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METALLURGY DIVISION

QUARTERLY PROGRESS REPORT

FOR PERIOD ENDING OCTOBER 31, 1951

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METALLURGY DIVISION
QUARTERLY PROGRESS REPORT
for Period Ending October 31, 1951

J. H. Frye, Jr., Director

EDITED BY
W. H. Bridges

DATE ISSUED

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ABSTRACT

Eleven thorium-carbon alloys in the range 0.02 to 2.0% carbon have been prepared by vacuum-arc melting. The hardness varied from 19 R_A for the 0.02% alloy to 108 R_B for the 2.0% alloy. The preparation of thorium-chromium alloy has been hampered somewhat by a reaction with the zirconia molds, which results in porosity in the casting. A study of the effect of radiation upon thorium has been undertaken. Tests before and after irradiation are planned to determine effects on tensile properties, dimensional stability, hardness, impact, resistance, and thermal and electrical conductivity.

The failure of aluminum-silicon bonded X-10 slugs after heat treatment for seven days at 400°C has been traced to two separate causes. The failure to remove tin after the tin dip causes diffusion bumps, and the presence of a gas during heat treatment leads to blisters.

Tensile tests have been made on extruded thorium, and no correlation was found with extrusion variables. A composition effect was found, however, which showed a linear relationship between carbon content and tensile strength. Creep tests on thorium, uranium, and Inconel continue.

The program of evaluating the variables in the fabrication of clad fuel elements, using MTR techniques, has been concerned mainly with the distribution of UO_2 in the core compact, the temperature of rolling, and the particle size of the metallic core powder. Progress is being made in the preparation of three-layer laminates without the use of a protective can during the hot-rolling operation but with the use of a hydrogen atmosphere during the preparatory heating. The "rubberstatic" technique is being tried as a fabrication method for the preparation of a seamless, tubular, solid-fuel element.

Molten hydroxides of barium, sodium, strontium, lithium, and potassium and molten sodium cyanide have been used as the corrosive media in numerous corrosion tests at 1500°F. Nickel, copper, and Monel appear to be the only commercially available materials that resist chemical reaction with the molten hydroxides and sodium cyanide at this temperature. Mass transfer, however, presents a difficult problem. Stainless steels coated with copper and nickel are a logical development to combine high-temperature strength with the necessary corrosion resistance. Thin coatings, however, are not effective in combating corrosion, but heavier coatings show promise. Tests are under way to determine the minimum required thickness and the factors governing adherence of coatings. Nitrogen and carbon diffusion into

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metal appears as a primary difficulty in containing sodium cyanide at 1500°F in a number of commercial alloys. Nickel and iron, however, give no evidence of interaction. The stainless steels vary in their tendency to interact.

Mass transfer in thermal convection loops continues to cause early termination of runs in which the liquid media is a hydroxide. A mixture of the fluorides of potassium, lithium, and sodium has been circulating in a loop for some time with no apparent signs of difficulty. The hoop stresses in tubes of various materials are being investigated. A tube under helium pressure is immersed into a bath of the desired liquid and left for 1000 hr or until failure. So far, type-316 stainless steel and Inconel have been tested. Early failure of the Inconel is believed to be due to faulty tubing, since tubing from a new batch has been under test for some time without signs of failure.

The diffraction of neutrons by liquid metals has been attempted in order to gain information on the structure of liquids. Both lead and bismuth have been run in a specially designed furnace that fits the neutron spectrometer. A mathematical analysis of the data is under way. The basic studies on the interactions of solid and liquid metals continue. By careful control of temperature gradients and of thermal cycling, mass transfer in copper-bismuth systems could be made either negligible or appreciable. The largest temperature deviation that can be tolerated in thermal cycling of a static system is 2°C. A basic study has been started to determine the effect of oxygen on the corrosion of metals by sodium hydroxide. When heated in the presence of atmospheric oxygen, sodium hydroxide contained in nickel turns quite dark in color and a black, flaky precipitate appears on cooling. When heated in a hydrogen atmosphere no such discolorations or precipitates occur. Thermal convection loops are being run to determine the effect of oxygen on a dynamic sodium hydroxide—nickel system. The first loop was stopped after over 300 hr of operation even though it was operating in a perfectly satisfactory way. Appreciable mass transfer had occurred in this loop, which had been loaded in a helium atmosphere and sealed off under vacuum. Subsequent loops will be hydrogen fired and operated under a hydrogen atmosphere.

The feasibility of cone-arc welding of tube-to-header joints has been demonstrated. Improvement in the consistency of results will come from proper control of welding variables. Spot welding has been shown to be a good method for joining tube-bundle straps to fuel tubes. Work on furnace brazing as a joining technique has been started.

METALLURGY DIVISION QUARTERLY PROGRESS REPORT

THORIUM RESEARCH

THORIUM-ALLOY DEVELOPMENT

Two distinct phases of study have been pursued on the thorium-alloy development program. The first of these pertains to the investigation of the solubility of elements such as carbon, oxygen, and nitrogen in the relatively pure thorium metal obtained through the iodide process. The second phase involves the preparation and subsequent study of the properties of alloys of Ames thorium with elements such as chromium, nickel, molybdenum, vanadium, and columbium.

For the investigation of solubility of carbon, oxygen, and nitrogen in the pure iodide thorium metal, 11 alloys of carbon and thorium have been prepared. The carbon additions ranged from 0.02 to 2.0% in these alloys. Six of these alloys received a homogenization treatment at 950°C for 12 hr in a vacuum of approximately 8 μ, in order to provide satisfactory uniformity of structure.

For the study of the effects of alloying additions on the mechanical properties of Ames thorium metal, three alloys of thorium with chromium were prepared. The chromium content in these alloys varied from 0.25 to 1.0%.

Experimental Work. *Method of Preparation of the Thorium-Carbon Alloys.* In the preparation of the thorium-carbon alloys a small vacuum-arc furnace was used. This furnace is of conventional design with the exception of the chamber, which is a pyrex-glass bell jar instead of the usual water-cooled copper chamber. The furnace is shown in Fig. 1.

Fig. 1. Small Vacuum-arc Furnace.

The charge (approximately 90 g of the iodide thorium crystal bar) and the pyrex-glass bell jar were placed on the water-cooled copper base block, and the system was evacuated to approximately 3 μ, as shown on the G-E vacuum gage. Since an arc could not be struck against the copper base block in such a low vacuum, argon gas[1] was passed into the chamber in

[1] Ordinary tank argon gas passed over zirconium chips that were heated in an enclosed chamber at 850°C to remove oxygen, nitrogen, and probably water vapor.

sufficient quantity so that the Ashcroft vacuum gage (attached to the system) indicated a pressure of 10 in. Hg. After the arc was struck, the metal charge was melted at 200 amperes. The melted charge was subsequently rolled into a sheet. The rolled sheet was sectioned into appropriate sections, which were then placed into the arc-melting chamber in alternate layers with the master alloy.[2] This mass was melted several times in order to insure thorough solution of the carbon in the thorium. After the final arc-melting, the alloy was placed into an evacuated desiccator for storage until further treatment.

Composition and Hardness of Alloys. The composition and the as-cast hardness values of the alloys prepared by the foregoing method are given in Table 1.

TABLE 1

Composition and Hardness of Thorium-Carbon Alloys

ALLOY NO.	INTENDED CARBON (%)	HARDNESS (Rockwell)
F-8	0.02	19 *A*
F-13	0.05	24 *A*
F-3	0.10	36 *A*
F-22	0.13	59 *A*
F-6	0.15	46 *A*
F-9	0.20	51 *A*
F-11	0.50	57 *A*
F-19	1.11	
F-17	1.54	106 *B*
F-20	2.00	108 *B*

Homogenization of the Alloys. In order to insure satisfactory homogeneity in the thorium-carbon alloys,[3] a homogenization treatment was carried out. This treatment consisted of heating the alloys at 950°C for 24 hr in a vacuum of about 10 μ. It was thought that such a treatment would probably provide an alloy possessing satisfactory microscopic homogeneity.

Method of Preparation of the Thorium-Chromium Alloys. The thorium-chromium alloys were prepared in a vacuum-induction furnace of the crucible-tilting type. The required charge was placed into a stabilized zirconia crucible retained in a conventional induction coil mounted on a micarta base. This micarta base also supported a stabilized zirconia mold mounted on an appropriate holder in a horizontal position, so that as the crucible was tilted the mold attained a vertical position and received the liquid metal. This whole assembly was bolted, with appropriate vacuum seals, to a water-cooled copper chamber.

The system was first roughed-down with a Kinney mechanical pump, type-DVD-8810. When a pressure of 200 μ was obtained (as measured with an N.R.C. gage), the three diffusion pumps were turned into the system. After a pressure of 25 μ was obtained on the furnace, heating at a low rate was started (5 kw). In order to prevent a rise in pressure to over 200 μ, the heating was continued only intermittently. The use of the cold trap made it possible to operate the furnace at pressures of approximately 25 μ, and the heating was carried out to approximately 1650°C, at which temperature the melt was poured.

Soundness of the Thorium-Chromium Castings. It was observed that during pouring, as the liquid alloy came into contact with the zirconia mold, a rather sudden reaction occurred that was indicated on the furnace vacuum gage. After removal from the mold,

[2] An alloy of thorium and 2.0% added carbon was prepared in this unit previously by repeated arc-melting in a graphite crucible.

[3] It is thought that rapid melting followed immediately by rapid cooling provides conditions that are ineffective in obtaining a homogeneous alloy.

the casting showed porosity of the "blow-in" type approximately 1/16 in. below the cast surface.

Future Work. The alloys of thorium and carbon will be homogenized thoroughly in vacuum at 950°C. Following this, the individual alloys will be sectioned into pieces of about ¼ by ¼ in. for solubility studies at various temperatures.

Additional alloys of thorium with chromium will be prepared and poured into a previously heated and outgassed mold. The pouring temperature will be investigated further in an attempt to prevent the "blow-in" type of reaction.

RADIATION DAMAGE

A study of the effect of radiation upon thorium has been undertaken. The initial work involved preparation and irradiation of a number of thorium and thorium-alloy specimens to determine whether samples could be handled outside of a hot cell after irradiation. The samples were exposed in the ORNL X-10 graphite pile for one month and it was found that the induced activity was essentially that of Pa^{233}. The activity of 1 kg of thorium exposed at Hanford for 125 days will be about 5 rep/hr at 1 ft after 300 days of cooling. Thus, the activity of irradiated thorium will be too great to allow testing except in hot laboratories. Several slugs of thorium irradiated at Hanford for another purpose have been made available for tests of radiation effect, but little information on the original properties and condition of the slugs is available. Attempts are being made to locate additional slugs from the same billets that were not irradiated. These would be desirable for comparison with the irradiated slugs, since the physical properties of Ames thorium are quite variable. Specimens of Ames thorium are being machined into tensile specimens, impact specimens, and other shapes for hardness and conductivity measurements. These specimens will be submitted for irradiation at Hanford. Tests before and after irradiation are planned to determine effects on tensile properties, dimensional stability, hardness, impact resistance, and thermal and electrical conductivity.

THORIUM FABRICATION

The drawing of thorium wire and rod has been investigated to a limited extent. Wire was drawn to 5-mil diameter by encasing 1/8-in.-diameter rod in copper tubing to provide a copper bearing. Rod was drawn bare from ½ in. diameter to ¼ in. diameter by the use of a mixture of "Molykote," fumed lead and zinc oxide, and lacquer. An extremely good surface was produced on the finished rod with no intermediate annealing and only one additional application of the lubricant.

Drawing speeds, alterations of the lubricant, and reductions per draft will be investigated now that a lubricant has been found that will permit drawing. Drawing with several recommended or suggested commercial lubricants was attempted with little or no success. Drawing appears possible with a lubricant composed of pulverized copper suspended in a lacquer to make it adhere to the metal.

A drawing speed of about 8 ft/min produced an excellent surface on a 0.226-in.-diameter rod with lubricant consisting of a mixture of two parts molybdenum sulphide to one part fused lead and zinc oxide suspended in a "Duco" lacquer binder.

X-10 SLUG PROBLEM

The use of aluminum-silicon bonded slugs in the X-10 graphite pile was recommended after an extensive study indicated that bonded slugs were much less susceptible to can failure by diffusion between the aluminum can and uranium than were the unbonded slugs. As a result, a production line was set up to manufacture triple-dipped bonded slugs for future use. The Hanford canning process was copied directly and no operating difficulties were anticipated.

After some 10,000 slugs were canned, it was found that when the slugs were given a 400°C heat treatment for seven days, extensive diffusion was taking place and causing "bumps" on approximately 20% of the cans. Since the Hanford process was copied so closely, it was felt that some minor deviation in the procedure was responsible for the failures. The canning process, in brief, is as follows: The alpha-rolled uranium slugs are machined to the proper size and placed into a bronze bath for 50 sec to give the uranium a beta heat treatment (bath temperature 720°C). The slugs are then given a dip in a tin bath (600°C) to dilute the copper coating on the slug and to quench the uranium from the beta temperature range. The slugs are removed and placed in a centrifuge to remove the excess tin. They are held in a holder consisting of two metal rings and a rod for bottom support. It should be pointed out that the contact between the rings and the slug affords a chance for a heavier deposit of aluminum-silicon at this point than on the remainder of the slug. The slugs are removed from the centrifuge and placed in a bath of eutectic aluminum-silicon for 6 sec to dissolve the tin and form a compound layer of aluminum-silicon-uranium, which acts as a barrier to diffusion between the aluminum can and the uranium slug. The slugs are canned in another aluminum-silicon bath and quenched in cold water. The slugs are tested in an autoclave at 130-psi steam pressure to detect faulty welds and diffusion resulting in bumps. The good slugs from the autoclave test are given a heat treatment of 400°C for seven days. The slugs showing small blisters or bumps after the heat treatment are rejected.

A metallographic examination of a rejected slug showed that the barrier had failed at numerous points and allowed rapid diffusion to take place between the aluminum can and the uranium. A rejected slug that showed extensive "bumping" was removed from the aluminum can and submitted for a spectrographic analysis to determine any gross differences in the areas where diffusion had taken place and areas where there was no diffusion. A high concentration of tin was found to be associated with the areas showing diffusion. No tin was detected on the remaining area where the bond was good, and no diffusion had occurred. Another rejected slug was given the same treatment; 12 separate bumps were checked as well as 12 regions where no bumping occurred. It was found that tin invariably was associated with the bumps, and it was concluded that the residual tin accelerated diffusion between aluminum and uranium and was responsible for the can failures.

To further prove that the tin is detrimental, the aluminum can was stripped from a rejected slug (400°C test for seven days) and the compound layer removed from the slug by a light machine cut in a lathe. The turnings were collected and submitted for quantitative analysis. The aluminum

can was scraped inside to remove any adherent compound. The total tin content of the turnings was 303 milligrams. The aluminum scrapings contained 101 mg of tin. Another slug that had passed the 400°C heat treatment and was considered to be satisfactory was given the same treatment and no tin was found either in the turnings or in the scrapings from the can.

The surfaces of ten more slugs were tested with the following results.

GOOD SLUGS		REJECTED SLUGS	
SLUG NO.	TIN CONTENT (mg)	SLUG NO.	TIN CONTENT (mg)
N-756	<1	N-761	~300
N-757	<1	N-762	~100
N-758	<1	N-763	~100
N-759	<1	N-764	~300
N-760	<18	N-765	~75

On the basis of the above results, it was recommended that the time in the aluminum-silicon bath be doubled to allow more time for the tin coating to be dissolved away from the slug. Subsequently, about 200 slugs were canned where the time in the aluminum-silicon bath was increased to 12 seconds. One hundred and forty of these slugs were tested for seven days at a temperature of 400°C and only four failures could be attributed to diffusion. This represented a tenfold improvement. Three of the four rejected slugs showed bumps that were located near the position of the ring on the holder used for handling the slugs. This suggests that a heavier deposit of tin was left on the slug at this point, and even the increased time in the aluminum-silicon bath was insufficient for complete dissolution of the tin.

It had been observed during the canning process that if the temperature of the bronze bath accidentally rose to a higher value than 720°C the slug would not be wetted uniformly and these non-wetted areas would result in bumps during the autoclave test. Because of this observation, one lot of slugs were canned with the temperature of the bronze bath reduced from 720 to 710°C. Eighty of these slugs were tested for seven days at 400°C, and only one slug was rejected because of bumps.

The Metallurgy Division was asked to heat treat some of the canned slugs at 550°C to further test the bonded slugs. Forty-four slugs that had looked good after a 400°C test for seven days were loaded in a furnace together with 44 slugs that had not been heat treated previously. These slugs were treated in a nitrogen atmosphere to prevent excessive oxidation of the graphite furnace and minimize oxidation of the uranium in case a can ruptured. Since the bumps which had been observed were due to diffusion and not faulty welds or holes in the can, the atmosphere was considered unimportant. The nitrogen was introduced into the furnace at the rate of 10 ft^3/hr. The slugs were held at 550°C for seven days with periodic inspection to detect obvious failures. At the end of seven days the slugs were inspected and measured and no bumps were found. Two slugs that had not been heat treated previously were larger in diameter than before the heat treatment. Possibly some general diffusion took place, but there was no evidence of localized attack such as causes bumps on the can.

Since a protective atmosphere was used in the first 550°C heat treatment, the same slugs were heated to 550°C in air and held at this temperature for seven additional days. Thus, the slugs received a total of 14 days of heat treatment at 550°C; seven days in nitrogen atmosphere and seven days in air. No bumps were observed on the

44 slugs that had been tested at 400°C; however, six of the slugs not previously tested showed bumps. These bumps were unlike the failures observed at 400°C in that they covered larger areas and seemed to be due to general diffusion instead of the localized attack that results in sharp pimples. Since an unbonded X-10 slug would fail by diffusion in one day or less at 550°C, there can be no question as to the efficiency of the aluminum-silicon bond.

Apparently the lot of 44 slugs that had not been given a prior heat treatment happened, by chance, to be a superior lot in which the tin was effectively removed and not much diffusion occurred. This is a reasonable assumption since another lot of 88 bonded slugs from the production line were given a 550°C heat treatment for seven days in a nitrogen atmosphere and ten slugs failed. Nine of these failures had developed after only 32 hr at temperature. Two more lots were tested at 400°C for seven days and it was found that no failures occurred in one lot while the other showed about 55% failures. This suggested that some operation was very near a critical condition where only a minor change would make the difference between a good slug and a bad slug. Approximately 535 slugs were canned after two changes were made in the canning procedure. The temperature of the bronze bath was lowered from 720 to 710°C and the time of dip in the aluminum-silicon bath was increased from 6 to 12 seconds. Both of these changes were designed to reduce the tin content of the slugs and eliminate the source of pimpling. Previous work had shown that when these changes were made independently, the rate of rejection dropped from about 20% to 2 or 3%. However, when the 535 slugs were tested for seven days at 400°C, it was found that 25% had blistered or pimpled. Metallographic examination as well as radiographs showed that the large blisters were voids and contained nothing more than a gas. Some of the slugs showed small pimples caused by diffusion. Thus, it is apparent that two entirely separate phenomona are present, and they probably have no connection.

Typical slugs showing the two characteristic types of failure are shown in Figs. 2 and 3. Photomicrographs showing sections through a diffusion bump and a gas blister are shown in Figs. 4 and 5.

An additional 500 slugs are being canned using the same technique except that the aluminum-silicon bath will be fluxed with a stream of argon gas in an attempt to remove gas from the bath. It is hoped that this will eliminate the source of blisters. All of the evidence to date has indicated that tin is responsible for the failures occurring by diffusion. Since the two changes made did reduce the tin content but did not entirely eliminate it, some consideration is now being given to an alternate plan for giving the alpha-rolled slugs a beta heat treatment. In this plan, the bronze bath and the tin bath would be completely eliminated.

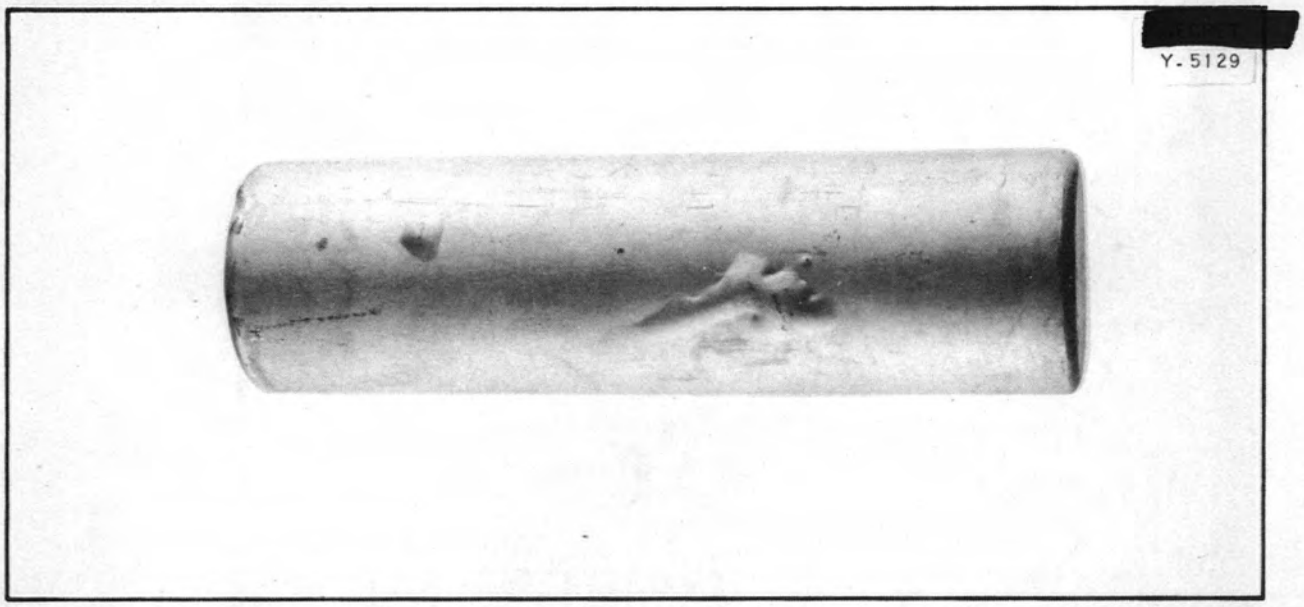

Fig. 2. Typical Gas Blistering of X-10 Slug.

