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COMPATIBILITY OF SNAP FUEL AND CLAD MATERIALS

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COMPATIBILITY OF SNAP FUEL AND CLAD MATERIALS

(Title Unclassified)

By
J. K. BALKWILL

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ABSTRACT

Sample capsules containing buttons of materials of interest for SNAP fuel elements were held at temperatures up to 1600°F, in hydrogen, under a 20-tsi load for 200+ hours.

The hydrided Zr-U alloys formed significant diffusion zones at 1200°F with Hastelloy-B, Inconel-X, Type 347 stainless steel, gold, and copper; but did not alloy with molybdenum, beryllium, or nickel at this temperature. At 1600°F, diffusion was also found between the fuel and molybdenum and nickel. The fuel did not react with the Type S-1435A Solaramic cladding coating at test temperatures of 1200 and 1500°F. At 1300°F, beryllium formed a diffusion zone with both Hastelloy-B and Inconel-X, and also reacted with the Solaramic, while BeO did not react with any of these.

I. INTRODUCTION

This compatibility program was established to investigate the behavior of materials of present and future interest as SNAP fuel, reflector, and cladding, when held in intimate contact at reactor operating temperatures. The experiments were designed to give qualitative answers only, with more refined studies of diffusion and reaction rates to be directed by these results. The main interest was in the barrier materials designed to contain the hydrogen, because a break in this barrier caused by alloying or reaction would cause serious loss of hydrogen from the element.

II. EXPERIMENTAL METHOD AND APPARATUS

The final furnace design is shown in Figure 1 in schematic. This includes several modifications from the original thinking on the apparatus, the most important of these being the necessity of limiting test temperatures to 1600°F, since at higher temperatures the pressure rods bent.

The sample sheath and end-caps were made of Inconel-X. The 1-in.-long sheath holds an adequate number of sample buttons if they are thin. (Only the first run was made with a 2-in.-long sheath.) The buttons were of the proper diameter to expand to a press-fit in the sheath, when the buttons reached test temperature. Assembly of the buttons in the sheath supported the top end-cap above the sheath, so that the pressure was transferred through the sample buttons and not through the sheath. Test buttons of the various hydrides were machined from specimens in the delta-hydride range (1.4 to 1.6 wt % H_2). Test samples of the hydrogen barrier material were prepared by coating buttons of Type 347 stainless steel with the standard SER application of two coats of Type S-1435A Solaramic to a total thickness of 2 to 3 mil.

The sample capsule was tested in hydrogen to simulate element-atmosphere in the reactor. To contain the hydrogen, the original design used a pressure-sleeve machined from Inconel-X bar stock with only assembly clearance for the pressure rods and sample sheath. During the first run, one pressure rod bent slightly, enough to jam the packing-nut and prevent its removal. The sleeve was cut open to recover the sample, and the design changed to increase the clearance. The sample container was changed to a length of schedule 80 stainless steel pipe with Conax seals, sized to allow significant clearance for the pressure rods. A nipple welded to the pipe leads to vacuum and hydrogen lines.

The pressure rods were Inconel-X or Hastelloy-X solution heat-treated for maximum strength. The original design, 3/8 in. in diameter and 8 in. long, survived tests at 1200 and 1500°F, but failed in bending in an 1800°F test. The diameter was increased to 3/4 in. with an accompanying increase in pipe diameter, but two of these assemblies failed in 1800°F tests when the pressure rods bent. A third assembly completed a 1600°F run successfully, and this is taken as the upper limit of the apparatus.

