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OBSERVATIONS ON THE IN-SODIUM CORROSION AND TRIBOLOGY OF ALUMINIDE COATINGS ON INCONEL 718

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ABSTRACT

In sodium cooled fast reactor systems, the ability of surfaces, which are in contact under sodium to slide relative to one another is critical to the successful operation of many components. Materials selected for application at these interfaces will be under load intermittently or continuously in high temperature liquid sodium for many thousands of hours. During operation, reactor mechanical components such as the duct load pads or core restraint mechanism, will be subject to movement resulting from normal operation, differential thermal expansion, and flow or mechanically induced vibrations. Design requirements for these components have necessitated materials with adequate long term sodium stability at ~1150°F, and thermal shock resistance coupled with good wear resistance and low coefficients of friction. This paper describes initial experimental investigations on the corrosion and tribological behavior of a nickel base alloy, Inconel 718, a material considered for use in these components. Subsequent studies have shown that the incorporation of an aluminide surface layer on Inconel 718, using a commercially available diffusion process, results in reductions in the corrosion rate and friction and wear coefficients.

After 2000 hours exposure to 1100°F, low oxygen flowing sodium, the average corrosion rate for the diffusion treated alloy was significantly lower than the 0.06 mpy value recorded for the untreated Inconel 718. Structural integrity of the aluminide layer was maintained even though some aluminum loss was detected. Average dynamic friction coefficients of ~ 0.25 were obtained for the treated material in 1000°F sodium pin on plate tests. No measureable wear of the aluminide coating against itself was observed in these tests. In contrast, Inconel 718 exhibited substantial adhesive type wear.

INTRODUCTION

Relative motion of one surface with respect to an adjacent contacting surface is a common operating feature of many mechanisms in a reactor design. In a sodium cooled system, materials selected for application at certain component interfaces may be under load intermittently or continuously in a high temperature corrosive environment before being required to slide with respect to one another. In addition to the required motion during normal operation, movement may also result from differential thermal expansion and flow or mechanically induced vibrations. Nuclear component design presently necessitates the inclusion of allowances for frictional loads and wear between contacting surfaces, and intensive efforts are underway with the objective of minimizing friction and wear.

Design requirements for such components, e.g., the core restraint mechanism, include materials with long term sodium stability at $\leq 1150^{\circ}$F (620°C), thermal shock resistance, high wear resistance and low coefficients of friction (≤ 0.4). Some seizing and galling has been encountered with the 300 series type stainless steels in sodium, with the result that interest has been stimulated in alternate structural materials such as Inconel 718 for sliding contact applications. Current materials development programs have as one of their objectives the upgrading of Inconel 718 and stainless steel by the use of chromium carbide coatings and aluminide diffusion coatings for necessary low friction interfaces.[1-6]

In this paper, some of the metallographic aspects of the sodium compatibility and friction and wear studies on Inconel 718 and on Inconel 718 with an aluminide coating performed as part of the above mentioned program are presented.

MATERIALS AND TECHNIQUES

The sodium compatibility testing was performed in a pumped sodium loop system constructed of stainless steel and designed for the exposure of 4-1/4 in. x 5/8 in. x 0.180 in. plate and 1/8 in. diameter pin samples to high temperature sodium. This loop system, which is of a type described in detail elsewhere,[7] was operated non-isothermally at test section temperatures of 1160 and 1100°F for 2128 hours at a sodium flow velocity of ~5 fps, and an oxygen impurity level in the sodium in the range 0.5 to 1.0 ppm. Average corrosion rates for the period 0 to 2128 hours were calculated from the change in weight of the test samples resulting from the sodium exposure.

The friction and wear test machines permit prototypic testing in sodium at temperatures up to 1200°F under controlled impurity contents (0.5-1.0 ppm, oxygen) and flow rates (~ 1 fps). The basic experimental approach is to press two diametrically opposed horizontal specimens (pins) against mating specimen surfaces (plates) mounted on a vertically reciprocating rod.[8] Tests are run under as prototypic-as-possible conditions of load and motion, simulating those anticipated in the reactor system. The contact loads (normal static loads), the force necessary to move the plates (frictional load), and the displacement are continuously measured and recorded on magnetic tape. Friction coefficients are calculated as the ratio of the frictional and the measured normal static loads on the pin-plate specimens. Wear data are obtained from weight changes and measured height changes of the pin and from quantitative analysis of wear scars on the plate specimens using a surface profile instrument.

