N71-19700

NASA TECHNICAL MEMORANDUM

NASA TM X- 52976

NASA TM X- 52976

HOT CORROSION OF TD NICKEL AND TD NICKEL CHROMIUM IN A HIGH VELOCITY GAS STREAM

by W. A. Sanders and H. B. Probst
Lewis Research Center
Cleveland, Ohio

TECHNICAL PAPER proposed for presentation at
Tenth National Conference on Environmental Effects of Aircraft and
Propulsion Systems sponsored by the Department of the Navy
Trenton, New Jersey, May 18-20, 1971

HOT CORROSION OF TD NICKEL AND TD NICKEL CHROMIUM IN A HIGH VELOCITY GAS STREAM

W. A. Sanders and H. B. Probst
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

E-6183

William A. Sanders

Hubert B. Probst

William A. Sanders received his BS in Metallurgical Engineering from the Case Institute of Technology. He has been associated with the Lewis Research Center of the National Aeronautics and Space Administration as a materials research engineer for 15 years. He is presently involved in the evaluation of nickel- and cobalt-base superalloys and non-metallic materials for advanced turbine engine applications. His previous work has involved studies of the compatibility of refractory metals and compounds, and the fabrication and mechanical properties of refractory compounds.

Hubert B. Probst received his BS, MSE, and Ph.D. degrees in metallurgy from the University of Notre Dame, University of Michigan, and Case Institute of Technology respectively. He is presently Head of the Oxidation and Refractory Compounds Section at the National Aeronautics and Space Administration, Lewis Research Center. His current interests include developmental efforts in both metallic and non-metallic materials for applications in advanced gas turbine engines and re-entry thermal protection systems. Since joining NASA (formerly NACA) in 1954 his work has been devoted to high temperature materials including superalloys, refractory metals, and refractory compounds.

ABSTRACT

TD Nickel (2 percent Thoria-dispersed nickel) and TD Nickel Chromium (2 percent Thoria-dispersed Nickel-20 percent Chromium alloy) 1.5 mm (60 mil) sheet specimens were cyclically tested for susceptibility to hot corrosion in a Mach 0.5 gas stream of Jet A-1 fuel combustion products containing

2 ppm sea salt. Tests as long as 500 one-hour cycles between room temperature and specimen hot zone temperatures of 899° C (1650°F), 982° C (1800° F), and 1149° C (2100° F) were performed. Evidence of hot corrosion was found for both materials in the 899° C (1650° F) and 982° C (1800° F) tests, but not at 1149° C (2100° F). It was concluded that because of high metal thickness losses neither alloy in sheet form is suitable for long time engine application in a hot corrosion environment at temperatures of 982° C (1800° F) or above.

SUMMARY

An investigation was conducted to determine the susceptibility of TD Nickel and TD Nickel Chromium 1.5 mm (60 mil) sheet to hot corrosion in a simulated gas turbine engine environment. A Jet A-1 fuel-compressed air burner was used to produce a Mach 0.5 hot gas stream to which could be added a synthetic sea salt solution. Cyclic tests varying from 100 to 500 hours were conducted at atmospheric pressure at specimen hot zone temperatures of 899° C (1650° F), 982° C (1800° F), and 1149° C (2100° F) with and without injection of 2 ppm synthetic sea salt solution. Susceptibility to hot corrosion was evaluated by weight change, metal thickness loss, x-ray diffraction, electron microprobe, and metallographic analyses.

Hot corrosion attack was found for both materials in the 899° C (1650° F) tests. Although metal losses for TD Nickel Chromium were not excessive in the 899° C (1650° F) test [approximately 0.025 mm (1 mil) per side in 500 hours] metallographic and microprobe analyses showed that hot corrosion attack was present. For TD Nickel, hot corrosion attack did not occur in the 899° C (1650° F) hot zone but rather in a region where the temperature was only 807° C (1485° F). At temperatures of 982° C (1800° F) and above, neither material in sheet form is suitable for long time engine applications without a protective coating. At 982° C (1800° F), both alloys give evidence of hot corrosion attack and exhibit excessive metal thickness losses; for example, TD Nickel Chromium lost 0.21 mm (8.1 mils) per side after 500 hours exposure.

At 1149° C (2100° F), no evidence of hot corrosion was observed; however, again, both alloys suffered excessive metal losses. TD Nickel Chromium, for example, lost 0.1 mm (4 mils) per side after 200 hours exposure.

There is some evidence that TD Nickel suffers premature fracture due to the combined effects of hot corrosion and alternating stresses.

INTRODUCTION

The dispersion strengthened alloys, TD Nickel (2 percent Thoria-dispersed Nickel), and TD Nickel Chromium (2 percent Thoria-dispersed Nickel-20 percent Chromium alloy) are of particular interest for high temperature application. These alloys exhibit melting temperatures over 110° C (200° F) higher than cast and wrought nickel-base superalloys, and as a result of the fine Thoria (ThO_2) dispersion are metallurgically stable above 1150° C (2100° F). Although TD Nickel and TD Nickel Chromium are not as strong as nickel-base superalloys below 1090° C (2000° F), they are stronger than most superalloys at this temperature and exhibit superior retention of strength at even higher temperatures. Because of these attributes, both TD Nickel and TD Nickel Chromium would appear to have a potential as materials for certain hot components such as stator vanes of gas turbine engines.

The operating environment in an aircraft turbine engine is, of course, an oxidizing one and this environment may contain sodium sulfate (Na_2SO_4) as a result of sea salt ingestion due to aircraft operation over oceans and along coastal regions. Some Na_2SO_4 is present in the ingested sea salt, but most of the Na_2SO_4 results from the reaction of the ingested sea salt with sulfur dioxide and sulfur trioxide (ref. 5). These gases are present as a result of the combustion of a jet fuel such as JP-5 which can contain up to 4000 ppm sulfur. A recent survey found the average sulfur content of JP-5 fuel to be 950 ppm (ref. 17). The presence of Na_2SO_4 is conducive to an accelerated attack which is called hot corrosion (ref. 13). The hot corrosion reaction is generally believed to occur rapidly due to the disruption of a normally protective oxide scale by the fluxing action of Na_2SO_4. A nickel-base superalloy showing hot corrosion attack would typically exhibit a heavy surface layer of oxides, an alloy-depleted nickel matrix region with intermingled oxides, and below this level a matrix containing chromium and nickel sulfides (refs. 4, 13, and 16).

In spite of the apparent potential of TD Nickel and TD Nickel Chromium and the importance of the hot corrosion problem, there is very little information available on the behavior of these two alloys in a hot corrosion environment. Data reported in a manufacturer's brochure (ref. 3) indicated that TD Nickel bar stock lost considerable weight in 100 hours at 1149° C (2100° F) in a hot corrosion environment but did not exhibit any signs of hot corrosion attack. Tests in a hot corrosion rig for five hours resulted in grain boudary sulfides in TD Nickel exposed at 1024° C (1875° F) and no evidence of hot corrosion attack in TD Nickel Chromium at about 1135° C (2075° F) (ref. 15). More information is available on the hot corrosion of pure nickel and nickel chromium alloys which do not contain the ThO_2 dispersion. Hot corrosion tests have been run by furnace heating of samples of these materials coated with Na_2SO_4, by immersing samples in molten Na_2SO_4, and by exposing samples in JP-5 fueled burner rigs with salt injection (ref. 1). In a recent investigation (ref. 10); pure nickel samples tested in a JP-5 fueled burner rig with 200 ppm salt injection disintegrated after 8 hours at 913° C (1675° F) and after 4 hours at 982° C (1800° F). Nickel-15.3 w/o chromium alloy samples tested for 100 hours in this same rig were attacked to depths of about 0.25 mm (10 mils) at 913° C (1675° F) and about 0.33 mm (13 mils) at 982° C (1800° F).