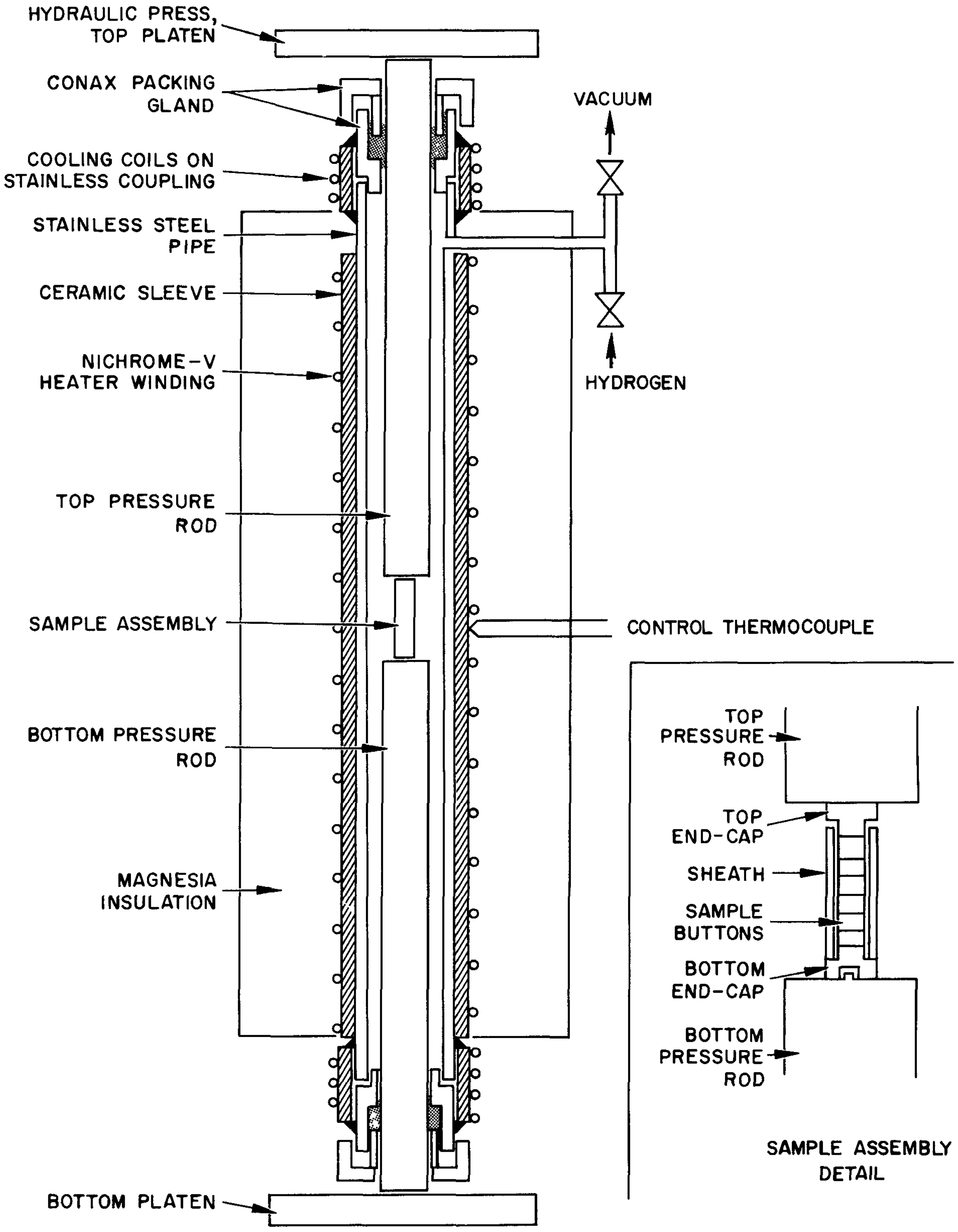


Figure 1. Compatibility Test Apparatus

The lower pressure rod had a locating pin machined on the end, to mate with the locating hole in the bottom end-cap, to facilitate assembly, and to ensure vertical alignment of the sample. A larger diameter sample, say 1/2 in., would give a sturdier sample capsule; the 1/4-in. diameter used in these experiments was fixed by the available sample material.

Pressure for the final run was applied by a 10,000-lb capacity hydraulic press with pressure automatically held constant by a pressure switch and pump. For the first runs, a standard hand-pumped hydraulic press was used, but the pumping unit soon developed a leak past the release valve, allowing the pressure to fall slowly to zero. It was then necessary to repressurize the capsule several times a day; and several runs were made in this manner, without constant pressure through the length of the run. A pressure of 40,000 psi nominal was used to ensure the intimate contact between buttons that would let temperature-induced diffusion proceed rapidly. High pressure also promoted mechanical button-bonding with the sheath, to give an improved mounted specimen; loose buttons could not be easily mounted, and any interface reaction was then lost in mounting and polishing.

The furnace was Nichrome-V wire wound over a ceramic sleeve, in a fashion to give a level hot-zone 2 in. long at its midpoint. The minimum furnace length to give this profile was found to be 10 in. If a commercially built furnace were available with a shorter heated length, it would be possible to reduce the length of the pressure rods accordingly. Power was supplied through a Powerstat and on-off controller with the control thermocouple placed at the midpoint of the windings.

After assembling a new furnace, the temperature profile was determined, and the control temperature required to produce a given temperature in the hot zone was established. The length and exact location of the hot zone were fixed. After cooling, the top pressure rod was lowered to the proper position and the packing (graphitized asbestos) rammed into place. The end-caps and sample buttons were cleaned and stacked in the desired order in the sheath, which was then centered on the bottom pressure rod and raised carefully into the furnace till the top end-cap touched the top pressure rod. The lower Conax seal could then be packed and tightened. The complete assembly was then supported between platens of the press, evacuated, and leak-checked. When leak-tight, a hydrogen atmosphere was established and an automatic regulator was set to feed hydrogen to maintain the desired pressure. Power was then turned on, with the controller and Powerstat set to give the test temperature. When this temperature was reached, the desired total force was applied by the hydraulic press. With the manually operated press, this load was reapplied as often as necessary during the day shift.

