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SPUTTERED PROTECTIVE COATINGS FOR DIE CASTING DIES

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INTRODUCTION

The manufacture of dies for aluminum castings is presently a two-billion dollar-per-year industry. Die casting dies are very expensive to fabricate and have a limited lifetime. For example, the cost of manufacture of even simplistic dies can be $50,000 or greater, and may be used to manufacture only 30,000 castings before replacement or repair is required. It is very desirable to increase the useful lifetime of a die both to reduce the cost per casting and to increase casting productivity.

Die casting dies are subjected repeatedly to large temperature extremes and associated severe mechanical stresses. As a result, the service life of such dies can be relatively limited. Die lifetime is shortened because of heat checking, corrosion of the die material by liquid aluminum, thermal

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stresses, and gross cracking because of the limited fracture toughness of the die material itself. Preliminary information from the Die Casting Research Foundation has indicated that a coating or combination of coatings sputter deposited on the die might be a possible technique to increase die life. If such a coating could increase die life by a factor of two, significant cost savings could be realized. A program was undertaken at NASA-Lewis Research Center and Case-Western Reserve University in cooperation with the Die Casting Research Foundation to improve die lifetime by sputter depositing coatings on a die surface using ion beam technology. The object of the program was to evaluate candidate materials and processes necessary to sputter deposit a coating on a die surface to prevent or retard heat checking and thus extend useful die lifetime. The program consists of three overlapping phases. In Phase I of the program, coatings of various candidate metal and metal oxides were sputter deposited on flat disk surfaces made of H-13 steel (die material) using an ion source for a preliminary evaluation of the adherence of various thickness coatings.[1] The best candidate coatings from these tests were than deposited on the corners of thermal fatigue test specimens, in Phase II for evaluation of their thermal fatigue resistance.[2] The specimens were alternately cycled between molten aluminum and an air/water-lubricant spray for 15 000 cycles. The coatings were than evaluated, in some cases with the aid of a metallograph, and the thermal fatigue resistance was measured. The results of these studies, which look promising enough to evaluate a coating on a die surface in Phase III of the program, are presented herein.

EXPERIMENTAL PROCEDURE

A 30-cm diameter argon ion source[3] with its ion optics masked down to an ion beam diameter of 10-cm was used for the sputter deposition of the metal and metal oxide materials. The ion source, developed from electric propulsion technology, uses argon gas in a hollow cathode in the main discharge chamber as well as in the neutralizer cathode. The vacuum facility is sufficiently large to minimize backsputtered material from contaminating the deposited films. To assure good adherence of the films to the H-13 steel substrates, the substrates were cleaned prior to insertion into the vacuum facility. This cleaning procedure included: cleaning with NA 500 (carbon tetrachloride), cleaning with a 2 percent Liquinox® soap solution, rinsing in distilled H_2O, and drying with nitrogen gas. The specimens were also sputter cleaned by the ion beam for 1/2 hour at an ion beam energy of 1000 eV and a current density of 2 mA/cm^2 prior to deposition.

During Phase I of the program the sputter technique, sputter rate, deposition energy level, and maximum thickness were determined for each candidate coating material. Adherence measurements, of the film to the disk substrate, for film thicknesses varying from 1 to 8 micrometers, were made and are presented in Ref. 1. For most of the films tested, the bond strength of the film to the substrate, exceeded the upper limit measuring capability of the adherence tester (approx. 9000 psi). To further evaluate the adherence of the coatings in Phase I at elevated temperatures, the H-13 steel disks were exposed to oven temperatures of 400° or 700° C for 15 minutes and allowed to cool to room temperature. The best candidate coatings were then sputter deposited on thermal fatigue test specimens[2] in the second phase of the program for further evaluation.