The program reported here was initiated to determine the suitability of TD Nickel and TD Nickel Chromium as materials for engine components in a hot corrosion environment. To this end we investigated the two alloys in sheet form, 1.5 mm (60 mil), under simulated gas turbine engine conditions, for long times (up to 500 hours).

A burner rig facility was employed for this evaluation. The rig was operated at a hot gas velocity of Mach 0.5 with Jet A-1 a kerosene-type fuel similar to JP-5 fuel. Jet A-1 contains 300 to 400 ppm sulfur. A synthetic sea salt solution could be added to the hot gas before it exited the burner and before the gas contacted the TD Nickel and TD Nickel Chromium sheet specimens. The salt addition resulted in a 2 ppm salt concentration in the hot gas stream. The materials were evaluated in six tests, three with salt and three without salt at 899° C (1650° F), 982° C (1800° F), and 1149° C (2100° F). Test durations varied from 100 to 500 hours. All tests were cyclic, i.e., after each hour at the test temperature samples were cooled to room temperature. The materials were evaluated on the basis of weight change, metal thickness loss, surface x-ray diffraction, metallography and electron microprobe.

MATERIALS

The compositional limits in w/o for the TD Nickel and TD Nickel Chromium materials used are as follows:

For TD Nickel (ref. 7):

ThO_2	1.8 to 2.6	Fe	0.05 max
C	0.02 max	S	0.02 max
Cr	0.05 max	Ti	0.05 max
Cu	0.15 max	Ni	by difference

For TD Nickel Chromium (ref. 6):

ThO_2	1.5 to 3.0
Cr	18.00 to 22.00
C	0.05 max
S	0.015 max
Ni	by difference

The test specimens were cut from 1.5 mm (60 mil) sheet as shown in Fig. 1. The 12.7 mm (0.50 in.) dimension was in the sheet rolling direction and the 76.2 mm (3 in.) long corners were beveled. After this preparation, the test specimens were cleaned in alcohol, dried, measured to the nearest 2.5 micrometers (0.1 mils) and weighed to the nearest 0.1 mg. The specimens were tested in the as-received condition, i.e., with ground surfaces.

APPARATUS AND PROCEDURE

A schematic of the high gas velocity cyclic hot corrosion apparatus is shown in Fig. 2 and has been described in detail in Ref. 9. The Jet A-1 fueled apparatus was operated at a combustion gas velocity of Mach 0.5 at the conditions given in Table I. Eight specimens at a time were rotated at 450 rpm in the air-rich combustion gas stream which contained 2 ppm synthetic sea salt. For the hot corrosion tests a commercial, granular sea salt mix was used with distilled water to prepare a solution which conformed to substitute ocean water specifications (ref. 2). A valve-controlled, pressurized flow of salt solution was fed into the burner and atomized with compressed air to effect mixing with the combustion gas before the gas left the burner.

For temperature calibration, a slip-ring assembly mounted in the lower end of the specimen holder shaft was used to provide an electrical circuit to a thermocouple mounted on a spare specimen. During tests, the temperature of the rotating specimens was monitored with an optical pyrometer. The burner gas temperature was controlled by an exhuast gas thermocouple downstream of the specimen. The nominal hot zone temperature of the specimens was maintained with ±8° C (±15° F).

In every test, the specimens were alternately heated for one hour at the test temperature and cooled for 3 minutes to room temperature. The materials were evaluated in six tests, three with salt injection, and three without at maximum specimen temperatures of 899° C (1650° F), 982° C (1800° F) and 1149° C (2100° F). All tests without salt injection were terminated at either 100 or 125 hours. At 25 hour intervals the samples were weighed, examined under a low power binocular microscope and photographed. The original intention was to reach a maximum exposure time of 500 hours in the hot corrosion environment; however, tests were often terminated at shorter times due to obvious rapid attack or fracture of samples. Some samples were removed from the tests short of 500 hours exposure for metallographic analysis. Such samples were sectioned transversely through the hot zone. Some longitudinal cross sections were also metallographically prepared. For the TD Nickel specimens an etchant consisting of 92 ml HCl, 5 ml H_2SO_4 and 3 ml HNO_3 was used. For the TD Nickel Chromium specimens an etchant to delineate chromium depletion regions as well as grain boundaries was used. This etchant consisted of 100 ml H_2O, 2 gm chromic acid and 10 ml H_2SO_4.

Selected metallographic samples were also analyzed by means of the electron microprobe.

RESULTS AND DISCUSSION

The principal types of observations made in this study were weight change, dimensional changes, macro appearance, and microstructure of samples. The results will be presented in that order. In addition, x-ray diffraction and electron microprobe were used to augment these observations and these data will be presented throughout the following sections as required.

Weight Change

899° C (1650° F) tests

The tests conducted at 899° C (1650°F) continued for approximately 500 hours. However, no meaningful weight change data could be obtained. All samples tested at 899° C (1650° F) with salt injection exhibited massive deposits, particularly in the cooler portions of the samples outside the direct path of the gas stream. These deposits precluded any consistent or meaningful measurements of weight change.

X-ray diffraction results indicated that these deposits were composed of Na_2SO_4 and the sodium oxide, Na_2O_2. The presence of Na_2O_2 is surprising since most investigators find another sodium oxide, Na_2O, in hot corrosion deposits. Some samples tested at 899° C(1650° F) also exhibit small glass nodules on their surfaces; these will be discussed in more detail later.

TD Nickel failed to reach the 500 hour target in the hot corrosion environment at 899° C (1650° F). Fracture of the one sample intended for 500 hour exposure occurred in 454 hours near the base where the temperature was about 807° C (1485° F).

982° C (1800° F) tests

The weight changes resulting from 982° C (1800° F) exposure are plotted in Fig. 3. TD Nickel underwent a continuous weight gain for 100 hours for both test conditions, with and without salt. The weight change data suggest that the salt addition had no effect on the behavior of TD Nickel at 982° C (1800° F) even though Na_2SO_4 was identified on the surface of these samples. However, there was an obvious difference in behavior in that TD Nickel samples tested under hot corrosion conditions (salt added) cracked after 25 hours exposure and fractured in 100 hours. Identical samples tested without salt showed no evidence of cracks after tests were terminated at 125 hours.

Weight change data for TD Nickel Chromium after 982° C (1800° F) exposure (fig. 3) suggest that the hot corrosion environment does cause accelerated attack. Samples tested with salt injection exhibited an ever-increasing weight loss with time compared to those tested without salt. Although not plotted, TD Nickel Chromium continued to lose weight at about the same rate to the termination of the hot corrosion test at 500 hours. Na_2SO_4 was found on the surfaces of the exposed samples.

1149° C (2100° F) tests

Fig. 4 shows weight change behavior resulting from 1149° C (2100° F) exposure. The data for TD Nickel indicate no difference between salt and no-salt exposure. The data for TD Nickel Chromium indicate that there might be an accelerating effect of the hot corrosion environment in relatively short times; however, this effect is diminished at longer times. At 125 hours the two data points for TD Nickel Chromium are coincident. The no-salt test was terminated after 125 hours, however, the TD Nickel Chromium sample in the hot corrosion test continued to lose weight until at 200 hours it had lost 1130 mg. X-ray diffraction indicated no Na_2SO_4 on the 1149° C (2100° F) samples, so it is doubtful that any hot corrosion occurred at this temperature.

At both 982° C (1800° F) and 1149° C (2100° F) in times up to 125 hours under both test conditions, TD Nickel exhibited weight gains while TD Nickel Chromium suffered weight losses (see figs. 3 and 4). TD Nickel did not spall under these conditions but TD Nickel Chromium appeared to spall in a continuous manner giving a fine flaky spall. X-ray diffraction results indicate the scales on TD Nickel Chromium are Cr_2O_3, NiO, and $NiCr_2O_4$ while only NiO was found on TD Nickel. These observed differences in spalling behavior can qualitatively account for the different weight changes; however, the loss of Cr_2O_3 from TD Nickel Chromium is also believed to contribute to the weight loss of TD Nickel Chromium. Cr_2O_3 is subject to evaporative loss by oxidizing further to the gaseous phase $Cr\ O_3$ (ref. 11). This evaporative loss can be accelerated by the high velocity gas stream.

