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CORROSION RESISTANCE OF NICKEL ALLOYS IN MOLTEN SODIUM HYDROXIDE

By H. B. Probst, C. E. May, and Howard T. McHenry

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

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SUMMARY

The corrosion resistance of 11 nickel-base compositions to molten sodium hydroxide at 1500° and 1700° F was studied in order to find a container material for the caustic at these temperatures. Although, in caustic, pure nickel is corrosion resistant (suffering only thermal-gradient mass transfer), it is structurally weak. Therefore, ways of strengthening nickel were sought that would not decrease its corrosion resistance. The materials selected for testing were as follows:

(1) Solid solutions: (nickel plus copper, nickel plus molybdenum, nickel plus zirconium, nickel plus tin, nickel plus columbium, nickel plus manganese, and nickel plus silicon)

(2) Two-phase materials: (a) materials containing mechanical dispersions of refractory particles (nickel plus titanium carbide, nickel plus magnesium oxide, and nickel plus aluminum oxide); and (b) precipitation-hardened alloy (nickel plus titanium)

At 1500° F only two nickel-base materials showed more than slight intergranular attack. This was a great improvement over commercial nickel-base alloys. However, other types of corrosion were still prevalent in some of the materials. The most common of these were the leaching of the solute, or second phase, and the formation of foreign nonmetallic phases within the alloy. In general, corrosion at 1700° F was only slightly more severe than at 1500° F. The only alloy that was as resistant to attack as pure nickel was the solid solution containing 30 percent copper. (This material still exhibited thermal-gradient mass transfer, however.) The materials containing molybdenum, zirconium, tin, titanium carbide, magnesium oxide, and aluminum oxide might be worthy of further investigation since corrosion was relatively slight. However, in the alloys containing columbium, manganese, silicon, and titanium the corrosion was sufficiently severe so that these materials should be given no further consideration.

INTRODUCTION

Two essential components of a thermal nuclear powerplant are a coolant and a moderator. If both functions, cooling and moderating, could be performed by a single material, reactor design might be considerably simplified (ref. 1). Molten sodium hydroxide is such a material. It also has the desirable properties of good resistance to radiation damage (ref. 2) and a wide temperature range between melting point (608° F) and boiling point (2534° F).

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In contrast to these advantages, molten sodium hydroxide is extremely corrosive to most materials at 1500° F (refs. 3 to 5). One of the most promising corrosion-resistant container materials for sodium hydroxide at high temperatures is pure nickel. Even this metal is subject to thermal-gradient mass transfer, which is the removal of metal from the hot zone and deposition in the cooler zone. Mass transfer of nickel has been somewhat inhibited by the use of additives to sodium hydroxide (ref. 6). Even if mass transfer were eliminated, pure nickel does not have sufficient strength at 1500° F to meet most design requirements. A material that is stronger than nickel and is as resistant as it is to corrosion by sodium hydroxide at elevated temperatures must be found if the unique properties of sodium hydroxide as both a moderator and a coolant are to be utilized.

Strong commercial nickel alloys such as the Hastelloys, Monels, Inconels, and Nimonics have previously been tested in molten sodium hydroxide up to 1500° F (refs. 4 and 5). All these materials were attacked more severely than pure nickel by molten sodium hydroxide; in most cases the corrosion was in the form of severe intergranular attack.

This investigation was conducted to study the corrosion resistance to sodium hydroxide of simpler nickel-base materials that were expected to be stronger than pure nickel. The materials selected for investigation were of two general classes, nickel-rich binary solid solutions and two-phase, nickel-base alloys strengthened by particles produced either by mechanical dispersion of refractory particles in the nickel matrix or by precipitation.

Solid-solution alloys were studied to determine if homogeneous single-phase alloys might be free from the severe intergranular attack observed in commercial nickel alloys. It was thought that the intergranular attack may have been due to concentration of alloying elements or second phases at the grain boundaries. Solid-solution alloying would be expected to strengthen nickel as a result of the lattice strain resulting from accommodation of foreign atoms into the nickel lattice. In addition, the presence of certain kinds of foreign atoms in solid solutions greatly alters the formation and effect of substructures. Such substructures have been shown to strengthen nickel solutions (ref. 7).

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Although commercial multiphase alloys generally had exhibited poor corrosion resistance to sodium hydroxide, two-phase alloys might be corrosion resistant if they possessed structures in which small, discrete particles of a second phase were isolated from each other by a corrosion-resistant matrix. If the second-phase particles were subject to attack by sodium hydroxide, such attack should largely be confined to the surface in contact with the hydroxide. Particles below the surface would effectively be shielded by the matrix. Such structures can be produced either by precipitation hardening or by mechanically dispersing small particles in nickel by powder-metallurgy means. Of the two methods, mechanical dispersion would be expected to be more successful, since it is probable that a pure nickel matrix known to have good corrosion resistance might be retained. In precipitation-hardened alloys, the particle is surrounded by a solid-solution matrix, which is likely to be less corrosion resistant than pure nickel. Both methods, however, are capable of producing substantial strengthening.

The materials evaluated and their methods of preparation are described herein and the results of static corrosion tests of the materials in sodium hydroxide at 1500° and 1700° F are presented.

MATERIALS

The materials investigated are listed in table I. Materials for all categories were selected on the basis of having low thermal neutron capture cross section, being readily available, and possessing desirable phase relations with nickel.

Solid-Solution Alloys

Nickel-base solid solutions containing the following percentages of alloying elements by weight were selected for study in this category: copper, 30; manganese, 15; molybdenum, 5; silicon, 1; tin, 5; and zirconium, 1. The amount of the alloying element was an arbitrary compromise between the maximum solute content for strengthening purposes and an alloy content which would ensure complete homogenization after heat treatment. Copper, which is completely soluble in nickel, was added to nickel as 30 percent by weight of the alloy. This alloy is comparable to the nickel-base alloy, Monel.

Nickel with Mechanically Dispersed Second Phase

Extremely fine dispersions of refractory particles have resulted in sizeable strength increases over that of the matrix material. For example, the tensile strength of commercially pure aluminum at 600° F is 2500 pounds per square inch (ref. 8, p. 810). A fine dispersion of 12 percent

aluminum oxide by weight throughout the aluminum matrix increases the tensile strength to 15,000 pounds per square inch at the same temperature (ref. 9). Increases in stress-rupture strengths have also been obtained with aluminum oxide dispersion in nickel matrices (ref. 10).

The materials selected for study in this category were nickel plus the following percentages by weight: titanium carbide, 12.0 or 8.2; aluminum oxide, 3.9; magnesium oxide, 4.3. The aluminum oxide containing material, which was supplied by Dr. N. J. Grant of the Massachusetts Institute of Technology, had fine particle size. No effort was made to obtain a particularly fine refractory particle size in the other materials. 4373

An addition of nickel oxide to nickel was not studied because of the instability of nickel oxide in hydrogen produced from the chemical reaction between sodium hydroxide and nickel (refs. 11 and 12).

Precipitation-Hardened Alloys

Alloys in the precipitation-hardened category were selected on the basis of favorable solubility changes with temperature and relatively low solubilities of the alloying element in nickel at the test temperatures of 1500° and 1700° F. Although there are a number of possibilities in this category, only nickel plus 15 percent columbium and nickel plus 11 percent titanium were selected for study. It was subsequently found that the nickel plus 15 percent columbium alloy could not be precipitation-hardened as will be discussed later.

EXPERIMENTAL PROCEDURE

Materials Preparation

A commercially pure nickel, "L" nickel having a maximum carbon content of 0.02 percent, was used for all alloys except for those that contained titanium carbide or aluminum oxide. Electrolytic nickel powder was used for the nickel plus titanium carbide compositions.

The nickel plus aluminum oxide material (obtained from M.I.T.) had been made from 10 micron "B" nickel powder and Alon C Al_2O_3 powder. The aluminum oxide powder originally had an average particle size of 0.03 micron. The particle size in the as-received bar is, however, much larger than this, approximately 0.1 micron (fig. 1(a)). Powder metallurgy techniques followed by extrusion had been used in making this composition.