The heat analysis for the Inconel 718 used in these studies is as follows (all values in weight percent): 53.05 Ni, 18.35 Cr, 18.18 Fe, 5.29 Cb +

Ta, 3.03 Mo, 0.95 Ti, 0.59 Al. The aluminide layer was applied by Chromizing Corporation and the process entails the packing of the part to be coated in a mixture of alumina, aluminum and chromium. After insertion of ammonium fluoride into the mixture at a temperature of 1800°F, aluminum fluoride is formed and adsorbed on the metal surface. Dissociation takes place with aluminum diffusing into the Inconel 718 forming NiAl and Ni_3Al, and typically results in a layer 2 mil thick after 10 hours at temperature. Following the coating process, the part is post heat treated (1 h. @ 1750°F, quenched and aged 8 h. @ 1325°F and 10 h. @ 1150°F) to regain the desired Inconel 718 properties.

Characterization of sodium exposed and friction and wear test samples included optical and scanning electron microscopy and x-ray diffraction. Metallographic specimen preparation consisted of: nickel plating for edge retention, conventional polishing and etching. An etch consisting of 1% chromic acid was used for the Inconel 718, while a mixture of glycerol, hydrochloric and nitric acids (6:5:4) was the preferred etchant for the aluminide layer.

RESULTS AND DISCUSSION

In the sodium compatibility section of this study, samples of Inconel 718 and aluminized Inconel 718 were exposed to sodium at 1150°F for 2128 hours, whereupon they were cleaned of any residual sodium by rinsing in alcohol and water. Both materials lost weight as a result of exposure to sodium with average corrosion rates of 0.24 mpy for the Inconel 718 and 0.03 mpy for the aluminized material being recorded. These corrosion rates probably reflect the ease with which nickel is removed from the surface. Previous work has shown that nickel alloys such as Inconel 718 lose weight during sodium exposure due to loss of nickel[7], but in the case of nickel aluminides, nickel is more tenaciously fixed in the matrix as Ni_3Al or NiAl and therefore lower corrosion rates would be anticipated.

Examination of the cross sectional microstructure of Inconel 718 revealed only a very shallow affected depth (1-2 μm) in the sodium exposed samples (see Figure 1). The matrix consists of uniform austenitic grains with extensive grain boundary precipitation (gamma prime phase and a Ni_3Nb phase).[9] The microstructural appearance of the aluminide layer similarly appeared little changed in cross section, but in this case there was no sign of an affected surface layer. Based on an optical metallographic examination, it appeared that the measured weight losses must be attributable to surface or near surface (< 1 μm) changes and this was partly confirmed in a scanning electron microscopical (SEM) examination. In Figure 2, some typical low power surface views before and after sodium exposure are depicted. The Inconel 718, as ground, unexposed surface became somewhat smoother during exposure and at higher magnification (not shown) the surface ridges were less pronounced suggesting that material removal consistent with the weight loss had occurred. The aluminide layer on Inconel 718; however, exhibited a form of pitting type attack rather than a general removal with the original surface ridges and pebbly areas still in evidence after sodium exposure. Surface x-ray analysis using the x-rays generated in the SEM revealed that the outermost layer (~1 μm in depth) of the Inconel 718 sample had lost nickel such that chromium became the major constituent in the layer. For the samples of Inconel 718 with an aluminide surface layer, no major change in surface chemistry was detected. However, from this work, there was a suggestion that the aluminum content of the surface layer may have decreased slightly. To complement the characterization studies, an x-ray diffraction examination using nickel filtered, copper radiation was conducted on both the Inconel 718 and the nickel aluminide layer. For the Inconel 718, the major phase was austenite with minor indications for carbides and gamma prime phase. Figure 3 schematically depicts the structure of the aluminide layer obtained by x-ray diffraction. No change in composition was detected as a result of sodium exposure for both materials which is consistent with indications of shallow affected depths discussed previously.

