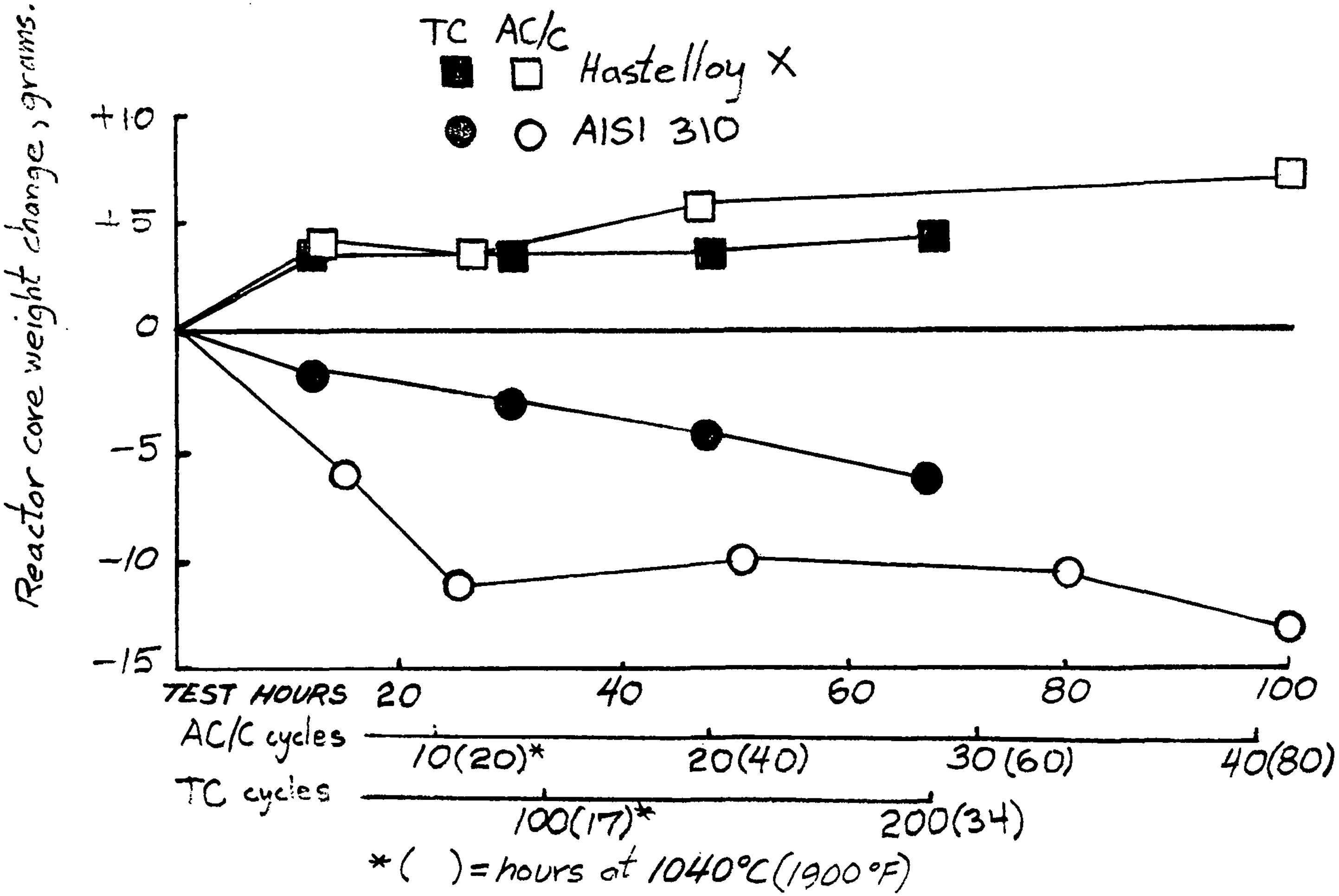


Figure 5.- Comparison of materials performance in accelerated creep and corrosion (AC/C) and thermal cycle (TC) screening tests.