The nickel plus magnesium oxide material was made from -325 mesh Plast nickel powder and analytical grade magnesium oxide powder. The

powders were mixed for 3 hours in a ball mill using no balls, hydrostatically pressed at 100,000 pounds per square inch, and vacuum sintered for 2 hours at 2500° F. The resulting density of the compact was 96.7 percent of theoretical density and the average magnesium oxide particle size was approximately 10 microns (fig. 1(b)).

All other compositions were melted under argon atmosphere in zircon crucibles and cast in an argon atmosphere into a $2\frac{1}{2}$-inch-deep by $7\frac{7}{16}$-inch-diameter copper mold. All heat treatments (see table I) were carried out in an argon atmosphere. The nickel plus 15 percent manganese and nickel plus 1 percent zirconium alloys required plastic deformation, as noted in table I, before complete solution could be obtained. The microstructure of the nickel plus titanium carbide materials is shown in figures 1(c) and (d).

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The nickel plus 15 percent columbium alloy showed no response to the aging treatments shown in table I. After these treatments, there was no definite metallographic evidence of any precipitate. Hardness measurements also indicated that no aging reaction had taken place. Thus, it is believed that this composition is in the solid-solution range at the aging temperature and that the equilibrium diagram in reference 8(p. 1187) is in error. As a result, this alloy will subsequently be classified as a solid-solution alloy.

Corrosion Test

The test specimens (11/32 in. square by 1/16 in., approximately 1 g) were prepared by surface grinding. The following data were obtained for each test material before the corrosion tests were made: microstructure, X-ray diffraction pattern from the surface of the specimen, and weight of the test specimen.

The corrosion tests were run in "L" nickel capsules of the geometry shown in figure 2. A specimen was placed at the bottom of each capsule. The components of each capsule assembly, that is, tube, top, bottom, and vent tube, were cleaned in modified aqua regia (ref. 12) before assembly for 2 minutes.

After the bottom was welded in place, the weighed test specimen was placed in the capsule and sodium hydroxide (13.0±0.1 g) was then added to the capsule in a helium dry box. The top and vent tube were then welded in place by means of a modified heliarc welding process. A welding atmosphere of helium plus 15 percent hydrogen was maintained while argon flowed through the assembly during welding.

Moisture and air were removed by evacuating to a pressure of 5 microns of mercury at 580° F (below the melting point of NaOH) and then evacuating to a pressure of 5 microns of mercury at 720° F (above the melting point of NaOH). The approximate times required for the two evacuations were 16 hours and 4 hours, respectively. Following the evacuation and cooling process, a cover gas of helium at a pressure of 2 pounds per square inch gage was admitted and the assembly sealed by crimping and welding the vent tube. Thermocouples were spotwelded to the capsules 1/8 inch from the bottom and also at the liquid level, $2\frac{1}{2}$ inches from the bottom.

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Eight such capsules were placed in a container which held the capsules in a circular arrangement, each being $1\frac{1}{4}$ inches from its nearest neighbor. The container was then placed in a specially designed Globar furnace (ref. 3) already at test temperature. In each group of eight capsules, two contained "L"-nickel specimens for control data.

Corrosion tests were run at 1500° and 1700° F. The temperature difference between the bottom of a capsule and the liquid level was approximately 45° F; the top of the liquid was always cooler than the bottom of the capsule in order to promote mass transfer by convective flow. The duration of all tests was 24 hours.

The oxide coating on the outside of the capsules was removed by vapor blasting. The capsules were then opened and their contents washed out with distilled water. This water solution and any accompanying precipitate were chemically analyzed for the alloying element of the test material. The specimens were reweighed after the corrosion run and were visually examined for severe attack. An X-ray diffraction pattern was taken from the top surface of each specimen and compared with the pattern obtained before the corrosion test. All materials were examined metallographically before and after the corrosion runs.

Testing various materials in nickel capsules is not the most desirable way to study the corrosion resistance of these materials, for the presence of dissimilar metals is known to affect the results of corrosion tests. A better procedure would be to use a system where both capsule and test piece are of the same material. However, since fabrication of capsules from the experimental materials would have been a major problem, nickel capsules were used for these first screening tests.

RESULTS AND DISCUSSION

The data obtained from the corrosion tests are entered in table II. In this table are given the "amount leached" data. These data indicate the percent of alloying element, or compound, found in the sodium hydroxide

in excess of the amount expected if only uniform surface removal had occurred. This excess amount could be due to either grain boundary attack, removal of solute atoms from solution, or attack of the second phase. The method used to calculate the leaching is presented in the appendix.

A summary of the results is given in table III. It can readily be seen that intergranular corrosion was not prevalent at 1500° F and only the zirconium and titanium alloys showed more than slight attack. In general, results at 1700° F indicated only slightly more attack. Of more concern than the intergranular attack were the leaching and foreign-phase formation observed for several of the materials.

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Solid-Solution Alloys

Corrosion of the solid-solution alloys can be classified into three groups: no attack, nickel plus 30 percent copper; slight attack, nickel plus 5 percent molybdenum, nickel plus 1 percent zirconium, and nickel plus 5 percent tin; and severe attack, nickel plus 15 percent columbium, nickel plus 15 percent manganese, and nickel plus 1 percent silicon.

Nickel plus copper. - The nickel plus copper alloy exhibited good resistance to attack by sodium hydroxide at both temperatures. No change in microstructure was observed and there was no leaching. Of all the compositions investigated, the alloy of nickel plus 30 percent copper showed the greatest resistance to attack.

These results for nickel plus copper are considerably more promising than those obtained with commercial Monel (refs. 4 and 5). Monel (Ni + 30 percent Cu + 1.5 percent Fe + 1 percent Mn) was attacked intergranularly by molten sodium hydroxide. The absence of intergranular attack in the nickel plus copper alloy investigated here may be due to one or both of the following factors: (1) The minor constituents, iron and manganese, contained in commercial Monel-type alloys probably decrease the corrosion resistance from that of simple nickel-copper compositions. (2) The homogenization heat treatment used in the present investigation may have reduced grain-boundary concentrations of minor constituents and thereby helped in preventing intergranular attack.

Nickel plus molybdenum. - No change was noted in the microstructure of the nickel plus 5 percent molybdenum composition after testing at 1500° F; however, slight intergranular attack resulted at 1700° F, as shown in figure 3. Leaching of molybdenum from the solid solution was slight at both temperatures, as measured by analysis of the sodium hydroxide.

Nickel plus zirconium. - The nickel plus 1 percent zirconium alloy was attacked intergranularly at both test temperatures. The attack at

1700° F, shown in figure 4, is about twice as deep as at 1500° F. The intergranular attack at 1700° F is characterized by a foreign phase in the grain boundaries; no such phase was observed after the 1500° F test. Leaching at 1500° and 1700° F was not severe, as indicated by chemical analyses. However, X-ray data indicated that there was at least surface leaching at both temperatures.

Nickel plus tin. - No change in microstructure was noted for the nickel plus 5 percent tin alloy after testing at 1500° F. Some intergranular attack was observed after the 1700° F test. A foreign phase was present in the grain boundary regions as shown in figure 5. Since the leaching appeared to be slight at 1700° F, according to chemical analysis, it was assumed to be slight at 1500° F as well. However, chemical analysis was lacking for the lower temperature sample. X-ray diffraction data showed similar changes in the lattice parameter after the tests at both temperatures, indicating at least surface leaching.

Nickel plus columbium. - The nickel plus 15 percent columbium alloy showed an unusual reaction to sodium hydroxide. The swelling that occurred on the surface at 1500° F and the microstructure of this swelling are shown in figures 6(a) and (b), respectively. The surface of the swell is a single phase, while the body of the swell contains a foreign phase. The microstructure of this composition after the 1700° F test is shown in figure 6(c). Grain boundary attack is evident with a foreign phase present in the grain boundary regions. While it is not evident in the photomicrograph, a slight surface swelling was present after the test at 1700° F. Both X-ray and chemical analysis data indicated that considerable leaching of columbium had taken place at both test temperatures.