In Phase II of the program a small scale thermal fatigue test[2] was employed which simulated the thermal cycle encountered in the most severe conditions during the aluminum die casting process. This test used an internally water-cooled 5.1-cm square x 17.8-cm long rectangular specimen, made of H-13 steel, that was alternately cycled for 15 000 cycles between a molten aluminum alloy bath and an air/water-lubricant spray. This thermal fatigue specimen (shown coated in Fig. 1) had sharp corners specially designed to initiate thermal cracks in relatively few cycles. The cracks propagate at right angles to the long axis of the corner or in the direction of axial stress, for it is at the corners of the specimen that the temperature fluctuations and geometrical constraints are the greatest. The best candidate coatings from Phase I were deposited on the corners. In the ion beam coating process each of the corners was coated with a different material using some rather unique fixturing[4] to manipulate the speicmen. One corner was left uncoated and served as a reference base for the other coatings during the thermal fatigue test.

After 15 000 cycles, the samples were removed from the thermal fatigue test apparatus. To allow evaluation of the cracks at the edges, each surface was ground (~0.002 cm) to remove the oxide film which was formed during testing. Only the central 7.6 cm of each edge was examined to eliminate possible end effects. Two parameters were used to characterize the degree of thermal fatigue cracking. The longest crack on each corner was noted; the second parameter considers "the total crack area;" it is defined to as "$\sum nd^2$," where n is the number of cracks of length d.

RESULTS AND DISCUSSION

The results of the thermal fatigue tests are shown in Fig. 2, where the ratio of $\sum nd^2 / \sum nd^2_{uc}$ (uc = uncoated corner) is plotted for the various 1 micron thick coatings (unless specified) tested. Some coatings were repeated more than once, and some scatter occurred in the data. The scatter probably occurred because of the inherent variables in the test. Values of $\sum nd^2 / \sum nd^2_{uc}$ less than one indicated less thermal fatigue than that of the uncoated corner. Coatings which resulted in reduced thermal fatigue were Platinum (Pt), Tungsten (W) (1 μm), Silicon Nitride (Si_3N_4), molybdenum (Mo), and a silver (Ag)/copper (Cu) combination. Some coatings such as gold (Au), Cobalt (Co), Chromium (Cr), Nickel (Ni), Silver (Ag), Tantalum Silicate (Ta_5Si_3), Tungsten (W) 1/2 micrometer thick, and composite coatings of 0.17 μm W/1 μm Pt and 0.17 μm Mo/1 μm Pt provided poorer thermal fatigue resistance than the uncoated corner. Three of the coatings, all 1 micrometer thick, W, Pt, and Mo looked promising and were repeated more than once on other thermal fatigue specimens.

None of the coatings tested remained completely intact for the 15 000 cycles. However, coatings such as W, Pt, and Mo do last a significant length of time and reduce the thermal fatigue, at least until the coating is fractured. In effect, the coatings inhibit oxidation[5] and suppress crack initiation as long as they remain intact.

Metallographic results of the 1 micrometer thick platinum and tungsten coated corners, which consistently reduced thermal fatigue are presented here. The control corner (without a deposited coating) was also examined as a comparison with the coated corners. Energy Dispersive Analysis of X-rays (EDAX) was employed, quantitatively or semi-quantitatively to determine changes in the chemical composition near the surface or in the crack area. From the EDAX data, it was determined that the metal surfaces at the cracks are completely covered with oxides. Figure 3 demonstrates the role of oxides in thermal fatigue cracking on an uncoated corner. This specimen has its plane of polish in a diagonal of the thermal fatigue test specimen, parallel to the long axis of the specimen. An oxide layer covers the surface and fills the cracks propagating into the steel. Thermal fatigue cracking is accompanied by oxidation[2] of the die steel. The oxide penetrates into thermal fatigue cracks and appears to increase propagation of the crack into the die steel. Analyses of the stresses during the thermal fatigue cycle have shown that the existing stresses are compressive.[5] The wedging action of the oxide filling the crack has the capability of promoting crack propagation by producing a tensile stress at the crack tip. Figures 4(a) and (b) are photographs taken at different locations of a diagonal view of a platinum-coated specimen. An oxide layer developed underneath the Pt coating in Fig. 4(a), but no cracks as yet have formed in the substrate. In Fig. 4(b) an oxide developed underneath the coating and small cracks began to penetrate into the steel at some fractured points of the coating. Many thin and shallow cracks occurred in the surface of the platinum coated corner, but not many large cracks (as exist on the uncoated corners). These microcracks on the surface can destroy the corrosion resistance of the platinum coating, but they may alleviate the stresses associated with the thermal cycling. These microcracks may have slower crack propagation and may be a reason why the platinum-coated specimen exhibited high thermal fatigue resistance.