Friction results from pin on plate tests for Inconel 718 and Inconel 718 with an aluminide surface layer (each rubbing against itself) at a 10,000 psi stress level are compared in Figure 4. The data, which are presented as average dynamic friction coefficients as a function of cumulative travel distance, illustrate the pronounced enhancement in frictional behavior resulting from the aluminide diffusion coating. In all three phases of testing, results for the aluminide material are significantly less than those for Inconel 718. In addition, the range in friction coefficients, reflecting changes in friction with cumulative rubbing, is also considerably smaller for the aluminide material. The highest friction coefficients were obtained at the end of the initial 450°F period for the aluminide material but at the end of the final 450°F period for the Inconel 718. The reason for this difference is not clear, but may be related to specimen wetting behavior at 450°F coupled with the presence of a sodium chromite lubrication film.

The aluminide diffusion treatment was also found to greatly increase the wear resistance of Inconel 718. Plate specimens were examined quantitatively for wear using a surface profiling instrument. Although light wear scars were visually evident on the plate specimens, no measurable wear could be detected. Tests involving Inconel 718 without an aluminide diffusion coating by comparison resulted in average wear coefficients (K) in the range of 12 in^3/psi-in $\times 10^{-13}$ and maximum wear depths of 1 to 2 mils. (See Table I). The typical surface appearance of the Inconel 718 and the aluminide pin specimens tested under conditions shown in Figure 4 are illustrated in Figure 5. Prior to testing the Inconel 718 pin sample had a ground finish and the aluminide pin was finished using an abrasive to an 100 μinch r.m.s. surface finish. The Inconel 718 material incurred moderate to severe surface damage, characteristic of an adhesive type wear process. Material transfer and smearing was evident, with debris buildup on regions of the pin edges. In contrast, the aluminide material, tested at the same conditions, incurred minimal surface damage with wear limited to a smoothing and polishing of surface asperities and unworn regions (light grey areas) are clearly evident on the pin surface. No evidence of extensive material transfer across the interface could be detected.

SUMMARY AND CONCLUSIONS

Results from an experimental program have indicated that an aluminide diffusion coating, consisting principally of an NiAl phase, on an Inconel 718 alloy substrate has several advantages over uncoated Inconel 718 for certain sodium cooled reactor applications. In compatibility and friction and wear testing, under sodium conditions typical of those to be experienced in reactor service, an aluminide layer on Inconel 718 has been found to possess a lower corrosion rate at 1160°F, and a lower friction and wear rate. No deterioration in the NiAl surface layer has been observed after greater than 2000 hours sodium exposure or 4000 inches cumulative rubbing distance under reciprocating conditions. Additional testing, presently in progress at Westinghouse Hanford Company has as its objective the qualification of coatings such as aluminides for in-reactor service and will supplement this work particularly in the area of irradiation behavior.[10]

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REFERENCES

(1) R. N. Johnson, S. L. Schrock and G. A. Whitlow, "Wear Resistant Coatings for Reactor Components in Liquid Sodium Environments," to be published, J. Vac. Sci. Technol., July/August (1974).

(2) W. J. Kurzeka, R. M. Oliva, and F. W. Poucher, in Atomics International Rockwell International, Annual Technical Progress Report, Sodium Technology and Cover Gas Seal Development Programs Government Fiscal Year 1972, AI-AEC-13037, 59, (1972).

(3) Liquid Metal Engineering Center (LMEC), Annual Technical Progress Report, January-December 1972, LMEC-73-1, 1, (1973).

(4) Westinghouse Advanced Reactors Division, Sodium Technology Program Quarterly Progress Report for Period Ending January 31, 1974, WARD-NA-3045-9, (1974).

(5) N. J. Hoffman and J. J. Droher, "A Review of Wear in Sodium" presented at NACE Meeting, CORROSION/74, Chicago, March 1974, Paper #15, (1974).

(6) G. A. Whitlow, R. L. Miller, S. L. Schrock and P. C. S. Wu, "Sodium Compatibility Studies of Low Friction Carbide Coatings for Reactor Application," presented at NACE Meeting, CORROSION/74, Chicago, March 1974, Paper #17, (1974) to be published in CORROSION (1974).