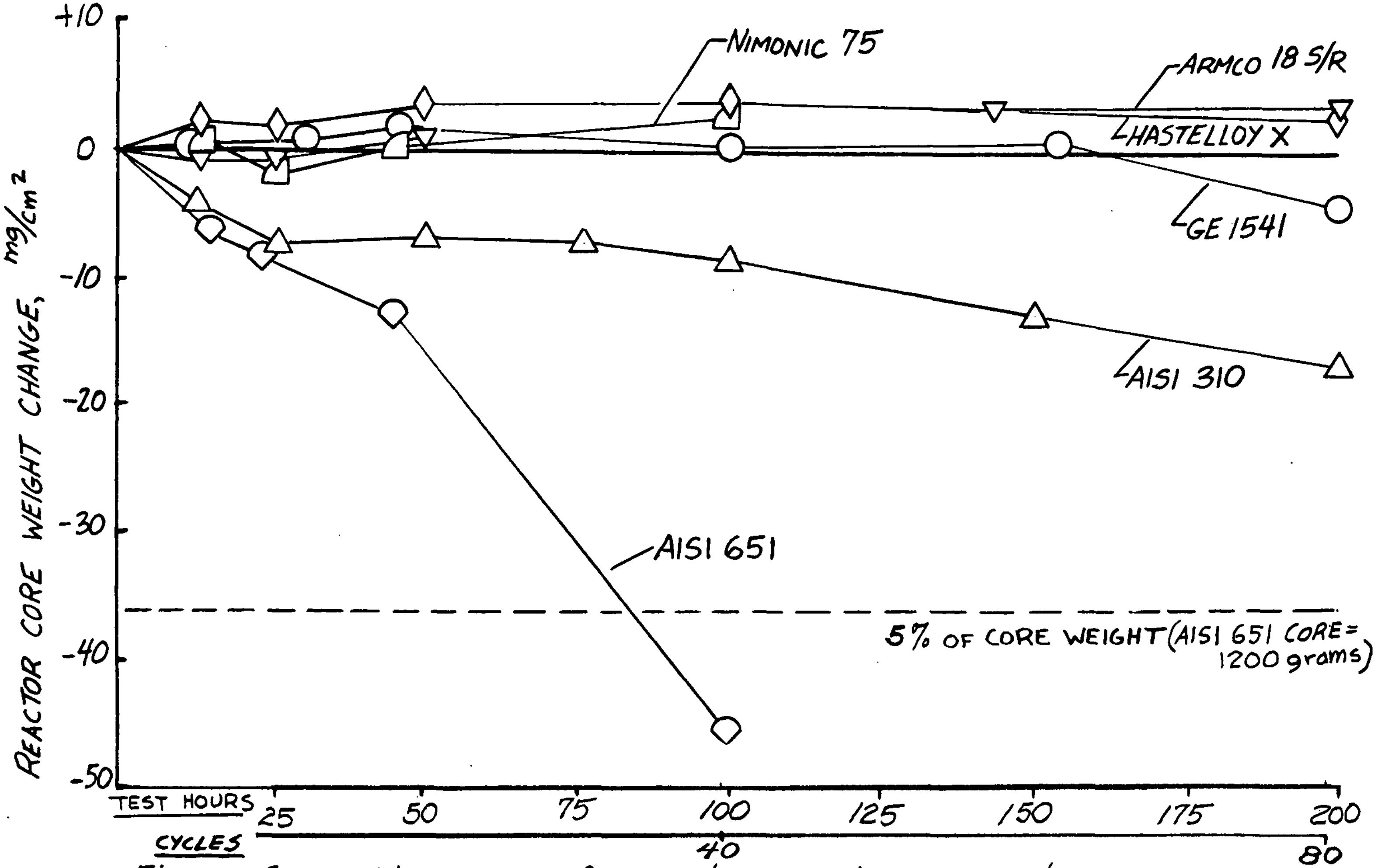


Figure 6.- Weight change of uncoated reactor cores during 200 hours of accelerated creep and corrosion (AC/c) screening tests. Cycle consisted of 2 hour exposure at 1040 °C (1900 °F) followed by 20 minute forced cooling to less than 70 °C (160 °F) and 5 minute return to peak measured reactor core metal temperature.