Dimensional Changes

As alluded to above, weight change measurements alone are insufficient to give an accurate description of true material behavior. They can be misleading due to scale vaporization, scale spalling, or deposits other than scale occurring on sample surfaces. Thus, a valuable adjunct to weight change data is the direct measurement of the amount of metal consumed, i.e., change in the dimensions of metal samples.

The dimensional changes resulting from edge and side attack are summarized in Fig. 5 for both alloys and for the two test conditions (salt and no-salt) at the three test temperatures. Cases in Fig. 5 where edge loss is not specifically noted indicate no observable difference between edge and side losses.

899° C (1650° F) tests

At 899° C (1650° F) for times of the order of 500 hours, both alloys undergo relatively minor metal loss of about .05 mm (2 mils) per side or less in the hot corrosion environment. Also, there appears to be no accelerating effect caused by the salt additions.

982° C (1800° F) tests

At 982° C (1800° F) there is little difference in the metal loss from TD Nickel after 100 hours with or without salt. This agrees with the general weight change behavior of TD Nickel and does not account for the premature fracture of TD Nickel in the salt exposure. Thus, the premature fracture would appear to have resulted from a combination of the salt environment and the alternating stress imposed on the samples by the gas pressure.

The thickness change data for TD Nickel Chromium at 982° C (1800° F) show an accelerated attack in the hot corrosion atmosphere comparing the metal loss in 125 hours in the two environments. This is to be expected from the weight change results. The 982° C (1800° F) data for TD Nickel Chromium also show an important observation, i.e., the more severe attack of sample edges compared to the attack on the sides (see sketch in fig. 5). All evidence in this study indicates that this is most likely a geometrical effect and not related to alloy composition nor test environment. Corners are generally more vulnerable to attack, and in thin sheet we believe the attack at two adjacent corners has overlapped and is observed as a greater gross attack at the edge of the samples. The differences between edge and side attack are in some cases quite significant--e.g., after 500 hours in the hot corrosion environment at 982° C (1800° F) TD Nickel Chromium shows 0.21 mm (8.1 mil) per side metal loss while the edge loss is 0.62 mm (24.5 mils).

1149° C (2100° F) tests

Comparisons of metal losses for both alloys after 1149° C (2100° F) exposure indicate no accelerated attack due to the hot corrosion environment. This is in agreement with the weight change data and the absence of Na_2SO_4 on sample surfaces after 1149° C (2100° F) exposure.

Macro Appearance

899° C (1650° F) tests

Samples exposed at 899° C (1650° F) showed no obvious change in shape. The deposits in the cooler portions of these samples have already been mentioned. The longer time samples (200 hours or more) of both TD Nickel and TD Nickel Chromium exposed at 899° C (1650° F) exhibited what appeared as glass-like beads or nodules on their surfaces. As illustrated in Fig. 6, these beads were usually concentrated near the edges of the samples. They were definitely glass-like inasmuch as under a binocular microscope their surfaces were extremely smooth as fire polished glass; they were translucent, and their shape suggested viscous flow of a high surface tension material. Qualitative spectrographic analysis of these beads showed them to be very high in sodium. The major constituent of the glass is no doubt a sodium oxide since an oxide of sodium was identified in the deposit on these samples. To our knowledge, this is the first time a glassy phase has been observed as a result of hot corrosion reactions.

982° C (1800° F) tests

After 982° C (1800° F) exposure TD Nickel Chromium samples exhibited obvious dimensional changes as illustrated in Fig. 7. In the hot zone the edges of samples have recessed so much that it is obvious to the naked eye. This large edge recession is also reflected in the quantitative measurements of metal loss reported in Fig. 5 and discussed earlier. Similar recession were not noted in TD Nickel at 982° C (1800° F), probably due to the premature fracture that occurred in 100 hours.

1149° C (2100° F) tests

Edge losses after 200 hours exposure at 1149° C (2100° F) are illustrated in Fig. 7. The general appearance of TD Nickel Chromium samples throughout this study, as exemplified by those shown leads one to believe that spalling occurred in a rather uniform manner. Surfaces usually appeared somewhat flaky on a fine scale, and there was little evidence of any large pieces of scale spalling sporadically. TD Nickel spalled only after longer times (>125 hours) at 1149° C (2100° F). In this case the spalling was rather sporadic with sizeable pieces of oxide being lost as is clearly apparent from Fig. 7.

Microstructure

Microstructure examination was made of selected samples in an effort to find any microstructural features that would indicate the presence or absence of hot corrosion reactions.

899° C (1650° F) tests

The microstructure of TD Nickel after 454 hours of hot corrosion exposure at 899° C (1650° F) is shown in Fig. 8. The microstructure is rather straight forward showing the elongated TD Nickel grain structure and a thick NiO scale. There is no evidence of hot corrosion. Microprobe analysis showed a Th enrichment in the inner portion of the NiO layer in comparison to the Th present in the matrix. This has been observed before (ref. 14). This enrichment may not be real but just a reflection of the lower absorption coefficients of NiO compared to Ni. Thus the Th content could be identical in scale and metal but appear high in the oxide due to the lower absorption coefficient of NiO. The absence of Th in the outer scale may indicate that this scale was formed above the original metal surface, i.e., the ThO_2 particles are acting as inert markers remaining in their original positions in the metal. Such apparent Th enrichment in the inner scale was observed to varying degrees in all samples examined. Generally, the apparent enrichment was more definite in TD Nickel samples and less obvious in TD Nickel Chromium samples. This may be the result of some dilution of Th in scales on TD Nickel Chromium brought about by oxide reactions, e.g. spinel formation and positive volume change.

TD Nickel Chromium is shown in Fig. 8 after 500 hours hot corrosion exposure at 899° C (1650° F). The most obvious feature of its microstructure is a zone immediately beneath the scale that etches light. Microprobe analysis confirmed that this zone is a Cr depletion zone. Within this zone microprobe traces give sporadic points of high Cr and high S intensity. These no doubt correspond to the particles in the Cr depleted zone, and suggest they are a chromium sulfide. Thus, although metal thickness measurements gave no indication of hot corrosion in 125 hours at 899° C (1650° F) in TD Nickel Chromium (fig. 5), there is microstructural evidence that hot corrosion is occurring in TD Nickel Chromium at 899° C (1650° F).

Fig. 9 shows the microstructure of TD Nickel in the cooler zone where fracture occurred in the 899° C (1650° F) tests. The localized very heavy scales suggest a crack oxidizing as it propagates. This is further evidence that these fractures in TD Nickel are probably the result of combined stress and corrosion. Again this region of the sample was subjected to maximum bending stresses by gas loading. The higher magnification and etched microstructure in Fig. 9 shows a grain boundary phase present. This could be a nickel sulfide; however, microprobe analyses were inconclusive as to whether or not these regions contained S.

982° C (1800° F) tests

Fig. 10 shows the microstructures of TD Nickel Chromium after 500 hours of hot corrosion exposure of 982° C (1800° F). Both regions of the sample, i.e., side and edge show Cr depletion. Within these Cr depleted zones microprobe analysis shows definite high Cr and high S contents associated with the small gray particles. This indication of hot corrosion attack agrees with the weight change data (fig. 3), and metal loss data (fig. 5), which also show accelerated attack caused by the presence of sea salt.

Fig. 10 shows that the Cr depletion zone is considerably thicker in the edge regions of the sample than along the side of the sample. This may be related to the grain structure of the sample. All samples were cut so that the direction of the elongated grain structure was perpendicular to the leading and trailing edges. Thus the great number of grain boundaries intersecting the edge of the sample could have provided fast diffusion paths for S and/or Cr leading to the deeper Cr depletion zone.

