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COMPATIBILITY OF CALCIUM-SILICATE INSULATING BOARD (JOHNS-MANVILLE PRODUCT HEAT-TREATED MARIMET-45) WITH ENGINEERING ALLOYS AT 1100 TO 1600°F

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SEPTEMBER 1973

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ABSTRACT

Calcium-silicate insulating board (Johns-Manville product "heat-treated Marimet-45") was tested as a candidate material for spacer bushings which support electric clamshell heaters on high-temperature piping systems. Compatibility tests were made in typical piping configurations at temperatures from 1100 to 1600°F. No detrimental effects were observed on three metal alloys after 5700 hr exposure to calcium silicate at high temperature. It was concluded that the test material is suitable for use as support spacers for clamshell heaters. The material appears useful for other applications where a readily machinable, load-bearing, electrical or thermal insulation must be in contact with the high-temperature alloys which were tested. The oxidation characteristics of the three alloys were noted at various positions along the tubes. Hastelloy N showed the most oxidation resistance followed by type 316 stainless with type 304 stainless steel showing the least oxidation resistance.

Key words: Hastelloy N, stainless steels, compatibility, oxidation, high temperature, electrical insulation, thermal insulation, calcium-silicate, hydrous aluminum-silicate.

INTRODUCTION

Lava, grade A (hydrous aluminum-silicate), has been used as a spacer material to support electric clamshell heaters on high-temperature piping systems at Oak Ridge National Laboratory (ORNL) over the past 20 years. These spacers or heater bushings locate the clamshell heater concentrically around the pipe and frequently provide piping support along horizontal runs. A heater bushing preferably should be an electrical insulator in case the electric heating element contacts the bushing accidently. Also the material must have considerable compressive strength to support the piping at elevated temperatures. The bushing material should be readily machinable to allow rapid fabrication of special sizes and, of course, must be compatible with the piping material.

Grade A Lava heater bushings have several disadvantages, the foremost of which are (1) relatively high material cost, (2) furnace firing is required to drive off volatile constituents prior to installation to reduce the probability of corrosive attack on the pipe material at high temperature, and (3) the material becomes so hard after firing that field modifications are impossible. There have been several instances of piping attack under grade A Lava bushings even though the bushings had been fired before use, suggesting that closer control of this operation may be necessary. Because of these problems, a test of calcium silicate heater bushings was initiated in an attempt to find a substitute material for grade A Lava.

Purpose of the Test

The purpose of this experiment was to compare the compatibility and behavior of fired and unfired grade A Lava heater bushings with calcium-silicate bushings under service conditions and at temperatures from 1100 to 1600°F. The alloys tested with the heater bushings were Hastelloy N, type 304 stainless steel, and type 316 stainless steel. These three alloys were chosen because they are frequently used in our experimental programs. The compositions and heat numbers of the three alloys are given in Table 1.

Our main basis of comparison was the effect of the bushing material on the alloy at elevated temperatures. Thus the appearance of the alloy after test was quite important.

Previous Compatibility Problems with Lava Heater Supports

Compatibility problems with grade A Lava bushings have previously existed, and several cases involving Hastelloy N and grade A Lava at ORNL have been reported. In 1964 ORNL conducted a corrosion program in support of the SNAP-8 space nuclear electrical-generating system.[1] A "chromized" Hastelloy N was used for the piping system which contained

[1] SNAP-8 Corrosion Program Summary Report, ORNL-3898, pp. 60–62 (December 1965).

Table 1. Composition of alloys

	Hastelloy N[a] IR-8402-2[d]	Type 304 SS[b] IR-9556-021[d]	Type 316 SS[c] IR 9697 Y-12 8010-1[d]
Ni	Balance	10.24	13.14
Fe	3.91	Balance	Balance
Cr	6.92	18.59	17.04
Mo	16.39		2.75
C	0.05	0.066	0.057
Mn	0.44	1.63	1.37
Si	0.63	0.49	0.37
P	0.001	0.026	0.027
S	0.009	0.006	0.015
Cu	0.01		
W	0.05		
B	0.007		
Co	0.09		
V	0.35		
Al + 1	0.02		

[a]Wall Tube Company, Heat No. 5101.

[b]Trent Seamless Tube Company, Heat No. 42672.

[c]Union Steel Company, Heat No. 83658.

[d]The IR number is the inspection request number for nondestructive testing.

sodium-potassium alloy (NaK). Several instances of excessive corrosion were noted on the exterior of the "chromized" Hastelloy N tubing underneath ceramic (grade A Lava) bushings. The first instance occurred after 552 hr of operation. The corrosion was found during replacement of heaters. No NaK leakage had occurred. Exterior pitting was found on the chromized 3/4-in.-OD, 0.072-in.-wall Hastelloy N tubing under the ceramic bushings (grade A Lava, unfired) used to space the electric heaters from the tubing. The attack was noticable on the tube wall at the location where the temperature was estimated to have reached 1250°F, and the attack increased toward the high-temperature end of the tubing,

where the temperature was calculated to have been approximately 1445°F. The maximum attack found near the hottest end of the tube was 0.037 in. deep, as determined by external measurements and x-rays of the area. While the exact nature of this attack was not determined, it was attributed to oxidation-inducing volatile material driven out of the ceramic bushings. Subsequent to this, all ceramic bushings of the grade A Lava type were fired at 2000°F prior to installation. The firing did reduce external tube attack in subsequent SNAP-8 test loops, except for the instance reported below.

The second case of exterior corrosion on chromized Hastelloy N happened later in the same program on another loop. The exterior corrosion occurred on the 3/4-in.-OD, 0.072-in.-wall of the hot-spot section, which was operating at 1450°F. A NaK leak developed after 1300 hr of operation. Visual inspection of the failed area showed that the Hastelloy N piping had been excessively oxidized in very localized areas under the fired ceramic insulator bushing in the heater section. The metallographic examination revealed a mixture of metal and oxides in the corrosion products, which was proved by x-ray diffraction analysis to be mostly nickel and nickel oxide.

The circumstances surrounding this incidence of catastrophic oxidation suggest that breakdown of the normally protective oxide layer on the pipe exterior surface was attributable either to contamination from some unknown element or compound in the ceramic bushing or to oxygen starvation in the stagnant area under the bushing. An Inconel 600 shim was placed between the ceramic bushings and the tubing to eliminate these conditions in the remaining loops.

The third case of exterior piping attack of Hastelloy N involved the failure of a thermal convection loop.[2] A salt leak ended the loop operation, and the leak was attributed to a reaction between the grade A Lava bushing and the standard Hastelloy N tubing. It is not known whether the bushings for this test were fired or unfired at the time of installation.

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[2] J. W. Koger, Evaluation of Hastelloy N Alloys after 9 Years Exposure to Both a Molten-Fluoride Salt and Air at Temperatures from 700 to 560°C, ORNL-TM-4189 (to be issued).