Nickel plus manganese. - Another material showing abundant formation of a foreign phase at 1500° F was nickel plus 15 percent manganese. In this case, however, the foreign phase was not localized and appeared to have formed with no volume change (fig. 7(a)). The surface of the sample was pure nickel as shown by X-ray diffraction data. This region may have been depleted of manganese rather than being plated as there is no evidence of any interface. Localized formation of the foreign phase with some surface swelling resulted after the 1700° F test, as shown in figure 7(b). Chemical analysis showed only minor leaching at both temperatures.

Metallographically, the foreign phases contained in the various materials appeared to be similar, that is, they had a dark gray to black appearance resembling the oxide. Of the materials containing the foreign phase, the sample shown in figure 7(a) (Ni + 15 percent Mn) was the best suited to identify the foreign phase by X-ray diffraction. The surface of the sample was ground to various depths that were within the region containing the foreign phase. X-ray patterns obtained from these newly ground surfaces gave only nickel lines. However, it was noted that after the specimen was left standing in air for 24 hours, the ground surface of

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the metal was covered with a gray film. This film, presumably formed by reaction of a foreign phase (possibly a sodium metallate) with moisture and carbon dioxide in the air, was identified by X-ray diffraction as $Na_2CO_3 \cdot H_2O$.

Nickel plus silicon. - There was no change in the appearance of the microstructure of the nickel plus 1 percent silicon alloy after testing at 1500° F, but slight intergranular attack resulted at 1700° F as shown in figure 8. This composition showed the greatest leaching effect of all the compositions studied. At both 1500° and 1700° F, virtually all the solute was leached by the sodium hydroxide. Thus, this alloy has no advantage over pure nickel, since the alloy is essentially changed to pure nickel when exposed to molten sodium hydroxide.

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Nickel with Mechanically Dispersed Second Phase

The group of materials with mechanically dispersed second phases possessed generally good corrosion resistance. There was no intergranular attack at either 1500° or 1700° F. Contrary to expectation, however, the attack of refractory particles was not confined to the particles exposed at the surface. Microscopic examination showed that particles below the surface had been removed, leaving voids. Figure 9 is a photomicrograph of the nickel plus magnesium oxide material after corrosion test at 1700° F. The nickel plus titanium carbide compositions showed a similar removal of titanium carbide particles. In addition to voids, the nickel plus 8.2 percent titanium carbide specimen tested at 1700° F contained a dark foreign phase near the surface similar to that noted in a number of the solid-solution alloys.

Metallographic examination of the nickel plus aluminum oxide material did not clearly indicate to what extent the aluminum oxide particles had been removed by the hydroxide. The size of the aluminum oxide particles in this material was too small to distinguish between particles and voids. However, the chemical analysis of the sodium hydroxide, shown in table II, indicated that considerable aluminum oxide had been leached from the sample.

Precipitation-Hardened Alloy

The nickel plus 11 percent titanium alloy was severely attacked by molten sodium hydroxide both at 1500° and 1700° F. As shown in figure 10, attack was predominantly intergranular. The grain boundary regions were depleted of titanium and showed the presence of a foreign phase. In addition, specimens tested at both temperatures also showed localized surface swelling with a foreign phase in the swells.

During the test at 1500° F, a nickel plate formed on the specimen (fig. 10(a)); this presumably came from the nickel capsule. In figure 10(a), the surface swells containing the foreign phase appear to have been formed after the deposition of the nickel plate. An accompanying positive volume change is suggested by the ruptured plate. The internal boundary regions of the swells in figure 10(c) appear to be depleted of Ni_3Ti. X-ray data of the alloy after the 1500° F test indicated only the nickel plate on the sample.

Nickel plus titanium was the only precipitation-hardened alloy tested. It is not reasonable to expect that the poor corrosion resistance of this alloy is representative of all aging alloys. Alloys containing spherical precipitates (although probably not as strong) might show better corrosion resistance than did the more continuous Widmanstätten type precipitate in the nickel plus titanium alloy. The composition of the solid-solution matrix for a precipitation-hardened alloy would also have an important bearing on the corrosion resistance.

SUMMARY OF RESULTS

At 1500° F, of 11 materials evaluated, only two nickel-base materials (Ni + Zr and Ni + Ti) showed more than slight intergranular attack. This was a great improvement over the commercially available nickel-base alloys. However, other types of corrosion were still prevalent in some of the materials. The most common types were the leaching of the solute, or second phase, and the formation of foreign nonmetallic phases within the alloy. In general, the corrosion of the materials at 1700° F was only slightly greater than that at 1500° F.

The only alloy as resistant to attack as pure nickel was the solid solution containing 30 percent copper. This material still exhibited thermal-gradient mass transfer as does pure nickel. Three solid solutions (Ni + Mo, Ni + Zr, and Ni + Sn) and all the materials containing refractory particles (Ni + TiC, Ni + MgO, and Ni + Al_2O_3) might be worthy of further investigation since the over-all corrosion resistance of these materials was good. However, for the remaining solid solutions (Ni + Cb, Ni + Mn, and Ni + Si) and the precipitation-hardening alloy (Ni + Ti), the corrosion was sufficiently severe that no further consideration should be given these materials.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, October 16, 1957

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APPENDIX - METHOD OF CALCULATING EXCESS REMOVAL OF ALLOYING ELEMENTS

The method of calculating the percentage of the alloying element, or compound, which was removed from specimens in excess of normal surface removal by mass transfer is described herein.

The quantity of the alloying element s removed as a result of the surface removal of nickel is calculated as follows:

$$s = \frac{ky}{1 - y}$$

where k is the amount of nickel removed from unalloyed samples ($k = 11.2$ mg at 1500° F and 40.1 mg at 1700° F; the assumption was made that k remains constant for all alloying additions; see table II) and y is the weight fraction of alloying element, or compound, in the sample.

The "amount leached" data (additional removal) were obtained from the following relation:

$$\text{Amount leached, percent} = \frac{100(x - s)}{t - s}$$

where x is the amount of alloying element, or compound, found in the sodium hydroxide after tests (in mg), t is the total amount of alloying element, or compound, in the samples (in mg), and s, as defined previously, is the calculated amount of alloying element or compound that should be in the sodium hydroxide if no leaching took place.

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TABLE I. - MATERIALS INVESTIGATED AND PREPARATION DATA

Alloying element or compound, nominal percent by weight	Purity of alloying element or compound, percent	Form of alloying element or compound	Chemical analysis after fabrication, percent by weight	Heat treatments before tests[a]	Resulting microstructures before tests
Solid solutions					
30 Cu	99.9	Shot	28.20 Cu	2350° F - 1/2 hr; air cool	Solid solution
5 Mo	99.0	Turnings	5.11 Mo	1800° F - 1/2 hr; air cool	Solid solution
1 Zr	----	Stick	1.05 Zr	Cold pressed to 216,000 lb/sq in. 1900° F - 22 hr; water quenched	Solid solution + impurity phase
5 Sn	99.8	Stick	4.99 Sn	1800° F - 1/2 hr; air cool	Solid solution + Ni_3Sn
				2000° F - 1/2 hr; water quenched	Solid solution
15 Cb	99.8	Powder	14.47 Cb	Solution annealed - 2320° F - 1/2 hr; water quenched	Solid solution; Rockwell A hardness = 52
				Aged - 1800° F - 2 hr; water quenched	No visible precipitate; Rockwell A hardness = 54
				Aged - 1500° F - 4 hr; water quenched	No visible precipitate; Rockwell A hardness = 54
15 Mn	96.0 + {2 Fe, 1 Si, 0.2 C}	Lump	14.30 Mn	Cold pressed to 216,000 lb/sq in. 1900° F - 20 hr; air cool	Solid solution
1 Si	99.9	Lump	1.05 Si	1800° F - 1/2 hr; air cool	Solid solution + second phase
				2000° F - 1/2 hr; water quenched	Solid solution
Materials containing refractory particles					
12 TiC	----	Powder	11.40 TiC	2250° F - 5 hr; argon cool	Uniform dispersion
8.2 TiC	----	Powder	7.20 TiC	2200° F - 3 hr; argon cool	Uniform dispersion
3.9 Al_2O_3	----	Powder	4.30 Al_2O_3	As received	Uniform dispersion
4.3 MgO	99.0	Powder	3.65 MgO	As sintered	Uniform dispersion
Precipitation-hardened alloy					
11 Ti	99.5	Rod	10.60 Ti	Solution annealed - 2350° F - 4 hr; water quenched	Solid solution + impurity phase; Rockwell C hardness = 23
				Aged - 1550° F - 24 hr; water quenched	Solid solution + impurity phase + Ni_3Ti; Rockwell C hardness = 33

[a]Alloys listing more than one heat treatment were treated in the order given.