Fig. 11 shows microstructures of TD Nickel after 100 hours of hot corrosion exposure at 982° C (1800° F). One view shows the microstructure in the hot zone while the other shows the fracture region. The hot zone shows porosity and some large, light appearing grains. Microprobe analysis suggests non-uniform Th distribution in this region and some indication of areas of high S content.

The microstructure of the fracture region shows a definite grain boundary phase appearing quite similar to that found near the fracture in TD Nickel samples tested at 899° C (1650° F) (fig. 9). Microprobe analysis of the fracture microstructure shown in Fig. 11 gives definite indications of regions of high S contents, indicating this grain boundary phase is a nickel sulfide.

1149° C (2100° F) tests

Fig. 12 shows the microstructures of both alloys after 200 hours of exposure in the hot corrosion environment at 1149° C (2100° F). The microstructure of TD Nickel shows no evidence of hot corrosion, however, a very thick NiO layer was formed. The TD Nickel Chromium microstructure shows the Cr depleted zone but no S was found by microprobe, thus there is no evidence of hot corrosion having occurred. The large particles in the surface region of the metal may be the result of internal oxidation and/or Kirkendall voids resulting from the Cr loss. The distinction between the two possibilities is difficult to make. Microprobe analysis was inconclusive whether or not the particles were high in Cr contents. Microprobe results do suggest that the minimum Cr content in this region is about 14 percent which would be somewhat high for internal oxidation to occur. Refs. 8 and 12 report internal oxidation can occur up to about 10 percent Cr.

SUMMARY OF RESULTS

In this investigation TD Nickel and TD Nickel Chromium sheet 1.5 mm (60 mil) samples were exposed to a simulated gas turbine engine environment. Cyclic tests were conducted at atmospheric pressure at specimen temperatures of 899° C (1650° F), 982° C (1800° F), and 1149° C (2100° F) with and without the injection of sea salt. The susceptibility of these materials to hot corrosion was judged by weight change, thickness change, visual appearance, metallography, microprobe, and x-ray diffraction. The results may be summarized as follows according to test temperature.

899° C (1650° F)

Combining evidence from metallography, x-ray diffraction, and microprobe it is concluded that TD Nickel Chromium suffered classical hot corrosion attack at 899° C (1650° F). However, thickness change data suggest that the metal loss resulting from this attack was not excessive, i.e., only about 0.025 mm (1 mil) per side after 500-hour exposure.

The results for TD Nickel are not as straightforward. Within the portion of sample at or near the 899° C (1650° F) temperature there was no evidence of hot corrosion and metal loss was not excessive. However, in a cooler region of the samples (807° C (1485° F)) there were indications of accelerated attack; a grain boundary phase was present, and Na_2SO_4 and Na_2O_2 were identified on sample surfaces.

982° C (1800° F)

At 982° C (1800° F) both alloys are subject to hot corrosion attack. In 500 hours TD Nickel Chromium losses were 0.21 mm (8.1 mils) per side and 0.62 mm (24.5 mils) per edge. These losses are considered to be excessive. The observed exaggerated edge losses are believed to be only a geometrical effect.

At 982° C (1800° F) TD Nickel suffered premature failure in 100 hours apparently as a result of the combined effects of hot corrosion and alternating stresses.

1149° C (2100° F)

After exposure at 1149° C (2100° F) for 200 hours there was no evidence to indicate hot corrosion had occurred on either alloy. The degree of oxidation attack is high, however. TD Nickel losses were 0.15 mm

(6 mils) per side and 0.23 mm (9 mils) per edge, while TD Nickel Chromium losses were 0.10 mm (4 mils) per side and 0.39 mm (15.6 mils) per edge.

CONCLUDING REMARKS

The primary objective of this study was to determine the suitability of TD Nickel and TD Nickel Chromium as materials for engine components in a hot corrosion environment. Prior testing by others of these materials in hot corrosion environments was very limited, although several hot corrosion investigations had been made on pure nickel and nickel chromium alloys which did not contain the ThO_2 dispersion. In these tests pure nickel and some plain nickel chromium alloys were found to undergo considerable hot corrosion attack.

In view of the behavior observed in this study it must be concluded that neither alloy in sheet form without some form of protection is suitable for long time engine applications in a hot corrosion environment at temperatures of 982° C (1800° F) or above. In order to use these alloys as engine components in this temperature regime, protective coatings or improved corrosion resistance brought about by alloy modifications would be required.

The metal loss measurements made in this study would seem to suggest that both alloys may have some potential for use at 899° C (1650° F).

The data showing exaggerated amount of attack at edges, may be of some significance. Edges of sheet components are often troublesome to coat and coated edges can display early coating failures. The observation in this study of exaggerated edge attack would suggest that continued attention should be devoted to solving the problems associated with coating edges. Also in the design of certain components it may be possible to thicken edges and/or apply generous radii to edges. Such approaches would lessen the vulnerability to exaggerated edge attack.

Results of this study suggest that TD Nickel might suffer early mechanical failure at relatively low stresses as a result of the combined effects of stress and hot corrosion. This possiblity of a stress-corrosion effect should be investigated. This also implies that hot corrosion testing of TD Nickel, and quite possibly other alloys, might be more relevant to engine development if such tests were conducted with an intentional stress imposed on samples.

REFERENCES

(1) American Society for Testing and Materials, Hot Corrosion Problems Associated with Gas Turbines, ASTM Special Technical Publication No. 421, 1967.
(2) American Society for Testing and Materials, Specification D1141-52, Substitute Ocean Water (1965).
(3) Berkley, S. G., Fansteel Metallurgy, No. 58 (April-May 1968).
(4) Bornstein, N. S., DeCrescente, M. A., Corrosion 26, 209 (1970).

(5) DeCrescente, M. A., Bornstein, N. S., Corrosion 24, 127 (1968).
(6) Fansteel Inc., Metals Division, Technical Data Bulletin S-TC-S-01-R-1, TD Nickel Chromium Sheet (December 1, 1969).
(7) Fansteel, Inc., Metals Division, Technical Data Bulletin S-TD-S-01-R-0 TD Nickel Sheet (April 1, 1969).
(8) Giggins, C. S., Pettit, F. S., Trans. AIME 245, 2495 (1969).
(9) Johnston, J. R., Ashbrook, R. L., NASA TN D-5376 (1969).
(10) Kaufman, M., ASM Trans. 62, 590 (1969),
(11) Kohl, F. J., Stearns, C. A., NASA TM X-52879 (1970).
(12) Kosak, R., Jr., Ph.D. Dissertation, Ohio State University (1969).
(13) National Materials Advisory Board, Publication NMAB-260 (May 1970).
(14) Pettit, F. S., Felten, E. J., J. Electrochemical Soc. 111, 135 (1964).
(15) Reid, J. A., Phillips Research Center, Bartlesville, Oklahoma, Letter Report No. Rd-899-64R.
(16) Sims, C. T., ASME Publication 70-GT-24 (May 1970).
(17) United States Bureau of Mines, Petroleum Product Survey, 1959-1968 (6).

TABLE I. - TYPICAL BURNER CONDITIONS FOR HOT CORROSION TESTS

[Specimen test cycle: 1 hour at maximum temperature,
3 minute forced air cool to room temperature]

Maximum specimen temperature, °C (°F)	899 to 1149 (1650 to 2100)
Burner gas temperature, °C (°F)	1316 to 1593 (2400 to 2900)
Gas velocity	Mach 0.5
Salt injection in burner gas	2 ppm
Burner pressure, MN/m^2 (psia)	0.12 (18)
Specimen rotational speed, rpm	450
Burner air flow, kg/sec (lbm/sec)	0.17 to 0.14 (0.37 to 0.32)
Cooling air flow, kg/sec (lbm/sec)	0.05 (0.1)
Air to fuel ratio	30 to 20
Burner nozzle diameter, cm (in.)	5.1 (2.0)

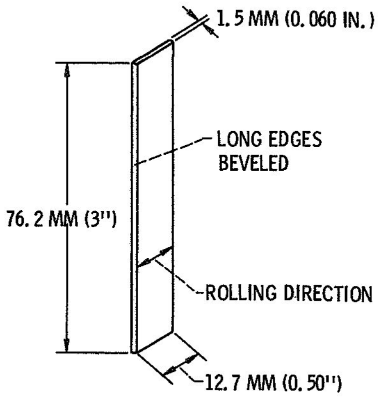

Figure 1. - Hot corrosion test specimen.