Corrosive attack of type 316 stainless steel systems has also been noted underneath grade A Lava bushings. Pipe damage was noted during operation of a series of high-temperature, boiling-potassium experiments which operated at temperatures up to 1560°F.[3] After piping attack was found underneath unfired grade A Lava heater bushings, it became standard procedure to fire all bushings at 1900°F before installation. As an additional safeguard, stainless steel or Inconel-600 shimstock was placed between each Lava spacer and the test piping. In spite of these precautions there was one additional incident of piping attack adjacent to a fired Lava spacer on the final experiment of the test series. During replacement of a failed clamshell heater, a sharp crater was found on the exterior of the drier inlet line (2 in., sched 40) which operated at a temperature of about 1560°F. The crater was about 3/4 in. in diameter and about 0.130 in. deep; that is, the attack had nearly penetrated the 0.154-in.-thick pipe wall. The experiment had operated more than 10,000 hr at the time the crater was found. The damaged section of piping was replaced, and the experiment completed 14,500 hr of operation without further incident.

The problem we fear with an interaction of a bushing and an alloy is catastrophic oxidation.[4] Catastrophic oxidation results in gross amounts of material removal and is caused by low-melting or volatile compounds which form during service. The compounds are formed from impurity oxides which combine with protective oxide films or alloy constituents. The impurity oxides may stem from the alloy itself but more often from external sources. The oxides MoO_3, V_2O_5, WO_3, Bi_2O_3, and PbO are the most serious offenders, and the temperature range in which the process is operative is 1100 to 1700°F. Generally, oxidation occurs at normal rates up to the melting point of the formed eutectic mixtures. Above this definite temperature, oxidation is catastrophic.

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[3] L. C. Fuller and R. E. MacPherson, Design and Operation of Stainless Steel Forced-Circulation Boiling-Potassium Corrosion-Testing Loops, ORNL-TM-2595, December 1969.

[4] J. H. DeVan, Catastrophic Oxidation of High-Temperature Alloys, ORNL-TM-51 (Nov. 10, 1961).

Experimental Procedure and Materials

Test description

The heater bushings were installed on four pieces of 3/4-in.-OD by 48-in.-long tubing in approximately the same configuration as a typical high-temperature piping system. Calcium-silicate bushings were installed on three different metal tubes to evaluate compatibility with type 316 stainless steel, type 304 stainless steel, and Hastelloy N. Both fired and unfired grade A Lava bushings were installed on another Hastelloy N tube for comparison purposes. Clamshell heaters, 6 in. long, with embedded heating elements were used to heat the tubing. Figure 1 shows one of the test tubes during installation of heaters and thermocouples. The partially assembled test is resting on a 1-in.-thick layer of thermal insulation. Note that each heater bushing was slipped over the end of the 3/4-in.-OD tube and moved axially to its proper position. We did not split each bushing in half, as is normally done to permit installation, because of the relatively short lengths and constant diameters of the tubing. Seven heaters were placed on each 48-in.-long tubing specimen along the six Chromel-Alumel thermocouples. Each thermocouple was located between the heater bushings at the end of the clamshell heaters. The thermocouples were 1/16-in.-OD stainless steel sheathed with insulated junctions and were clasped against the exterior diameter of the tube with special stainless straps, as shown in Fig. 2. After installation of heaters and thermocouples was completed, each assembly was insulated with two layers of 1-in.-thick Kaowool insulation (manufactured by Babcock and Wilcox) and placed in steel mesh support trays for testing.

Test temperatures from 1100 to 1600°F were selected as typical of those to be expected in our future experimental work. Therefore, this temperature gradient was maintained along each of the four tube specimens. Variable voltage transformers were manually adjusted to obtain the proper temperature levels. Thermocouple output was continuously recorded on two multipoint strip chart recorders. The inside of the tubing was exposed to stagnant air throughout the test period.

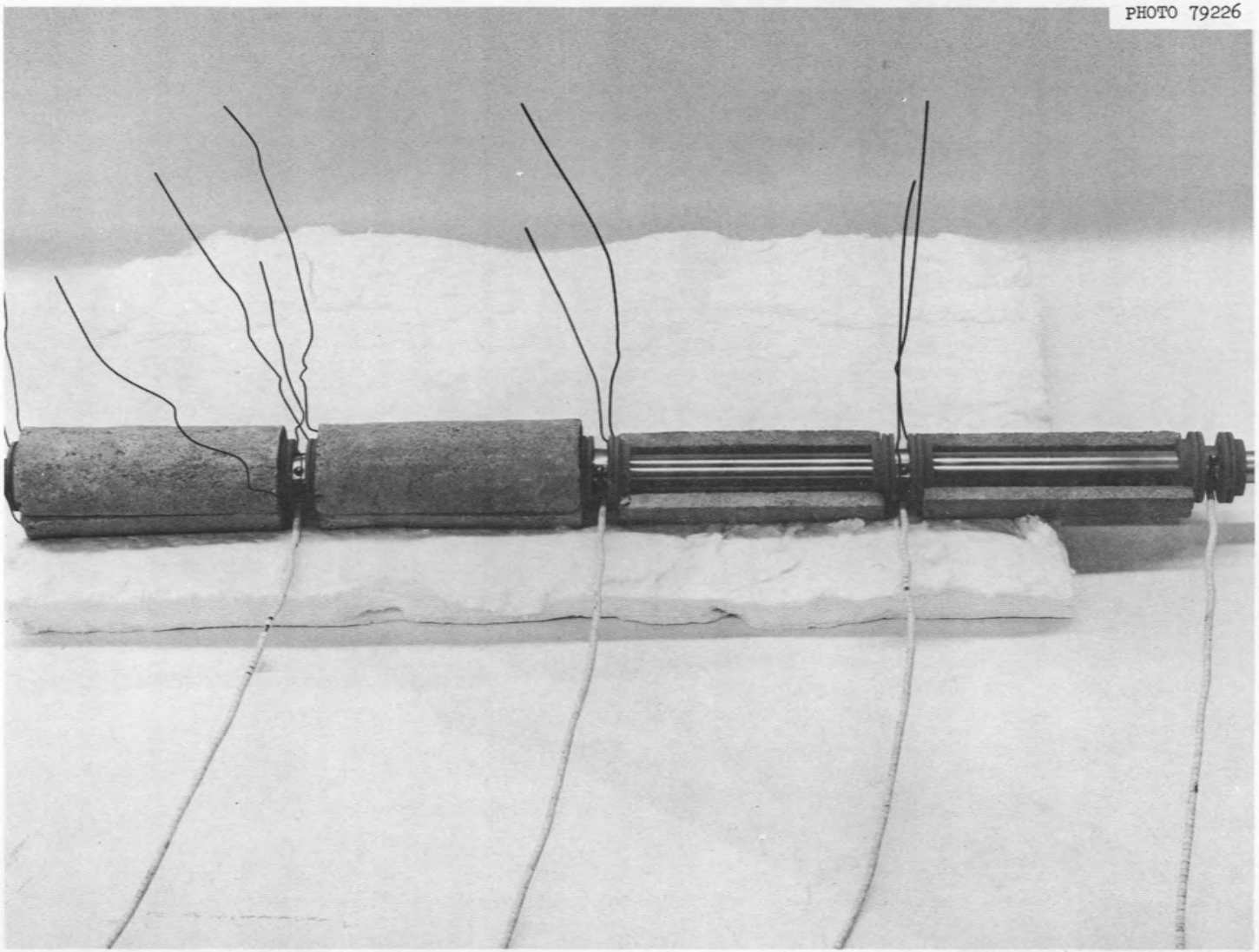

Fig. 1. Test tubing during installation of heaters and thermocouples.