The transverse (normal to the long axis) and diagonal views of a tungsten-coated corner from the thermal fatigue specimen are illustrated in Figs. 5(a) and (b). Figure 5(a) shows the W coating intact on the H-13 surface after 15 000 cycles of exposure to molten aluminum and water. No oxide layer is observed under the tungsten coating in Fig. 5(a). The crack shown in Fig. 5(b) initiated and propagated from a fractured location of the coating, but no oxide developed underneath the coating near the crack (i.e., the W coating is intact at the surface). The crack in Fig. 5(b) is filled with oxide. Fewer cracks occurred on the W coated surface than on the uncoated surface, but they were relatively large.

From these preliminary studies it is not clear whether Pt or W is the preferred coating to reduce thermal fatigue on a die surface. Both coatings reduce thermal fatigue on thermal fatigue test specimens, but in a somewhat different way. The W coatings protect the H-13 from surface oxidation better than any other coating. Once the W coating has been breeched however, the oxidation at the crack proceeds, and the strain and stress may be concentrated at fewer stress locations, which enhance crack propagation. Compared to the Pt coating the W coated corner accentuates the stress concentration effect because of the reduced number of cracks. However, the high corrosion resistance and superior physical properties (i.e., high yield strength and ductility), probably allow W to last for a longer period of time before cracks are initiated.

Because of the nature of the tests it was impossible to observe crack initiation or propagation as a function of the thermal cycling (due to the build up of oxide on the specimen, it is not possible to observe the cracks without interupting the tests). Before a decision can be made on using either Pt or W as a protective coating on a die surface, further investigation is needed.

In the third phase of the program, one cavity of dual cavity die is to be coated with the coating and techniques found to best protect the thermal fatigue test specimen. The die selected, typically has a major failure or degradation in 1 year or less. The die produces at least 100 000 aluminum castings per year. At periodic intervals during the casting production (approx. 20 000 castings) both the coated and uncoated cavity will be inspected for thermal fatigue and the relative protection of the coating evaluated.

CONCLUDING REMARKS

The results of this investigation indicate that some ion beam sputter deposited coatings reduce thermal fatigue on the corner of H-13 steel thermal fatigue test specimens. One micrometer thick W and Pt coatings reduced the thermal fatigue more than any other coating tested and are candidates to be used on a die surface to increase die life. The mechanism for alleviating stress may have been different, for W and Pt coatings. There were many thin and shallow cracks on the platinum coated corner. These microcracks on the surface may have alleviated the stresses associated with the thermal cycling.

The tungsten coated corner remained intact on the surface after thermal fatigue testing. No oxide formed under the coating, although few large cracks formed on the W coated surface, where the coating broke down. The good corrosion protection afforded by the W coating at other locations reduced thermal fatigue.

Further thermal fatigue testing is planned before a die surface coating is closen for production evaluation.

REFERENCES

1. M. Mirtich, NASA TM-81585, Oct. 1980.
2. J. C. Benedyk, D. J. Moracz, and J. F. Wallace, 6th Society of Die Casting Engineers International Die Casting Congress, Paper No. 111, Nov. 1970.
3. J. S. Sovey, AIAA Paper 76-1017, Nov. 1976.
4. M. Mirtich, AIAA Paper 81-0672, April 1981.
5. Personal Communication, J. Wallace, 1980.