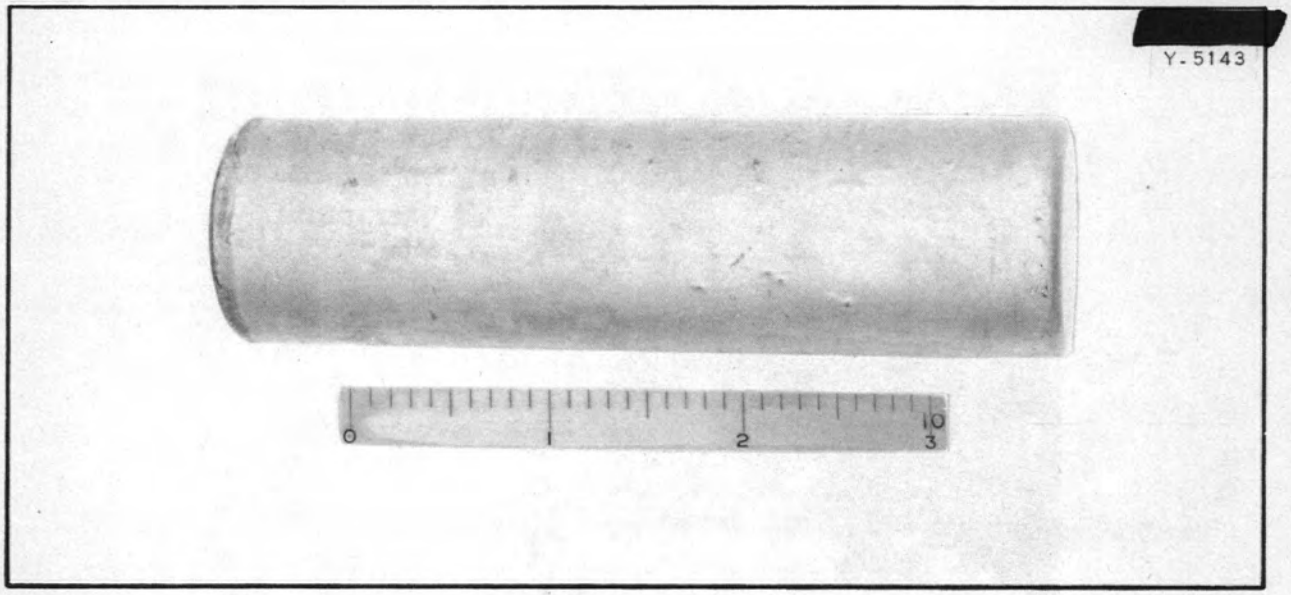

Fig. 3. Typical Diffusion Pimples of X-10 Slug.

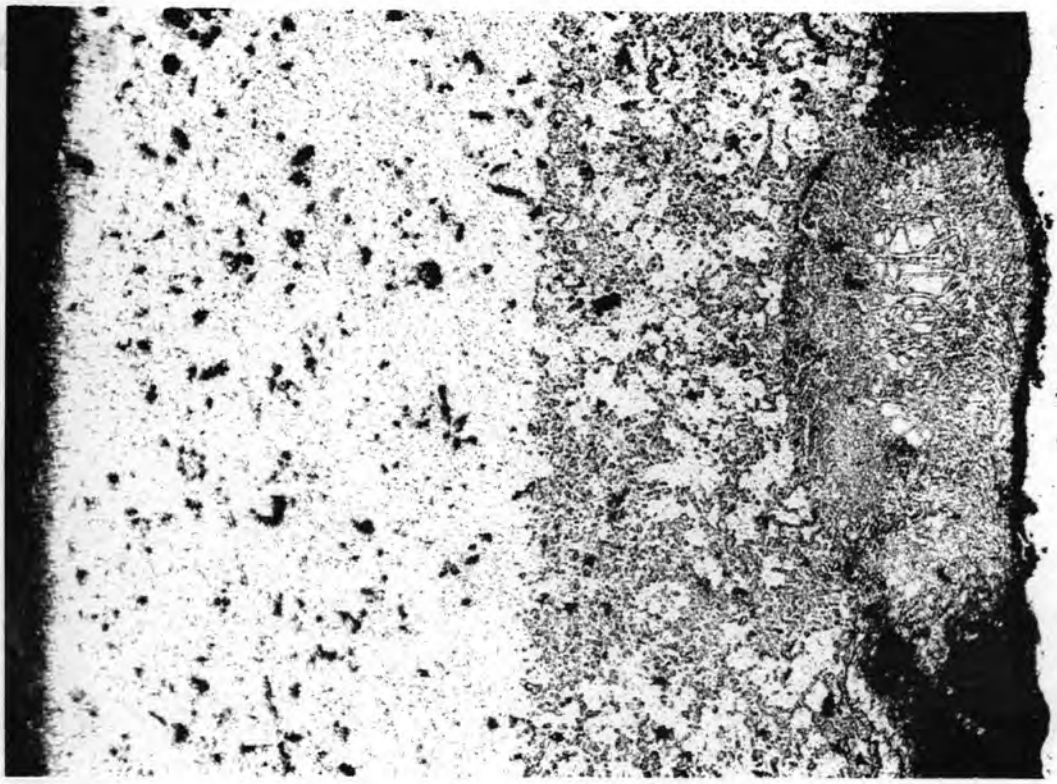

Fig. 4. Section Through a Diffusion Bump. 100 X.

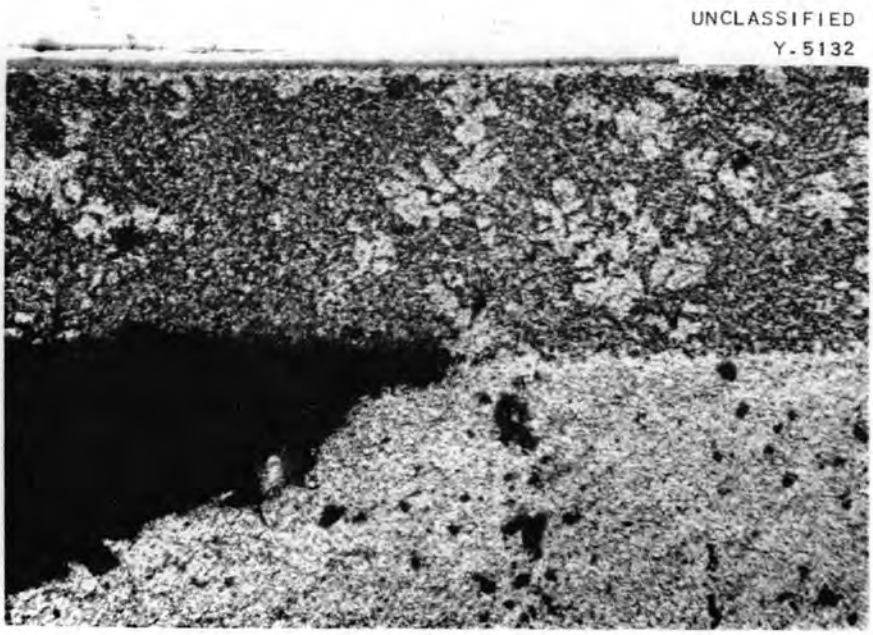

Fig. 5. Section Through a Gas Blister. 100 X.

MECHANICAL PROPERTIES OF METALS

THORIUM

During the past quarter and part of the previous quarter 14 thorium creep tests in vacuum at 300°C have been concluded. Stress levels from 2,000 to about 27,000 psi were used with test durations up to 1600 hours. The data obtained cannot be specifically interpreted at present since the several samples were of widely varying chemistry; the carbon plus beryllium content varied from less than 0.05 to about 0.12% and room-temperature tensile strengths varied from 27,000 to 39,000 psi. In the absence of more suitable extensometers, the extensions were measured with external dial gages. Thus, these measurements include all of the stretch and slack in the loading system.

However, several general statements based on this data can be made. Within the limits of the carbon and beryllium content encountered, both the room temperature and elevated temperature strengths noticeably increase with increased total carbon plus beryllium content. Prior cold work, up to 5% plastic deformation, results in a slightly decreased creep rate. For thorium with a carbon plus beryllium content greater than about 0.095%, the increase in creep rate as a function of increasing stress (up to about 19,000 psi) is smaller than would be expected.

Tensile-test specimens were prepared from each rod extrusion made during a recent investigation of the fabrication qualities of extruded thorium. At the time it was thought that it might be possible to correlate mechanical properties with extrusion variables. The specimens were tested in a 120,000-lb Baldwin-Southwark tensile-testing machine at a constant strain rate of 0.1 inch per minute. Examination of the data failed to show any correlation with extrusion variables. It was noted, however, that the values tended to fall into three distinct groups. These groups could be correlated to original ingot numbers, indicating a composition effect. A complete analysis was obtained of each specimen and it was found that the carbon content (possibly carbon plus beryllium) has considerable effect upon the mechanical properties of thorium. This tendency has been noted in the past, but the present series of samples shows the linear relationship between carbon content and tensile strength. The mechanical properties of the extruded thorium and the analysis of each specimen are shown in Table 2.

URANIUM

An attempt was made to obtain qualitative information on the effect of thermal cycling on the dimensional change of uranium during creep testing. A test bar was placed in the furnace and a positive stress of less than 200 psi was applied. The dimensional changes were observed on an external dial gage (smallest division, 0.001 in.). The temperature was held to within a ±0.5°C cycle with a 24-hr period. During about 300 hr with this temperature control (preceded by some temperature cycling) the specimen showed a small but detectable growth. When the cycle was changed to about ±4°C with a 10-sec period there was increased and erratic growth. It was also noted that a period of about 25 hr elapsed before the increased growth rate caused by the thermal cycling started. This experiment is to be repeated using an internal extensometer so that quantitative data can be obtained.

INCONEL

Sixteen tests have been run on bar and sheet specimens with both fine- and coarse-grain sizes. These tests

TABLE 2

Mechanical Properties of Extruded Ames Thorium

SPECIMEN NO.	ELONGATION IN 2-in. GAGE LENGTH (%)	REDUCTION IN AREA (%)	YIELD POINT (psi)	TENSILE STRENGTH (psi)	BREAKING STRENGTH (psi)	ANALYSIS			
						Th (%)	C (%)	Be (%)	Si (%)
A223A	50	75	22,100	30,500	58,000	99.5	0.045	0.017	0.27
A222C	52	71	20,750	30,300	60,300	99.9	0.053	0.017	0.35
A227A	51	74	21,100	29,750	52,000	99.6	0.051	0.022	0.18
A234A	52	74	22,700	31,050	58,000	99.6	0.050	0.014	0.11
Average values	51	74	21,600	30,400	57,000	99.7	0.05	0.017	0.28
A231A	54	74	27,970	34,200	72,500	99.8	0.070	0.021	0.14
A231B	48	74	27,500	34,750	75,000	98.9	0.076	0.021	0.19
A231C-1	50	71	27,550	34,300	64,300	98.4	0.074	0.018	0.20
A231C-2	52	73	27,400	34,500	72,300	99.4	0.062	0.034	0.20
Average values	54	74	27,600	34,400	71,000	99.1	0.071	0.24	0.18
A239C-1	48	70	33,100	38,200	77,500	99.0	0.095	0.029	0.26
A239D-2	46	70	31,900	38,900	79,400	98.8	0.084	0.028	0.22
A239D	49	72	31,400	37,750	73,300	98.5	0.085	0.031	0.30
A239B	48	65	32,050	38,750	70,500	98.1	0.088	0.029	0.12
A239A	49	69	31,250	39,000	78,500	98.5	0.098	0.016	0.37
Average values	48	69	31,900	38,500	75,800	98.6	0.09	0.026	0.25

were conducted at 815°C in an atmosphere of purified argon and at stress levels from 3000 to 8000 psi. At all stress levels, the bar specimens exhibited a far greater creep resistance than the sheet specimens. Coarse-grained sheet specimens had about one-half the creep rate of fine-grained sheet specimens at stresses below 5000 psi. At stresses above 5000 psi the fine-grained specimens had the greater creep resistance. Rather massive edge cracking was observed on all fine-grained sheet specimens.

Z-NICKEL

A program was initiated to study the creep properties of Z-nickel. In the solution-treated and aged condition the creep properties in argon at 815°C and 2000-psi stress appeared to be good enough to warrant further investigation if the corrosion properties had been acceptable. Since corrosion tests show Z-nickel to be unsuitable for containing hydroxides, these tests have been discontinued.

Considerable time has been devoted to calibration of the lever-arm testing machines using SR-4 type A-12 strain gages. The lever-arm counter weights have been shifted so that the change in tare weight of the lever arm as it swings through its usable arc is equal and opposite to the spring rate of the sealing bellows on the testing chamber.

ANP PROGRAM

SOLID FUEL ELEMENT FABRICATION

Fuel Element Fabrication Variables. Work has continued on evaluating the variables in fabrication of clad fuel elements using techniques characteristic of the MTR fuel element fabrication.

Earlier efforts toward producing a UO_2-stainless steel core clad with stainless steel indicated that the distribution of UO_2 was dependent on several factors. Three of these factors were the UO_2 and core metallic particle sizes and the temperature at which the rolling operation was carried out. Effects of variation in size of the metal particles used to make the core were shown in a previous report.[1]

Figure 6 illustrates the effect of UO_2 particle size on distribution. The range of particle sizes includes those measuring 250 μ to particle size smaller than 10 microns. Estimates of the range of fission fragments indicate that the optimum particle size for minimum damage to the metallic network lies within this spread of sizes. Confirmation must await the results of inpile tests.

The effects of the temperature chosen for rolling of the clad cores are shown in Fig. 7. An increase in the processing temperature results in greater uniformity of oxide distribution. This improvement in core quality, however, is accompanied by a greatly increased rate of oxidation of the protective capsule in which the laminate is contained. Deterioration of the capsule is followed by roughening of the surface of the cladding.

Elimination of Capsule. The sequence of operations, as described in previous reports,[1,2] for producing the laminated sheets discussed above includes a canning operation that is quite troublesome and time consuming. Production of solid fuel plates in quantity would almost necessitate elimination of this step. Two laminates that were prepared without benefit of a capsule show considerable promise toward doing away with the protective can. The service performed by the capsule in the original method of fabrication was the exclusion of air from contact with the cladding and core during hot rolling. Thus, the essentially oxide-free surfaces were preserved and a maximum opportunity for bonding was assured.

It was reasoned that the cladding layers could be made to serve as a protective envelope for the core during rolling if the edges could be effectively sealed and if excessive oxidation of the cladding could be prevented during rolling. The first sample was prepared by arc-melting the edge of a three-layer laminate in a dry-box containing a dried helium atmosphere. The second sample was sealed by the use of stainless steel powder in the following manner: A thin layer of the powder suspended in Nicrobraz cement was painted in a narrow strip about the outer edges of the picture frame, after which the sample was assembled and sintered overnight at 1250°C. Sintering was followed by a hot-forging operation in which the laminate was removed from the furnace at 1000°C and pressed at 5 tons per square inch. A protective hydrogen atmosphere was used for heating preparatory to rolling. Oxidation of the cladding due to contact with air while rolling was quite mild and did not reach the scaling stage.

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[1] *Metallurgy Division Quarterly Progress Report for Period Ending April 30, 1951,* ORNL-1033.

[2] *Metallurgy Division Quarterly Progress Report for Period Ending January 31, 1951,* ORNL-987.

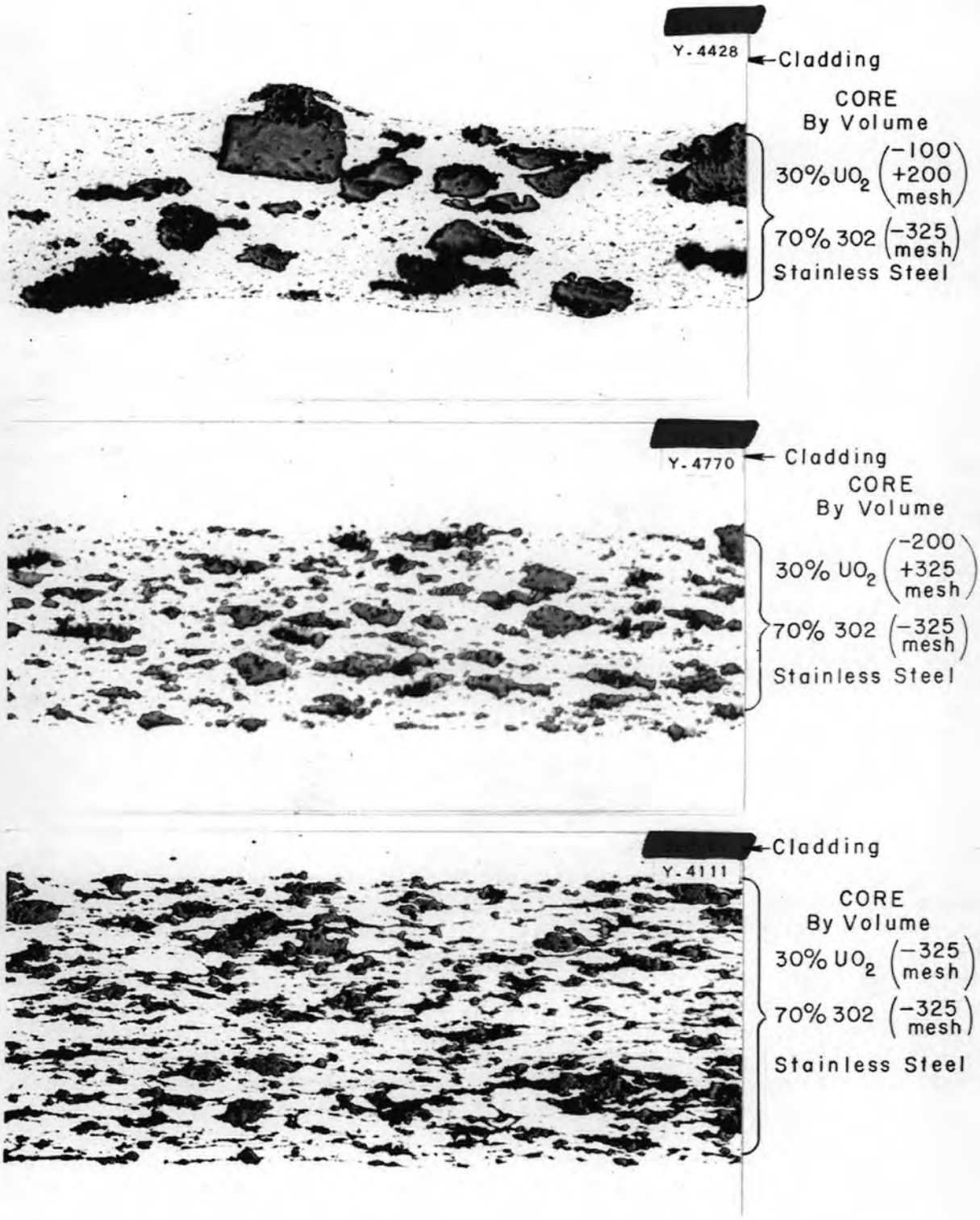


Fig. 6. Effect of UO_2 Particle Size on Distribution. 175 X.