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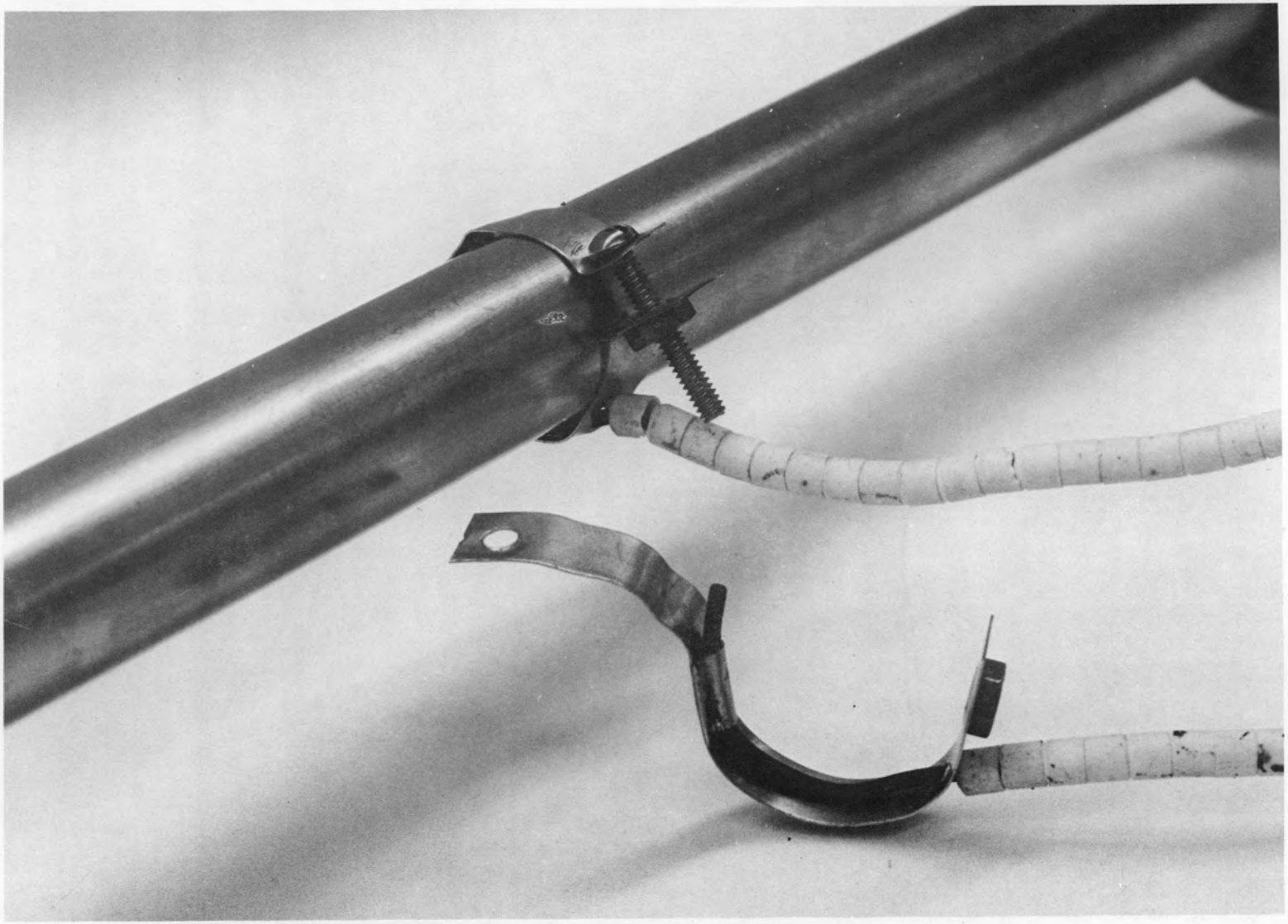

Fig. 2. Method of strapping thermocouples to test tubing.

Tube preparation prior to testing

Each of the 48-in.-long metal tubes was cleaned prior to test assembly to insure that no foreign material was present to cause attack or corrosion during high-temperature operation. The tubes were first polished with sandpaper (400 mesh grit) and wiped clean with acetone. The outer diameter of each tube was then measured at 8-in. intervals. Stencil marks were placed on the outer surface at each end of the tubes to insure positive identification throughout testing and post-run inspection. Final cleaning was done by again wiping with acetone-soaked cheesecloth on both the inner and outer tube surfaces. This was followed by similar wiping with an alcohol-soaked cloth. White gloves were used in subsequent test assembly to preclude surface contamination by fingerprints. The inspection request number and heat number for each of the tube materials were recorded, and a 2-in.-long archive sample of each tube was stored for future reference.

Characteristics and description of calcium-silicate material

The calcium-silicate insulating board used to fabricate the heater bushings was manufactured by Johns-Manville and is sold as "heat treated Marimet-45." The material is heat treated at 1100°F during its manufacture. The board was purchased in a 4-ft × 8-ft × 1-in.-thick sheet. Physical and thermal properties listed by the manufacturer are shown in Table 2.

The electrical resistivity of the calcium-silicate board was checked prior to high-temperature operation using a 500-V dc megohm tester. The test meter indicated an infinite resistance through a 1-in. thickness. The resistance of a heater bushing was rechecked after operating for 5700 hr at 1600°F. The resistance of a 1/4-in. thickness was indicated as infinite.

Calcium-silicate board is attractive as a replacement for Lava insulator bushings under clamshell heaters because it is readily machinable and does not need the expensive firing process now used to remove impurities from Lava. Another advantage of calcium silicate is that it remains machinable after service at high temperatures which allows easy field modification, if needed. The cost of heat-treated Marimet-45 is about two cents/in.^{3} when purchased in a 4-ft by 8-ft sheet. The cost of grade A

Table 2. Physical and thermal properties of heat-treated Marimet-45

Density, lb/ft^3		44 average
Modulus of rupture, psi		900 minimum
Screw holding strength, lb/screw		
(withdrawal load for type A, No. 8 sheet metal screw in a No. 20 drilled hole)		
1/2-in. penetration		100 minimum
7/8-in. penetration		275 minimum
Dimensional change after reheating, % shrinkage		
24 hr at: 1400°F – length and width		0.8 maximum
thickness		1.5 maximum
1700°F – length and width		1.5 maximum
thickness		3.0 maximum
1800°F – length and width		2.0 maximum
thickness		10.0 maximum
Durometer hardness, units		50 minimum
Thermal conductivity, $Btu/hr\text{-}ft^2\text{-}°F/in.$		
Mean temperature, °F	Average	
200	0.75	
400	0.77	
600	0.79	
800	0.81	
1000	0.84	
Size		4 ft by 8 ft
Thicknesses		Up to 2 in.