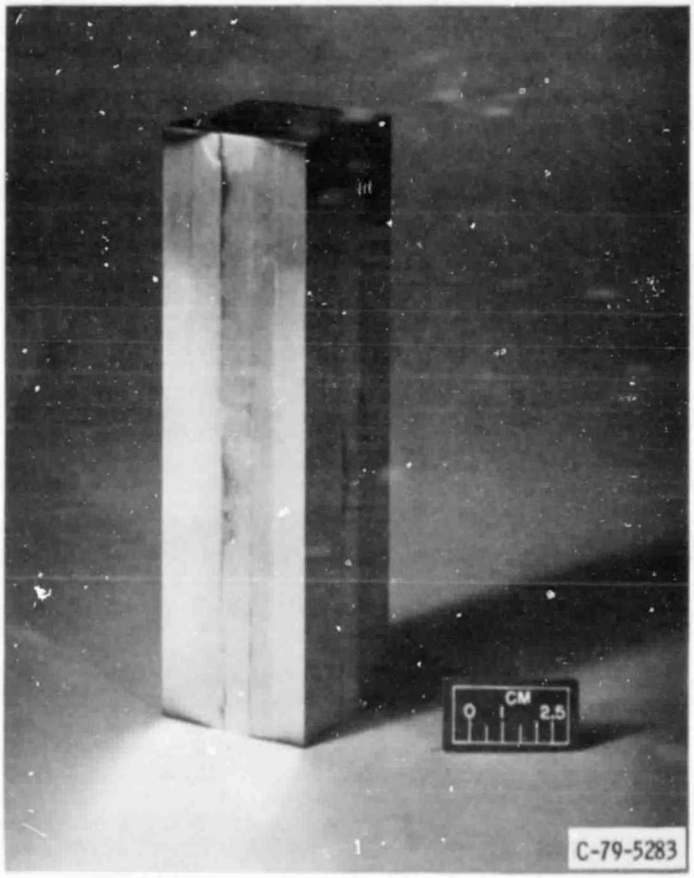


Figure 1. - A thermal fatigue specimen coated with (from left to right) cobalt, gold, and tungsten.