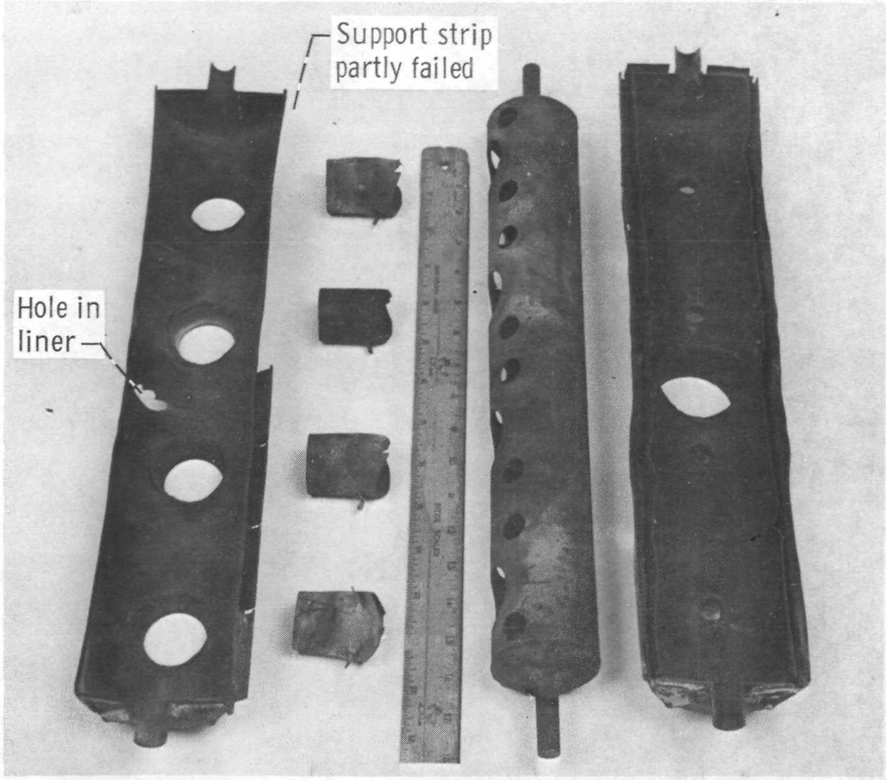
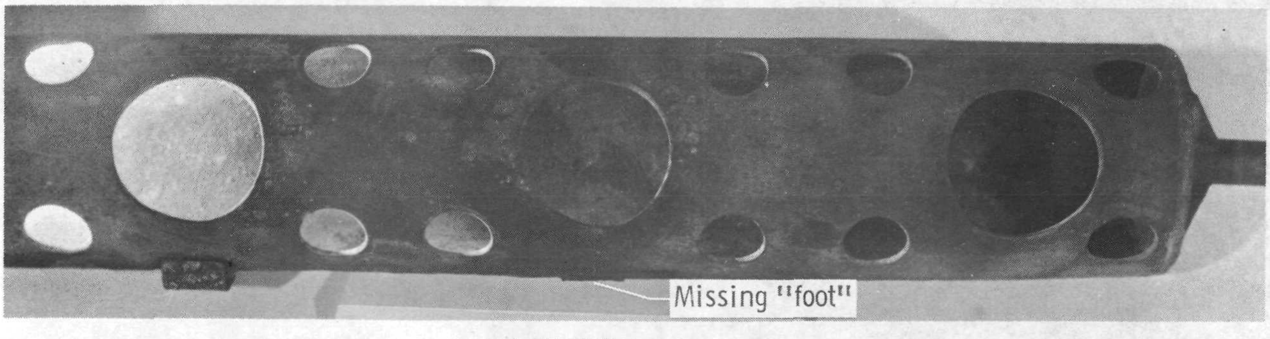
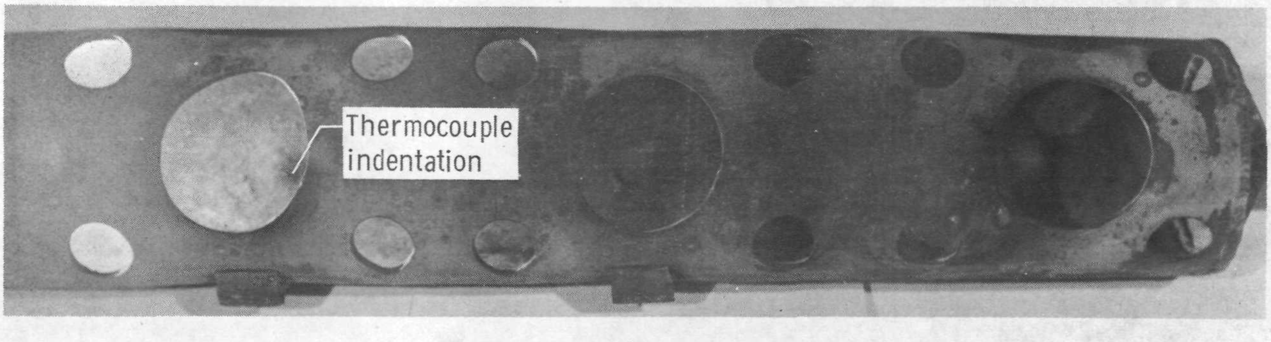