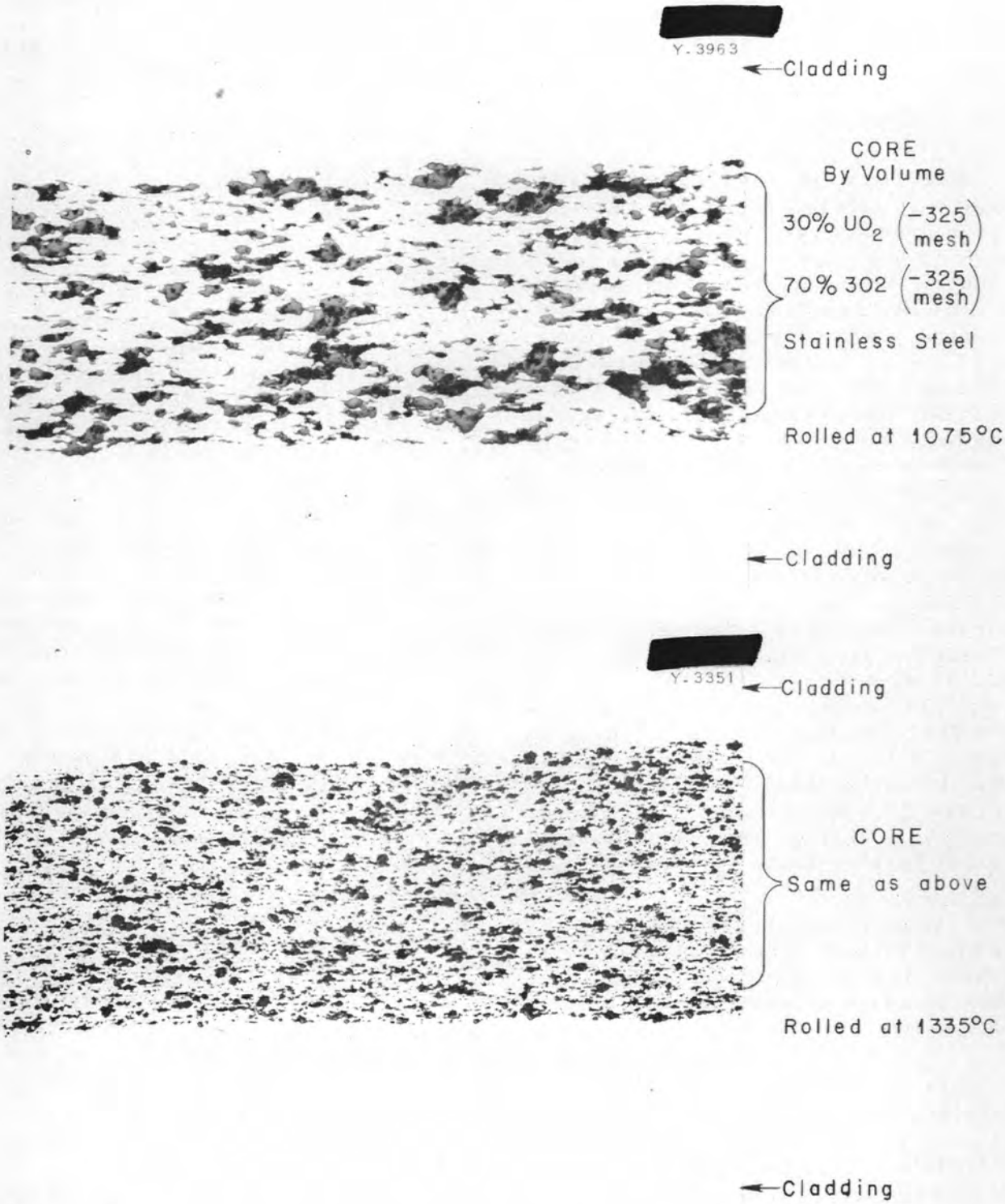


Fig. 7. Effect of Rolling Temperature on Distribution. 175 X.

Figure 8 includes pictures of the two specimens and shows that region where the cladding and picture frame meet. The integrity of the bond is evident, showing that air did not find its way between the sheets.

Seamless Element Fabrication. Seamless, tubular, solid fuel elements may be needed, and efforts are being made to work out a fabrication technique. The method being investigated at present involves pressing a fuel-bearing powder layer along the inner surface of a tube and sintering, followed by pressing a second tube against the surface of the sintered powder layer. A second sintering operation completes the schedule.

A distribution of pressure approaching that of a hydraulic system is obtained along the inner surface of the powder layer by use of a rubber insert inside the tube. The rubber insert is slightly smaller than the inside diameter of the tube and the resulting annulus serves to receive the fuel-bearing powder. Loading is applied to the ends of the rubber insert. A hardened steel back-up die serves to support the tube during pressing. This pressing operation is called "rubberstatic" pressing.

Figure 9 illustrates the type of bond obtained after sintering the powder layer. A better quality of bond is obtained when the UO_2 used is of a coarse fraction.

Cold Drawing of Tubular Laminates. Tubular elements formed from two flat plates pressed into semicircular cylinders and welded together at the two seams are being cold drawn to smaller diameters and wall thicknesses.

A tube fabricated by the "rubberstatic" technique is also being drawn. Subsequent metallographic examination will tell whether this program will yield sound tubular elements of small diameter and usable length.

Fuel Element Testing. Nondestructive checks of the quality and extent of bonds in a solid fuel element after processing are very desirable. Two of several check methods that may yield usable information were observed at the Westinghouse Power Division Laboratories. One involved use of electrical contacts that measured the variation in resistance across a laminate. The other method involved use of a double-crystal Sperry Reflectoscope.

Of the two systems, the resistance measuring device showed a greater sensitivity in picking up known imperfections in a prepared laminate. Use of the Sperry Reflectoscope for this type of examination would require a semiskilled operator for operation and interpretation. However, neither of the systems would lend itself to examination of fuel plates in quantity without an extensive development program.

Thermal Cycling of Fuel Plates. Three plates clad with type-316 stainless steel and prepared by the hot-rolling procedure have been cycled alternately between a temperature of 1000°C and back to room temperature ten times each without external signs of blistering. The compositions of the cores (volume %) are as follows:

30% UO_2 (-100 mesh)	70% Fe (10 μ)
50% UO_2 (-100 mesh)	50% Fe (10 μ)
30% UO_2 (-100 mesh)	70% type-302 stainless steel (-325 mesh)

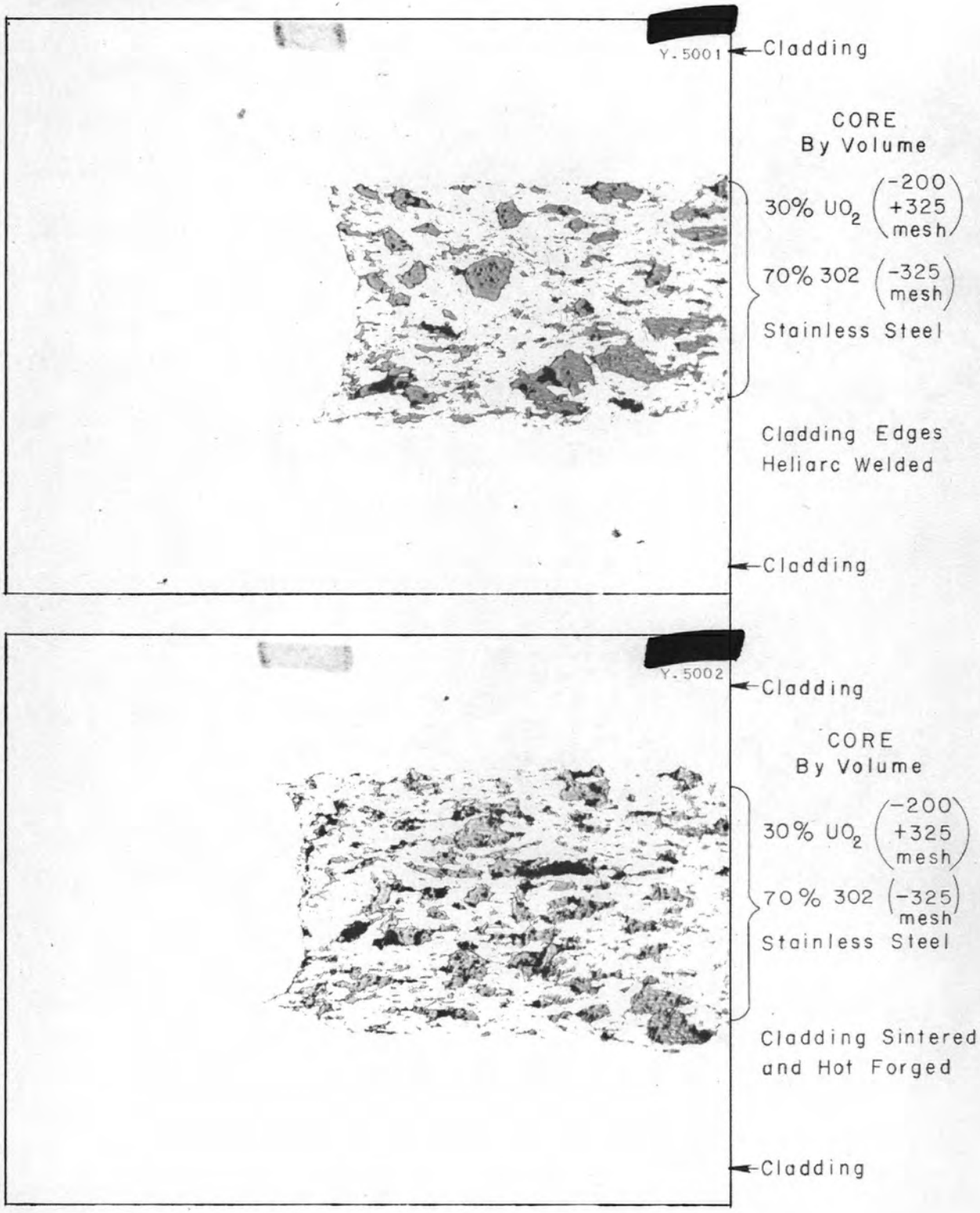
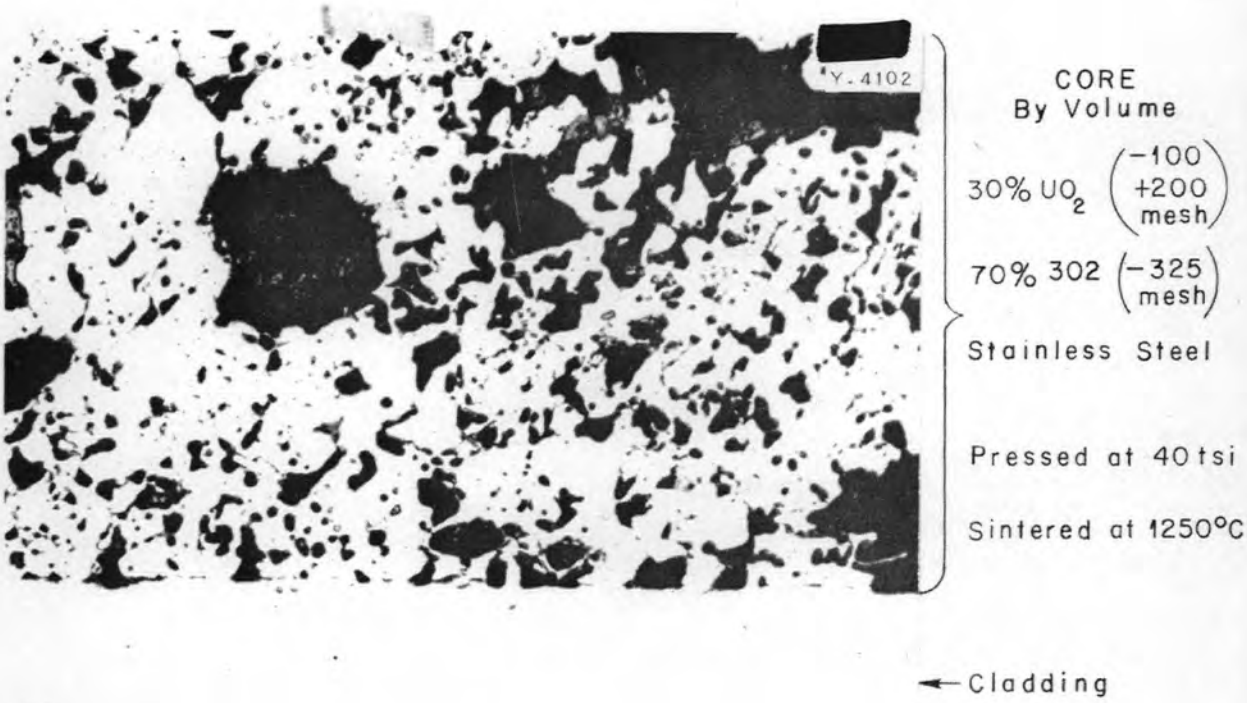
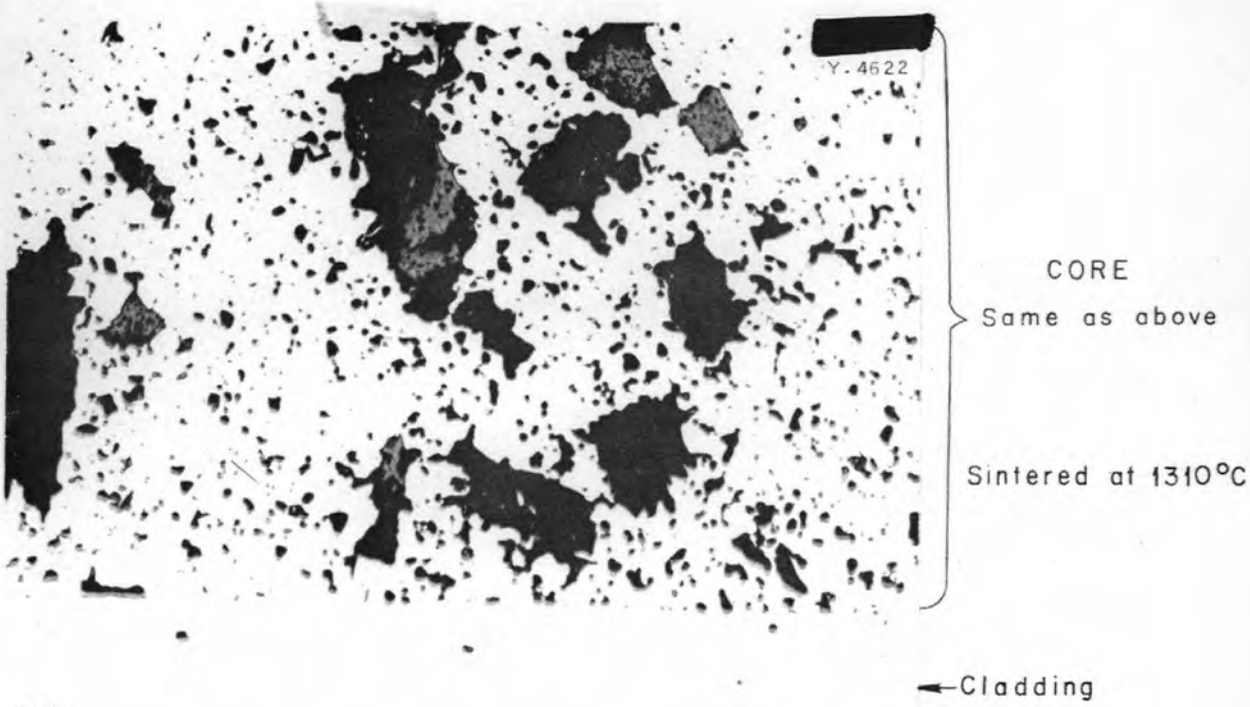


Fig. 8. Elimination of Capsule. 150 X.



(a)



(b)

Fig. 9. Samples Pressed by "Rubberstatic" Method. (*a*) 250 X, (*b*) 175 X.

CORROSION STUDIES

Numerous corrosion tests have been made at 1500°F in two types of nonmetallic media which may find possible uses as reactor moderators or coolants, namely, molten hydroxides of barium, sodium, strontium, lithium, and potassium and molten sodium cyanide. The reactions between these various hydroxides and metals at high temperatures do not seem to be very sensitive to the nature of the positive ion. Nickel, copper, and Monel are the only commercial metals that appear to resist chemical reaction with all molten hydroxides and sodium cyanide at this temperature. The phenomenon of crystal formation, however, still persists as an objectionable problem.

Stainless steels coated with copper and nickel were therefore a logical development to combine high-temperature strength with the necessary corrosion resistance. However, thin coatings are not effective in suppressing corrosion, whereas heavier coatings definitely show promise. Tests are under way to determine the minimum required thickness and the factors governing adherence of coatings.

A number of commercial alloys tested in molten sodium cyanide at 1500°F indicate that nitrogen and carbon diffusion into metal is the primary reaction, which frequently results in embrittlement. However, several metals appear to be quite inert to molten cyanides. Nickel and iron gave no evidence of interaction, whereas stainless steels exhibited varying degrees of tendency for reaction.

Corrosion by Molten Hydroxides. Corrosion tests have been made at 1500°F for 100 hr with molten hydroxides on nickel-plated, nickel-clad, copper-plated, and unplated stainless steel specimens. In a few instances partial and complete protection from corrosive attack was effected by these noble-metal coatings. They involved nickel coatings above 0.003 in. in thickness and copper coatings of undetermined thickness. The use of a hydrogen atmosphere above molten hydroxide was found to result in considerably less corrosion than similar instances in which vacuum or air was used. Mass transfer effects under the influence of a thermal gradient were also minimized in the few tests thus far made.

Coated Specimens. Nickel and copper are two of the few metals known to be resistant to reaction with molten hydroxides (Monel and silver are also relatively inert at 1500°F). The application of these materials as protective coatings on stainless steels for use in contact with hydroxides was therefore undertaken. Although this work is still in progress, preliminary results indicate that corrosion protection can be effected in this manner.

Type-304 stainless steel was found to be severely attacked by sodium hydroxide at 1500°F in a 100-hr test. The depth of metal affected because of oxidation by the molten caustic was measured and found to be 0.025 inch. Hence, it was decided that protective coatings of nickel and/or copper could be evaluated by depositing them on this easily oxidized steel.

Electroplating. A 0.003-in. copper-plated coating on type-304 stainless steel reduced attack to 0.005 inch. A 0.003-in. nickel plate also resulted in reduced attack (0.009 in.) on the underlying stainless steel. Copper-plated specimens were tested in copper capsules, whereas nickel-plated specimens were tested in nickel capsules. Electroplated specimens

that were immersed in molten sodium hydroxide were observed to have poor plate adherence; whereas the coatings on similar specimens immersed in molten barium hydroxide were very adherent and generally resulted in improved corrosion protection. The results of these 100-hr tests at 1500°F are summarized in Table 3.

nickel coating was approximately 0.005 in. thick at the top and bottom and appreciably heavier at the sides. Figure 11 illustrates the results. The thin sections of nickel were attacked, whereas the thicker side sections were not attacked. There is no satisfactory explanation for this behavior at the present time.

TABLE 3

Results of 100-hr Corrosion Tests on Electroplated Type-304 Stainless Steel

COATING	CAUSTIC	DEPTH OF METAL ATTACK (in.)	WEIGHT CHANGE (g/in.^{2})	REMARKS
Unplated	NaOH	0.025 uniform	+7.5	Uniform attack
0.003-in. Cu	NaOH	0.005 uniform	+0.094	Copper plate not adherent
0.003-in. Ni	NaOH	0.009 uniform	-0.094	Nickel plate not adherent
Unplated	$Ba(OH)_2$	0.029 uniform	>1.0	Uniform attack
0.003-in. Cu	$Ba(OH)_2$	0.094 maximum	-1.92	Adherent copper plate
		0.027 average		
		0.001 minimum		
0.003-in. Ni	$Ba(OH)_2$	0.001 uniform	+0.002	Adherent nickel plate

In barium hydroxide, the 0.003-in. nickel plate offered good protection. There was, however, a small amount of oxidation observed, as shown in Fig. 10. In the case of the 0.003-in. copper plate, severe attack was noted on the specimens, however, the corners were relatively unattacked. This may possibly be attributed to the heavier electroplate that occurs at corners. Heavier copper coatings will be tested in barium hydroxide in an attempt to ascertain this possibility.

Cladding. Nickel-clad type-316 stainless steel and nickel-clad Inconel were prepared using the picture-frame technique and tested in sodium hydroxide for 100 hr at 1500°F. The

A heavier nickel cladding (0.010 in.) on Inconel and type-316 stainless steel produced from nickel powder gave excellent protection from corrosion in sodium hydroxide at 1500°F for 100 hours. No evidence of oxidation was noted, as may be seen in Fig. 12.

Uncoated Specimen. A number of metals were tested in molten hydroxides of sodium, potassium, barium, and strontium at 1500°F for 100 hours. These were conducted in containers that were the same composition as the specimen. The results of these tests are summarized in Tables 4, 5, and 6. Figures 13 through 22 illustrate the nature of attack.