Lava runs from five to ten cents/in.^{3} depending on the size of block purchased. An important savings in our experimental usage would come from the time and labor saved by deletion of the overnight firing process now used for Lava heater bushings.

Fifty-two heater bushings were machined from a portion of the 4- by 8-ft sheet of insulating board. Each bushing was scribed with an identifying number and the inside diameter of the bushing was recorded for later use in determining the amount of shrinkage during high-temperature service. Measurements made after 5700-hr operation at temperatures from

1100 to 1600°F showed that the inside diameters shrank less than 1% in all cases. This is consistent with the manufacturer's data in Table 2 which indicate a maximum length and width shrinkage of 1.5% at 1700°F.

The load-bearing capability of calcium silicate was not demonstrated in this test because the weight of the test apparatus was quite small. However, the material has been used in other applications in the Reactor Division where a considerable compressive load was imposed on the insulating board. In one instance, a 1-in.-thick layer was used to support and thermally insulate heavy steel boxes at temperatures up to 800°F. The calcium-silicate board was subjected to compressive loads up to 200 $lb/in.^2$ with no apparent damage.

Description of Lava heater support material

The ceramic bushings for this test were machined from an American Lava Corporation product, "Lava grade A." The machining was done before the Lava bushings were fired, as is the normal procedure. After firing, the Lava develops into a good electric insulator and becomes extremely hard. The firing process also reduces the potential for piping attack at high temperature and, therefore, firing is routinely done for most of our applications. The firing procedure for the Lava used in this test consisted of preheating at 300 to 1500°F, holding for about 1 hr, and then increasing the temperature at 300 to 1900°F. The bushings were then furnace cooled. A portion of the bushings was left unfired to see if this caused a difference in test performance.

Test operation

The test operated almost continuously for 8 months (5700 hr) and was then shut down for destructive examination. The test was stopped at this time because this represents a normal operating time for most of our experimental tests. The experiment had only one sharp temperature cycle during operation due to a power outage which occurred after 400 hr of operation. Since an operator happened to be present at the time of the outage, temperatures were restored before changes greater than 30°F could occur.

Precise temperature control of each bushing location could not be obtained because the clamshell heater circuits were hooked in parallel to limit the number of variable transformers required. However, the temperature recorders indicated readings within 40°F of a desired level, e.g., a bushing temperature designated as 1200°F should be considered as 1200 ±40°F. Day-to-day temperature variations due to line voltage changes and building temperature changes were less than 15°F and are included in the 40°F tolerance.

Post-test Examination of Tubing

Figure 3 gives the overall view of the tubes after test. The widely spaced areas were the locations of the heaters. The heater bushings had been moved a small distance axially into the space occupied by the heaters to look for tube damage at the time the picture was taken. Lava bushings were installed only on the upper Hastelloy N tubing specimen and alternate sets were fired and unfired, i.e., unfired bushings were located at the left end and at the 1200, 1400, and 1600°F positions, while fired bushings were located at the right end and at the 1100, 1300, and 1500°F positions. All unfired Lava bushings, with the exception of the bushing on the far left, were partially fired during the test as indicated by color change from gray to pink.

Note the failure of the type 316 stainless steel and the heavy scaling of the type 304 stainless steel under the last heater. Metallographic specimens of the tubing were taken from underneath each heater (except the last heater) and from underneath one bushing for each temperature level. The outside and inside surfaces were examined. Figure 4 shows a close-up of the outer surfaces after the heaters were removed. Figure 5 shows another view of the heavily oxidized stainless steel tubes.

Tubing beneath heaters. Figure 6 shows micrographs of the tubing beneath the heaters. Table 3 summarizes the amount of oxide and grain boundary penetration observed. The type 304 stainless steel showed the worst behavior, with the type 316 stainless next, and Hastelloy N showing the most resistance to oxidation.