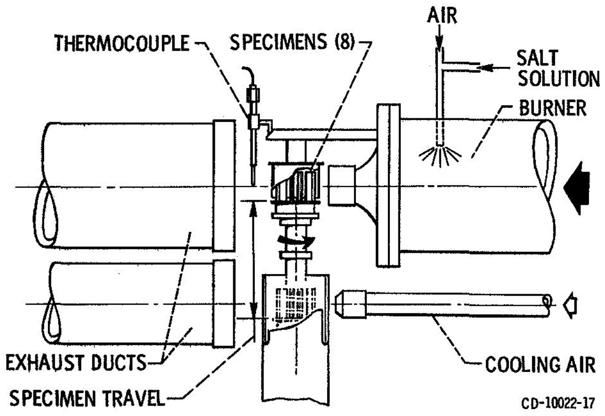

Figure 2. - High-gas-velocity hot corrosion apparatus.

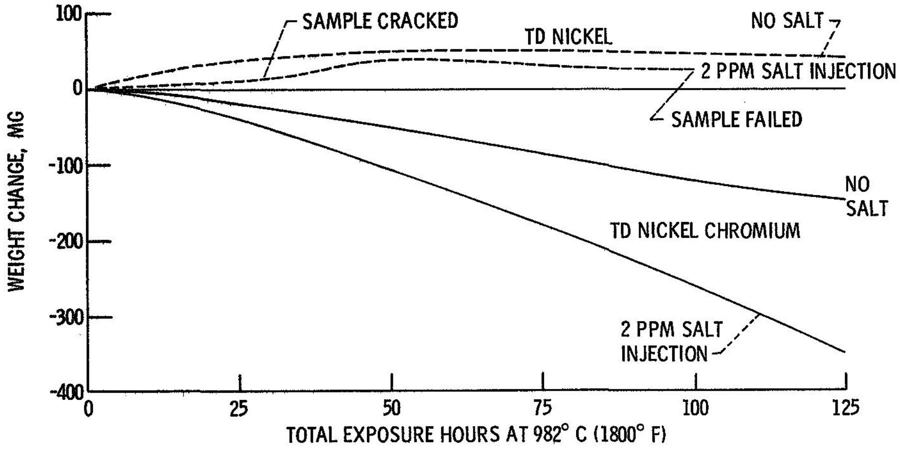

Figure 3. - Weight change of TD Nickel and TD Nickel Chromium resulting from 982° C (1800° F) cyclic exposure in simulated engine environment (Jet A-1 fuel). Test cycle: 1 hour at 982° C (1800° F), 3 minutes forced air cool to room temperature.

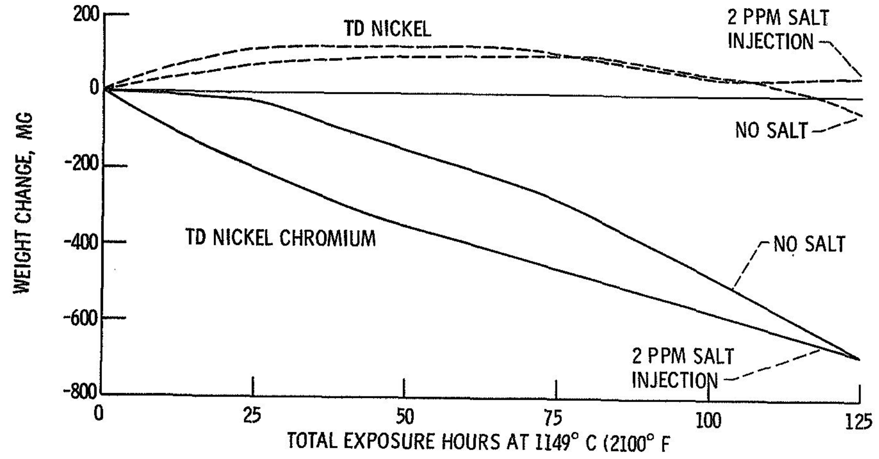

Figure 4. - Weight change of TD Nickel and TD Nickel Chromium resulting from 1149° C (2100° F) cyclic exposure in simulated engine environment (Jet A-1 fuel). Test cycle: 1 hour at 1149° C (2100° F), 3 minutes forced air cool to room temperature.

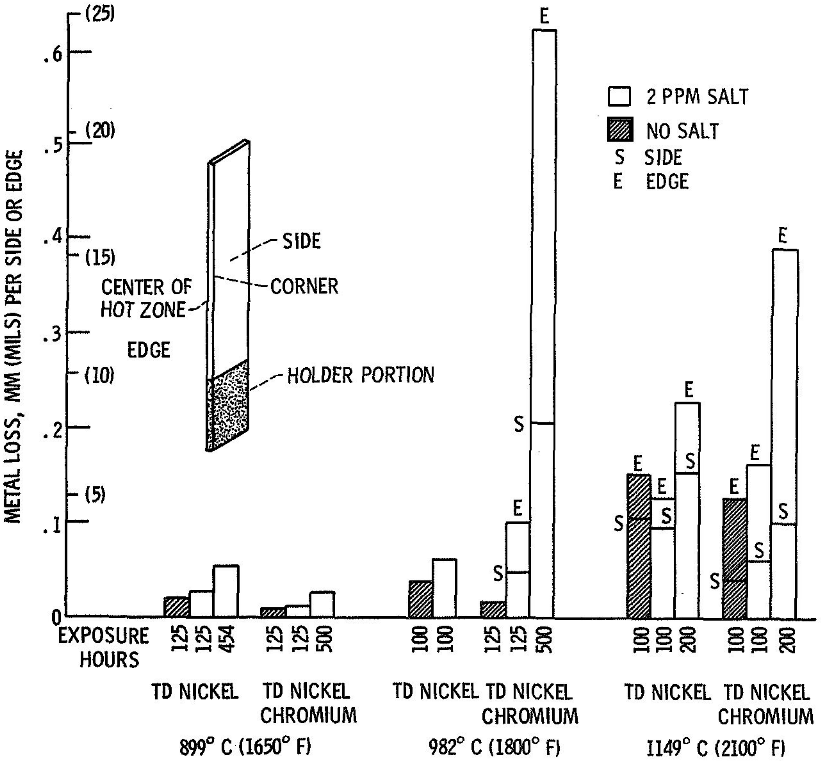

Figure 5. - Hot zone metal losses for TD Nickel and TD Nickel Chromium resulting from 899° C (1650° F), 982° C (1800° F), and 1149° C (2100° F) cyclic exposure in simulated engine environment (Jet A-1 fuel). Test cycle: 1 hour at test temperature, 3 minute forced air cool to room temperature.