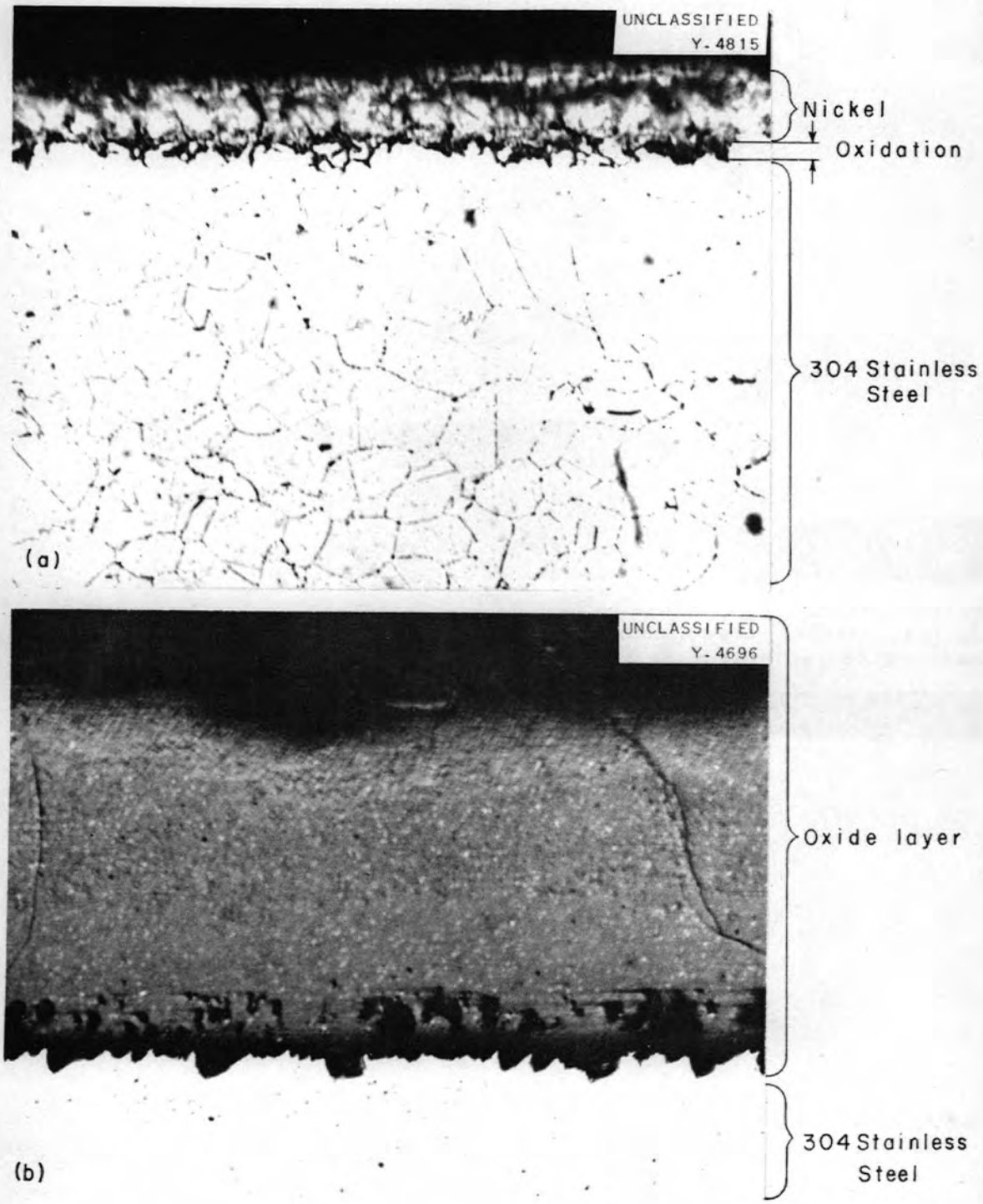


Fig. 10. (*a*) **Type-304 Stainless Steel Coated with 0.003-in. Nickel Plate After 100-hr Exposure to Barium Hydroxide at 1500°F.** 500 X. (*b*) **Uncoated Type-304 Stainless Steel After 100-hr Exposure to Barium Hydroxide at 1500°F.** 250 X.

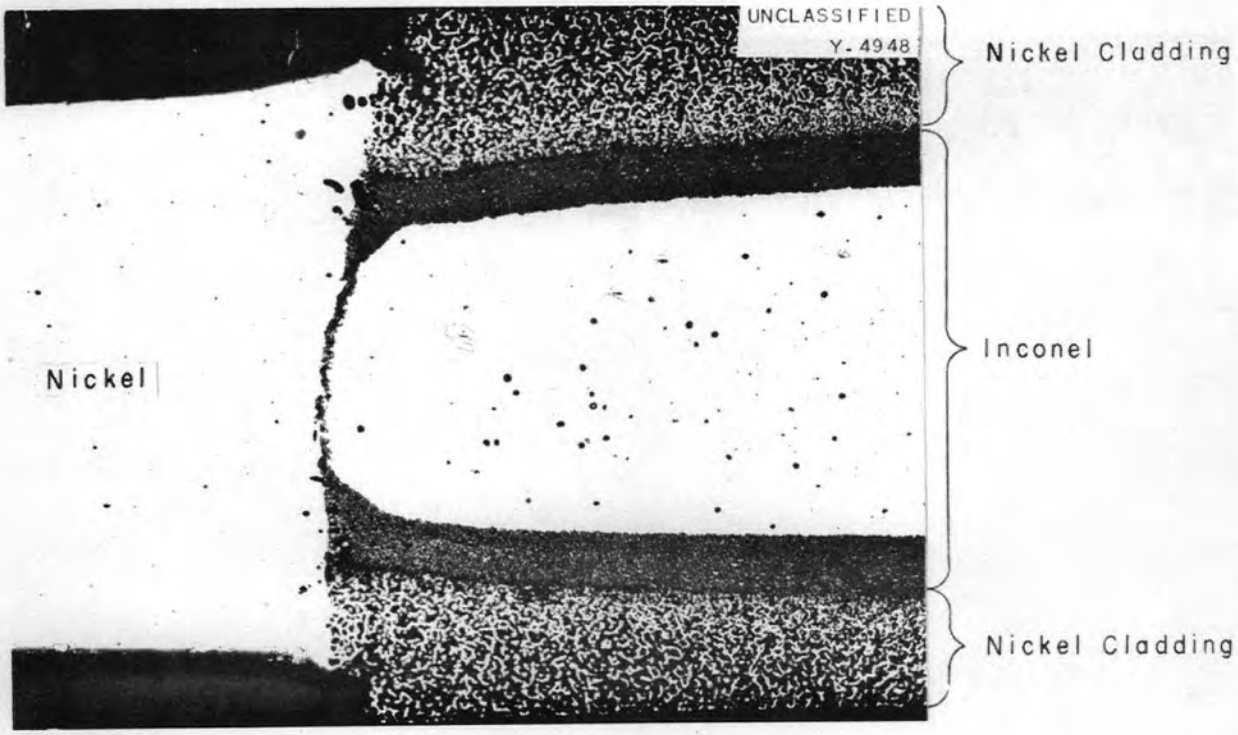


Fig. 11. Nickel-clad Inconel (Prepared Using Nickel Sheet) After 100-hr Exposure to Sodium Hydroxide at 1500°F. 50 X.

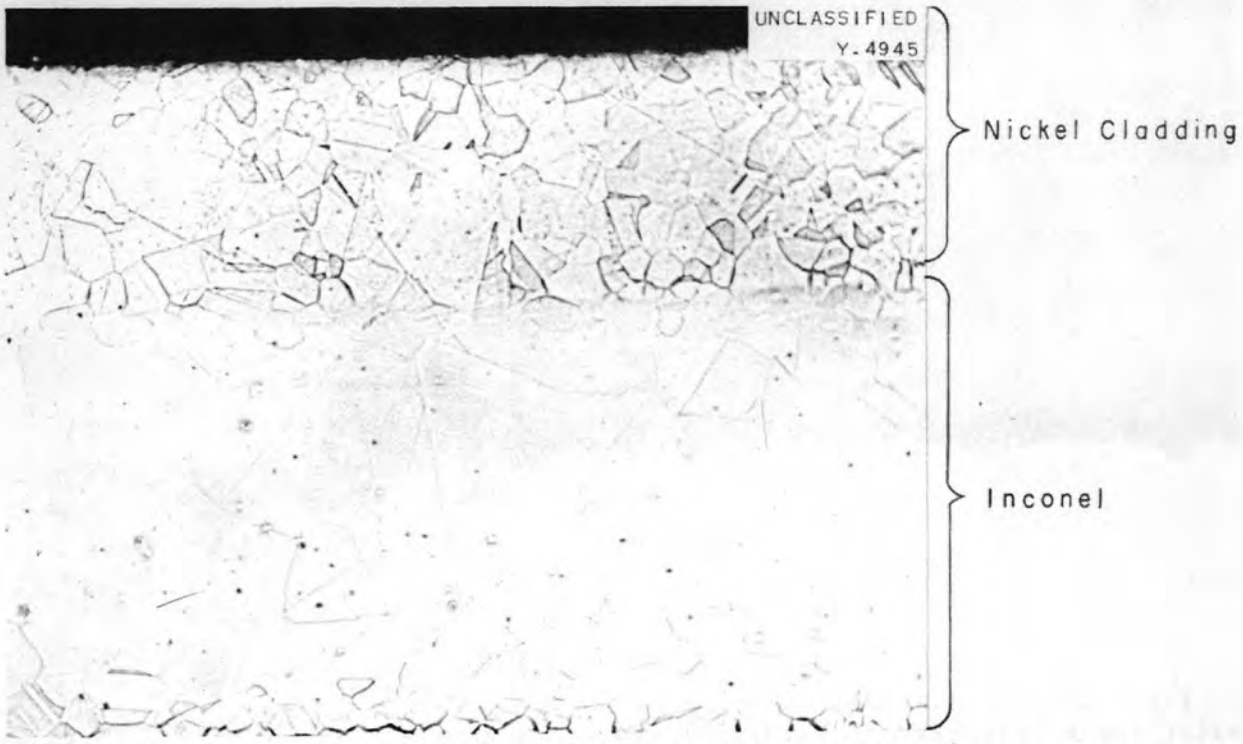


Fig. 12. Nickel-clad Inconel (Prepared Using Nickel Powder) After 100-hr Exposure to Sodium Hydroxide at 1500°F. 100 X.

TABLE 4

Summary of Corrosion by Barium Hydroxide at 1500°F in 100 Hours

Specimens tested in evacuated capsules of like material

MATERIAL	WEIGHT CHANGE (g/in.^{2})	DEPTH OF METAL AFFECTED (in.)	METALLOGRAPHIC NOTES
304 stainless steel		0.0055	0.010-in. oxide layer
310 stainless steel	+0.046	0.0035	0.005-in. oxide layer
316 stainless steel		0.0090	0.010-in. oxide layer
318 stainless steel	-0.061	0.0045	0.006-in. oxide layer (Fig. 13)
321 stainless steel	-0.740	0.0045	0.005-in. oxide layer
347 stainless steel	-0.083	0.0047	0.006-in. oxide layer
446 stainless steel		0.0050	0.008-in. oxide layer (Fig. 14)
Copper (OFHC)	-0.003	0.0005	No evidence of attack
Iron (Globe)	-0.994	0.0085	Heavy oxide formed
Zirconium	-0.221	0.0090	0.001-in. gray oxide layer, voids observed to a depth of 0.004 in.
Hastelloy B	-1.109	0.0095	Heavy oxide formed
Inconel X		0.0065	0.020-in. oxide layer (Fig. 15)
Z-nickel		0.0060	Erratic attack; 0.002-in. average 0.006-in. maximum (Fig. 16)
Monel	-0.005	0.0000	No evidence of attack

Most metals and alloys are susceptible to hydroxide corrosion in varying degrees, with the exception of nickel, copper, and Monel (an alloy of nickel and copper). Work at Battelle[3] indicates that zirconium is also unattacked by sodium hydroxide at 1500°F. Our tests, however, do not agree with this result; zirconium was found to be very severely attacked by sodium hydroxide as reported in Table 6.

A series of corrosion tests are in progress using potassium hydroxide at 1000 to 1500°F in an attempt to determine the extent of reaction at these temperatures and the effect of the type of atmosphere above the hydroxide on the corrosion reaction. Vacuum, air, and hydrogen are being used currently.

In air and vacuum, preliminary results indicate that both type-316 stainless steel and Inconel are attacked. The depth of attack at 1500°F is of the same order of magnitude as that noted at 1000°F (Fig. 23). In hydrogen, however, no corrosion product was noted on either type-316 stainless steel or Inconel at 1000°F. At 1300°F, shallow corrosion had occurred. The determination of the extent of this reaction, and a verification of the above preliminary results are being made.

Metal-Crystal Formation. Nickel crystals have been observed to form

[3] G. P. Smith and A. deS. Brasunas, *Battelle Trip Report*, September 21, 1951.

TABLE 5

Summary of Corrosion by Strontium Hydroxide at 1500°F in 100 Hours

Specimens tested in evacuated capsules of like material

MATERIAL	DEPTH OF METAL AFFECTED (in.)	METALLOGRAPHIC NOTES
304 stainless steel	0.0120	Heavy oxide layer, specimen cracked on bending
310 stainless steel	0.0047	
316 stainless steel	0.0030	0.003-in. oxide layer
318 stainless steel	0.0029	0.006-in. oxide layer (Fig. 17)
321 stainless steel	0.0045	0.006-in. oxide layer
347 stainless steel	0.0070	0.020-in. oxide layer
446 stainless steel	0.0040	0.011-in. oxide layer (Fig. 18)
Iron (Globe)	0.0020	Heavy oxide layer (0.017 in.) on specimen (Figs. 19, 20)
Inconel X	0.0030	0.007-in. oxide layer on specimen
Hastelloy B	0.0090	Specimen heavily oxidized and embrittled
Copper (OFHC)	0.0000	No evidence of attack
A-nickel	0.0000	No evidence of attack
Z-nickel	0.0015	0.002-in. layer of attacked zone
Zirconium*	0.0075	0.001 oxide layer observed

*Tested in nickel capsule.

frequently on the walls of metal containers of molten hydroxide. In thermal convection loops containing sodium hydroxide, nickel crystals generally, but not always, collect on the cold zone walls. The polished internal pipe surfaces in the hot zone suggest strongly that dissolution occurs at that point. In static-corrosion-test capsules and other molten-hydroxide containers constructed of nickel and maintained at approximately constant temperature, crystalline-nickel deposits have been observed on the container walls at the hydroxide-atmosphere interface. These areas were not necessarily the coolest portion of the system, and suggest that an explanation of this phenomenon by thermal gradients is an oversimplification of the problem.

Several thermal-gradient "standpipe" corrosion tests have been made to evaluate the effect of a thermal gradient on metal-crystal formation. In these tests, nickel tubes containing hydroxide were heated so that a temperature of 800°C was maintained at the center, and a temperature of about 500°C was attained at both the top and bottom of the tube. In the first test, barium hydroxide was used in an air atmosphere. A black, crystalline deposit formed that analyzed 42.6% nickel and corresponded to the compound $BaNi_2O_5$. The second test involved potassium hydroxide in

TABLE 6

Summary of Corrosion by Sodium Hydroxide and Lithium Hydroxide at 1500°F in 100 Hours

Specimens tested in evacuated capsules of like material

MATERIAL	WEIGHT CHANGE (g/in.^{2})	DEPTH OF METAL AFFECTED (in.)	METALLOGRAPHIC NOTES
In Sodium Hydroxide			
304 stainless steel		0.0245	0.025-in. oxide layer
Chromium	-0.1	0.003	No evidence of attack, uniform solution only (Fig. 21)
Zirconium*	-2	0.082	Very severe attack, specimen almost completely consumed
A-nickel	+0.001	0.0000	Slightly attacked
Z-nickel	+0.6	0.020	Severely attacked
Al_2O_3 (Sapphire)		0.0023	Uniform solution, not severely attacked
In Lithium Hydroxide			
304 stainless steel	-0.185	0.0045	0.014-in. oxide layer (Fig. 22)
Inconel	+0.043	0.0040	0.006-in. oxide layer
A-nickel	-0.001	0.0000	No evidence of attack

*Tested in nickel capsule.

air, and again a black, crystalline substance formed. In each of the above tests, the caustic appeared dark when cooled. In the third test, sodium hydroxide was used in a hydrogen atmosphere. Under these circumstances no crystals were formed; the nickel tube and the caustic gave no evidence of interaction, that is, the caustic was not discolored.

Corrosion by Molten Cyanide. Numerous molten salts have been considered for possible use in a reactor, but cyanides do not appear to have been mentioned. This nonoxidizing salt is quite stable at high temperatures and has been used widely in the heat-treating and carburizing industry for many years.

Pure sodium cyanide melts at 564°C (1047°F), however, suitable additions can undoubtedly lower this temperature. The specific heat of sodium cyanide is reported as 0.25 Btu/lb (solid) and 0.40 Btu/lb (liquid); the heat of fusion is 135 Btu/lb. Uranium is appreciably soluble in sodium cyanide at 1500°F.

Static-corrosion tests in vacuum were made at 1500°F for 100 hr on numerous metals and alloys. These are listed in Table 7 together with the data obtained in the tests. Figures 24 and 25 illustrate the attack. The cyanide reaction results in carbon and/or nitrogen absorption by the metal, which frequently embrittles the material, as determined by a simple

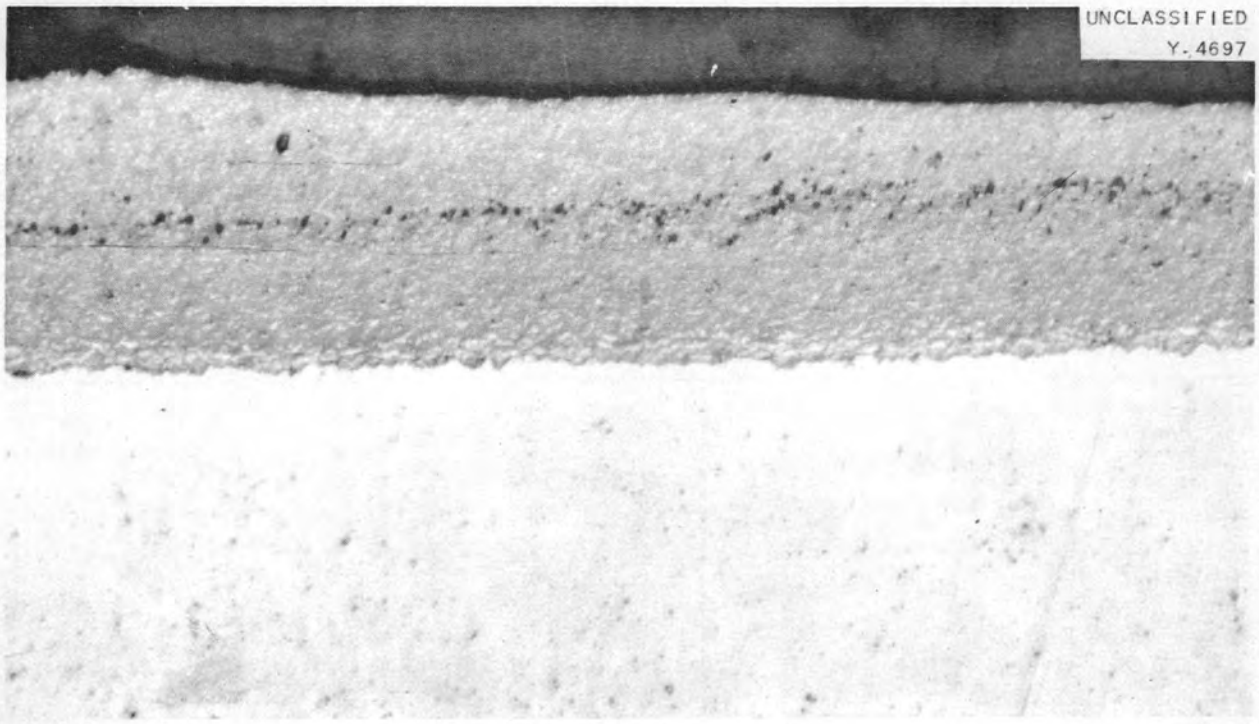


Fig. 13. Type-318 Stainless Steel After 100-hr Exposure to Barium Hydroxide at 1500°F. Unetched. 250 X.

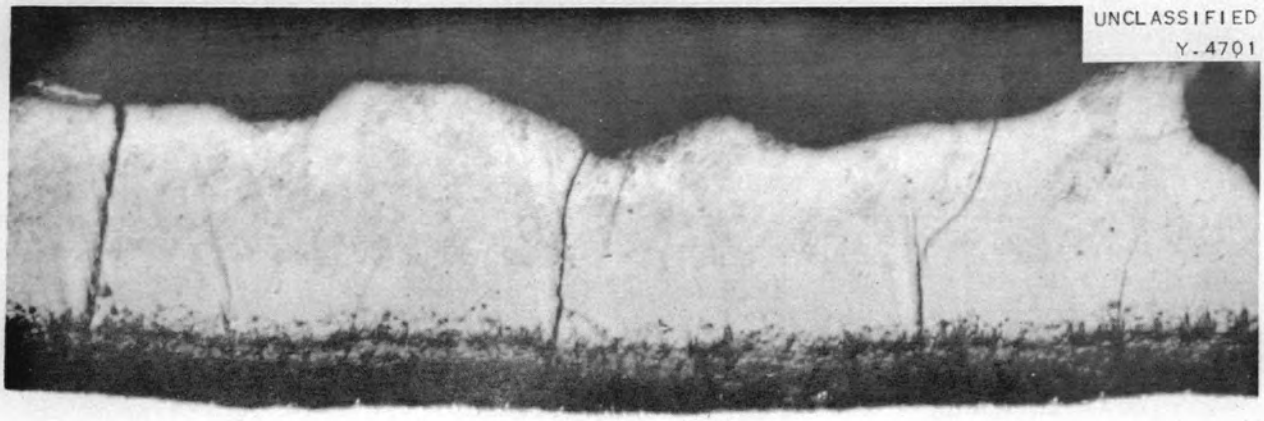


Fig. 14. Type-446 Stainless Steel After 100-hr Exposure to Barium Hydroxide at 1500°F. Unetched. 100 X.