Figure 7. - Hastelloy X reactor components after 200 hours accelerated creep and corrosion testing (83 test cycles).



(a) GE 1541 reactor core.



(b) Armco 18 S/R reactor core.

Figure 8. - Ferritic iron alloy reactor cores of GE 1541 and Armco 18 S/R after exposure to 83 accelerated creep and corrosion test cycles (200 hours total exposure).

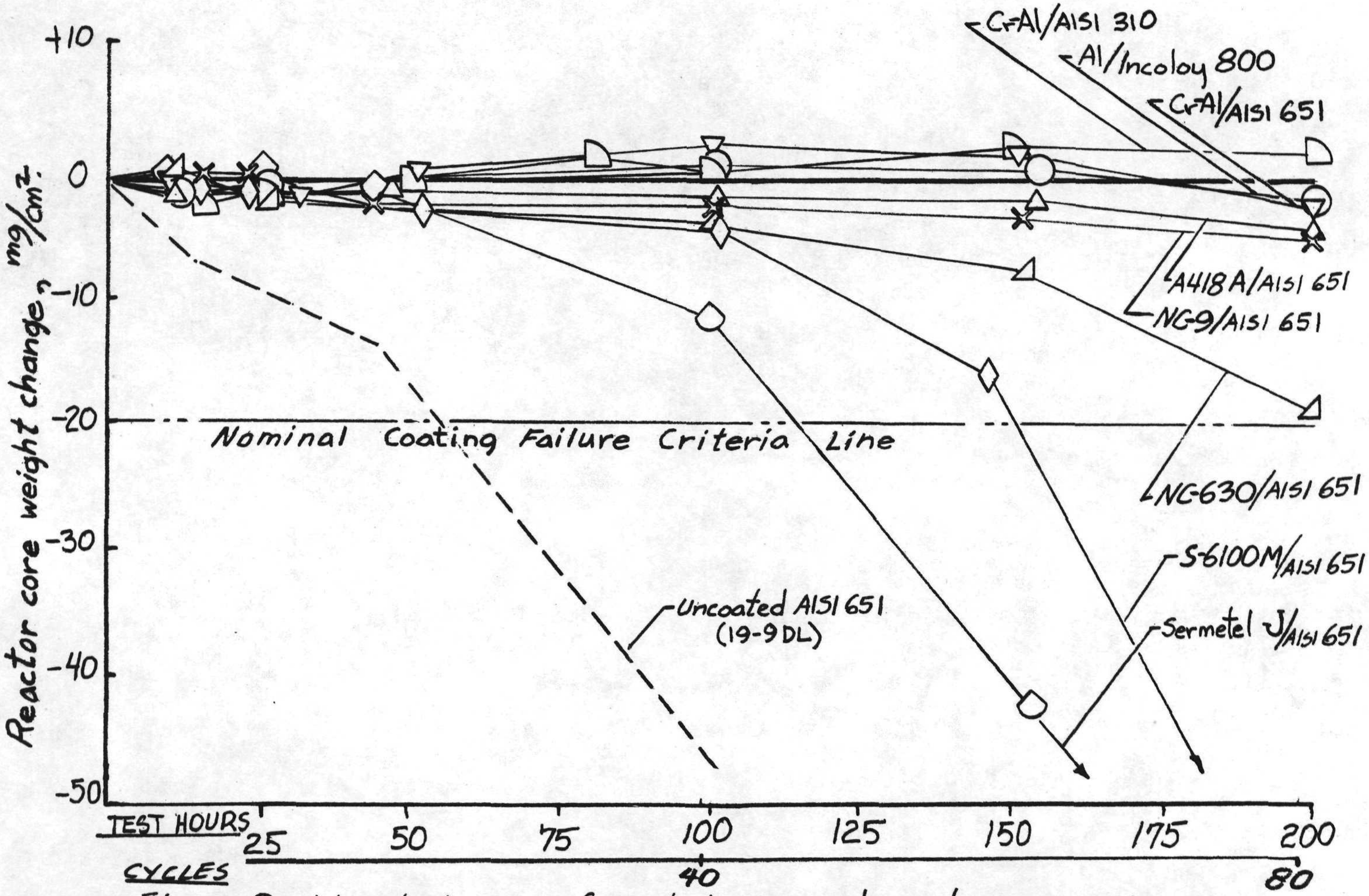


Figure 9.- Weight change of coated thermal reactor cores during 200 hours of accelerated creep and corrosion (AC/C) screening tests. Cycle consisted of 2 hour exposure at 1040 °C (1900 °F) followed by 20 minute forced cooling to less than 70 °C (160 °F) and 5 minute return to peak measured reactor core metal temperature.