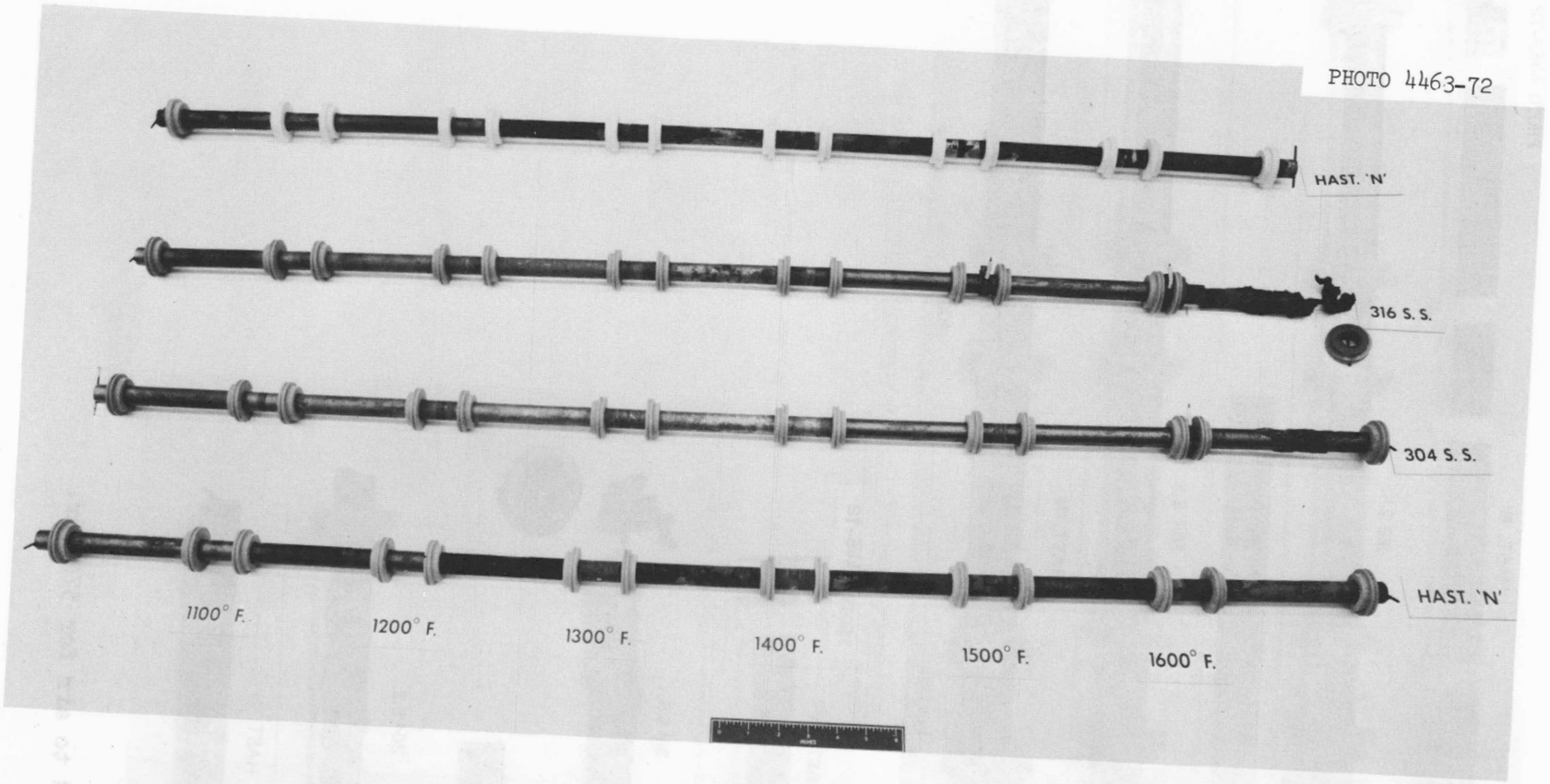

Fig. 3. Tubing and bushings after 5700 hr exposure to air.

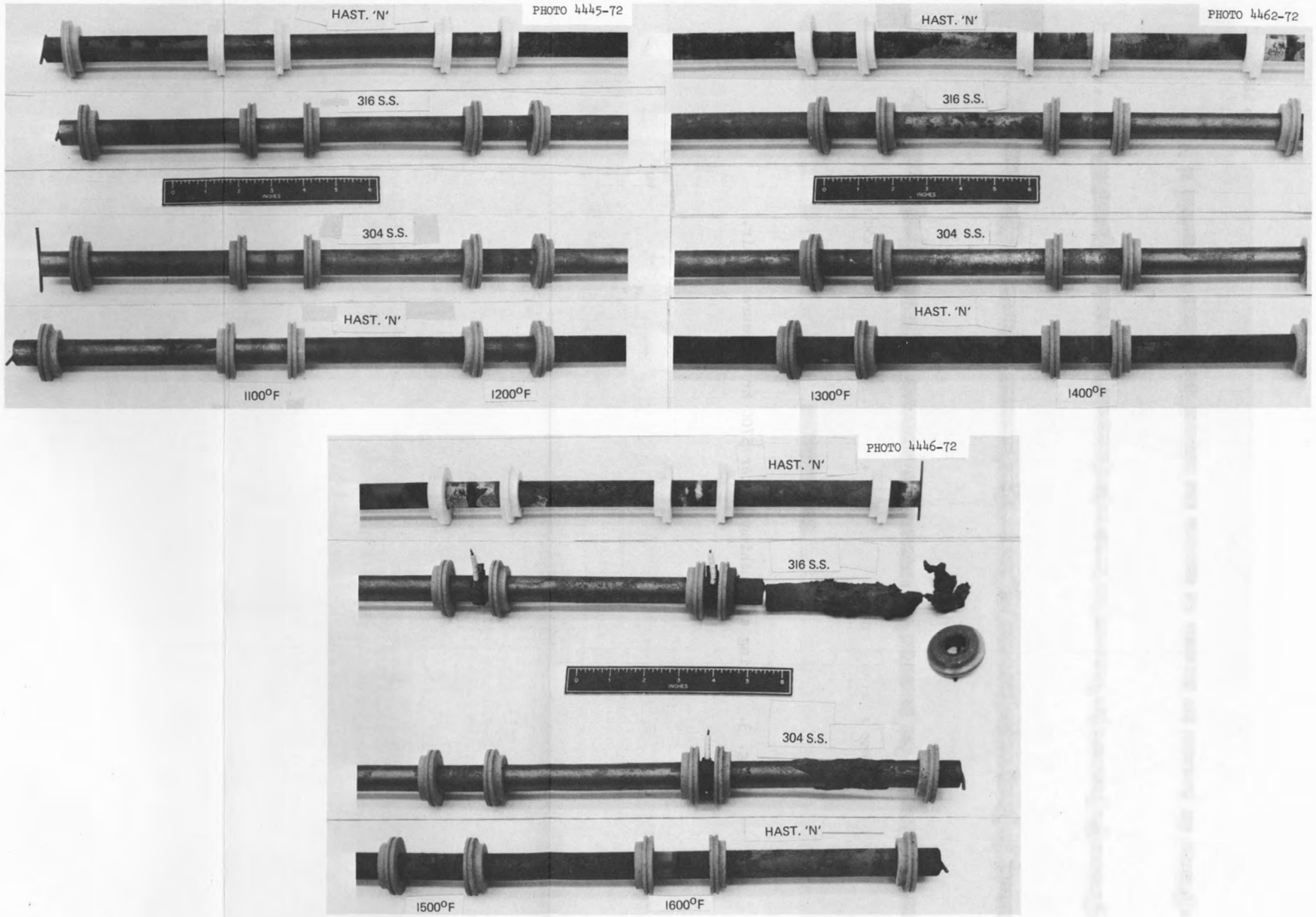

Fig. 4. Outside surface of tubing exposed to air for 5700 hr.