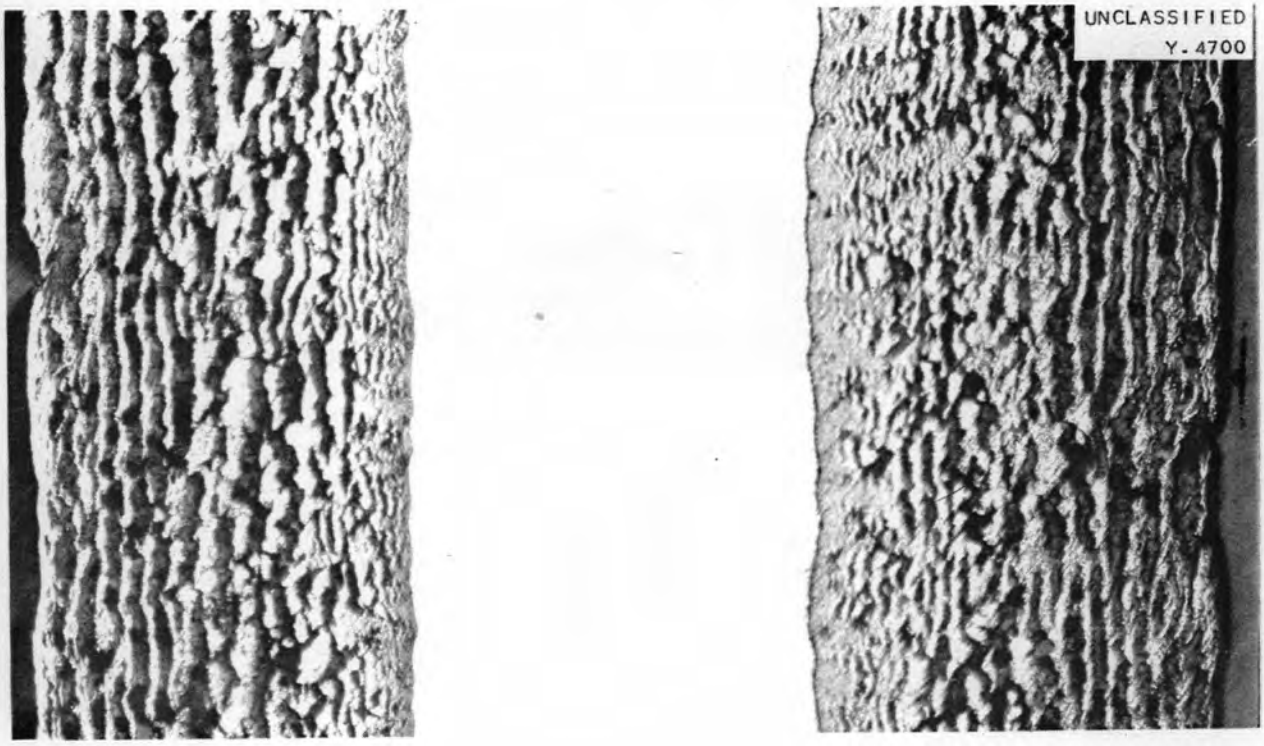


Fig. 15. Inconel X After 100-hr Exposure to Barium Hydroxide at 1500°F. Unetched. 100 X.

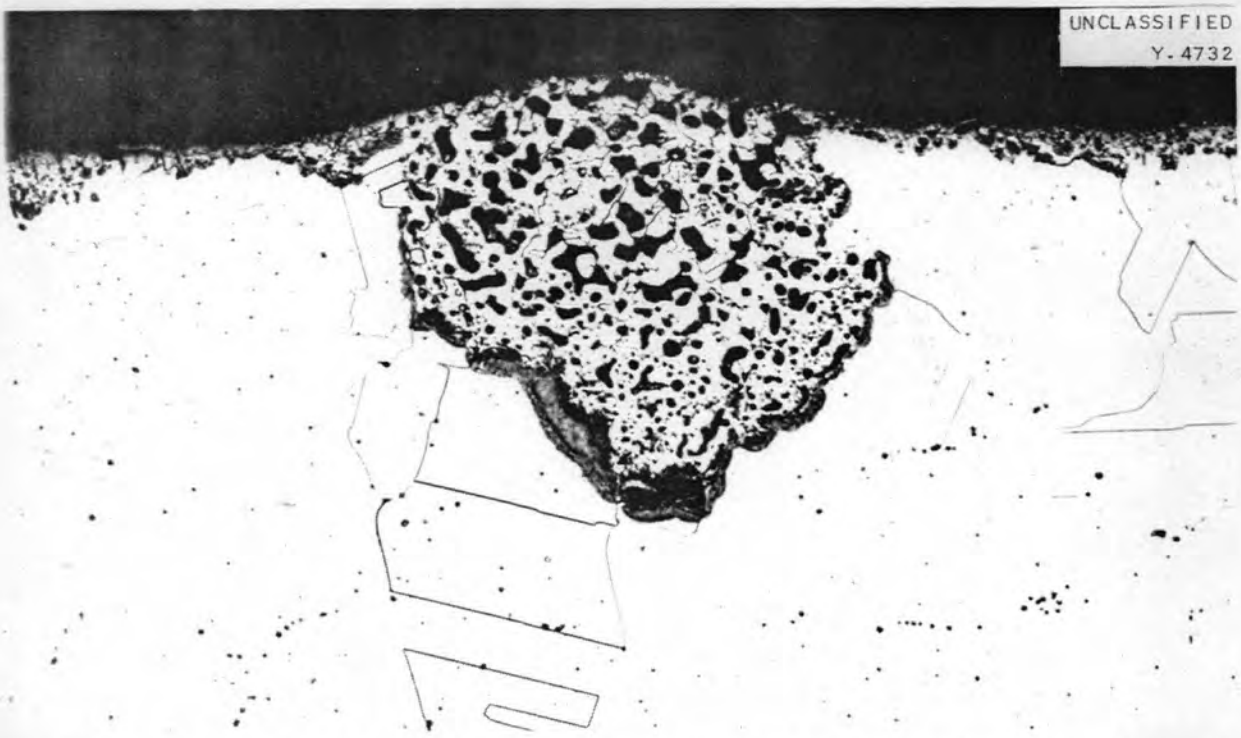


Fig. 16. Z-Nickel After 100-hr Exposure to Barium Hydroxide at 1500°F. Note erratic behavior. 200 X.

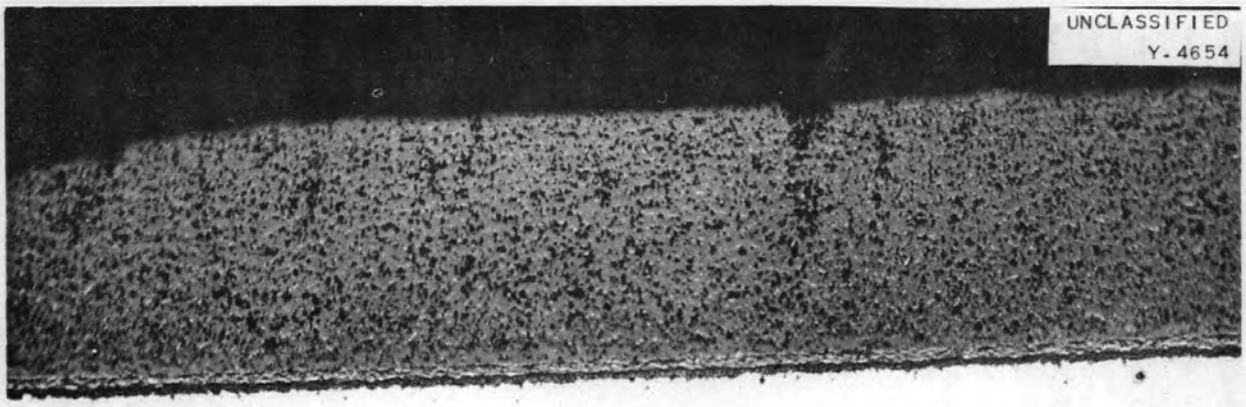


Fig. 17. Type-318 Stainless Steel After 100-hr Exposure to Strontium Hydroxide at 1500°F. Unetched. 175 X.

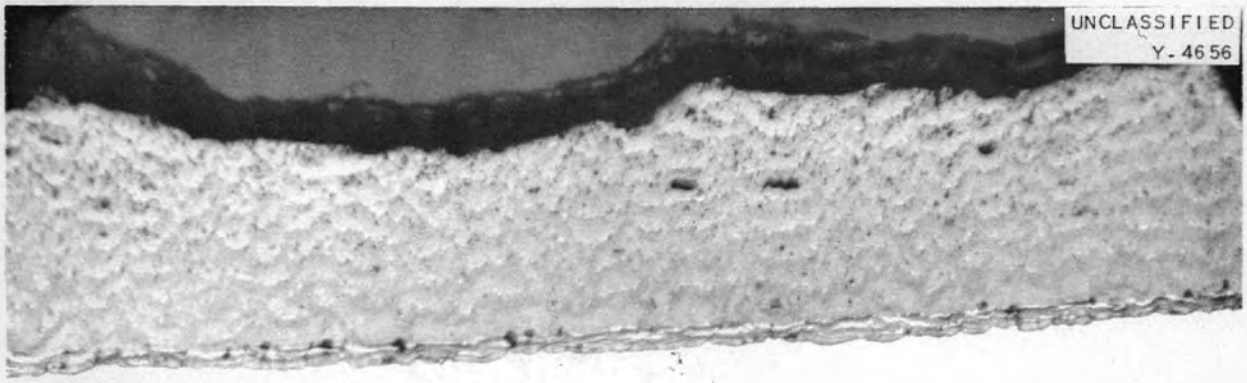


Fig. 18. Type-446 Stainless Steel After 100-hr Exposure to Strontium Hydroxide at 1500°F. Unetched. 150 X.

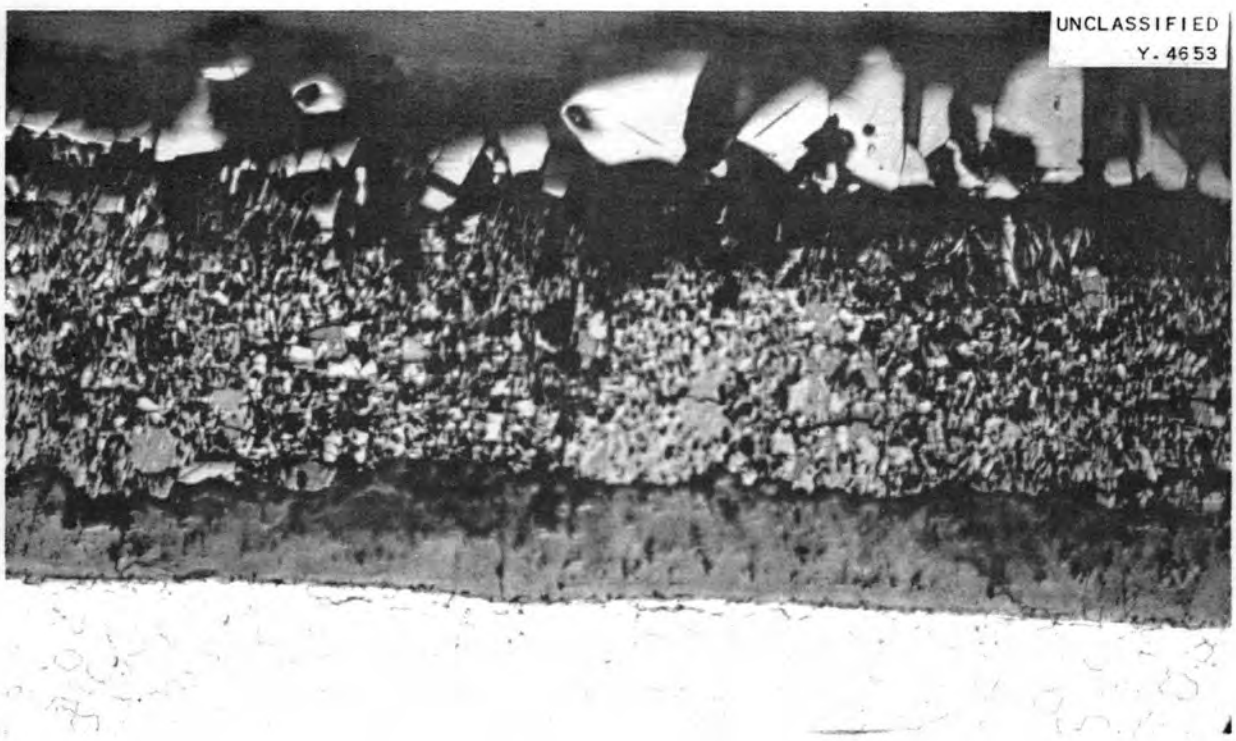


Fig. 19. Iron (Globe) After 100-hr Exposure to Strontium Hydroxide at 1500°F. 175 X.

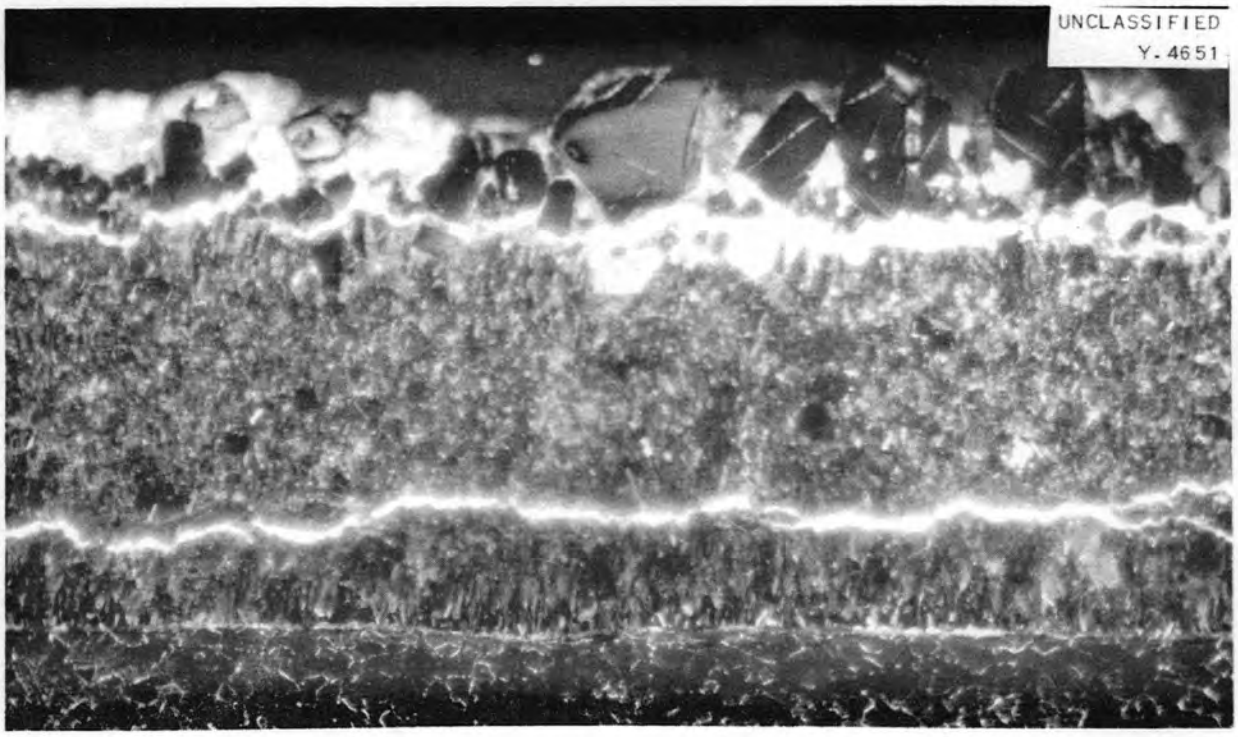


Fig. 20. Iron (Globe) After 100-hr Exposure to Strontium Hydroxide at 1500°F. Note columnar crystals on innermost layer. Polarized light. 175 X.

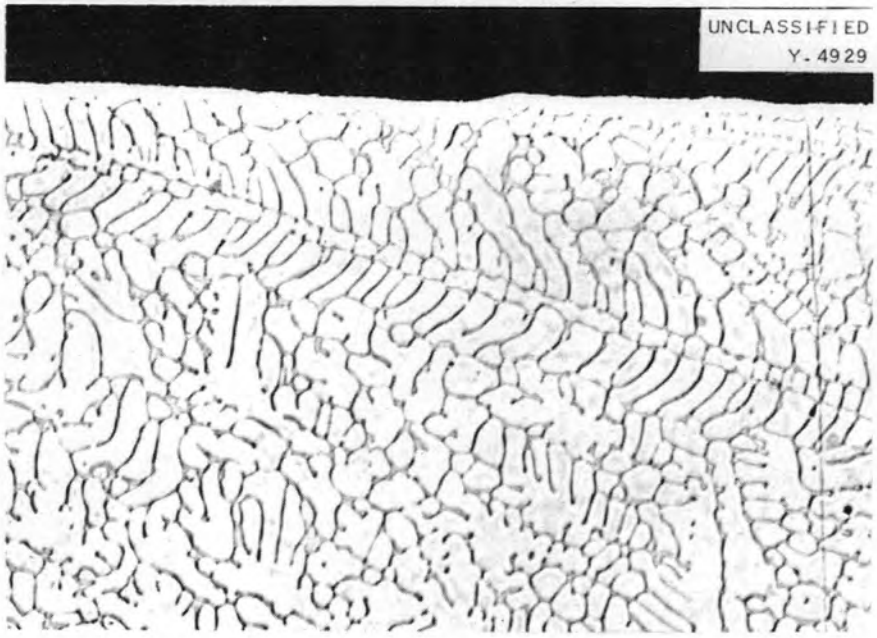


Fig. 21. Chromium After 100-hr Exposure to Sodium Hydroxide at 1500°F.

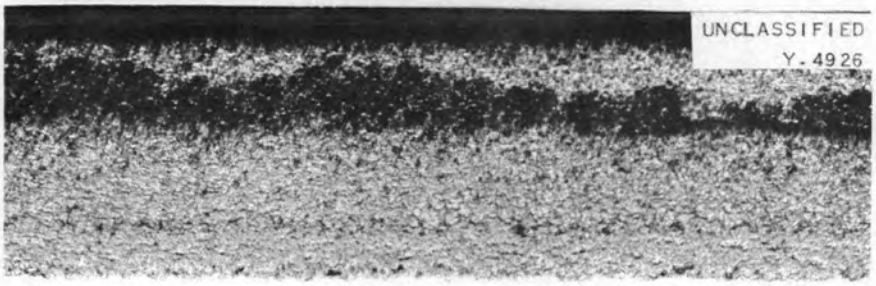


Fig. 22. Type-304 Stainless Steel After 100-hr Exposure to Lithium Hydroxide at 1500°F.

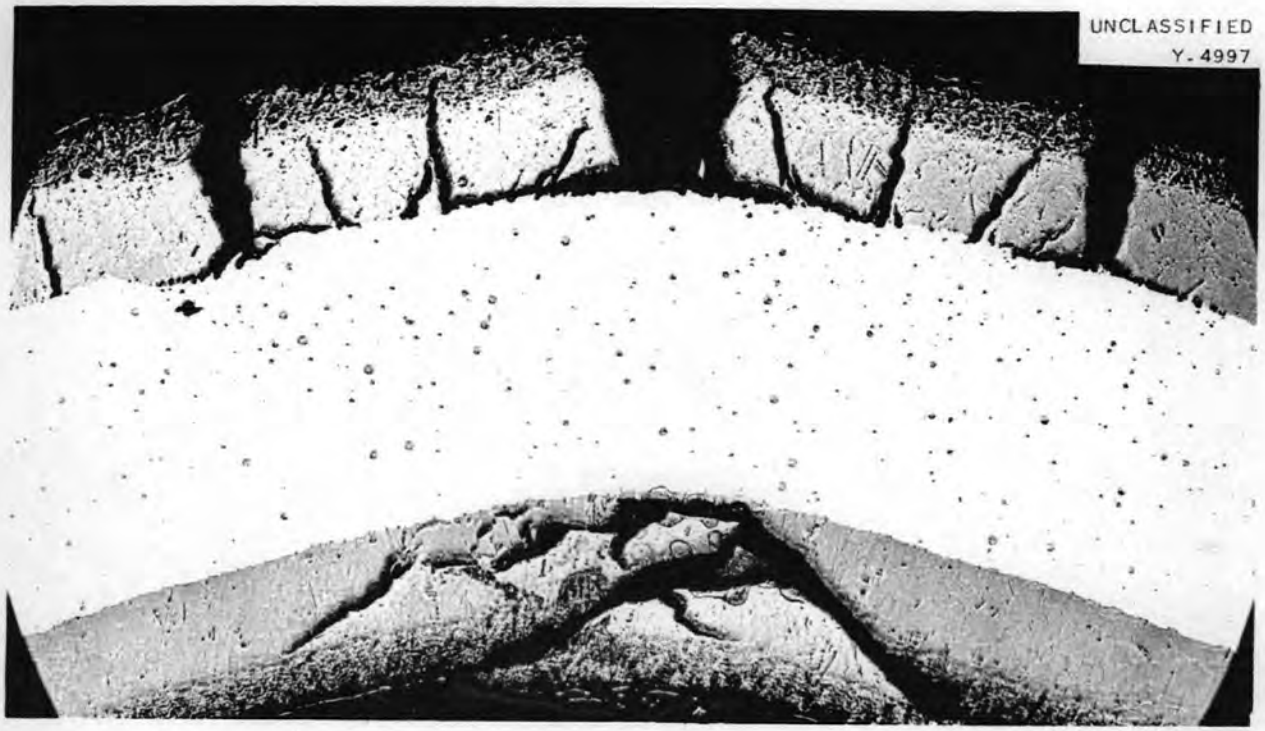


Fig. 23. Attack on Inconel by Potassium Hydroxide in an Evacuated Container at 1000°F in 100 Hours. Note severe embrittlement of 0.010-in.-thick hydroxide affected layer. Unetched. 80 X.

bend test at room temperature. There is, of course, the possibility that embrittlement does not occur at high temperature but is merely a room-temperature phenomenon.