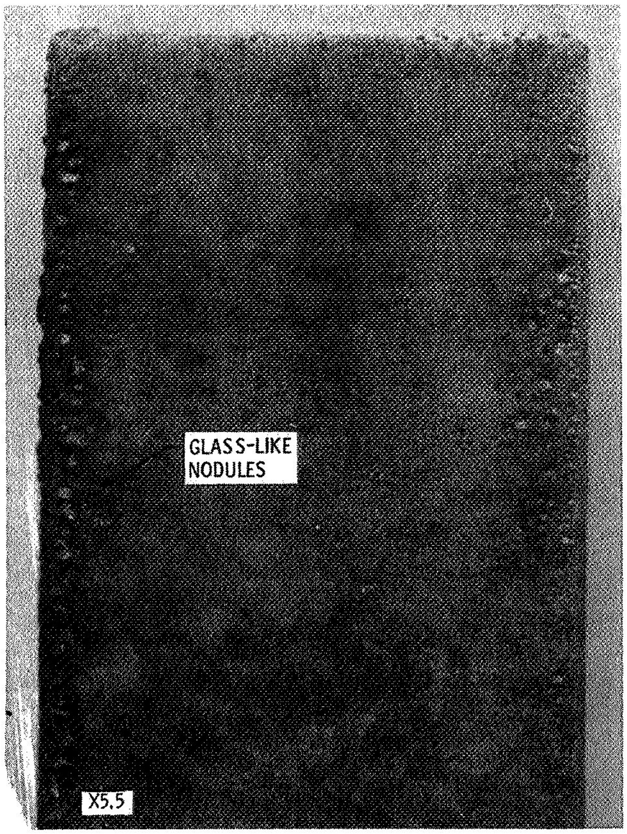

Figure 6. - Glass-like nodules which appeared on TD Nickel and TD Nickel Chromium after 200 hours exposure in 899° C (1650° F) test. Two ppm salt injection.

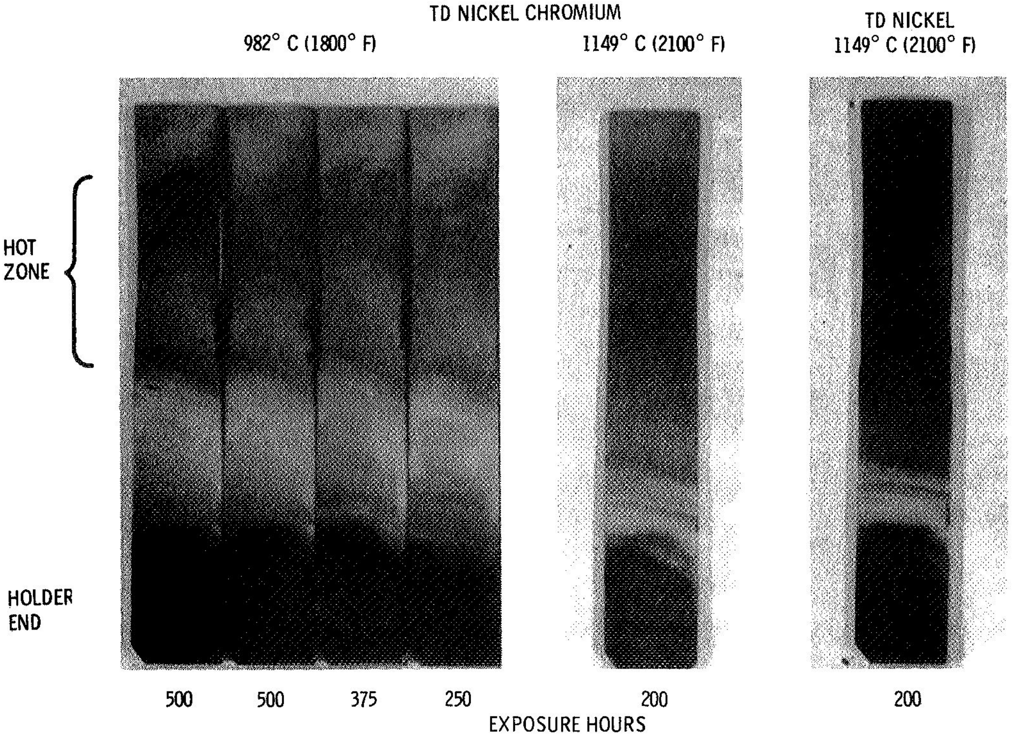

Figure 7. - TD Nickel Chromium and TD Nickel specimens exposed at 982° C (1800° F) and 1149° C (2100° F). two ppm salt injection.

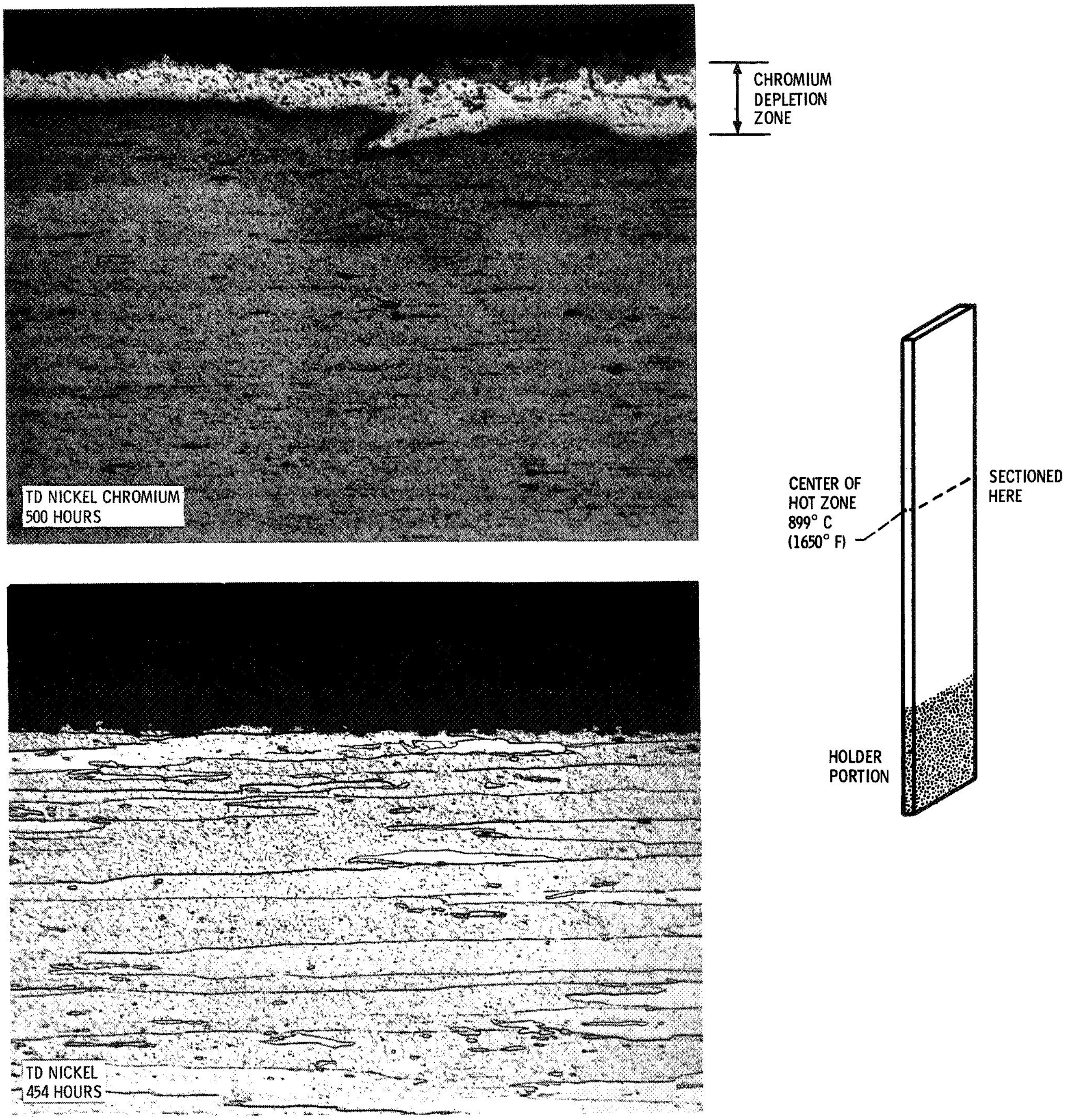

Figure 8. - Microstructures of TD Nickel Chromium and TD Nickel specimens exposed at 899° C (1650° F). Two ppm salt injection. X250.