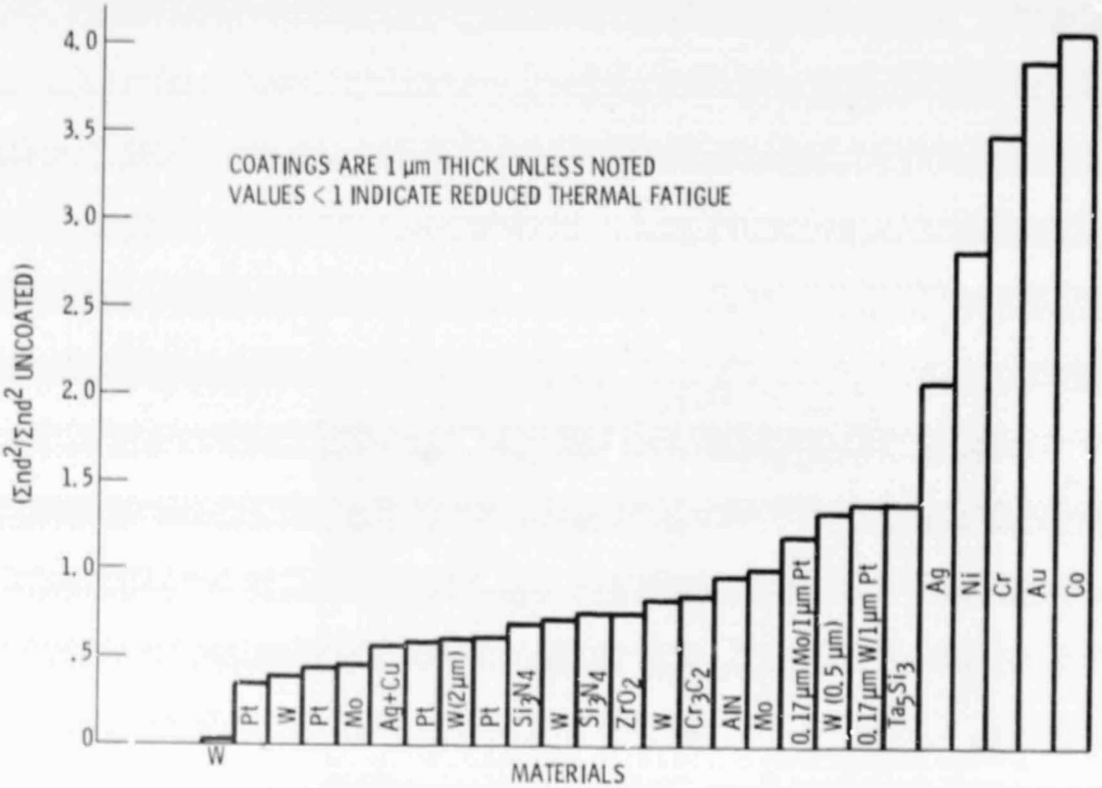


Figure 2. - Plot of the ratio of "cracked area" of a coated corner of a thermal fatigue specimen normalized to an uncoated corner versus various materials.