The relative superiority of nickel and high nickel alloys (Inconel and Nichrome V) in resistance to carburization is well known. The behavior of iron, however, is very surprising. This could be caused by the absence of oxygen, which must be present[4] in a small amount in order that carburization can occur.

At present, no further work on cyanide corrosion is contemplated.

Corrosion by Liquid Metals. Long-time tests in liquid metals initiated some time ago have recently been evaluated. These were made with type-316 stainless steel in contact with sodium up to 1000 hr at 815 and 1000°C. The attack was negligible at both temperatures. Corrosion tests have also been made on type-347 stainless steel, type-316 stainless steel, and Inconel in sodium at 650, 704, 815, and 1000°C. The attack was again negligible, although some erratic embrittlement was noted.

Coated Specimens. Stainless steel specimens (type-304) were thinly coated with molybdenum and chromium by the Linde Air Products Company. It was hoped that these coatings of metals that are relatively inert to lithium would give protection to the underlying metal, which is very susceptible to attack. These specimens were tested in lithium at 1000°C in

[4] *ASM Handbook,* 1948, p. 694.

TABLE 7

Corrosion Data Obtained at 1500°F Using Molten Sodium Cyanide for 100 Hours

METAL	DEPTH OF METAL AFFECTED (in.)	EMBRITTLED	REMARKS
Iron (Globe)	0.000	No	No evidence of attack
A-nickel	0.000	No	No evidence of attack
Z-nickel	0.005	Yes	0.002-in. outer layer; gray constituent formed to depth of 0.005 in.
Inconel	0.003	No	Intergranular penetration of gray constituent to 0.003 in.
Hastelloy B	0.020	Yes	0.002-in. outer layer; nitride needles throughout
Hastelloy C	0.003		0.003-in. outer layer only (nitride?)
Nichrome V	0.005	No	0.001-in. outer layer; carburized to 0.005 in.
Uranium	0.01		Appreciable solution
Beryllium	0.002		Gray layer formed to depth of 0.002 in.
405 stainless steel	0.002	Yes	0.002-in. layer of scattered voids
430 stainless steel	0.020	Yes	Completely carburized; voids to depth of 0.002 in.
446 stainless steel	0.003	Yes	0.001-in. outer layer; 0.002-in. carburized layer beneath
304 stainless steel	0.011	Yes	0.001-in. outer layer; 0.011-in. carburized layer beneath
316 stainless steel	0.007	Yes	0.002-in. outer layer; 0.007-in. carburized layer beneath (Fig. 24)
347 stainless steel	0.007	Yes	0.002-in. outer layer; 0.007-in. carburized layer beneath
310 stainless steel	0.012	Partly	0.002-in. outer layer; 0.012-in. carburized layer beneath (Fig. 25)

the usual manner. The attack of the underlying metal although somewhat minimized was not eliminated as had been hoped. It is believed that thicker coatings would be necessary to effect the desired protection.

Dynamic Corrosion. Under the joint sponsorship of the Metallurgy Division and the Experimental Engineering Section of the ANP Division a program to study the effects of liquid metals on materials has been initiated. This project uses equipment and manpower of both groups. As originally planned most of the program was to determine the high-temperature effects of molten sodium on possible reactor materials. Recently the emphasis has been changed to molten fluorides and hydroxides. As rapidly as possible all test apparatus discussed in this section is being modified for use with these two materials.

Thermal Convection Loops. One of the major questions that must be answered before any circulating system

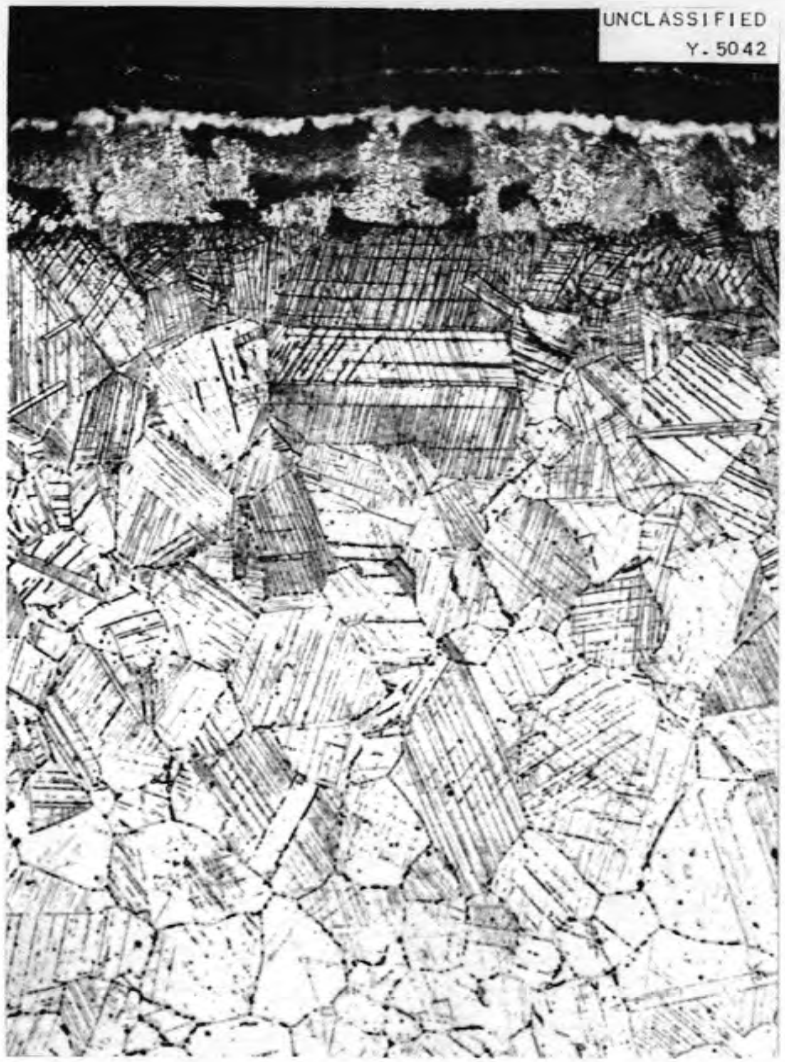


Fig. 24. Type-316 Stainless Steel After 100-hr Exposure to Sodium Cyanide at 1500°F. 250 X.

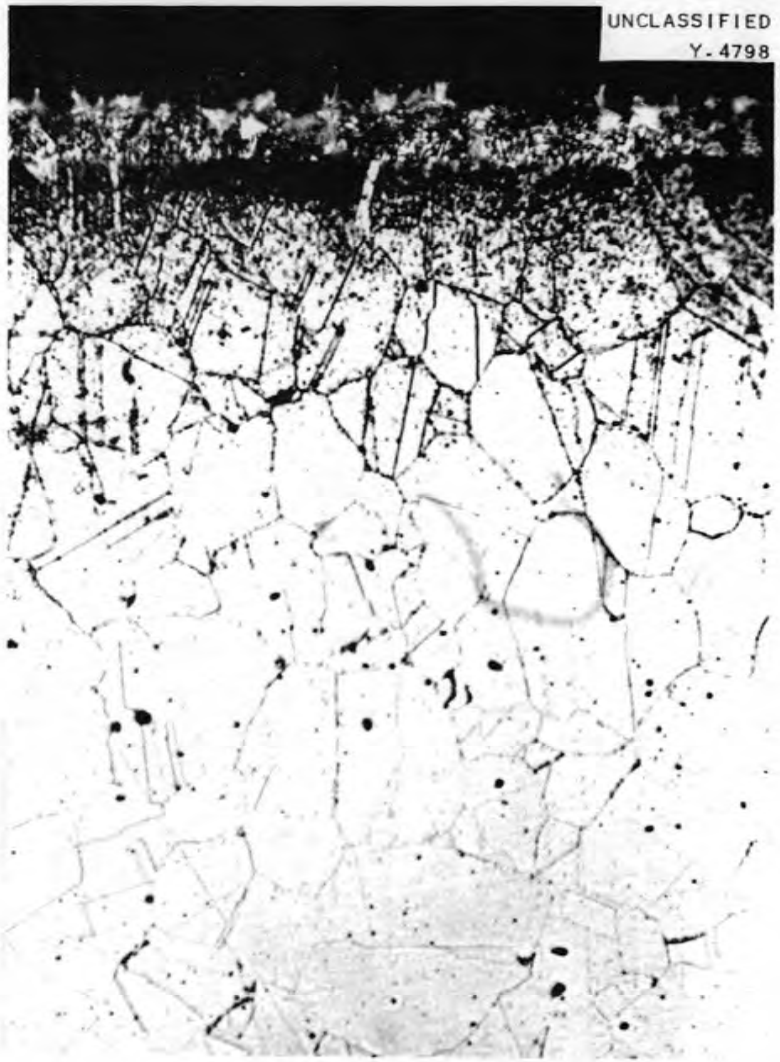


Fig. 25. Type-310 Stainless Steel After 100-hr Exposure to Sodium Cyanide at 1500°F. 250 X.

may be used is mass transfer. In this phenomena material is dissolved from one section of a system and precipitated in another, and plugging may take place. The simplest dynamic system is a thermal convection loop. With this apparatus a liquid may be circulated at low velocity without the necessity of solving pumping or seal-problems. This apparatus has been adopted for the preliminary tests to determine whether mass transfer takes place and to determine other effects in dynamic systems.

Various hydroxides have been circulated in nickel loops having a hot-leg temperature of 1400°F. In every case mass transfer was encountered. The loop circulating sodium hydroxide plugged completely in 54 hours. The plug was found to be a mass of dendritic nickel crystals located in the horizontal section of the hot leg. Similar masses were also found in the lower portion of the vertical hot leg. Single crystals were found attached to the walls in all portions except the hottest part of the hot leg. The section where crystals were not found had a polished surface and 0.008 in. of wall had been etched away. Some unevenness was found in the cold leg but the wall in most sections measured about 0.002 in. thinner than originally. A layer of fine equiaxed crystals was found over most of the surface. The layer varied in thickness from 0.0001 to 0.002 in. and usually had a rough surface. No intergranular type of attack was found in any section of the loop.

Nickel loops running at 1400°F plugged after 51 and 317 hr of operation with potassium hydroxide and lithium hydroxide, respectively. Examination of these loops has not been completed. In general the potassium hydroxide loop seemed to have plugged in the same areas and in the same manner as the sodium hydroxide loop. A much smaller volume of crystals seemed to be present in the lithium hydroxide loop, and they were located in the lower portions of both the hot and cold legs.

Most of the current interest is centered in a circulating fuel consisting of a eutectic mixture with a composition of 59.1 wt % potassium fluoride, 29.2 wt % lithium fluoride, and 11.7 wt % sodium fluoride, with 1 to 2 mole % of uranium fluoride added. Because of procurement and filling difficulties the first two loops are being run without the uranium fluoride. An Inconel loop has operated 100 hr at 1500°F with no falling-off of the temperature. A type-316 stainless steel loop is ready to be filled.

Inconel loops have operated for 1000 hr with sodium at 1600°F and with NaK at 1500°F. Neither loop developed serious amounts of mass transfer or corrosion. Small amounts of pitting were found, but no intergranular type of attack.

Stress-Rupture Tests. Very little stress-rupture data is available for materials at temperatures of 1500°F and above in nonoxidizing conditions. Work is now being done at other laboratories to obtain such data in inert atmospheres. Tests of a "quick-and-dirty" type are being run here to determine whether data obtained in sodium or other liquids is comparable to that obtained in inert gases or vacuum. Many of the stresses found in a reactor will be hoop stresses, and, since this is the simplest type of test to set up, it was the first type to be investigated. The wall of a tube of the desired material was machined over a 2-in. length to the desired thickness and one end was welded closed. The other end was

attached to a high-pressure helium system and pressure applied to the inside of the tube. The tube was then immersed in a high-temperature bath of the desired liquid for 1000 hr or until it ruptured. A type-316 stainless steel tube lasted 1000 hr in 1500°F sodium with a hoop stress of about 1900 psi. Another type-316 stainless steel tube has been under a stress of 2600 psi for 650 hours. One Inconel tube failed after 750 hr in 1500°F sodium at a stress of 1600 psi, whereas another failed in 99 hr while stressed to 1450 psi. Examination of both these tubes showed that serious intergranular attack had taken place on the inside of the tube. Failure had obviously been caused by this attack rather than by the sodium on the outside. A similar tube, which had not been tested, was examined and areas were found in which an impurity in the grain boundary extended through three-fourths of the wall. It seems obvious that a bad lot of tubing was used in machining these test sections. An Inconel sample from a new lot of tubing has now been under a stress of 1200 psi for 450 hours.

As a supplement for the tests described, a series of stress-rupture tests with flat pieces has been set up. It will be necessary to use very thin plate, about 0.020 in., so any values obtained will be approximate. They should, however, provide figures and curves to compare with those obtained in helium. The stress will be a dead load transmitted to the sample through a bellows. Values have been obtained for Inconel with stresses of 13,000 and 10,000 psi and for type-316 stainless steel at 7000 psi. These values seem to indicate that 1500°F sodium will not have a large effect on stress-rupture properties. To simplify the filling operation the apparatus is now being modified to provide a larger bath of liquid.

Self-welding Tests. In any system using a circulating coolant and metal-to-metal contacts, self-welding is always a possibility. In a reactor, places such as pumps and valve seats would be very susceptible to this difficulty. To obtain preliminary data on this phenomena, a series of simple tests is being carried out. A series of three cylinders with diameters of 1/8, 1/4, and 3/8 in. was machined from one material to be tested. A series of three cylinders 1/2 in. in diameter was then machined from either the same material or an entirely different one. The faces of the two sets were then polished and placed in contact in a bath of the desired liquid. A load of 75 lb was applied to the pieces for 100 hours. The following materials have been tested:

MATERIAL	RESULTS
Inconel to Inconel (polished, 120 grit)	Welding at a few points; broke during handling
Inconel to Inconel (metallographic polish)	Welding had just started but pieces could be handled
Inconel to zirconium	No welding
Type-347 to type-347 stainless steel	Welding at a few points on two small cylinders
Stellite to Stellite	No welding in 65 hr

PHYSICAL CHEMISTRY OF LIQUID METALS

Neutron Diffraction by Liquid Metals. The diffraction of neutrons by liquid metals is of interest both as a fundamental phenomenon and as an important source of information on the structure of liquids. A knowledge of the structure of a liquid is basic to a consideration of its properties in terms of atomistic forces and events. An equation equivalent to the one applied in the case of X-ray diffraction by liquid elements[5] has

[5] N. S. Gingrich, "The Diffraction of X-rays by Liquid Elements," *Rev. Mod. Phys.* 15, 90 (1943).

been used recently to analyze neutron-diffraction patterns to obtain atomic distribution functions.[6]

The use of neutron diffraction may prove to have certain advantages over X-ray diffraction for studies of liquids. One advantage results from the fact that neutron-diffraction cross sections and scattering amplitudes are essentially spherically symmetrical. This is in contrast to the rapid manner in which the X-ray atomic-scattering factors fall off with angle or more appropriately with the variable $\sin \theta/\lambda$. This means that the intensity of the diffraction pattern using neutrons should not fall off at large scattering angles as is the case with X-ray diffraction.

A second advantage in some cases results from the magnitude of the absorption and scattering coefficients. Some materials that have been difficult to study using X rays, because of their high absorption coefficients, can be studied with samples of convenient diameter using neutron diffraction.

The Physical Chemistry of Liquid Metals Group, with the very generous assistance of H. A. Levy and S. W. Peterson of the Chemistry Division, has been studying the diffraction of neutrons by liquid lead and liquid bismuth at two temperatures.

Bismuth and lead were chosen for study because the Physical Chemistry of Liquid Metals Group is currently investigating other properties of these metals, and a knowledge of their atomic distribution functions in the liquid state and the change in these distribution functions with temperature would be useful. At the present time no study has been made of the change with temperature in the atomic-distribution functions of liquid lead and liquid bismuth.

Neutron diffraction, rather than X-ray diffraction, was chosen for this study for two reasons. First, the neutron-absorption cross sections of these metals are of a convenient magnitude, whereas the X-ray absorption cross sections are not. Second, in the only previous neutron-diffraction study of liquid lead, an atomic-distribution function was obtained that differs appreciably from that obtained by X-ray diffraction methods. It is desirable to determine whether this may be attributed to the error of measurement, which was large, or to more fundamental causes.

The equipment used in these studies consisted of a neutron spectrometer[7] and recording system and a special furnace designed for this work. Five satisfactory diffraction patterns were obtained with liquid bismuth and three with liquid lead. Four of the patterns obtained with bismuth were at 300°C and one was at 550°C. Two of the patterns obtained with lead were at 350°C and one was at 550°C. The results obtained at the two temperatures are sufficiently different so that analysis will be carried out at each temperature to see what differences in the liquid structures can be detected.

A mathematical analysis of the diffraction patterns is now under way and the results will be reported as they are obtained.

Interactions of Liquid and Solid Metals. The purpose of this research was to study the fundamental aspects

[6] O. Chamberlain, "Neutron Diffraction in Liquid Sulfur, Lead, and Bismuth," *Phys. Rev.* 77, 305 (1950).

[7] Made available by H. A. Levy and S. W. Peterson, similar to that described by E. O. Wollan and C. G. Shull, "The Diffraction of Neutrons by Crystalline Powders," *Phys. Rev.* 73, 830 (1948).

of liquid metal—solid metal interactions. Solution-deposition interactions were chosen for investigation. This type of interaction includes the solution of the solid metal in the liquid metal and the inverse process of the deposition of the solid metal from the liquid metal solution. Solution-deposition interactions have the advantage of being comparatively simple from a fundamental standpoint, as well as being extremely important in connection with both mass transfer and simple corrosion in liquid-metal heat-transfer systems.

For the sake of simplicity in interpreting results, it was desirable to choose a liquid metal—solid metal system in which no compounds were formed and in which the liquid metal was virtually insoluble in the solid metal. It was also desirable for the oxides of both the liquid metal and the solid metal to be easily reducible with hydrogen. The system copper-bismuth, which is one of several systems having the above characteristics, was chosen for this work.

The experiments carried out in the previous quarter were of an extremely preliminary nature. They showed that at 400°C a considerable amount of mass transfer occurred in a copper-bismuth system in the presence of a small temperature gradient and cycling temperature control. It has been proposed that this mass transfer might be attributed either to thermal gradients or to thermal cycling.[8]

In order to test this hypothesis several experiments were performed in which thermal gradients and cycling were either controlled or else greatly reduced. The apparatus used was as follows: The metal samples were contained in glass apparatuses called "nucleation tubes." Each nucleation tube consisted of a pyrex tube divided into three compartments by fritted glass disks. Each compartment was connected to hydrogen and vacuum lines. It was found that the surface tension of liquid bismuth was sufficiently high to allow the bismuth to be contained in any compartment, as long as all the compartments were at the same pressure. However, the proper application of a vacuum to the lower compartments permitted the liquid bismuth to be transferred from a higher compartment to a lower one, whereas any solid particles were retained by the fritted disk.

The nucleation tubes were heated in a stirred salt bath. Although the bath was of very simple design, the temperature gradient was no more than 0.25°C, and the maximum extent of thermal cycling was 1°C.

Several experiments were performed with this apparatus. In all experiments the bismuth and copper samples were first deoxidized in separate compartments of the nucleation tube; then, the bismuth was brought into contact with the copper. In one series of experiments the copper and bismuth were allowed to interact at 400°C under conditions that were as nearly isothermal as the salt bath allowed. After various periods of time up to 100 hr the bismuth was drained from the copper and filtered through a fritted glass disk. In no case was there any evidence of the crystalline deposits or mass transfer that was previously reported for specimens interacted under conditions which were not nearly so isothermal. A second and unexpected result was obtained — the surface of the original polycrystalline copper specimen was roughened. That portion of the specimen which was immersed in the

[8] *Metallurgy Division Quarterly Progress Report for Period Ending July 31, 1951*, ORNL-1108.

bismuth had crystal facets formed on the surface.

In a second set of experiments the copper-bismuth system was subjected to controlled thermal cycling in the neighborhood of 400°C. Only a small thermal gradient existed in these experiments, but the thermal cycling amounted to 18°C. When the bismuth was filtered, a large quantity of copper crystals was retained by the fritted disk, and the original copper specimen was severely attacked. The deposit consisted of polyhedral crystals and dendritic crystals.

A third set of experiments was conducted to determine the degree of supersaturation necessary to produce the crystallization of copper from its solution in bismuth in the absence of previously undissolved copper, which might act as a nucleating surface. The bismuth was first saturated with copper at a selected temperature above 400°C, filtered through a fritted disk to remove any particulate copper, cooled to 400°C to produce a supersaturation, and finally filtered again to observe any crystals that had precipitated. The smallest supersaturation was produced by cooling 2°C (from 402 to 400°C). Precipitation and growth occurred in all cases.