E-6183

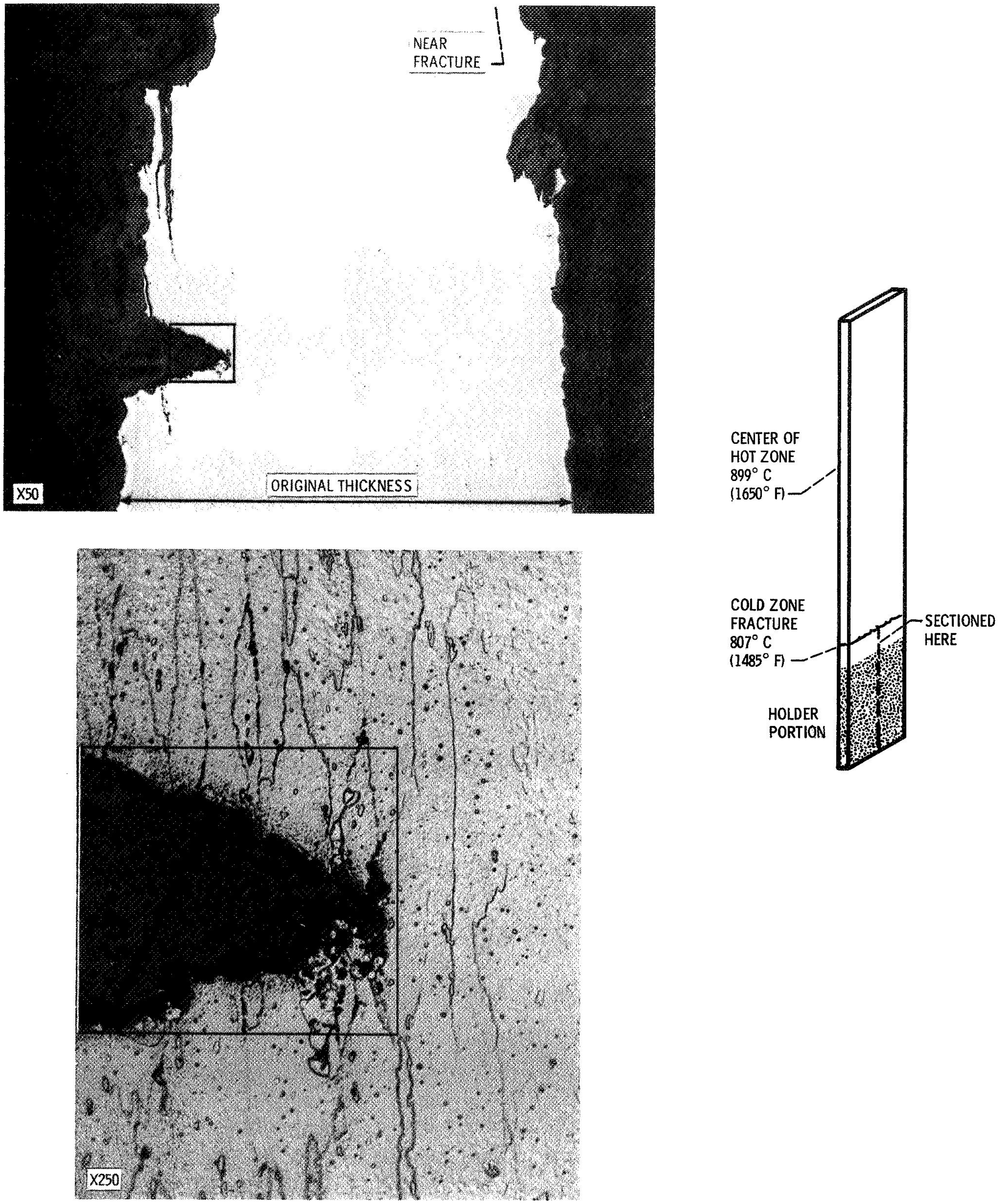

Figure 9. - Microstructures of TD Nickel near cold zone fracture; temperature, 807° C (1485° F). Specimen exposed at 899° C (1650° F) for 454 hours. Two ppm salt injection.

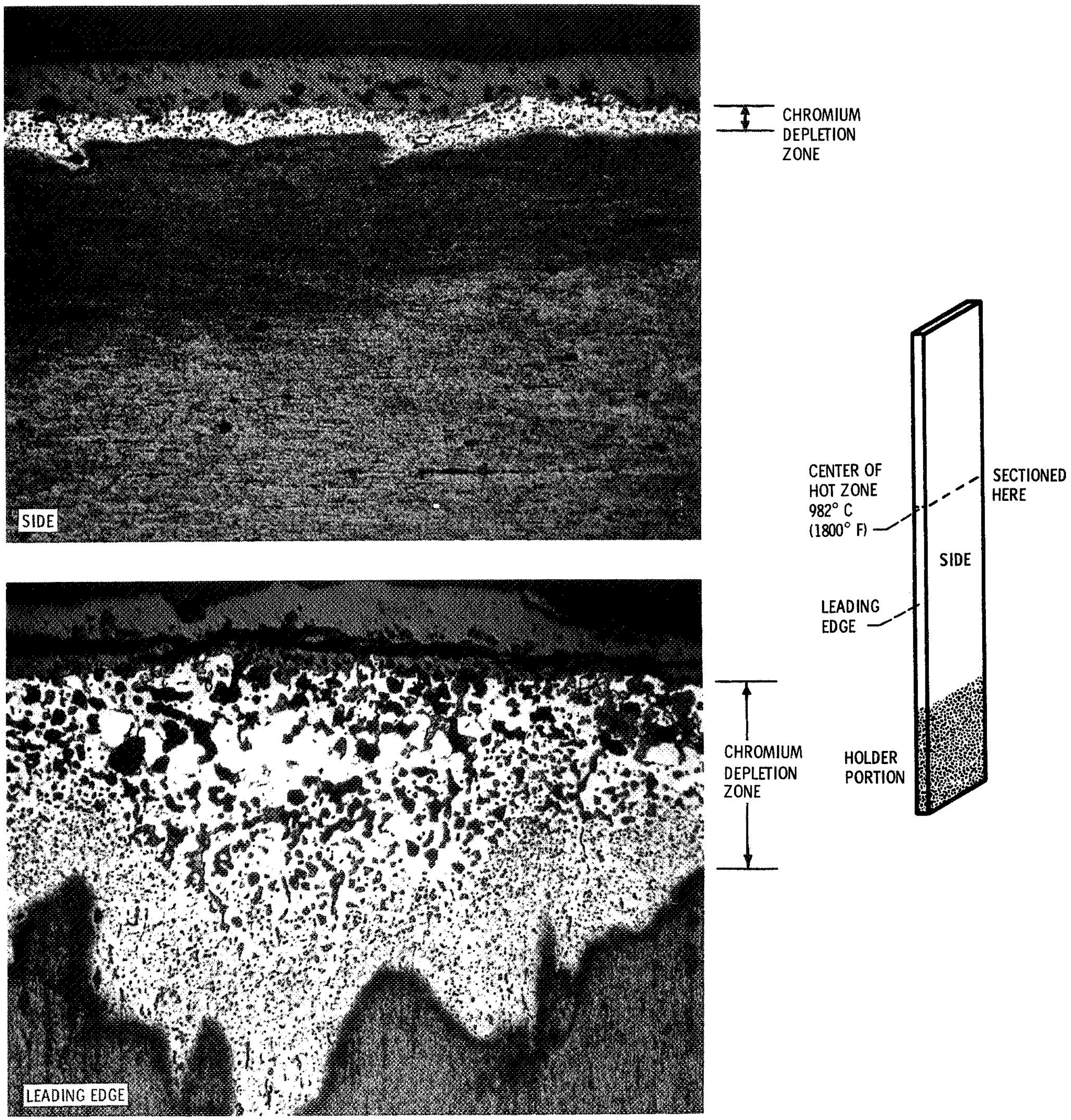

Figure 10. - Microstructures of TD Nickel Chromium exposed at 982° C (1800° F) for 500 hours. Two ppm salt injection. X250.