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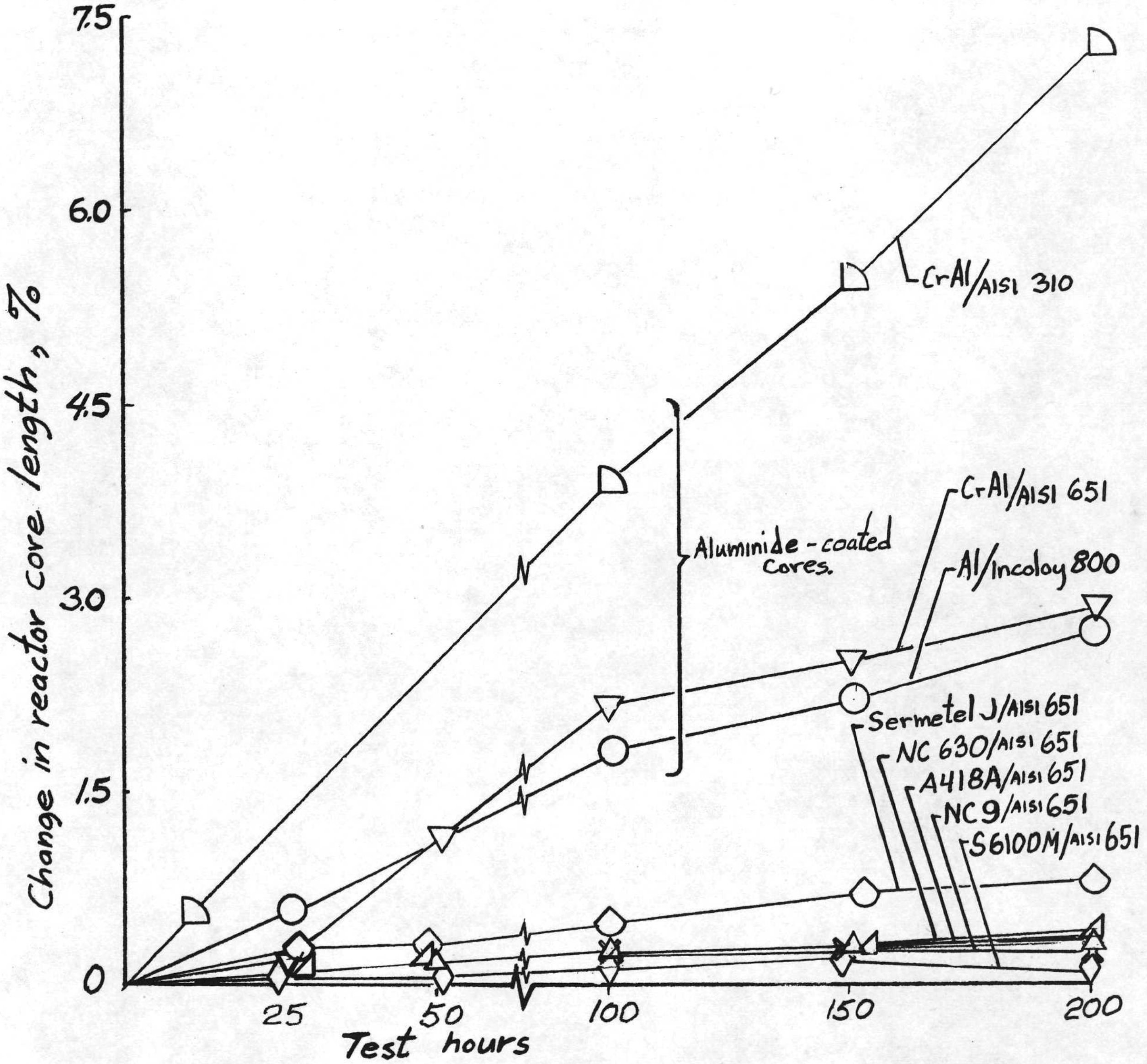