(7) G. A. Whitlow, J. C. Cwynar, R. L. Miller, and S. L. Schrock, "Sodium Corrosion of Alloys for Fast Reactor Applications," Proc. Symp. Chemical Aspects of Corrosion and Mass Transfer in Liquid Sodium, 1971 Meeting of AIME, Detroit, 1, (1973).

(8) W. L. Wilson, B. Minushkin and E. J. Chelen, "Friction and Wear Testing in Support of FFTF Components - Interim Summary Report, Westinghouse Electric Corporation Report WARD-NA-3045-13, August, (1974).

(9) H. J. Wagner and A. M. Hall, "Physical Metallurgy of Alloy 718," DMIC Report-217, June (1965).

(10) G. L. Guthrie, "Irradiation of Fuel Duct Load Pad Coatings" in Quarterly Progress Report, Irradiation Effects in Reactor Structural Materials, May-July, 1973, HEDL-TME-73-67, 62, (1973).

Note: References 2, 3, 4, 8 and 10 may be obtained from:

USAEC Technical Information Center
P.O. Box 62, Oak Ridge, Tenn. 37830

TABLE I

THE EFFECT OF AN ALUMINIDE DIFFUSION COATING ON THE CORROSION AND WEAR BEHAVIOR ON INCONEL 718 IN SODIUM

MATERIAL	CORROSION* RATE (mpy)	MAXIMUM WEAR DEPTH (mils)	WEAR COEFFICIENT** K (in^3/psi-in x 10^{13})
Inconel 718	0.24	2.1	12
Aluminide Layer On Inconel 718	0.03	No measurable wear	

*Average rate over time period 0-2128 hr. in 1160°F, 0.5 - 1.0 ppm O_2, 1 fps sodium.

$$**K = \frac{\text{volume of material worn away}}{\text{contact stress x travel distance}}$$; for details of test cycle see Figure 4.