TABLE II. - RESULTS OF SODIUM HYDROXIDE TESTS

(a) Temperature, 1500° F

Alloying element or compound, nominal percent by weight	Specimen weight change, mg	Change in lattice parameter from that of pure nickel, percent		Amount of alloying element or compound in NaOH after run, mg	Amount leached, percent	Microstructure after test
		Before test	After test			
0 (Pure Ni)	-11.2	0.0	0.0	0.0	0.0	No change
Solid solutions						
30 Cu	-16.4	0.61	0.60	3.3	-0.47	No change
5 Mo	-15.7	0.41	0.21	1.7	2.1	No change
1 Zr	-10.3	0.20	0.04	0.26	1.24	Intergranular attack
5 Sn	-19.8	0.69	0.34	(a)		No change
15 Cb	+0.6	1.80	0.09	45.0	31.8	Localized surface swelling with foreign phase in swell
15 Mn	+62.4	1.40	(b)	5.4	2.5	Ni plate; foreign phase below plate
1 Si	+1.3	0.00	-0.08	11.0	98.2	No change
Materials containing refractory particles						
12 TiC	- 6.5	1.02	0.03	9.14	6.7	TiC removed from surface region
3.9 Al_2O_3	-16.8	0.05	-0.03	7.65	16.6	No change
4.3 MgO	-13.1	0.03	0.02	2.82	7.0	MgO removed from surface region
Precipitation-hardened alloy						
11 Ti	+21.0	1.28	(b)	7.3	6.42	Ni plate; surface swelling with foreign phase in swells; grain boundaries depleted of Ni_3Ti; foreign phase in grain boundaries

[a]Chemical analysis was in error.

[b]Not possible to determine because of Ni plating on sample.

TABLE II. - CONCLUDED. RESULTS OF SODIUM HYDROXIDE TESTS

(b) Temperature, 1700° F

Alloying element or compound, nominal percent by weight	Specimen weight change, mg	Change in lattice parameter from that of pure nickel, percent		Amount of alloying element or compound in NaOH after run, mg	Amount leached, percent	Microstructure after test
		Before test run	After test			
0 (Pure Ni)	-40.1	0.0	0.0	0.0	0.0	No change
Solid solutions						
30 Cu	-62.4	0.61	0.67	11.5	-1.76	No change
5 Mo	-45.1	0.41	0.38	3.5	2.66	Slight intergranular attack
1 Zr	-27.7	0.20	0.00	0.1	-3.26	Foreign phase in grain boundaries resulting from intergranular attack
5 Sn	-41.7	0.69	0.36	0.2	-3.9	Foreign phase in grain boundaries resulting from intergranular attack
15 Cb	-38.1	1.80	0.11	39.0	24.3	Foreign phase in grain boundaries resulting from intergranular attack
15 Mn	-22.2	1.40	0.98	7.4	0.5	Localized foreign phase in surface region
1 Si	-40.0	0.00	0.00	9.0	80.5	Slight intergranular attack
Materials containing refractory particles						
6.2 TiC	+8.9	0.64	0.04	2.4	-1.06	TiC removed from surface region; foreign phase present
3.9 Al_2O_3	-46.2	0.05	0.00	13.3	27.2	No change
4.3 MgO	-38.8	0.03	0.00	5.8	12.9	MgO removed from surface region
Precipitation-hardened alloy						
11 Ti	-27.8	1.28	1.00	7.3	2.56	Surface swelling with foreign phase in swells; grain boundaries depleted of Ni_3Ti; foreign phase in grain boundaries

TABLE III. - SUMMARY OF REACTIONS WITH SODIUM HYDROXIDE

[Undetermined, u; reaction observed, x; little or no reaction, no symbol.]

Alloying element or compound, nominal percent by weight	Intergranular attack		Second-phase attack		Formation of foreign phase		Nickel plating on sample		Leaching of solute			
									X-ray diffraction		Chemical analysis	
	Temperature, °F											
	1500	1700	1500	1700	1500	1700	1500	1700	1500	1700	1500	1700
Solid solutions												
30 Cu												
5 Mo									x			
1 Zr	x	x				x			x	x		
5 Sn		x				x			x	x	u	
15 Cb		x			x	x			x	x	x	x
15 Mn					x	x	x		u			
1 Si									u	u	x	x
Materials containing refractory particles												
12 TiC		u		u		u		u	x	u		u
8.2 TiC	u		u		u	x	u		u	x	u	
3.9 Al_2O_3			u	u					u	u	x	x
4.3 MgO			x	x					u	u		x
Precipitation-hardened alloy												
11 Ti	x	x	x	x	x	x	x		u			

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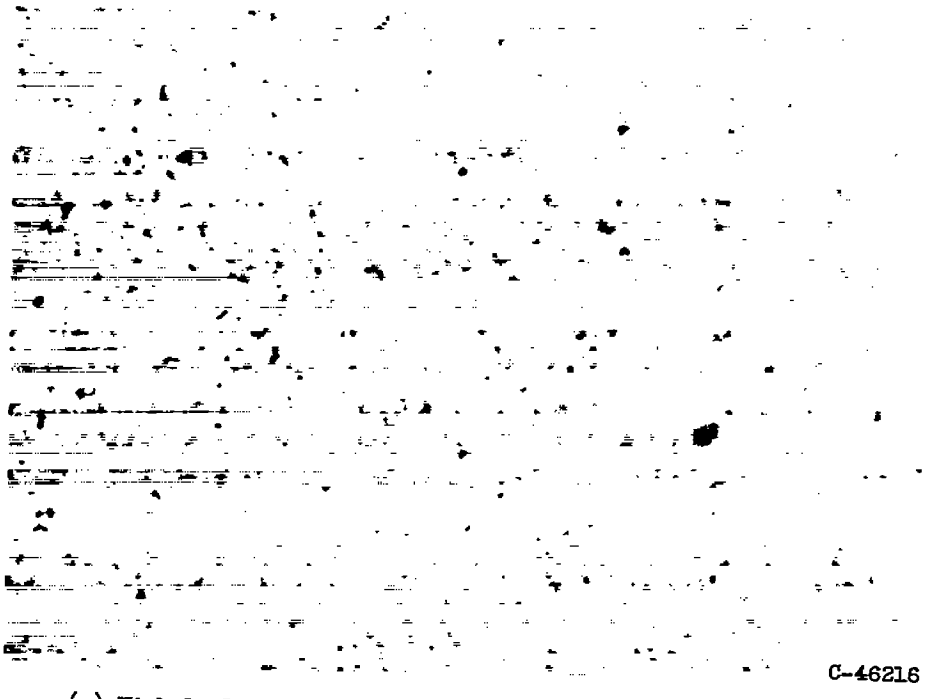
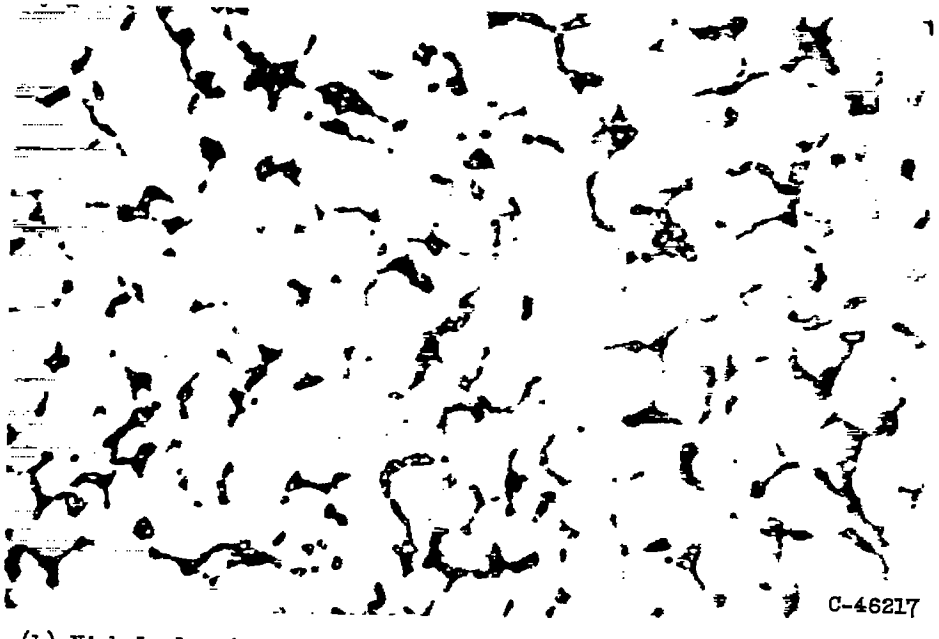