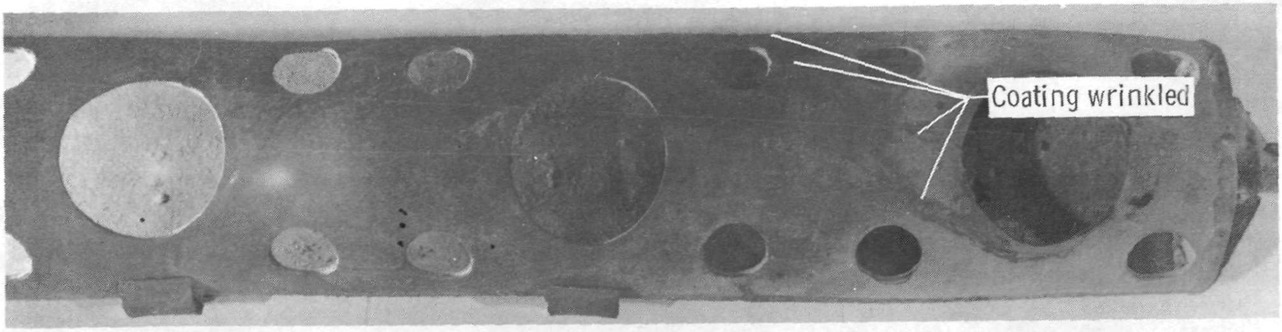
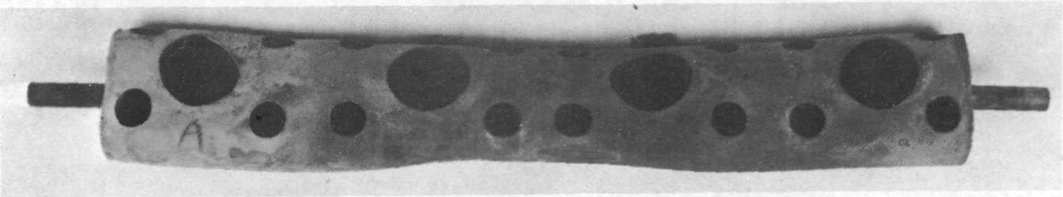