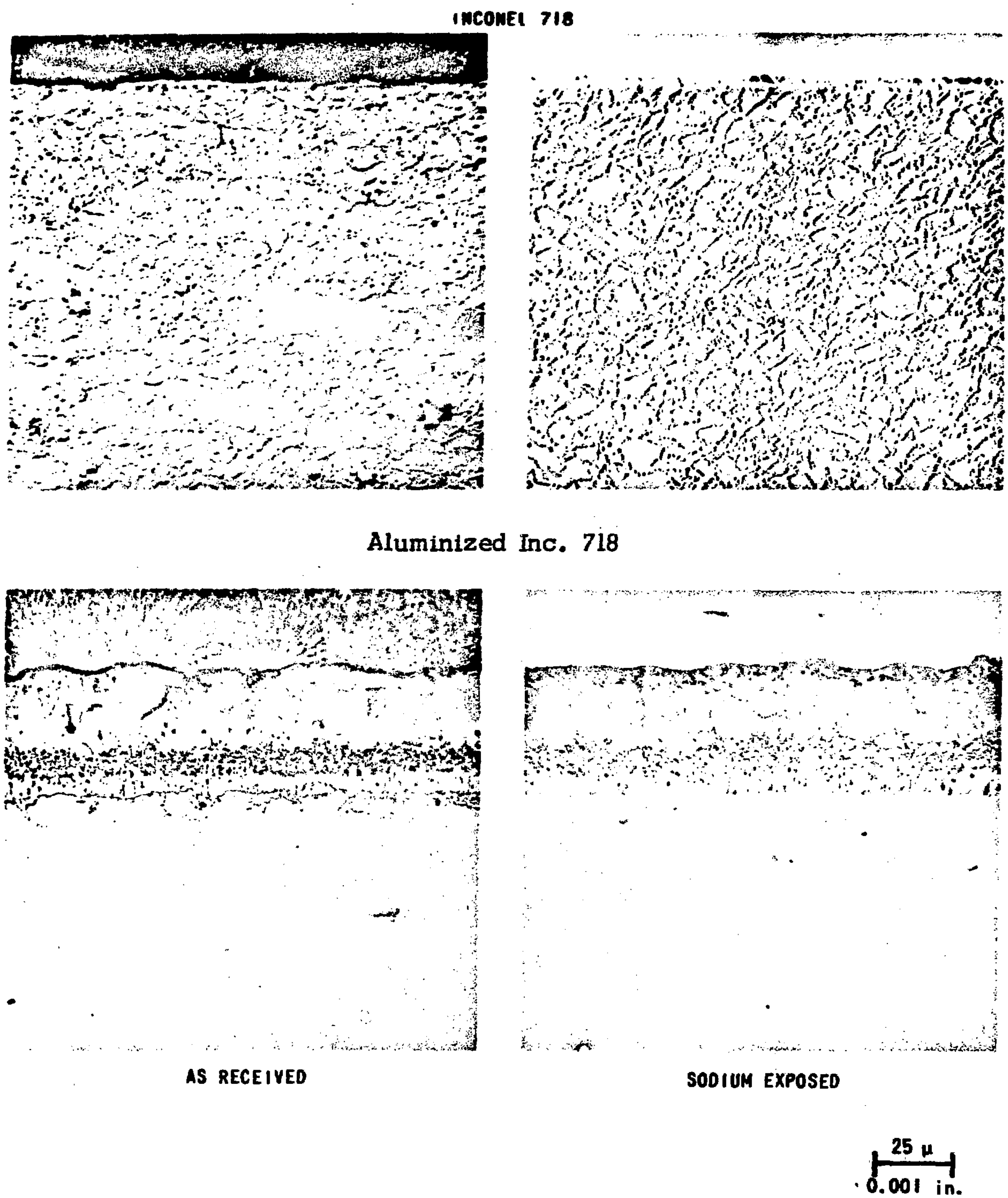

Figure 1. Effects of Sodium Exposure (2128 hrs. @ 1160°F) on the Cross Sectional Microstructure of Inconel 718 and Aluminized Inconel 718