(a) Nickel plus 3.9 percent aluminum oxide. Unetched; X1000.

Figure 1. - Refractory dispersions in nickel matrix.



(b) Nickel plus 4.3 percent magnesium oxide. Etch: 92 percent HCl, 5 percent HNO_3, and 2 percent H_2SO_4; X250.

Figure 1. - Continued. Refractory dispersions in nickel matrix.

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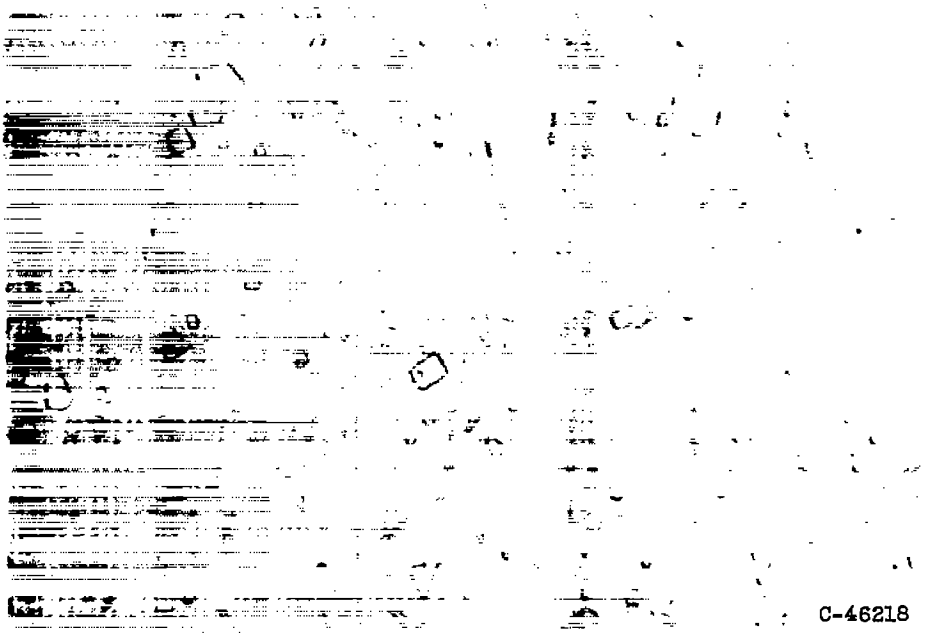
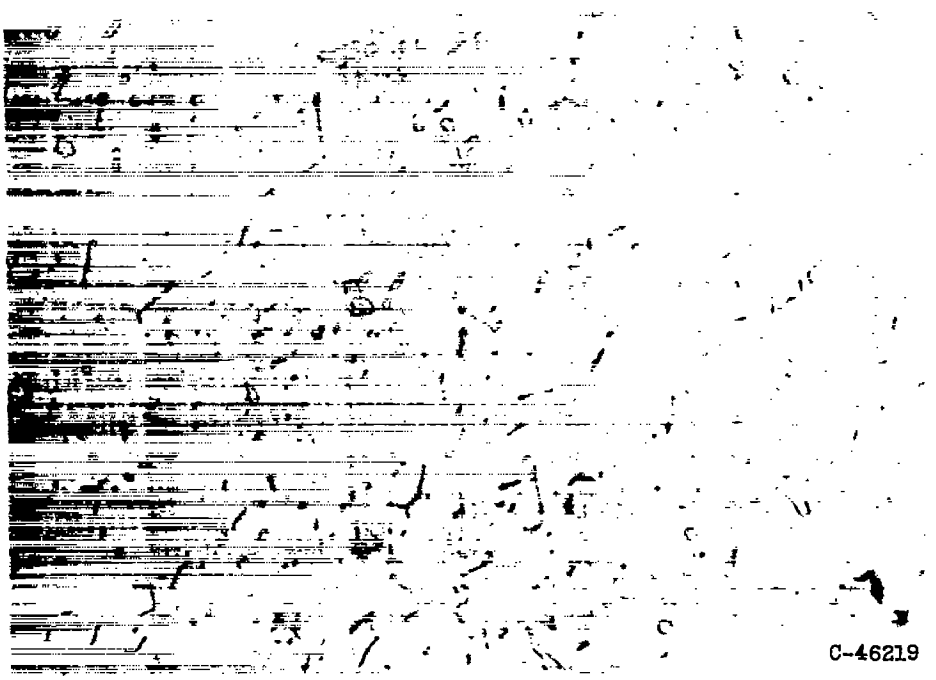


(c) Nickel plus 12 percent titanium carbide. Unetched; X750.

Figure 1. - Continued. Refractory dispersions in nickel matrix.



(d) Nickel plus 8.2 percent titanium carbide. Unetched; X750.

Figure 1. - Concluded. Refractory dispersions in nickel matrix.