Figure 10.- Change in coated thermal reactor core length during 200 hour accelerated creep and corrosion (AC/C) screening tests.

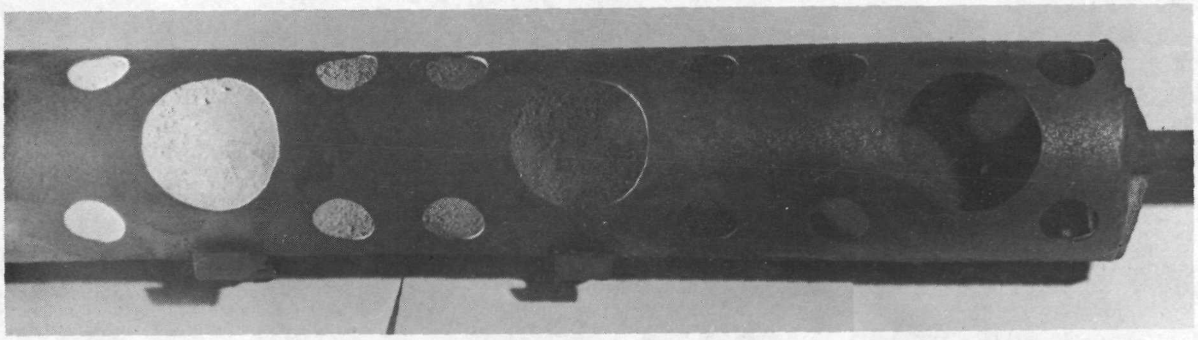


(a) Al-coated Incoloy 800 reactor core after 200 hours test exposure.

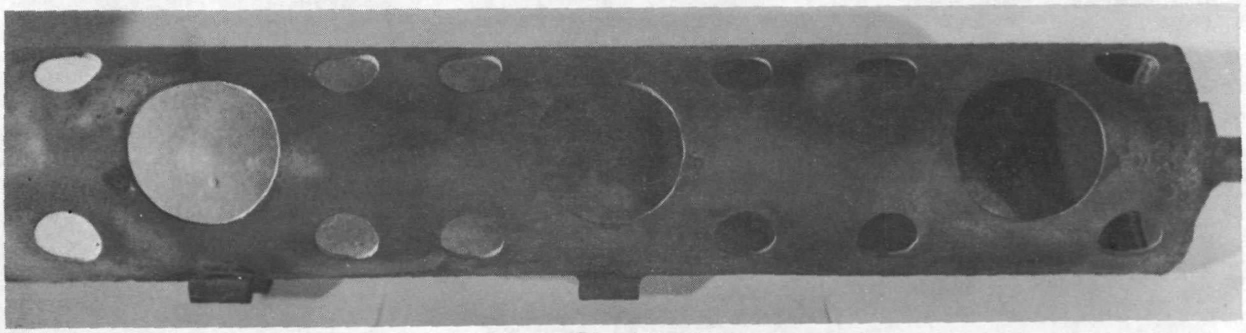


(b) Cr-Al-coated AISI 310 reactor core after 200 hours test exposure. X1/3.

Figure 11. - Aluminide-coated Incoloy 800 and chromium-aluminide-coated AISI 310 reactor cores after exposure to 83 accelerated creep and corrosion test cycles (200 hours total exposure).