[illegible]-2

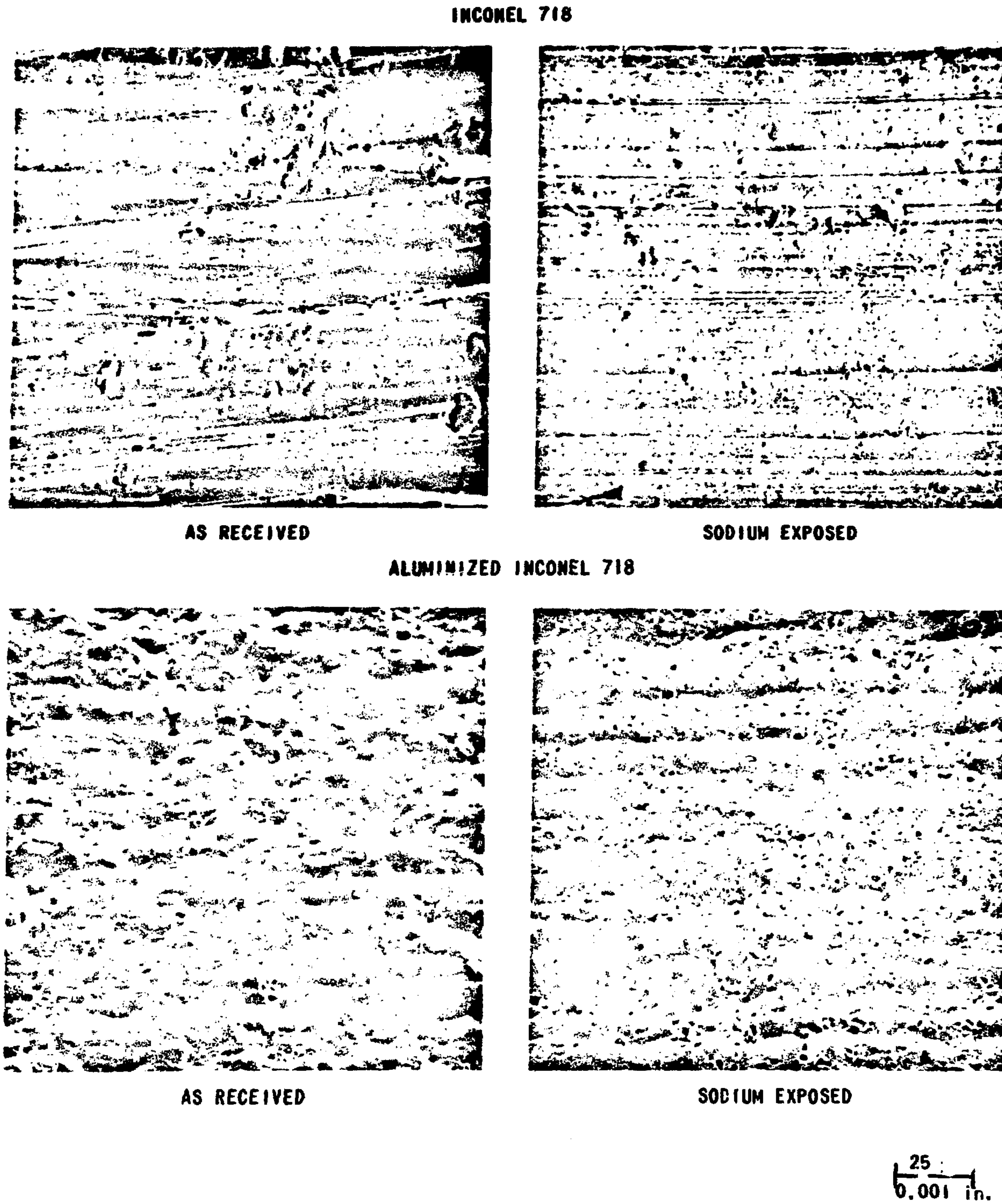

Figure 2. Scanning Electron Micrographs Showing Effects of Sodium Exposure (2128 hrs. @ 1160°F) on the Surface Appearance of Inconel 718 and Aluminized Inconel 718

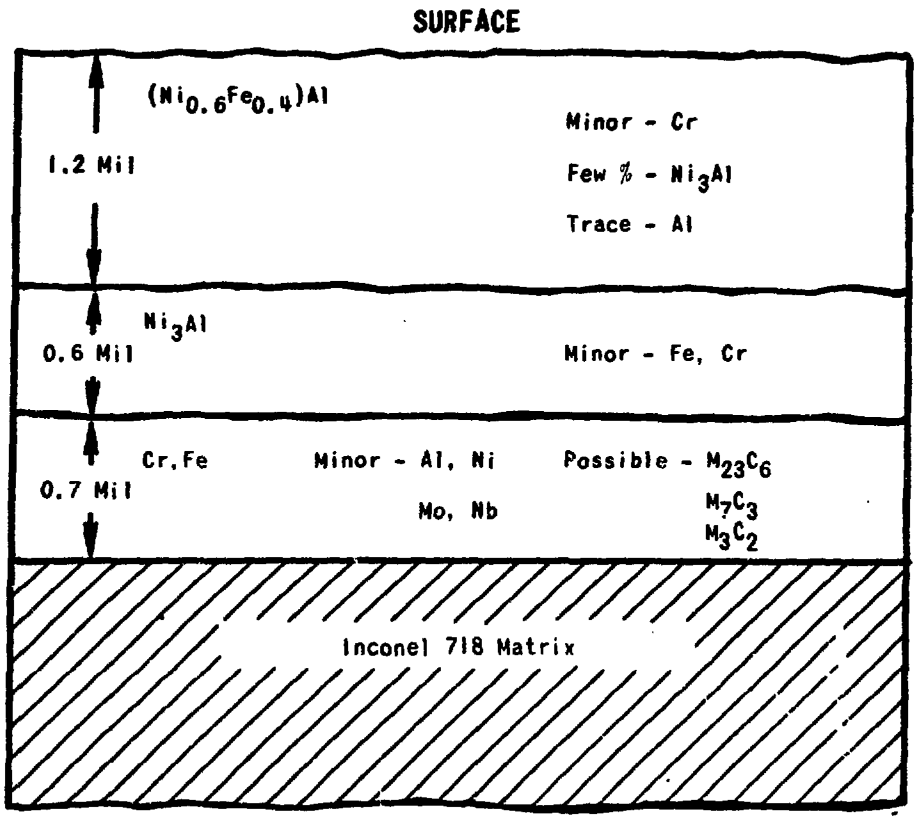

Figure 3. Schematic Depiction of the Composition of the Aluminized Layer on Inconel 718 Obtained by X-Ray Diffraction