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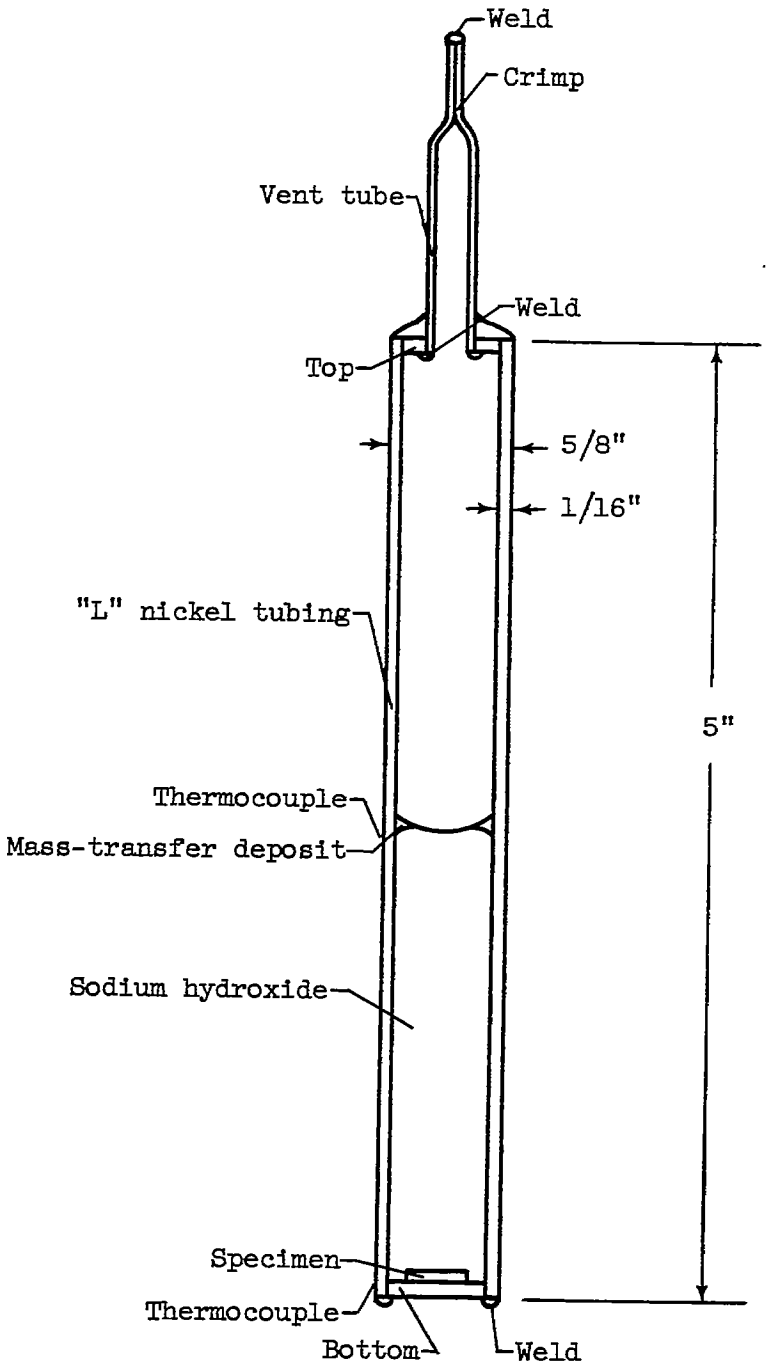

Figure 2. - Corrosion test capsule assembly.

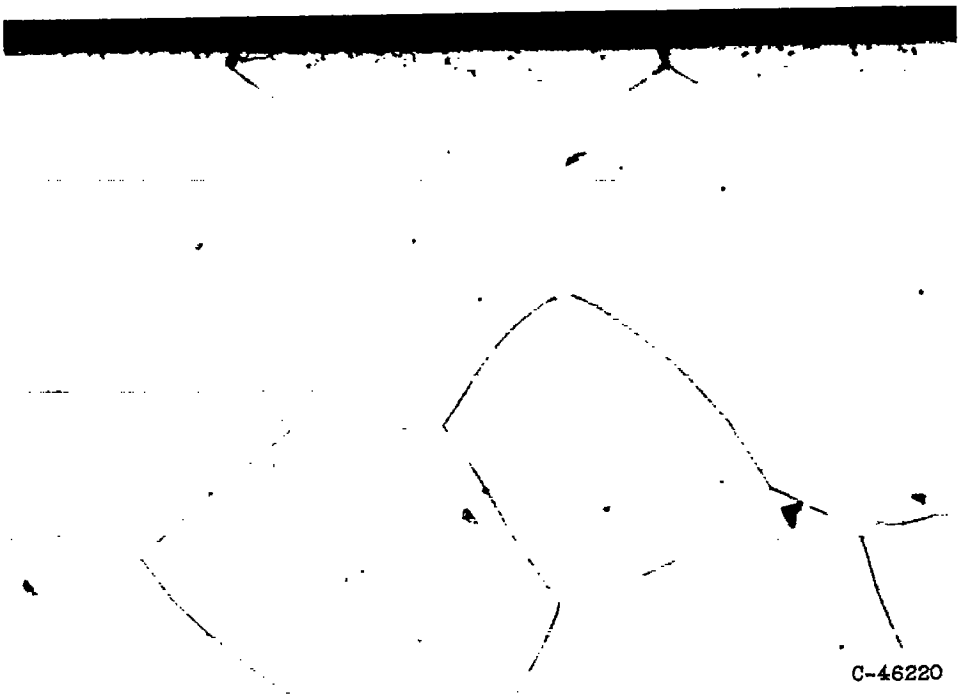

Figure 3. - Nickel plus 5 percent molybdenum after corrosion test at 1700° F. Etched; X250.

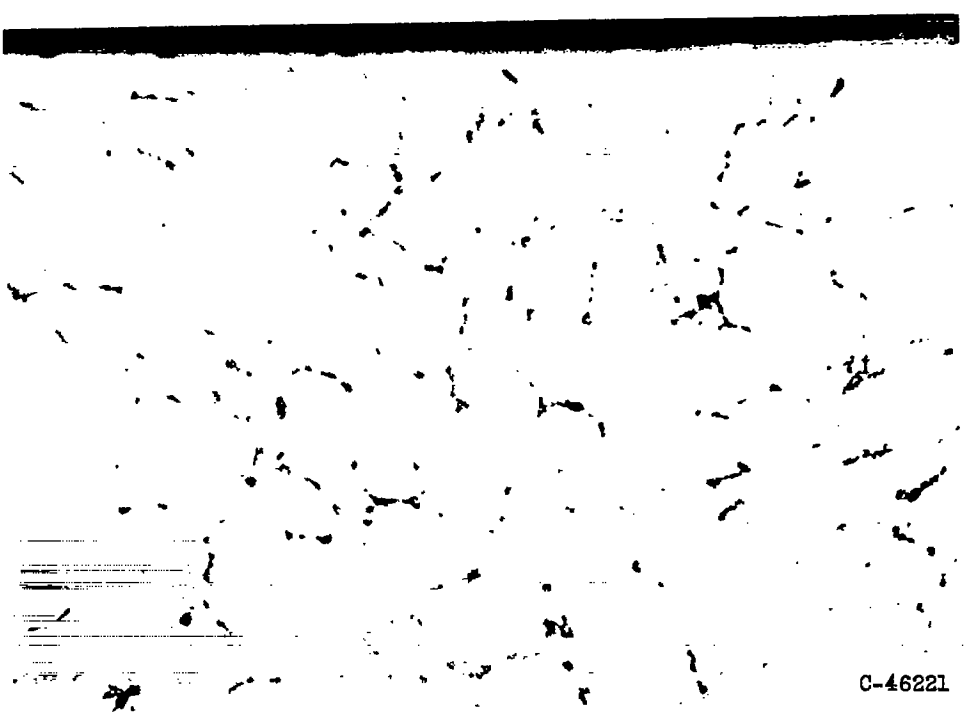

Figure 4. - Nickel plus 1 percent zirconium after corrosion test at 1700° F. Unetched; X250.