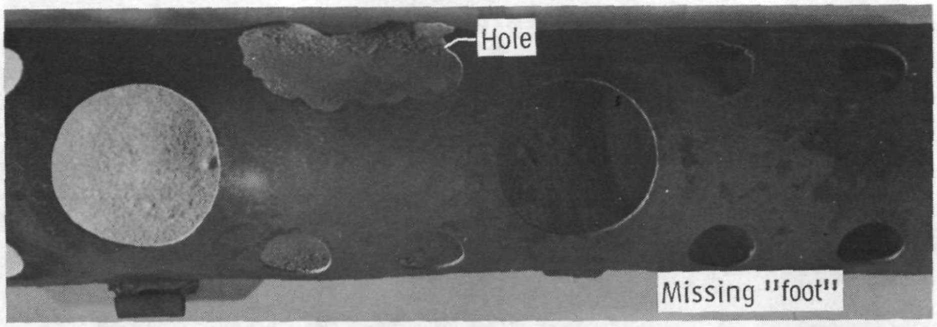


(a) Type S-6100M coated AISI 651 reactor core after 200 hours test exposure.

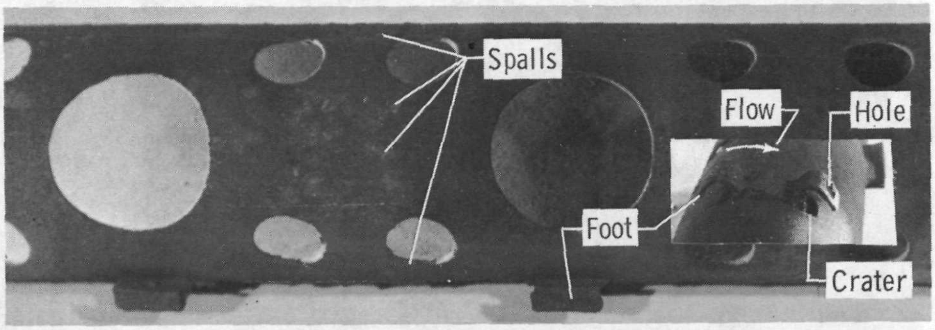


(b) Type A418A coated AISI 651 reactor core after 200 hours test exposure.

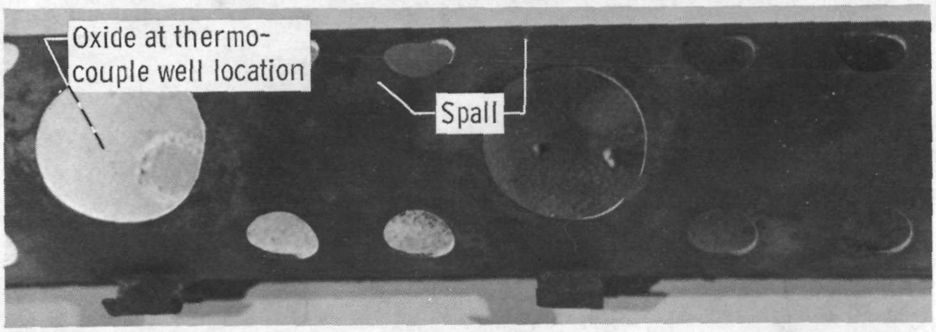
Figure 12. - Glass-ceramic type S-6100M and type A418A coated AISI 651 reactor cores after exposure to 83 accelerated creep and corrosion test cycles (200 hours total exposure).



(a) Type Sermetel J coated AISI 651 reactor core after 200 hours test exposure.



(b) Type NC-630 coated AISI 651 reactor core after 200 hours test exposure.



(c) Type NC-9 coated AISI 651 reactor core after 200 hours test exposure.

Figure 13. - Slurry metal types Sermetel J, NC-630, and NC-9 coated AISI 651 reactor cores after exposure to 83 accelerated creep and corrosion test cycles (200 hours total exposure).