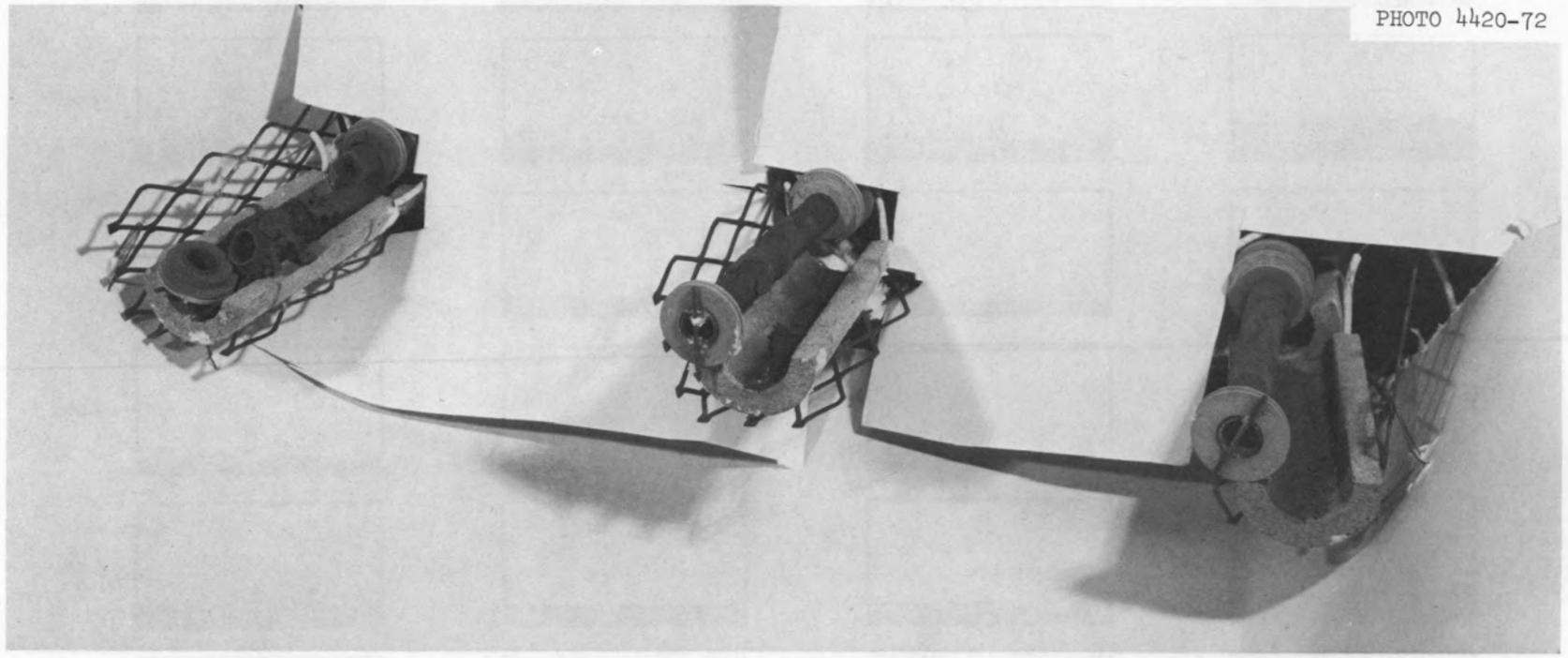

Fig. 5. Tubing exposed to air for 5700 hr at temperatures greater than 1600°F. Left to right, type 316 stainless steel, type 304 stainless steel, and Hastelloy N.

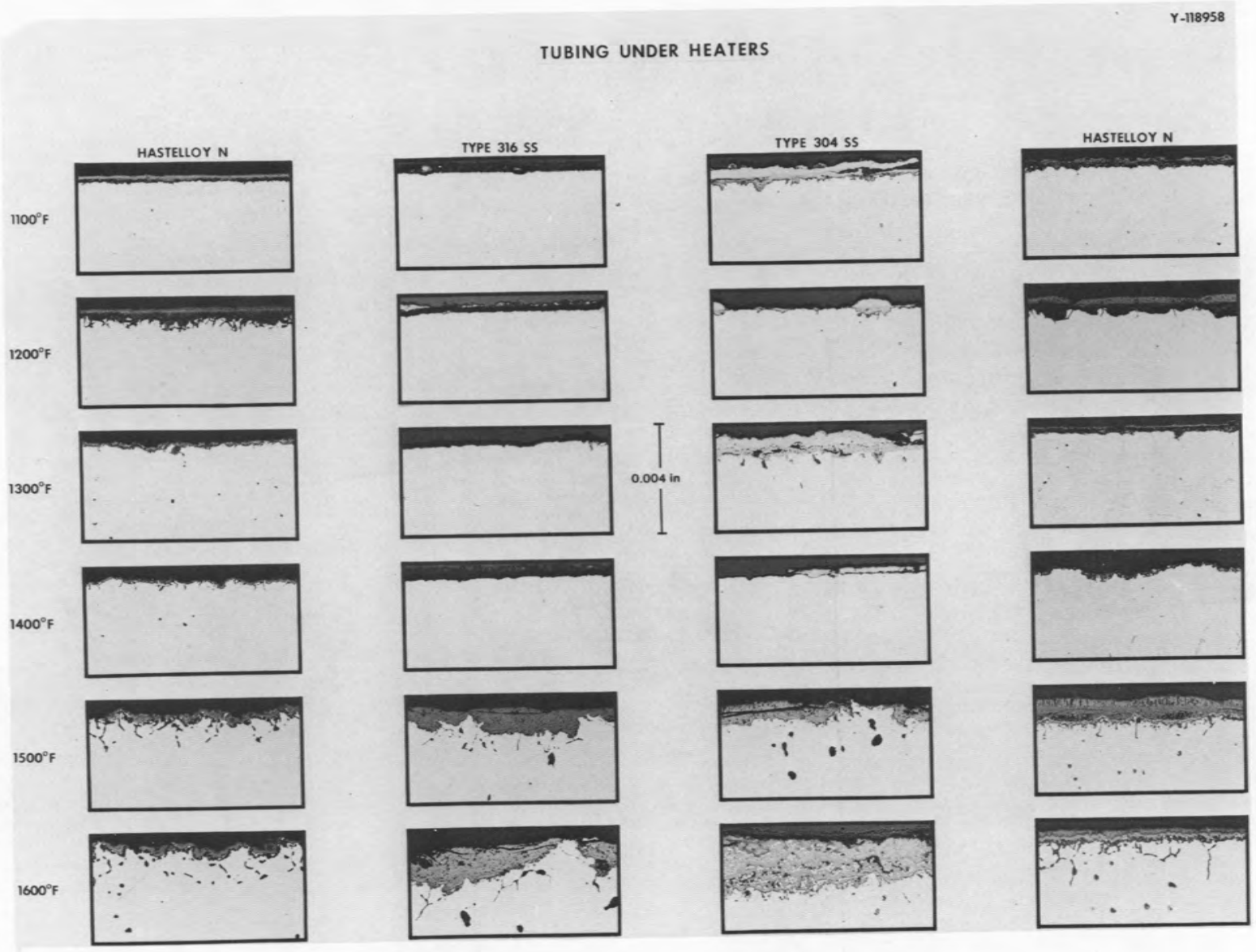

Fig. 6. Exterior surface of tubing under heaters.

Table 3. Oxide thickness on tubing under heaters exposed to air for 5700 hr

Temperature (°F)	Oxide thickness, microns			
	Hastelloy N	Type 316 stainless steel	Type 304 stainless steel	Hastelloy N[c]
1100	4	2	10	4
1200	8	2	15	8
1300	2	2	20	2
1400	2	2	7	2
1500	6[a]	10–20[a]	10–20[a]	20[a]
1600	6[b]	0–40[b]	50	10[b]

[a]Grain boundary penetration of 20 microns.

[b]Grain boundary penetration of 30 microns.

[c]Lava bushings were installed on this tube.

The temperature levels listed in Fig. 6 and Table 3 are approximate, since there were no thermocouples located directly under the clamshell heaters where the metallographic samples were removed. In all cases the metallographic samples were taken within 1 in. of the end of the heater where thermocouples were attached and therefore the temperature difference resulting from the axial gradient along the tube is small. However, when combined with the previously mentioned ±40°F maximum error in controlled temperature levels at the bushing locations, tubing specimen temperatures are only accurate to ±60°F.

The scaling temperature for type 304 and type 316 stainless steel is 1650°F, and it appears that the tubing at the high-temperature end of the specimen string was operating in this temperature range where heavy oxidation would be expected of the iron-base alloys.