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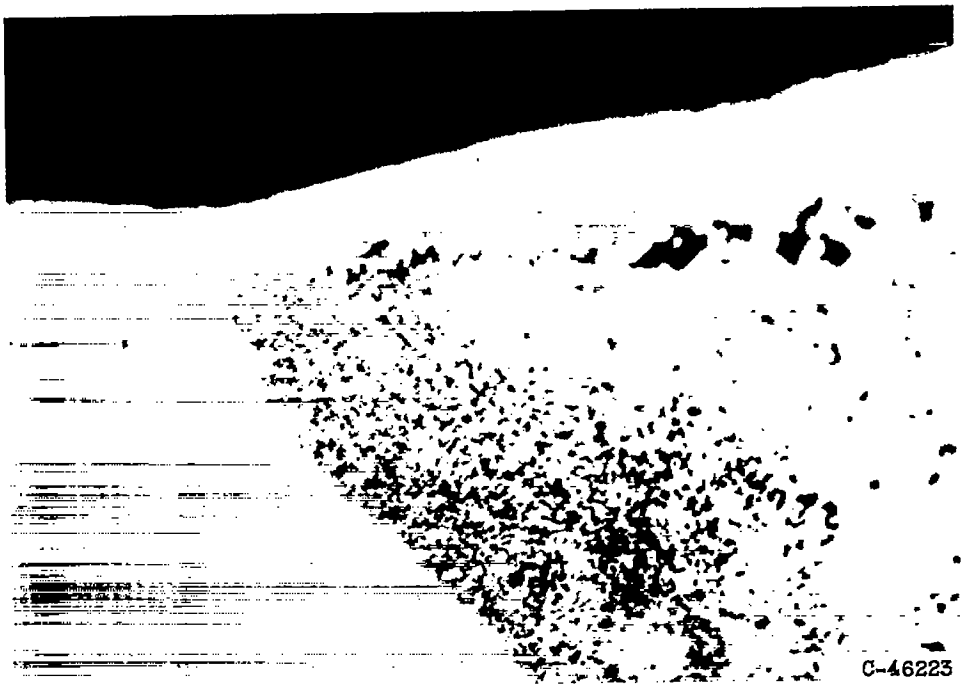
Figure 5. - Nickel plus 5 percent tin after corrosion test at 1700° F. Etched; X250.

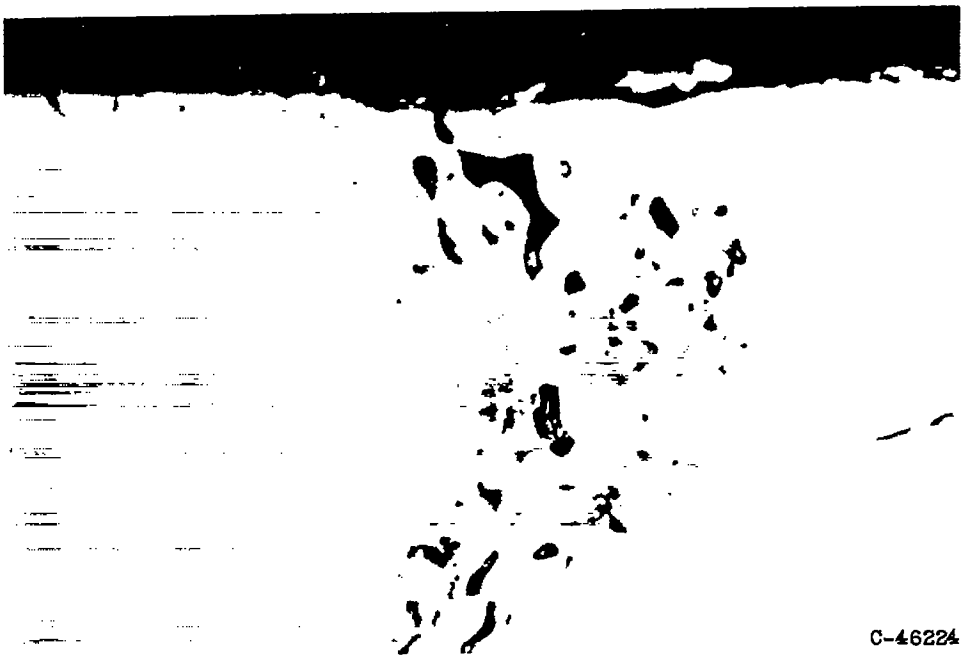
(a) Surface swelling. Temperature, 1500° F.

Figure 6. - Nickel plus 15 percent columbium after corrosion test.



(b) Microstructure of swelling. Temperature, 1700° F. Unetched; X750.

Figure 6. - Continued. Nickel plus 15 percent columbium after corrosion test.



(c) Microstructure. Temperature, 1700° F. Unetched; X500.

Figure 6. - Concluded. Nickel plus 15 percent columbium after corrosion test.

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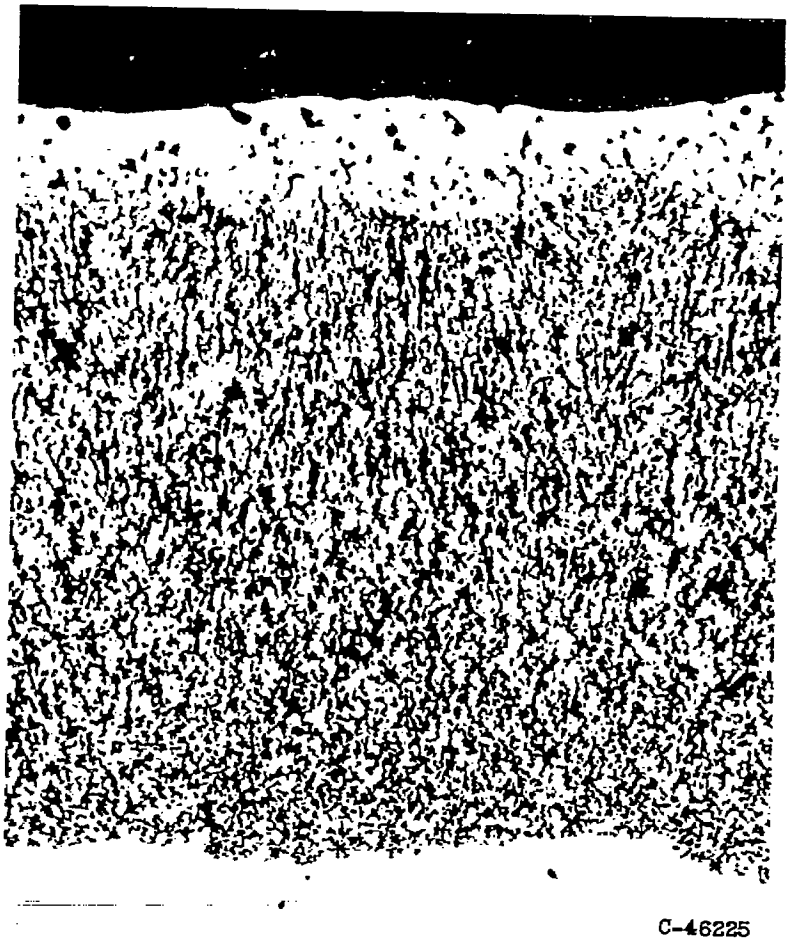
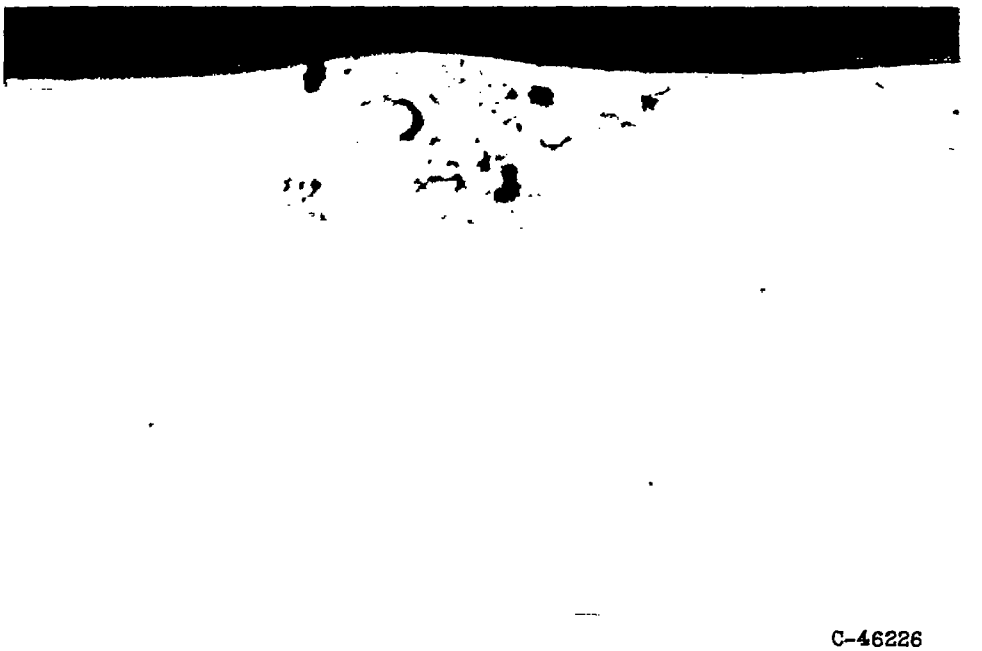
CU-3 back



(a) Temperature, 1500° F. Unetched; X250.