The experiments cited above clearly show that a devastating degree of mass transfer can occur in static systems. The only conditions required are a thermal gradient or the presence of thermal cycling. These conditions are of particular significance in static corrosion tests with liquid-metal systems. Thermal gradients are almost always present in an ordinary tube furnace, and thermal cycling will occur when an "on-off" type of temperature controller is used. Unless proper precautions are taken in static corrosion tests at elevated temperatures, the extent of corrosion in these tests is likely to be completely masked by mass-transfer effects.

Any future research on solution-deposition interaction should include a study of the isothermal-facet formation on a copper surface in a copper-bismuth system, as was observed in the above experiments. It is possible that this phenomenon is the first direct experimental evidence in support of the Curie theory of crystal habit.[9] Curie proposed that a crystal in contact with its saturated solution would assume an equilibrium form such as to minimize the total surface of the crystal. His theory predicts just such facet formation as mentioned above. This type of facet formation has never been observed on crystals in contact with a saturated aqueous solution.

It has been necessary to discontinue the liquid metal—solid metal studies because of the need for a study of mass transfer in sodium hydroxide—nickel systems. However the liquid metal—solid metal studies may be resumed in the next quarter.

Effect of the Atmosphere on Corrosion in a Sodium Hydroxide—Nickel System. Considerable interest has been evidenced recently in the possibility of constructing a sodium hydroxide—cooled reactor. Because the work of others has indicated the possibility that the presence of small quantities of gaseous contaminants, probably oxygen and water, might accelerate corrosion and mass transfer in such a reactor, a study is being made of the effect of atmosphere on corrosion of nickel by sodium hydroxide.

Experiments have been conducted with atmospheres of air and hydrogen.

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[9] H. E. Buckley, *Crystal Growth*, p. 105, Wiley, New York, 1951.

The apparatus used was very simple, consisting only of a nickel crucible containing sodium hydroxide. In the runs made in air, the open nickel crucible was heated with a burner to a temperature of about 700°C. The initial heating caused only a slight discoloration of the sodium hydroxide, but when the crucible was allowed to cool slightly, a black, flaky precipitate appeared. This precipitate redissolved when the temperature was again raised, the process being reversible with temperature. There was no perceptible increase in the amount of the precipitate when the heating and cooling cycle was repeated several times. When examined under a microscope the precipitate was shown to consist of two parts: thin, silvery plates that proved to be nickel, and a black powder that was nickel oxide.

When the sodium hydroxide that had been heated in air was allowed to solidify and then dissolved in water, a gas was evolved. This gas was qualitatively identified as oxygen.

For the studies in hydrogen, the nickel crucible containing sodium hydroxide was placed in a glass capsule, and after the sodium hydroxide was dehydrated in a vacuum at 250°C, hydrogen was let into the system. The hydrogen used was purified by passing it over hot (450°C), reduced copper oxide and then dried with potassium perchlorate and Ascarite (NaOH). The sodium hydroxide, melted in the hydrogen atmosphere, was a water-clear liquid until a temperature of 490°C was reached, at which time the molten sodium hydroxide took on a faint greenish tinge. As the temperature was raised, the intensity of this coloration increased until at 550°C the sodium hydroxide was a definite green reminiscent of the color of dilute solutions of nickelous compounds. The appearance of this coloration was reversible with temperature. The nature of the substance causing this phenomenon has yet to be determined. No appreciable attack on the nickel crucible was noted in a run that lasted 50 hr at 550°C.

It appears that hydrogen has a decided effect in reducing the corrosion of nickel by sodium hydroxide although the extent of its influence has not been determined. On the other hand, corrosion is very marked in the presence of air. No definite conclusions can be drawn at this time regarding the effect of hydrogen on mass transfer in a sodium hydroxide—nickel system, although there are indications that a hydrogen atmosphere is beneficial in this case also.

Mass Transfer in Sodium Hydroxide. There is experimental evidence that oxygen may contribute significantly to mass transfer in a sodium hydroxide—nickel system, and a study is being made to determine the extent. For this purpose, several small thermal-convection loops are being operated under closely controlled conditions. The first loop has been run. Its purpose was to provide preliminary data to guide future experiments and to develop handling techniques. After 316 hr its operation was stopped even though no failure had occurred. The loop was constructed of 1/2-in. nickel tubing bent into rectangular shape (Fig. 26). It was cleaned with dilute hydrochloric acid and loaded with dehydrated sodium hydroxide in a helium atmosphere. The loop was then evacuated and sealed by double crimping and welding.

The loop operated at a hot-leg temperature of 705°C and a cold-leg temperature of 465°C. The distribution of temperature can be seen in Fig. 27, which also shows the loop as reconstructed after the removal of the sodium hydroxide and longitudinally

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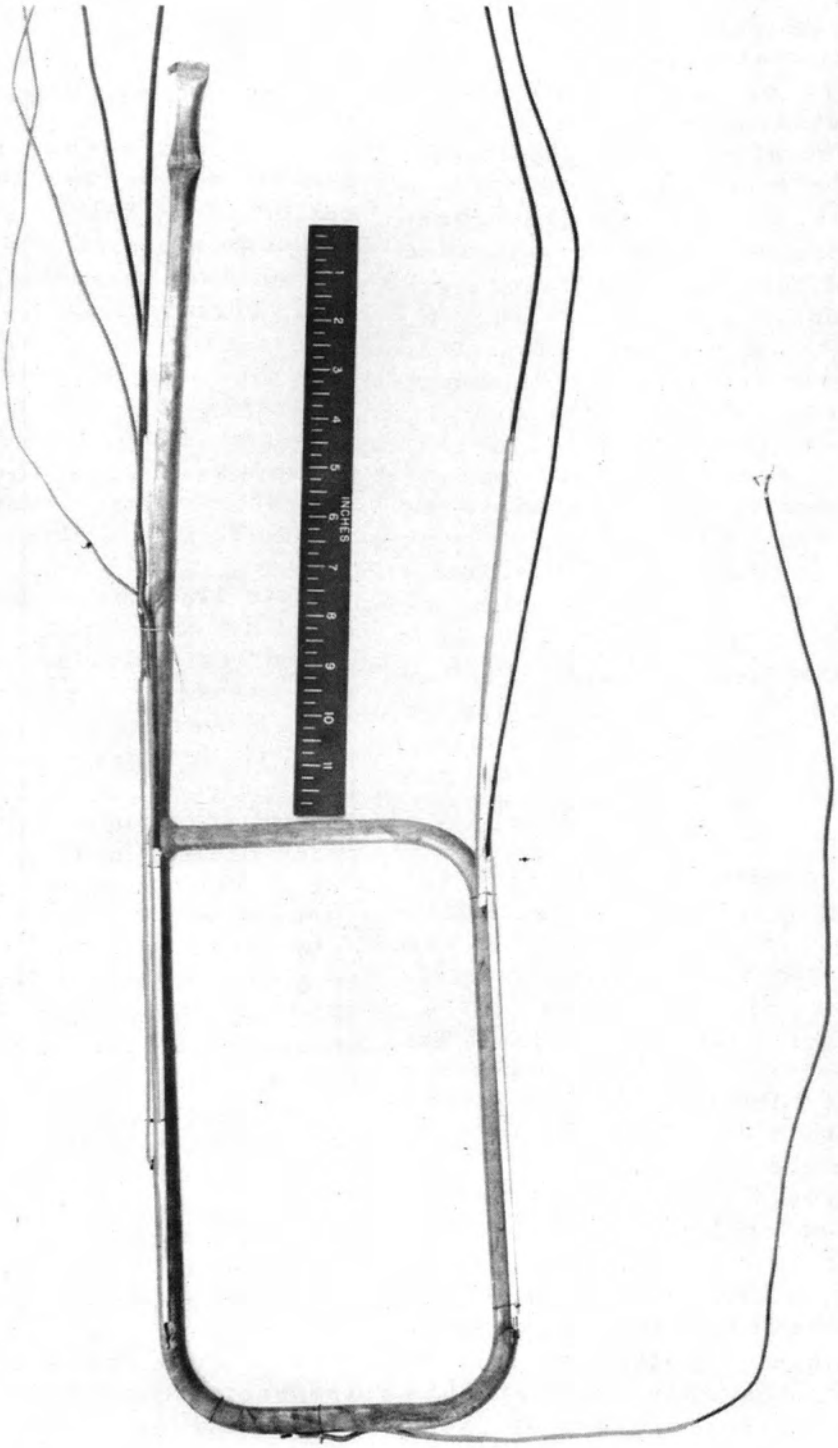

Fig. 26. Sodium Hydroxide—Nickel Thermal Convection Loop.

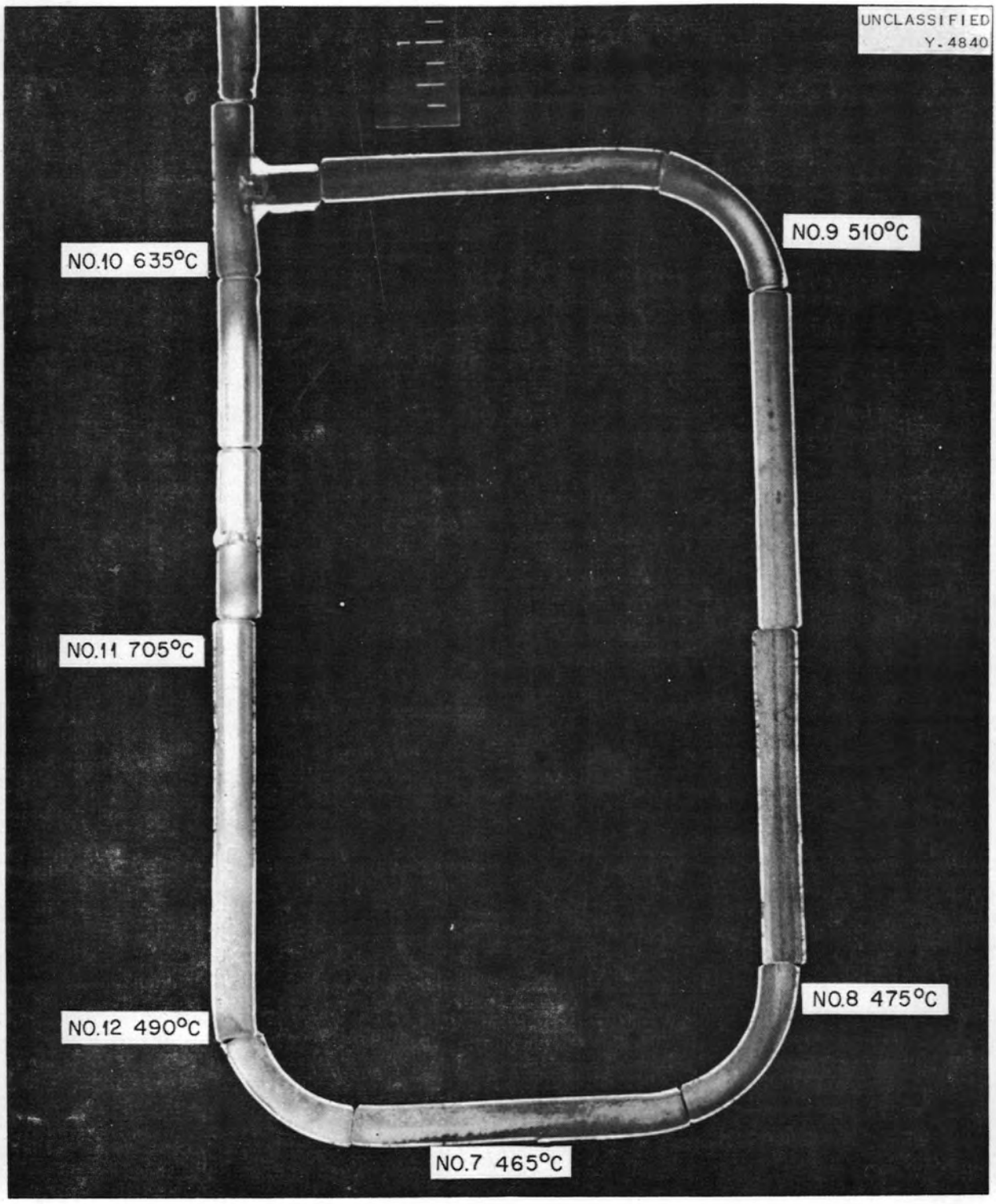

Fig. 27. Longitudinal Section of Sodium Hydroxide—Nickel Thermal Convection Loop After 316 hr of Operation.

splitting the individual sections. Although the loop was operating in a most satisfactory manner at the time the run was terminated, it was found that a considerable amount of mass transfer had taken place. Dendritic crystals were found in all portions except the very hottest part, and they were very dense in the cold zone. The crystals were identified as nickel by X-ray analysis. The microstructures of the different zones around the loop are currently being examined to determine the nature and extent of any changes that may have taken place.

Subsequent loops will be loaded by better techniques after having been hydrogen fired to remove surface oxides, and will be operated under a hydrogen atmosphere. This should answer the question of whether or not oxygen contributes to the mass-transfer phenomenon.

WELDING RESEARCH

Cone-arc Welding. Cone-arc welding apparatus, which incorporates a fixed-magnet nozzle on an inert-arc welding torch, has been used with moderate success to fabricate typical Inconel tube-to-header assemblies. The Inconel tubes were 0.188 in. o.d. with a 0.025-in. wall thickness. Headers were stamped from 0.0625-in.-thick Inconel sheet.

Although the feasibility of tube-to-header welded construction was demonstrated by these experiments, results were somewhat inconsistent. Apparatus has been completed that incorporates a Selsyn stator as part of the magnetic, inert-arc welding torch nozzle as described by previous investigations.[10] It is felt that the proper control of the additional welding variables introduced by this method will improve the consistency of results.

An investigation of the effect of these parameters on the type and quality of welded tube-to-header joints will be conducted concurrently with fabrication of full-size tube-to-header assemblies for testing by the Experimental Engineering Group.

Resistance Welding. Experiments have been conducted, using available equipment, which have demonstrated the feasibility of joining tube bundle straps to fuel tubes by spot welding. A typical spot weld is presented in Fig. 28, which shows a 0.016-in.-thick Inconel strap spot welded to an Inconel tube 0.188 in. o.d.; 0.025-in. wall thickness. It may be seen that penetration and soundness of the spot weld are excellent.

A preliminary investigation has been conducted to determine the feasibility of spot welding clad fuel elements together. The fuel elements consisted of a mixture of UO_2 and either stainless steel or iron powder. A sound spot weld could be made that apparently bonded the two sheets together without any gross macroscopic movement of the powder layers. This is illustrated in Fig. 29. Defects such as the crack in the fuel layer may be attributed to a lack of precise control of the welding variables with the available equipment.

A 20-kva combination spot-and-projection welder with suitable controls has been ordered for further experiments. It is expected that the production of a multitude of resistance welds with consistent results will require precise mechanical and and electronic control.

[10] E. R. Mann, *Means for Making Uniform Circular Heliarc Welds by Deflecting the Ion Beam Continuously*, ANP-63, April 9, 1951.

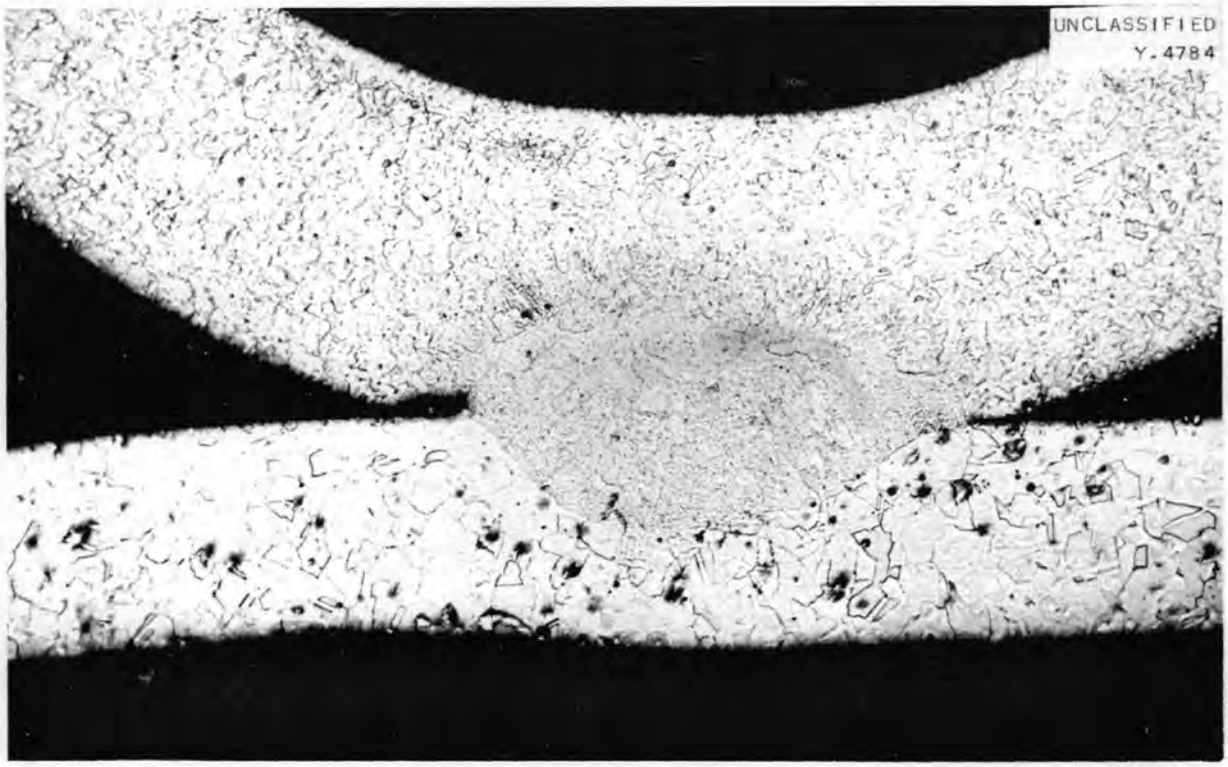

Fig. 28. Transverse Section of a 0.015-in.-thick Inconel Sheet Spot Welded to a 0.188-in.-o.d. Inconel Tube of 0.025-in. Wall Thickness. Etched with aqua regia and glycerin. 75 X.

Brazing. An introductory study has been made of furnace brazing under a controlled hydrogen atmosphere. The use of a commercial high-temperature brazing alloy (Nicrobraz) has been investigated and the effects of some of the brazing variables have been studied. The influence of brazing time on joints made in type-316 stainless steel can be seen in Figs. 30 and 31. Figure 30, which illustrates a joint held for 5 min at the brazing temperature, shows somewhat less diffusion of the brazing alloy into the base metal than Fig. 31, which was held at the brazing temperature for 30 min, indicating that time at temperature is not critical within these limits.

A short investigation was conducted on the high-temperature ductility of Nicrobrazed joints. Nicrobrazed T-joints on type-304 stainless steel were made and then struck at an elevated temperature with a sharp hammer blow. Excellent results were obtained as indicated by Fig. 32, which shows a joint tested at 1500°F.

A preliminary investigation has been conducted on the brazing of clad fuel elements. From the rather limited amount of work that has been done on this problem, it seems entirely feasible that these elements can be brazed either to a stainless steel sheet or to another clad fuel element. Figure 33 shows a clad fuel element Nicrobrazed to a stainless steel sheet. As can be seen, the brazing alloy wets the stainless steel very well and apparently has little tendency to float any of the fuel powders to the surface.

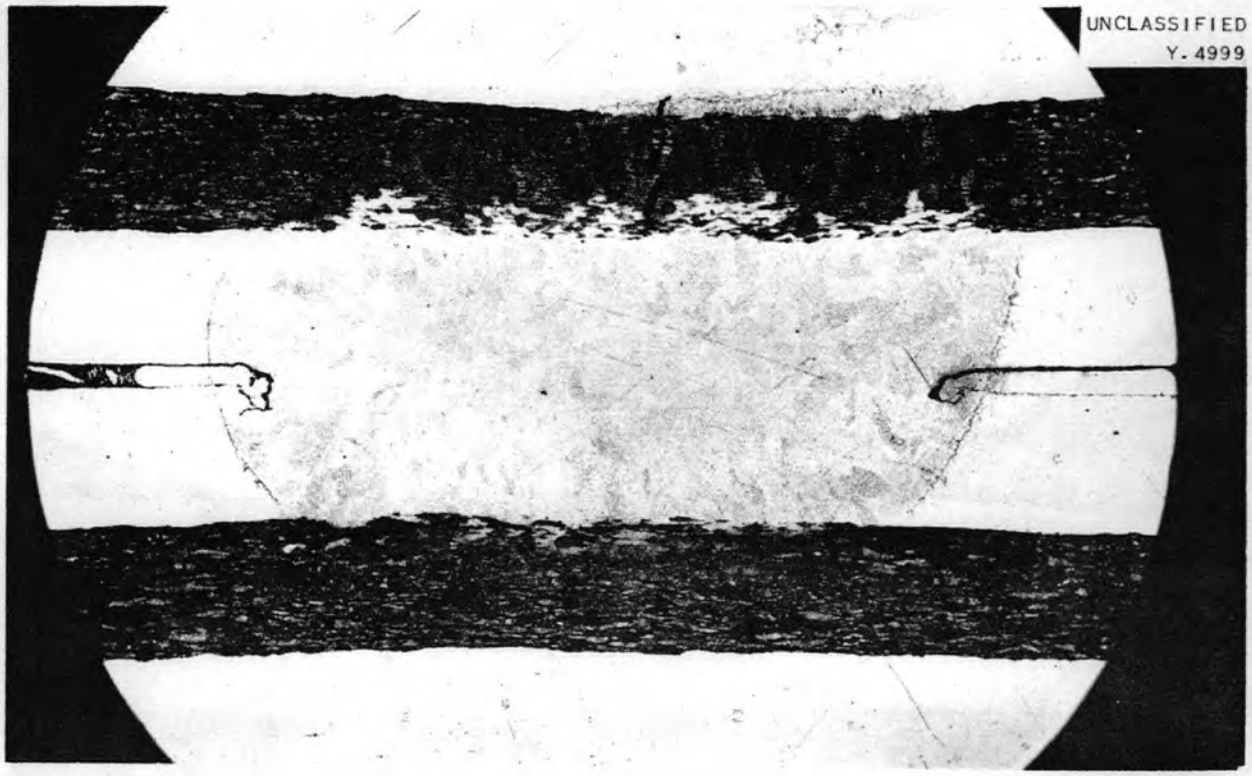

Fig. 29. Section of a Spot Weld Joining two Stainless-Steel-Clad Fuel Elements. Etched with aqua regia and glycerin. 60 X.