Tubing beneath bushings. Figure 7 shows micrographs of the tubing beneath the bushings. Table 4 summarizes the amount of oxide and grain boundary penetration observed. Again, the type 304 stainless steel was oxidized the most, followed by the type 316 stainless steel, with the

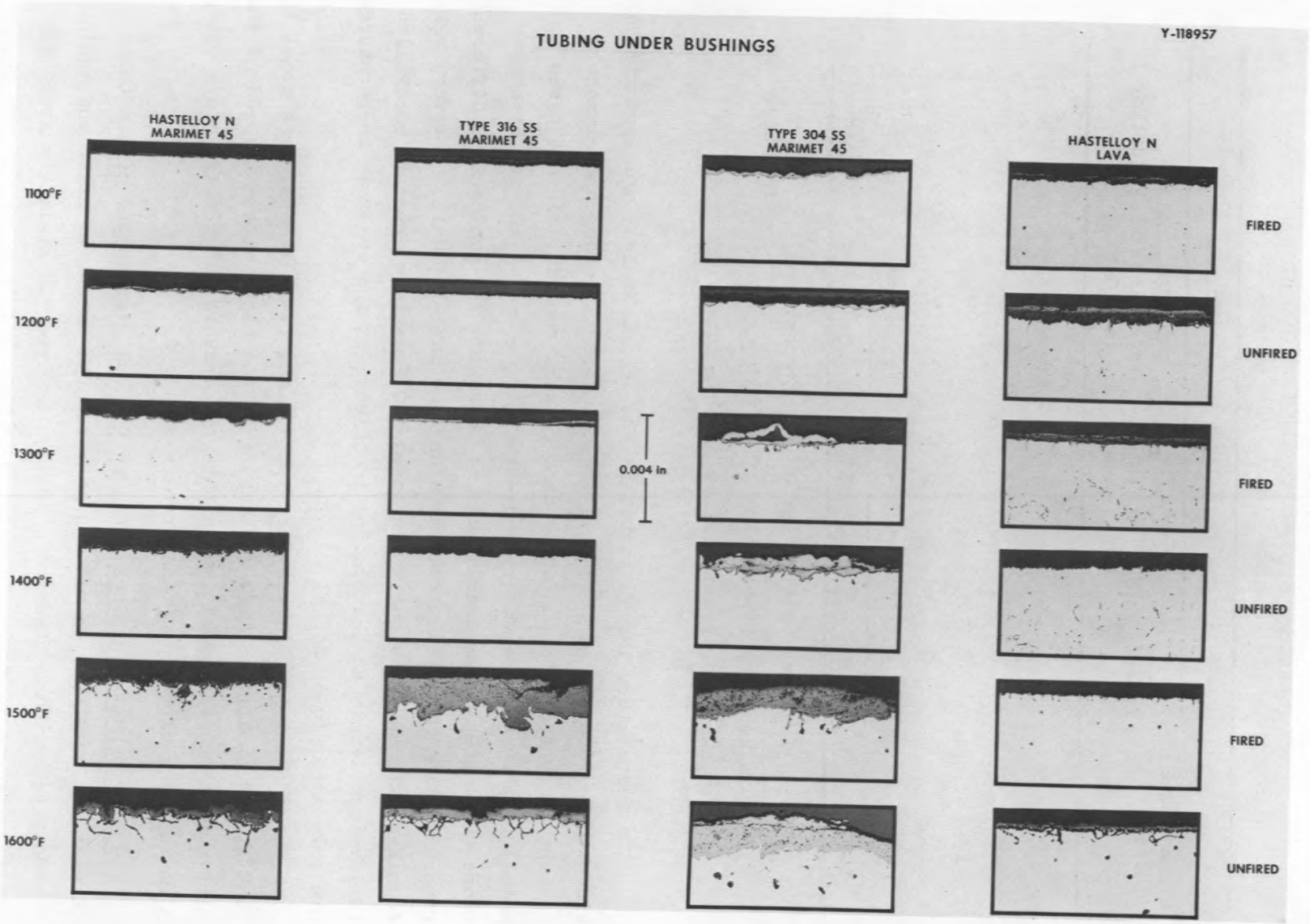

Fig. 7. Exterior surface of tubing under bushings.

Hastelloy N showing the most oxidation resistance. At 1500°F and above the oxide on the iron-base alloys spalls and penetrates the matrix. We see an example of a blister (without film rupture) on the type 304 stainless steel exposed at 1300°F. There is less oxide on the stainless steels under the bushings at 1600°F than on the stainless steels under the heaters at slightly higher temperatures. Thus the bushings may have had a protective effect.

Table 4. Oxide thickness on tubing under bushings exposed to air for 5700 hr

Temperature (°F)	Oxide thickness, microns			
	Hastelloy N	Type 316 stainless steel	Type 304 stainless steel	Hastelloy N[c]
1100	1	1	2	4
1200	2	1	2	8
1300	2	2	4	6
1400	4	1	15	2
1500	8[a]	20–30[b]	20[b]	2
1600	10[b]	10[b]	20[b]	4[a]

[a]Grain boundary penetration of 10 microns.

[b]Grain boundary penetration of 20 microns.

[c]Lava bushings were installed on this tube.

In terms of compatibility under the conditions of our experiment, there was no difference in the behavior of Marimet-45 and grade A Lava. Both fired and unfired grade A Lava bushings were tested on one Hastelloy N tube and no difference was observed in tubing-bushing interaction. The behavior of the grade A Lava in our experiment is not inconsistent with our past experience on experimental equipment since only occasional piping attack has occurred from both fired and unfired material. The reason for the random nature of pipe attack from grade A Lava bushings is not known but may be due to natural variations which are present in the material as mined or perhaps due to variations in our firing or fabrication procedures.

The Marimet-45 did not promote excessive oxidation and was compatible with the two types of stainless steel and Hastelloy N. Based on this limited, but highly successful experience, the material should be favored for future heater bushing applications.

Inside surface of tubing. Figure 8 shows micrographs of the inside surface of the tubing. Table 5 summarizes the amount of oxide and grain boundary penetration observed. Oxidation of the inside surface was a little worse than oxidation of the outside surface, both under the heaters and the bushings. Again the most oxide was found on the type 304 stainless steel, followed by the type 316 stainless steel, with the Hastelloy N showing the most resistance to oxidation. An unbroken oxide layer was found on all alloys at 1100°F. Above 1100°F some spalling was noted on the type 316 stainless steel. On the type 304 stainless steel, the oxide penetrated into the matrix at 1500°F and above. On the type 316 stainless steel, voids began forming at 1400°F and the oxide remained on the surface.

Table 5. Oxide thickness on inside surface of tubing exposed to stagnant air for 5700 hr

Temperature (°F)	Oxide thickness, microns			
	Hastelloy N	Type 316 stainless steel	Type 304 stainless steel	Hastelloy N[e]
1100	2	2	6	4
1200	4	4	4	4
1300	6	4	4	4
1400	4	10–20[a]	10[a]	20
1500	10[a]	15[c]	10–60[a]	20–30
1600	10–20[b]	20[d]	100	10[c]

[a]Grain boundary penetration of 20 microns.