Figure 7. - Nickel plus 15 percent manganese after corrosion test.



(b) Temperature, 1700° F. Unetched; X500.

Figure 7. - Concluded. Nickel plus 15 percent manganese after corrosion test.

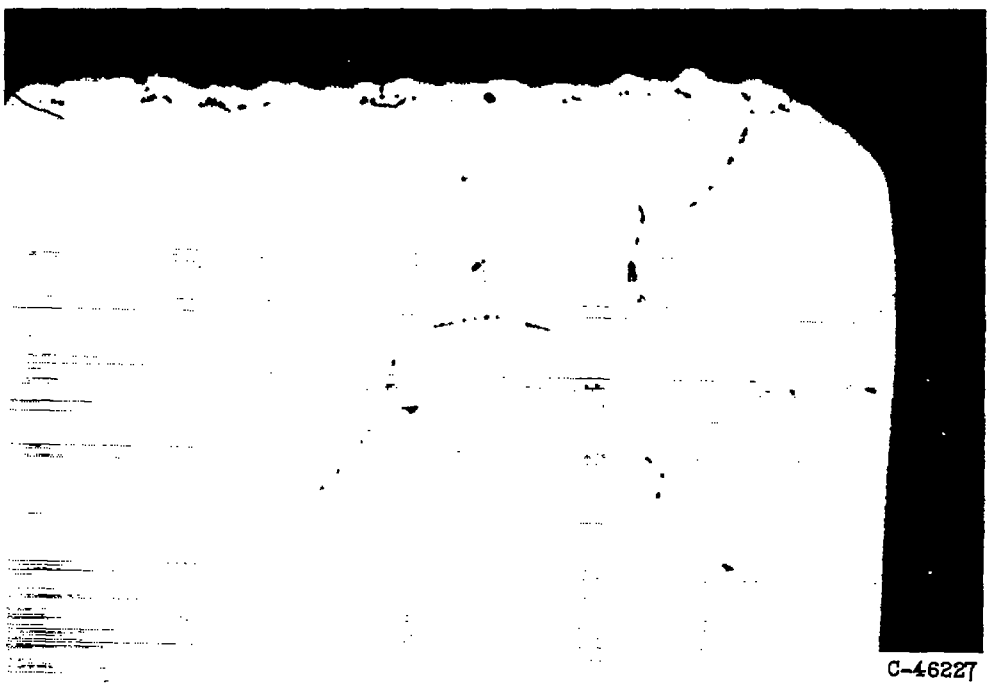


Figure 8. - Nickel plus 1 percent silicon after corrosion test at 1700° F. Etched; X100.

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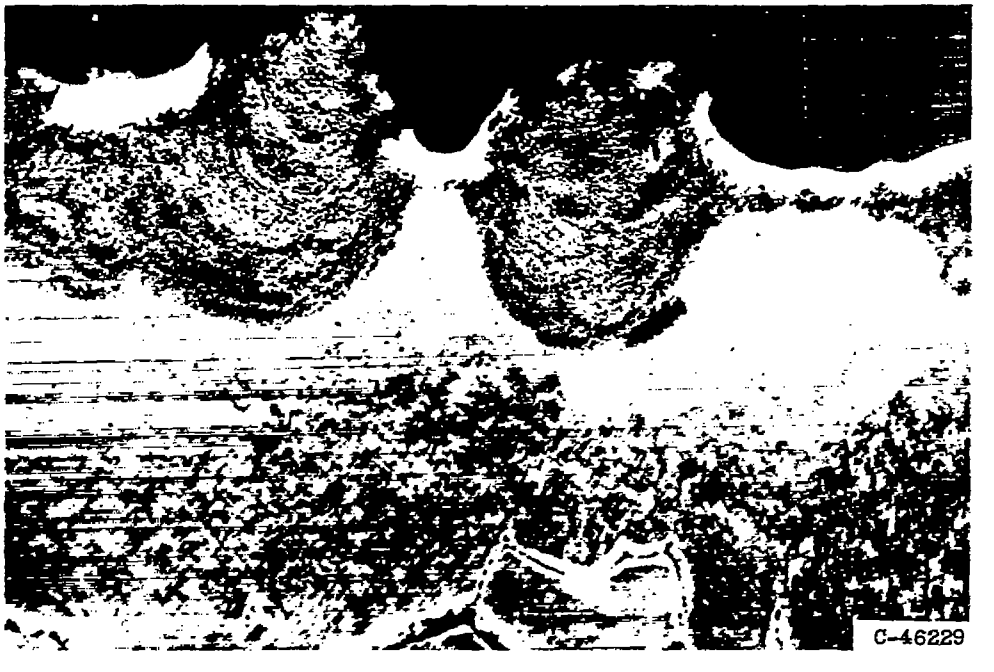
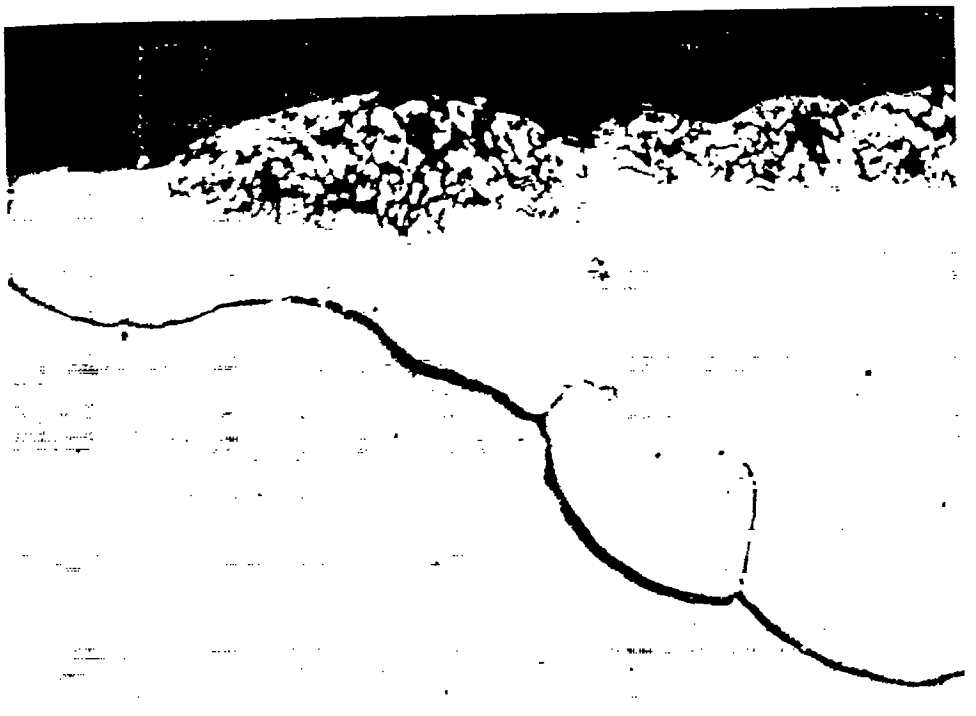
Figure 9. - Nickel plus 4.3 percent magnesium oxide after corrosion test at 1700° F. Etched; X500.



(a) Temperature, 1500° F. Etched; X100.

Figure 10. - Nickel plus 11 percent titanium after corrosion test.



(b) Temperature, 1700° F. Unetched; X200.

(c) Temperature, 1700° F. Etched; X250.

Figure 10. - Concluded. Nickel plus 11 percent titanium after corrosion test.