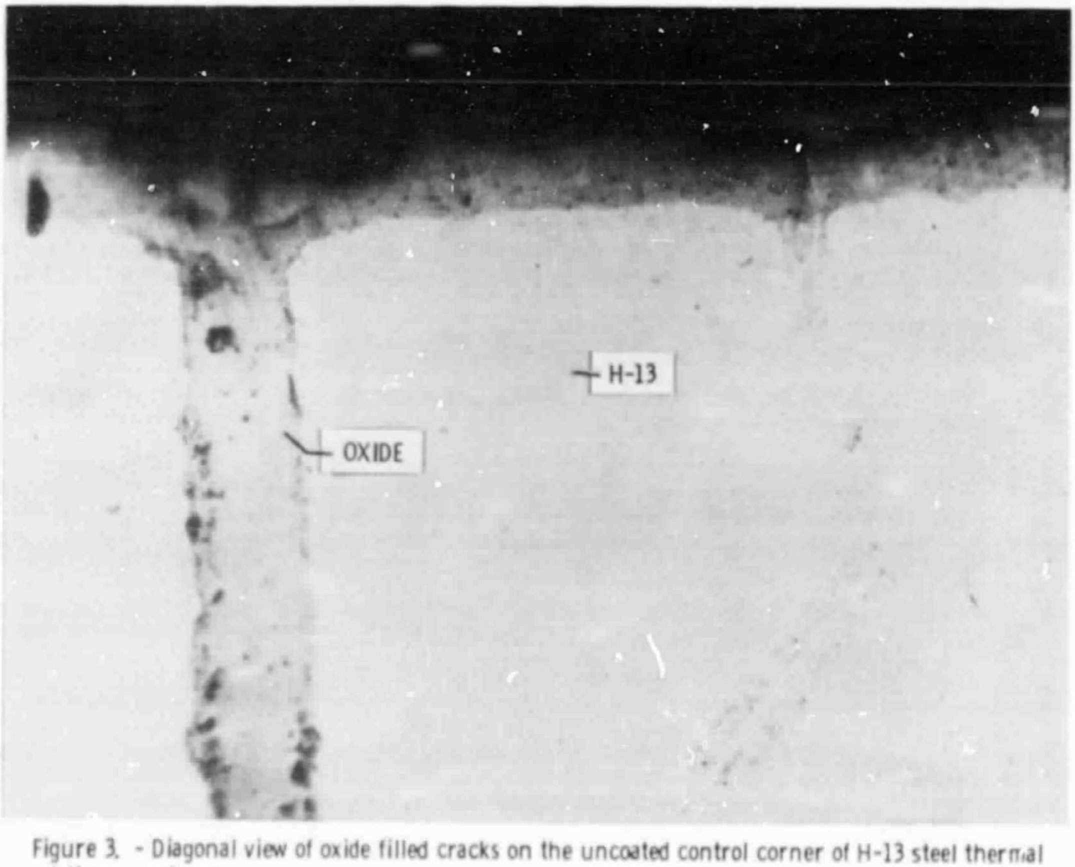
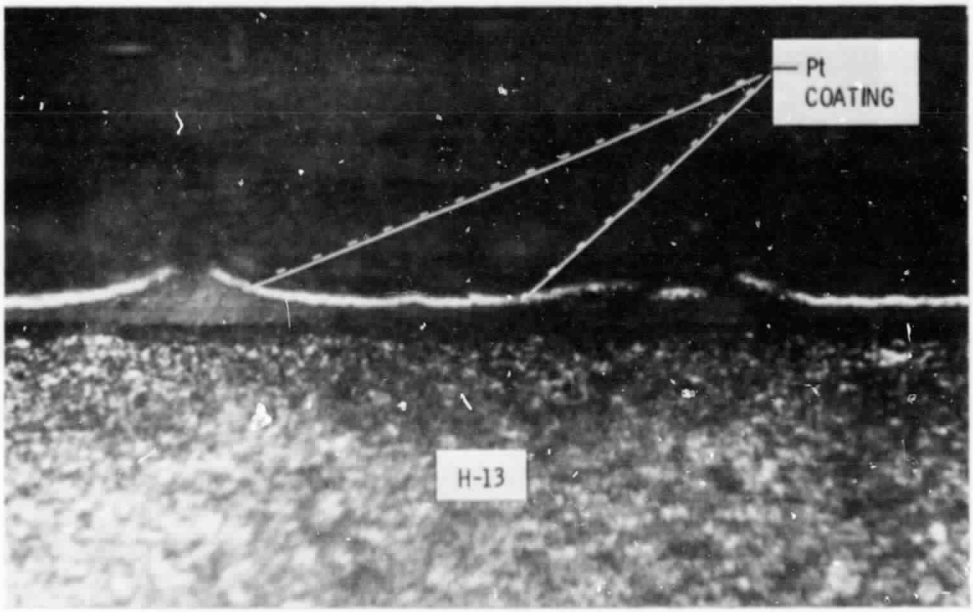
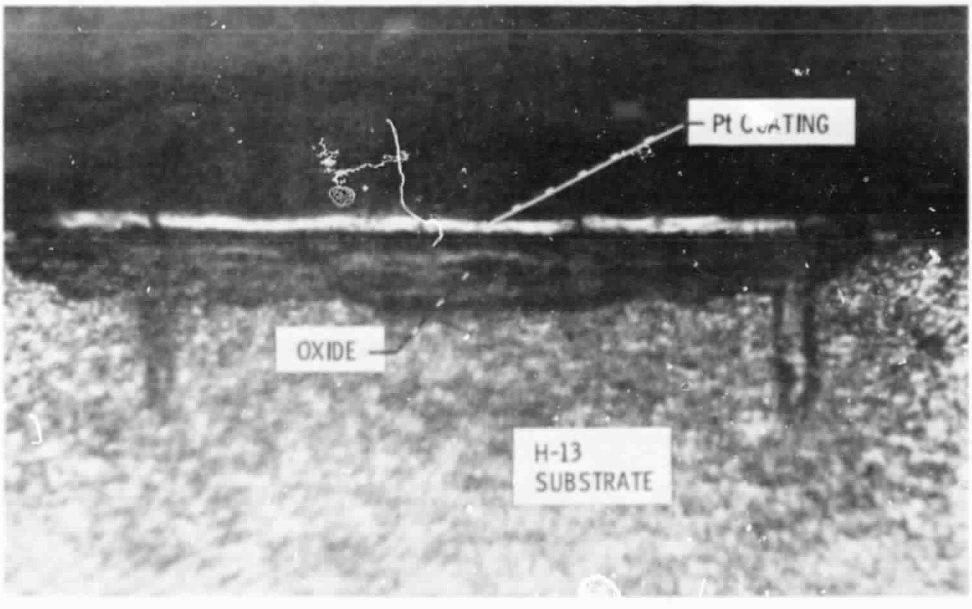


Figure 3. - Diagonal view of oxide filled cracks on the uncoated control corner of H-13 steel thermal fatigue specimen.