[b]Grain boundary penetration of 30 microns.

[c]Grain boundary penetration of 40 microns.

[d]Grain boundary penetration of 50 microns.

[e]Lava bushings were installed on this tube.

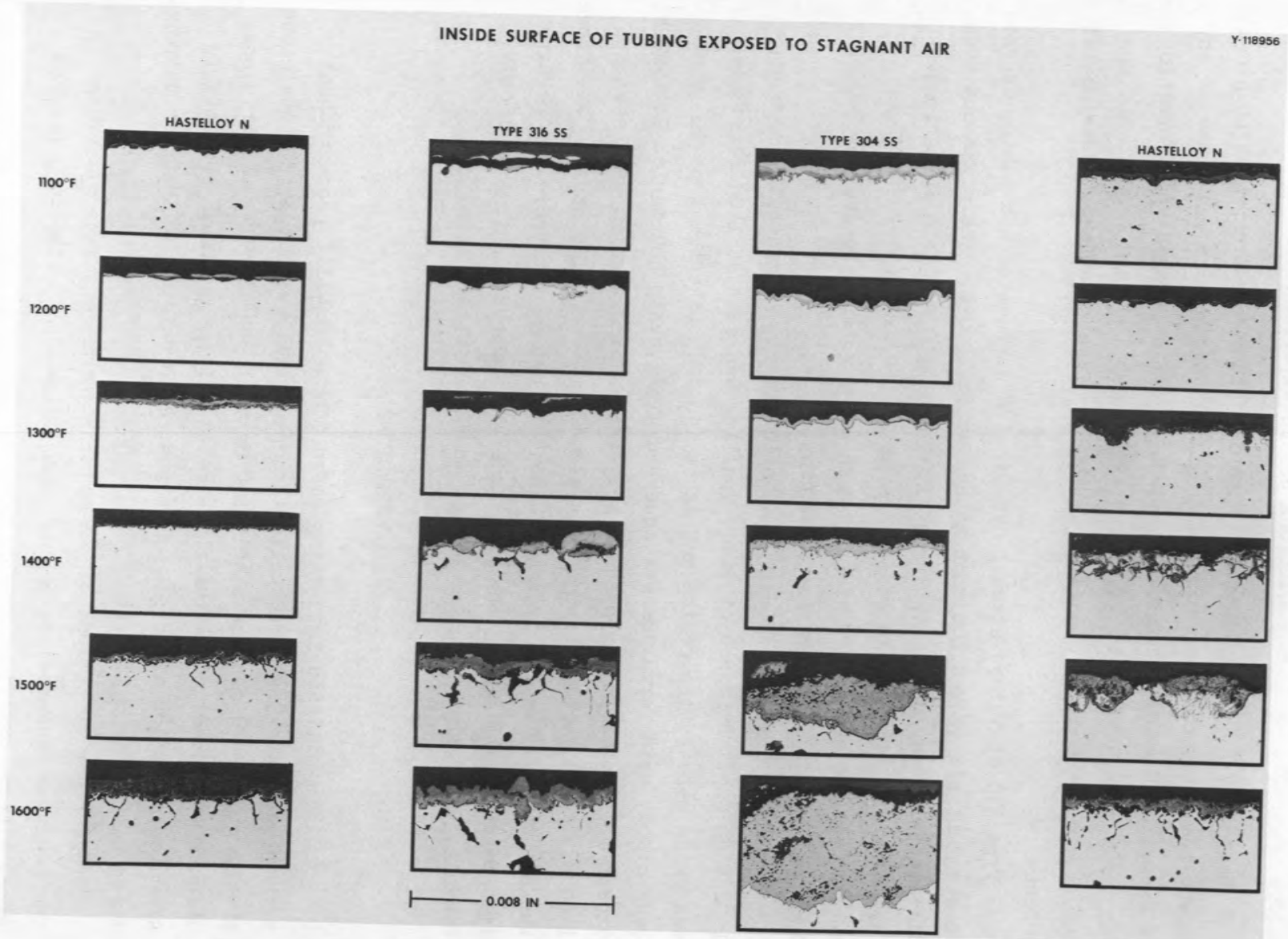

Fig. 8. Inside surface of tubing exposed to stagnant air.

Behavior of the individual alloys. The behavior of the individual alloys under the various conditions of the experiment is discussed below:

Hastelloy N. – The Hastelloy N tubing under the Marimet-45 bushings showed the least oxide up to 1300°F. Little difference was noted on any of Hastelloy N tubing from 1400 to 1600°F. In all cases void formation was first seen at 1400°F, was quite evident at 1500 and 1600°F, and was mainly along the grain boundaries. The bushings produced no detrimental effects.

Type 316 stainless steel. – The inside surface of the tubing exposed to stagnant air showed the most attack up to 1400°F. There was not much difference in any of the tubing at 1500 and 1600°F. Void formation was first evident along grain boundaries at 1500°F except for the inner tubing surface exposed to stagnant air where voids were seen at 1400°F. The bushings did not increase oxidation.

Type 304 stainless steel. – An oxide layer was generally seen at 1100°F and up. Evidence of oxide spalling was seen at 1400°F and there was oxide penetration of the matrix at 1500°F. Like the type 316 stainless steel, void formation was observed along grain boundaries at 1500°F except for the tubing exposed to stagnant air where voids were seen at 1400°F. Metal particles were seen mixed with the oxide at 1600°F. There was not much difference in any of the 304 tubing, but there was perhaps less oxide on the tubing that was under Marimet-45 bushings at 1600°F. Overall, the bushings had no effect on the oxidation behavior.

CONCLUSIONS

Calcium-silicate insulating board (Johns-Manville product "heat treated Marimet-45") has excellent compatibility with type 316 stainless steel, type 304 stainless steel, and Hastelloy N alloy. No detrimental effects were observed in the three metal alloys by visual and metallographic inspection after 5700-hr exposure to calcium silicate at temperatures ranging from 1100 to 1600°F.

Under the conditions of our experiment there was no difference in the behavior of Hastelloy N which was exposed to Marimet-45, unfired grade A Lava, and fired grade A Lava.

Calcium-silicate insulating board is recommended for use as spacer supports for clamshell heaters on high-temperature piping systems because of the low cost and easy machinability of the material. This test indicates that prefiring of Marimet-45 bushings is not required prior to use on high-temperature stainless steel or Hastelloy N piping systems. The material would appear useful in many high-temperature applications where a readily machinable, electric, or thermal insulator of modest load bearing capability must be in contact with high-temperature alloys.

Hastelloy N showed the most oxidation resistance overall, followed by type 316 stainless steel, and then type 304 stainless steel. In most cases voids were first seen in all the alloys at 1400°F with grain boundary attack at 1500 and 1600°F.

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