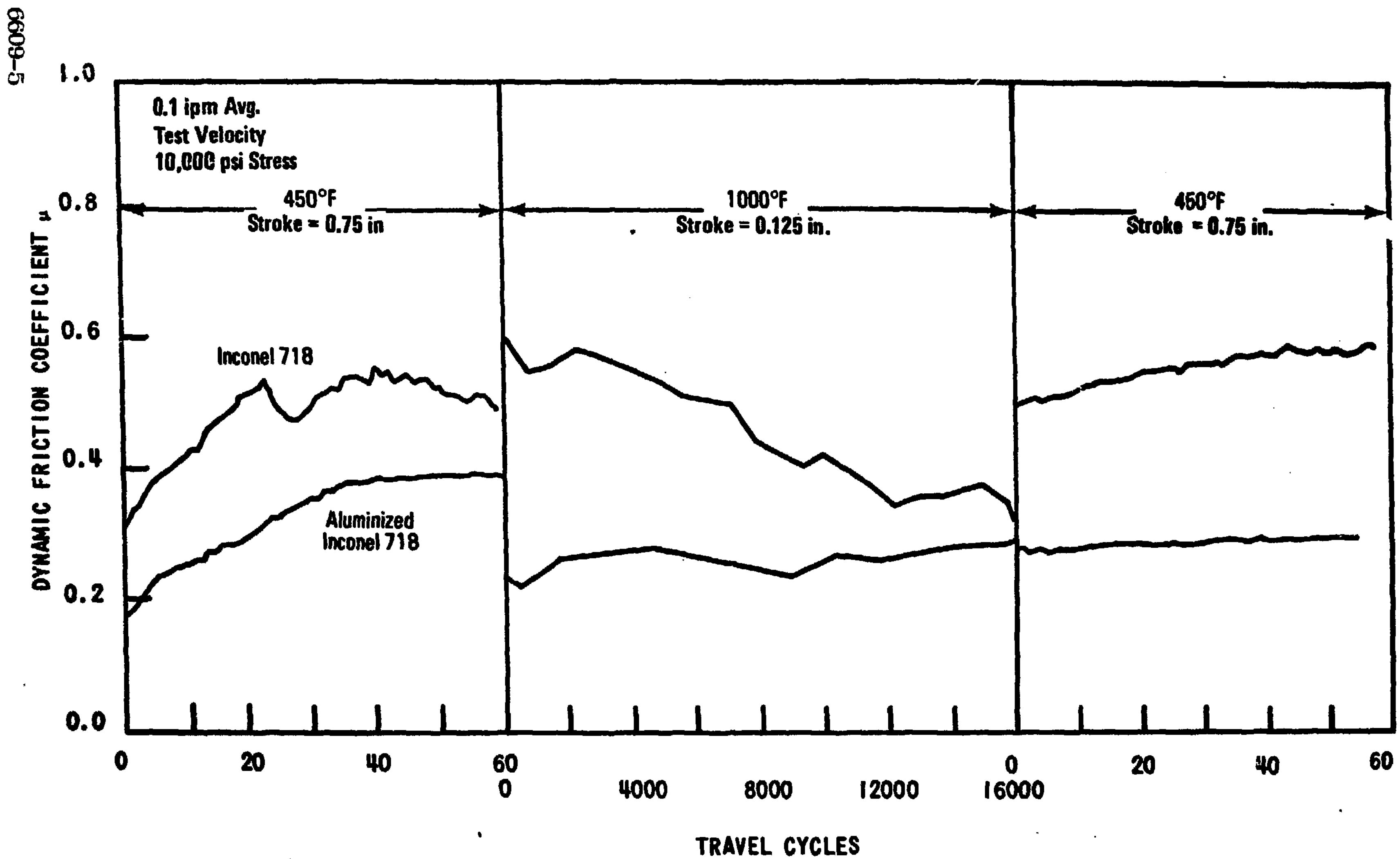

Figure 4. Effect of a Surface Aluminide Layer on the Frictional Behavior of Inconel 718 in Liquid Sodium