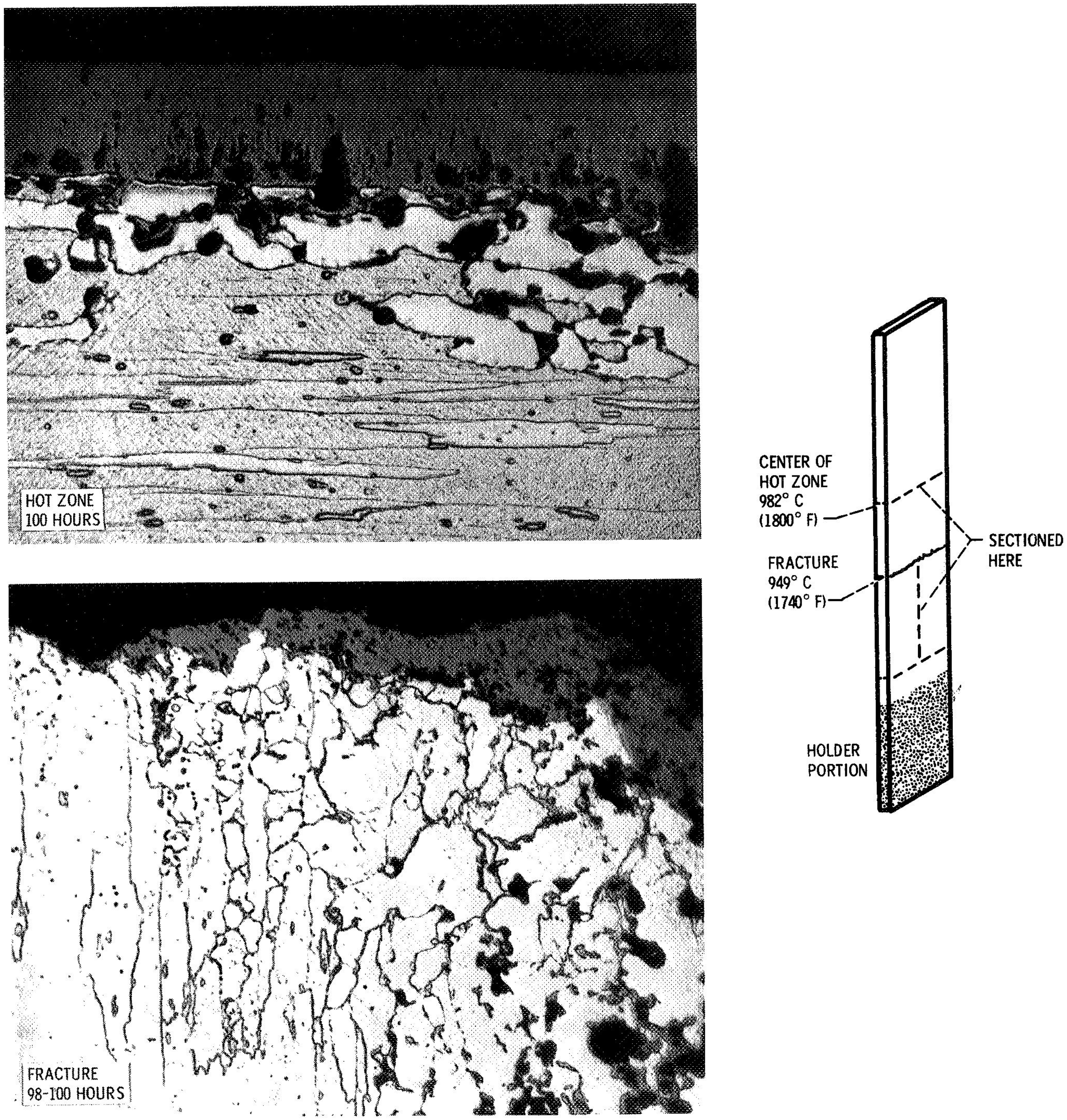

Figure 11. - Microstructures of TD Nickel in hot zone at 982° C (1800° F) and at fracture at 949° C (1740° F). Two ppm salt injection. X250.

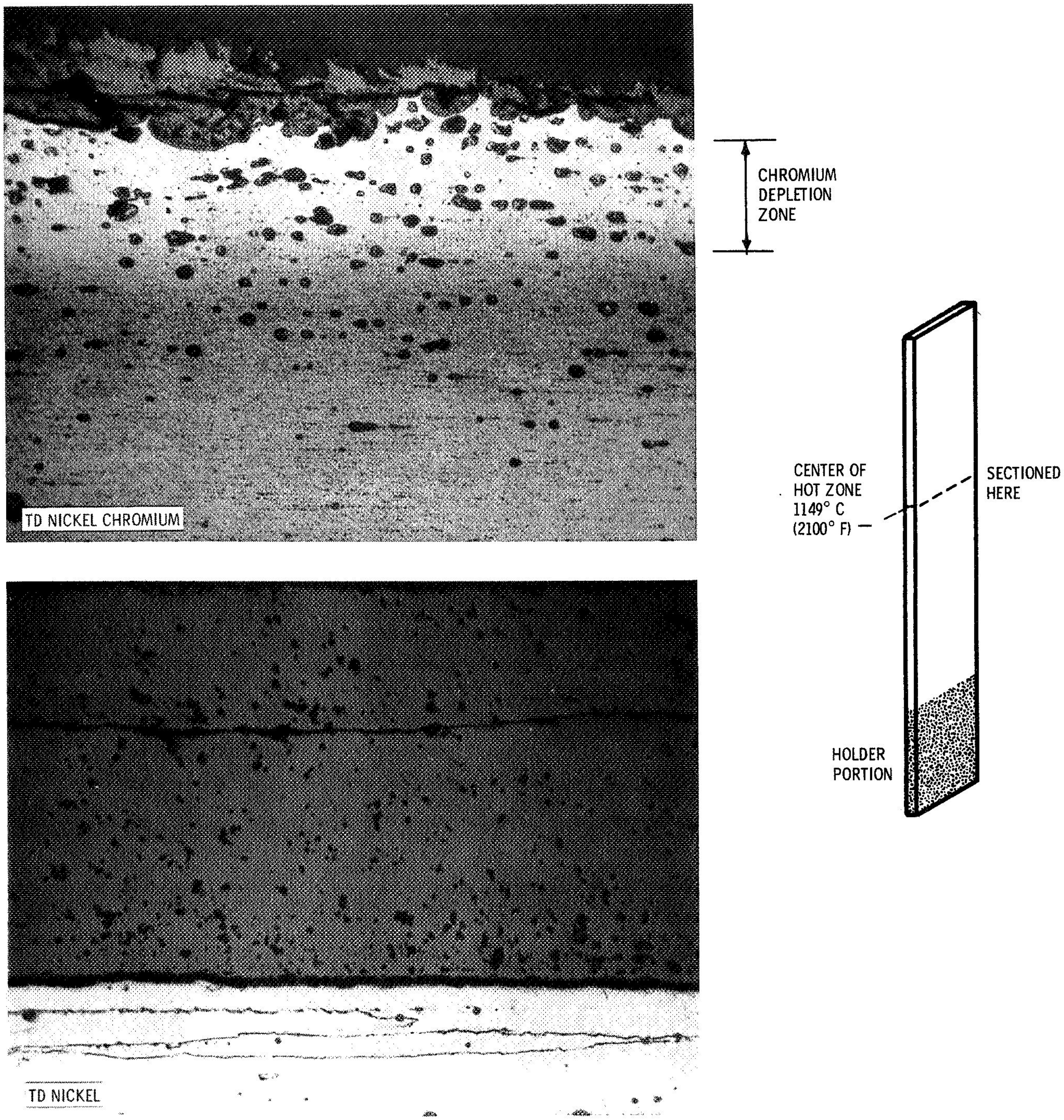

Figure 12. - Microstructures of TD Nickel Chromium and TD Nickel specimens exposed at 1149° C (2100° F) for 200 hours. Two ppm salt injection. X250.

E-6183