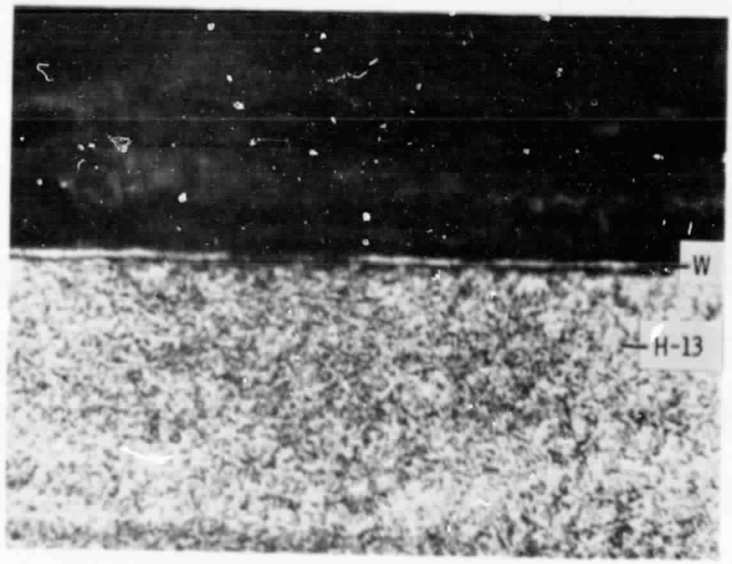


(a) DIAGONAL VIEW OF PLATINUM COATED CORNER. OXIDE DEVELOPED UNDER THE COATING BUT NO CRACKS IN THE H-13.

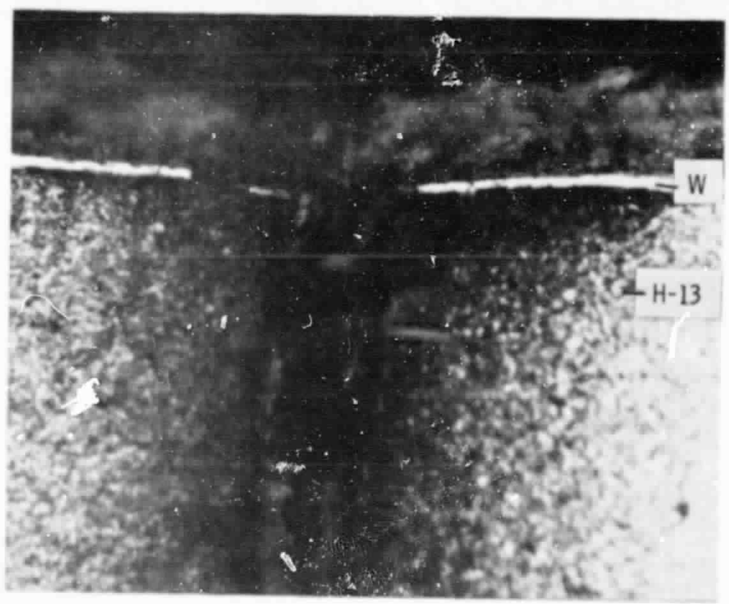


(b) THE DIAGONAL VIEW OF PLATINUM COATED SPECIMEN. OXIDE DEVELOPED UNDERNEATH THE COATING AND SMALL CRACKS BEGIN TO CUT INTO THE MATRIX AT SOME FRACTURED POINTS OF THE COATING. (MAG 500X.)

Figure 4.



(a) THE TRANSVERSE VIEW OF TUNGSTEN COATED SPECIMEN.



(b) THE DIAGONAL VIEW OF TUNGSTEN COATED SPECIMEN. IT IS NOTED THAT NO OXIDE ARE DEVELOPED UNDERNEATH THE COATING. (MAG 500X.)

Figure 5.