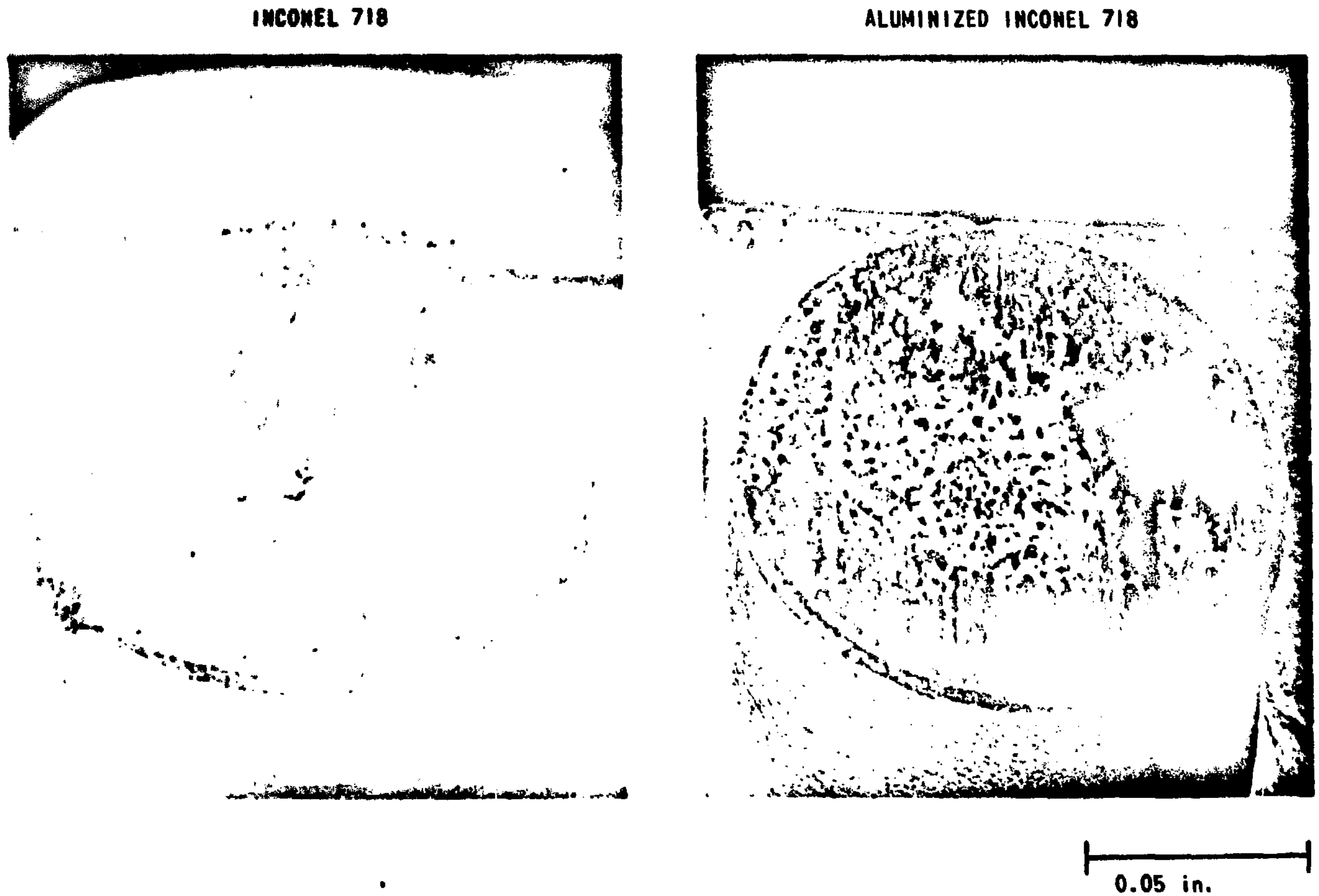

Figure 5. Surface Appearance of Pin Specimens (0.125 in. diameter) of Inconel 718 and Aluminized Inconel 718 After Friction and Wear Test in Sodium