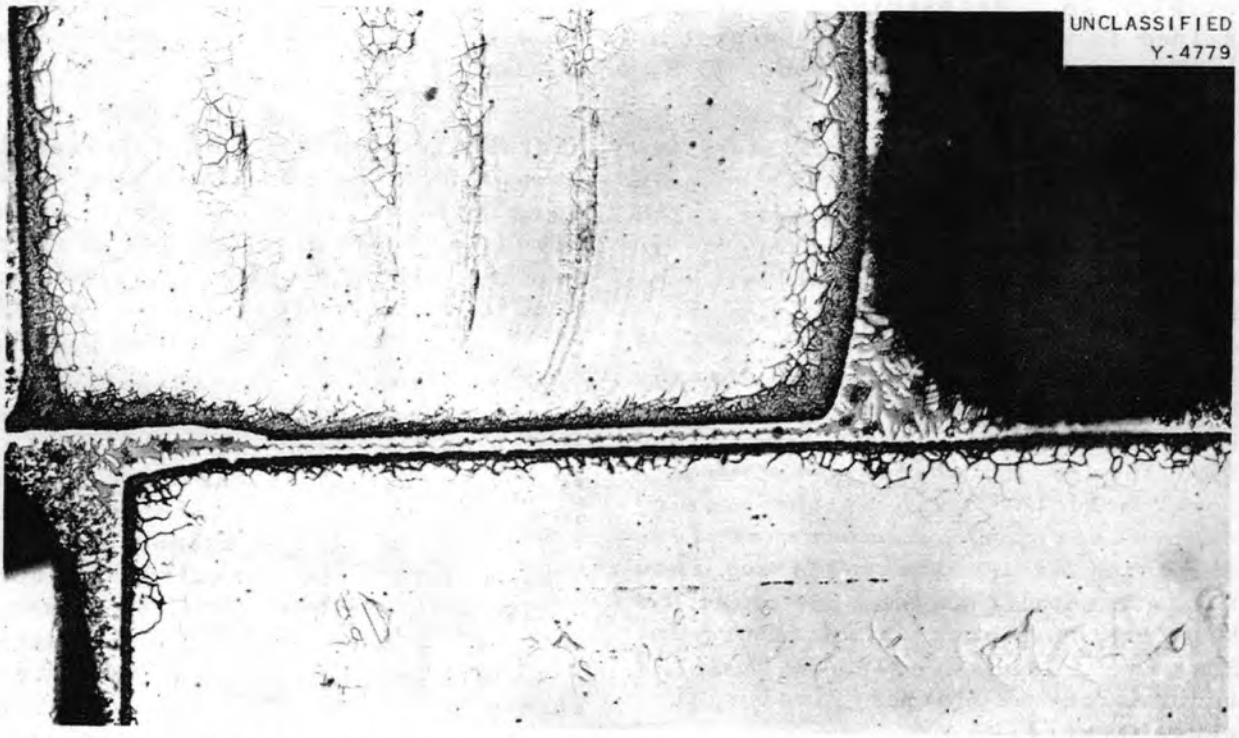

Fig. 30. Longitudinal Section of a Type-316 Stainless Steel Tube-to-Header Joint Nicrobrazed in a Dry Hydrogen Atmosphere for 5 min at 1120°C. Etched with aqua regia and glycerin. 75 X.

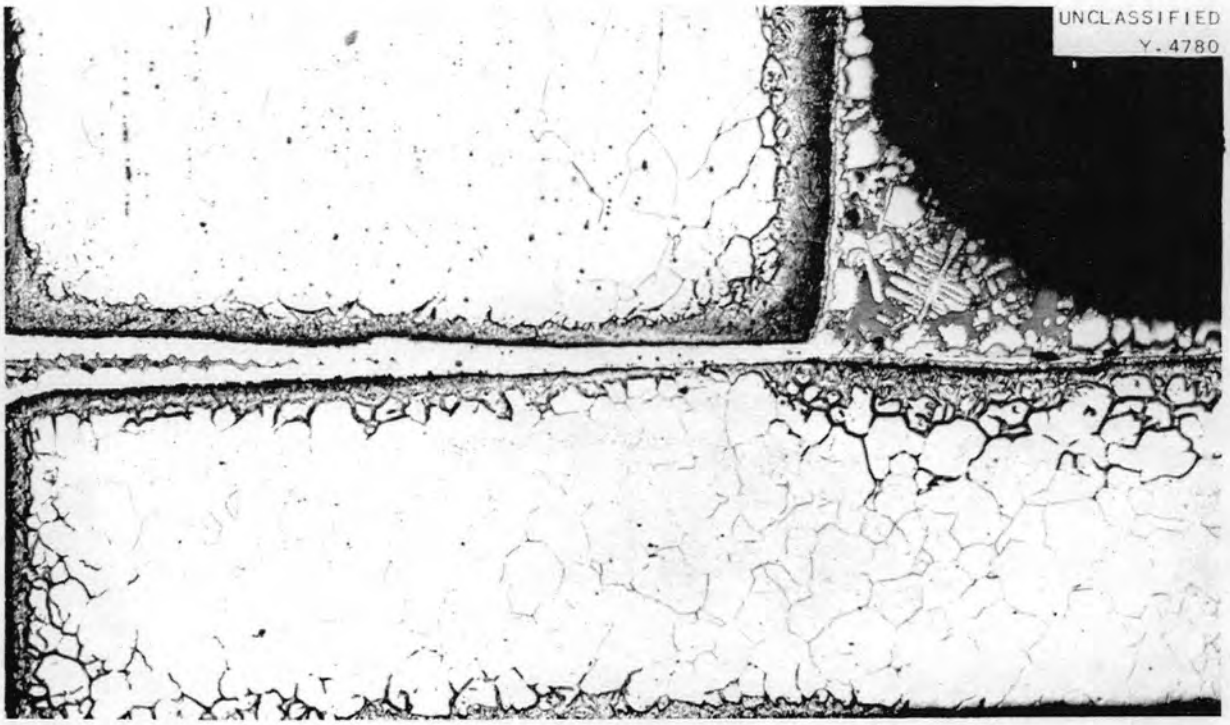

Fig. 31. Longitudinal Section of a Type-316 Stainless Steel Tube-to-Header Joint Nicrobrazed in a Dry Hydrogen Atmosphere for 30 min at 1120°C. Etched with aqua regia and glycerin. 75 X.

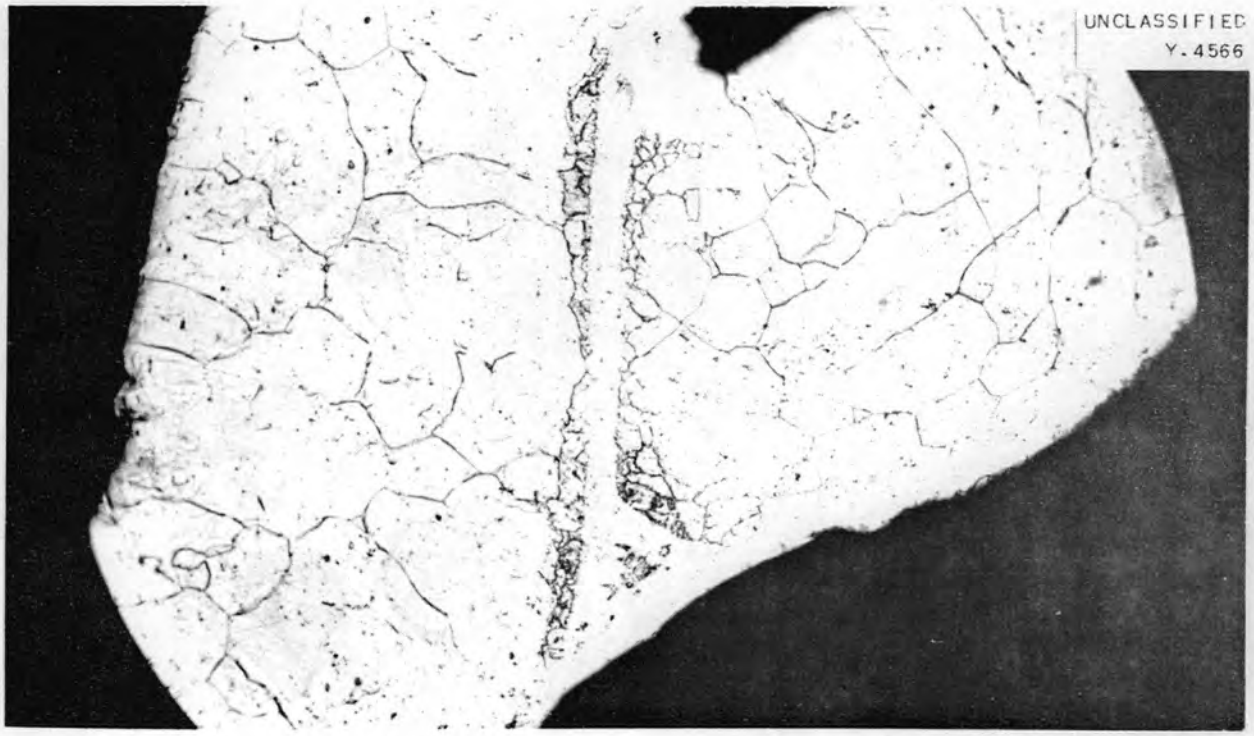

Fig. 32. Nicrobrazed T Joint on Type-304 Stainless Steel After Being Struck a Sharp Hammer Blow at 1500°F in a High-Temperature Ductility Test. Etched with aqua regia. 150 X.

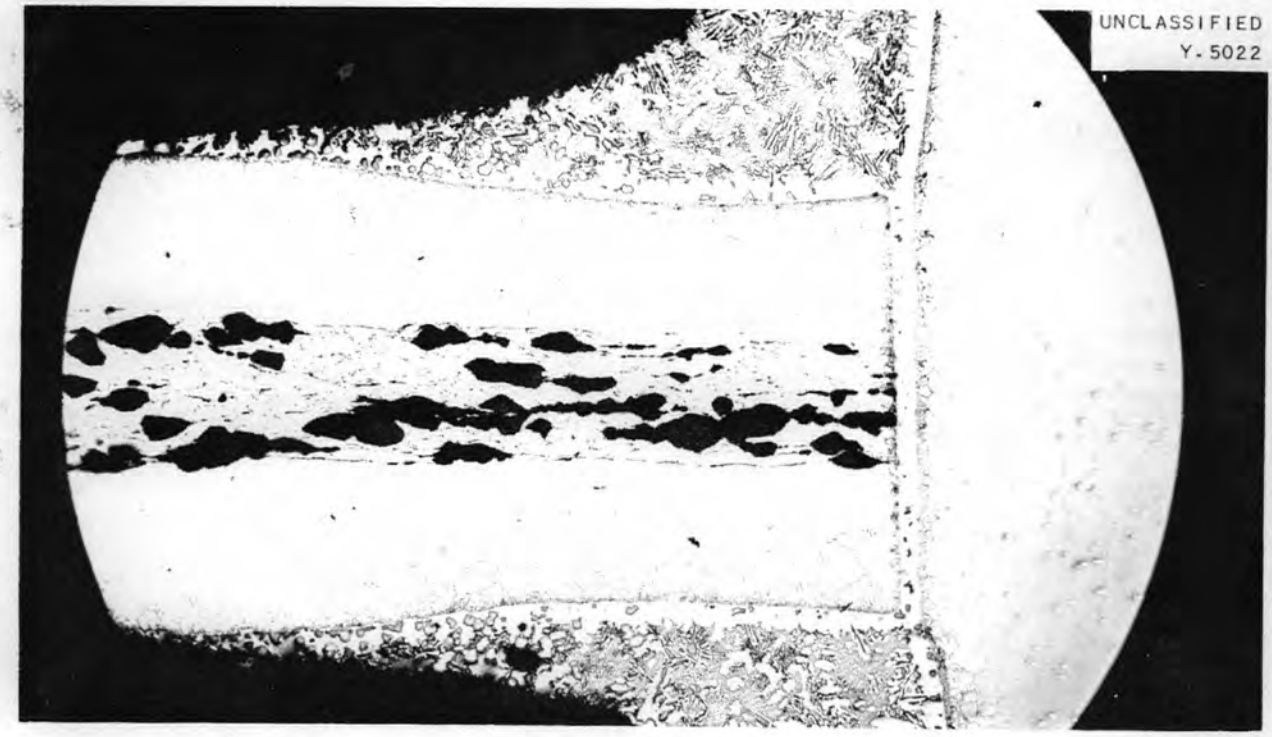

Fig. 33. Transverse Section of a Stainless-Steel-Clad Fuel Element Nicrobrazed as a T to a Stainless Steel Sheet. Unetched. 40 X.

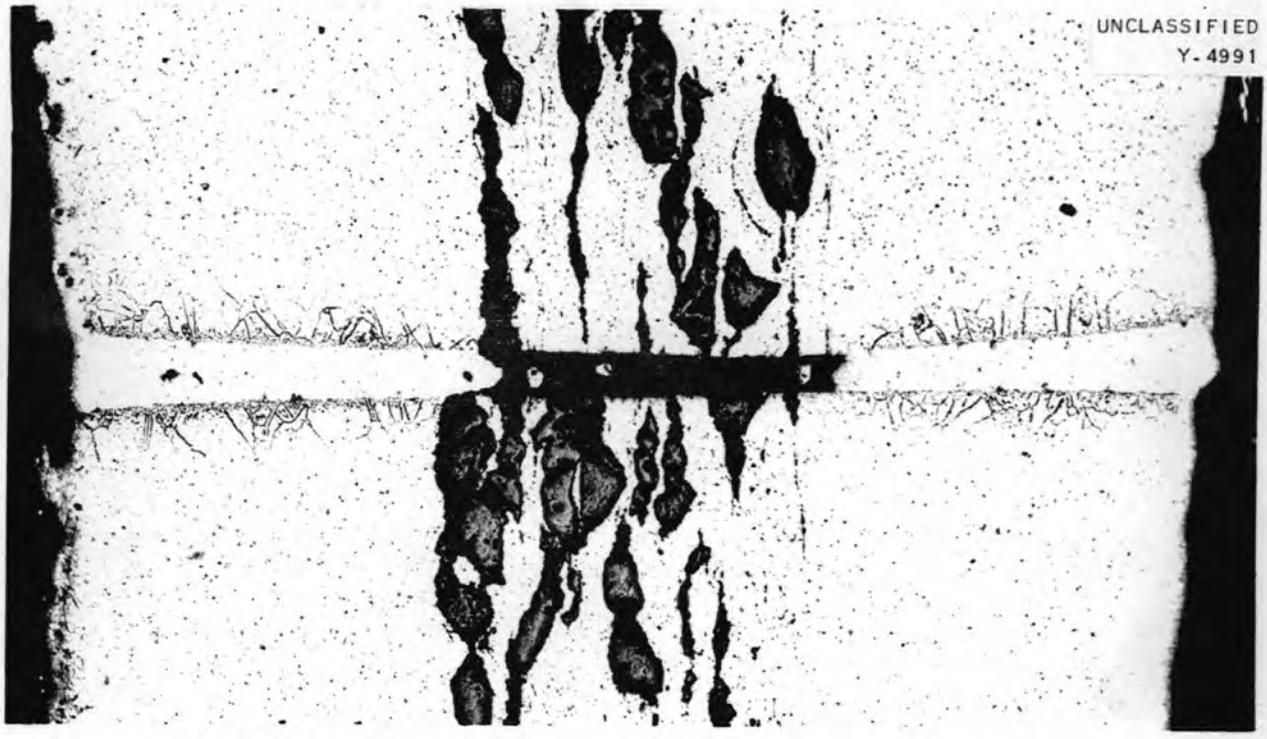

Fig. 34. Transverse Section of a Nicrobrazed Butt Joint of two Stainless-Steel-Clad Fuel Plates. Unetched. 120 X.

Fig. 35. Transverse Section of an Inconel Tube-to-Header Joint Brazed with a 40% Nickel—60% Palladium Alloy in a Dry Hydrogen Atmosphere at 1270°C for 20 Minutes. Etched with aqua regia and glycerin. 75 X.

Figure 34 is a photomicrograph of a Nicrobrazed butt joint of two clad fuel elements. The stainless steel in this case is wet very well by the brazing alloy, but the powder apparently is not. Again there seems to be no fuel powder entrapped in the brazing alloy. A more thorough investigation of the problems associated with the brazing of these clad components will be continued.

Since Nicrobraz contains about 4% boron, the use of this brazing alloy for fabrication of reactor components is somewhat limited. Experiments have been conducted using Nicrobraz modified by substituting beryllium for boron. However, the wetting properties of this modified alloy were poor, and further research is necessary to completely evaluate this approach to the problem.

In another attempt to find a brazing alloy with a more favorable nuclear cross section, consideration has been given to the nickel-palladium system with minor additions of silicon for melting-point control. Figure 35 is a photomicrograph of an Inconel tube-to-header joint brazed with a 40% nickel—60% palladium alloy at 1270°C in dry hydrogen for 20 minutes. As can be seen, the flow characteristics and wetting properties are apparently good.

Room-temperature tube-to-header tensile strengths were in the order of 80,000 psi. Further consideration will be given to this alloy system pending results of corrosion tests being conducted in molten coolants and fuels.

MATERIALS TESTING REACTOR PROGRAM

FUEL UNITS

Melting, cladding, and brazing work on the enriched-uranium fuel assemblies for IDOO continues at a steady pace. Approximately 9 kg of enriched-uranium metal has been converted to 14.2 wt % uranium-aluminum alloy and fabricated into the standard Alclad plates. A total of 35 fuel units and 5 fuel sections for shim-safety control rods are completely assembled except for attachment of end boxes and accessories. Two additional units were rejected because of improper plate spacing, and have been recycled to the melting operation. A normal reactor loading for MTR startup requires 23 fuel and 4 control rods.

Several sample castings of both the upper and lower adapters (end boxes) have been received from the Chemalloy Foundry Company and inspected. The semipermanent mold (sand core) for the upper adapter is now satisfactory and production has been authorized. The lower adapter mold and core print box are not yet producing acceptable castings and have been returned to the pattern shop for slight alterations.

CLADDING OF THORIUM FOR IRRADIATION IN THE MTR

During the next quarter, development work will be initiated on the cladding of thorium metal for production of U^{233} by irradiation in the MTR. The more desirable claddants, needed for corrosion protection and fission-product containment, are aluminum and zirconium.

Aims of the initial cladding studies will be to:

1. Confine work to only aluminum even though current fabrication work has demonstrated that zirconium can be metallurgically bonded to thorium. It is felt that the introduction of a new metal into the MTR, like zirconium, would likewise introduce a host of new corrosion as well as chemical separation problems not necessarily encountered with aluminum.

2. Concentrate on the production of a basic Alclad plate suitable for assembly into a beryllium A-piece reflector geometry rather than on the cladding of rods or slugs. Should it prove desirable to use rods or slugs, efforts can then be shifted to this somewhat less difficult job. Some experience is already available at Hanford on the canning of thorium slugs with aluminum.

3. Develop suitable techniques for hermetically sealing thorium in aluminum to give adequate heat transfer across the thorium-aluminum interface.

4. Determine the minimum cladding thickness necessary to do the job.

A 100-lb shipment of Ames thorium metal is expected to arrive shortly from Argonne National Laboratory for this work.

SERVICE WORK

Fifty feet each of 10-, 15-, and 20-mil diameter wire of crystal-bar zirconium, thorium, and type-347 stainless steel were prepared by cold drawing and shipped to California Research and Development Company for radiation-damage studies.

The 2- and 35-mil-thick enriched metal foils requested on KAPL orders were prepared and shipped.

Numerous samples of Inconel were cold and hot worked for the Physics of Solids Institute for ANP radiation-damage studies.

Twelve of the 20 Alclad Boral (50% mixture of boron carbide and aluminum) shielding shapes requested by Hanford were hot rolled to a finished size of 26 by 108 by 0.250 in. in thickness.