On completion of the time-at-temperature required, the furnace was cooled, the sample removed and sectioned longitudinally, mounted, and polished for metallographic examination. No attempt was made to show grain structure at the interfaces; the multitude of dissimilar metals present in each sample made uniform polishing and etching impossible. Some of the brittle intermetallics that formed at interfaces, and much of the Solaramic, tended to polish out of the sample.

III. RESULTS

Eight runs were completed in this qualitative series. A summary of operating conditions is given in Table I following.

Table II lists the materials assembled in each capsule. Each of these buttons was also in contact with the Inconel-X sheath.

A description of the interfaces of each capsule follows; photomicrographs of the more significant interfaces are included.

Run Number 1 – 216 hr at 1200°F

1. Solaramic (Type S-1435A) on Type 347 stainless steel vs hydrided zirconium – 10 wt % uranium (Figure 2): no diffusion zone or reaction is found in either stainless or hydride button. The Solaramic layer extruded somewhat from the interface to the sides of the button; this is typical of all Solaramic samples. An excellent mechanical bond exists between the Solaramic and both stainless and hydride buttons; any separation crack in cooling came in the Solaramic layer. Most of the Solaramic was lost in the polishing.

2. Solaramic vs hydrided zirconium – 7 wt % uranium and Solaramic vs zirconium hydride: Same as 1. above.

3. Type 347 stainless steel, surface oxidized, vs hydrided zirconium – 7 wt % uranium (Figure 3): a diffusion zone formed on both sides of the original interface, leaving the oxide layer unchanged. This layer, prepared by heating the button in air, is porous and only 2 μ thick.

4. Hastelloy-B, surface-oxidized, vs hydrided zirconium – 10 wt % uranium (Figure 4): a diffusion layer formed. This oxide layer was discontinuous and did not offer a good barrier.

5. Inconel-X vs hydrided zirconium – 10 wt % uranium (Figure 5): a diffusion zone formed, bonded strongly to both buttons; the hydride button cracked parallel to the bond on cooling. The same diffusion was found with the 7 wt % uranium alloy and with zirconium hydride.

TABLE I

SUMMARY OF OPERATING CONDITIONS FOR COMPATIBILITY TESTS

Run	Sample Temperature (°F)	Sample Pressure Load (psi)	Time at Temperature (hr)	Metal. Lab. Specimen Number
1	1200	40,000 (constant)	216	964-1 964-2
2	1200	40,000 (constant)	210	---
3	1500	40,000 (intermittent)	190	---
4	1600	40,000 (intermittent)	65	1052
5	1600	40,000 (intermittent)	360	1065-1
6	1300	40,000 (intermittent)	235	1091
7	1300	40,000 (intermittent)	494	1142
8	1300	40,000 (constant)	334	1227

TABLE II

MATERIALS ASSEMBLED IN TEST CAPSULES

Run	Button Assembly
1	ZrH_x/Solaramic on Stainless/Oxidized Stainless/U-ZrH_x/Solaramic on Stainless/U-ZrH_x/Oxidized Hastelloy B/U-ZrH_x/Solaramic on Stainless/ZrH_x
2	Molybdenum/ZrH_x/Copper/ZrH_x/Gold/ZrH_x/Nickel
3	Nickel/Solaramic on Stainless/U-ZrH_x/Molybdenum/U-ZrH_x/Hastelloy B
4	Not completed
5	Nickel/U-ZrH_x/Molybdenum/U-ZrH_x/Nickel/U-ZrH_x/Molybdenum
6	U-ZrH_x/Beryllium/Solaramic on Stainless/Beryllium/Hastelloy B
7	Not completed
8	Beryllium/Hastelloy B/Beryllium Oxide/Solaramic on Stainless/Beryllium Oxide/Type 347 Stainless Steel

Run Number 2 – 210 hr at 1200°F

1. Molybdenum vs hydrided zirconium – 10 wt % uranium: no diffusion or reaction took place.

2. Gold vs hydrided zirconium – 10 wt % uranium (Figure 6): an extremely wide diffusion zone formed – 115 μ wide. The zone cracked somewhat across its width on cooling, but remained bonded to both buttons. A similar, slightly thicker diffusion zone, 130 μ, was found at a second gold-zirconium hydride interface.

3. Copper vs hydrided zirconium (Figure 7): a diffusion zone 35 μ wide was formed. It cracked badly across its width on cooling.

4. Nickel vs zirconium hydride: no diffusion or reaction took place.

Run Number 3 – 190 hr at 1500°F

1. Molybdenum vs hydrided zirconium – 7 wt % uranium: no diffusion or reaction was found.

2. Hastelloy-B vs hydrided zirconium – 7 wt % uranium (Figure 8): a diffusion zone 45 μ wide formed, then split along its length on cooling. The zone may have been composed of two layers, parting at their interface.

3. Solaramic vs hydrided zirconium – 10 wt % uranium (Figure 9): no diffusion or reaction was found.

4. Solaramic vs nickel: no diffusion or reaction was found.

5. Inconel-X vs hydrided zirconium – 10 wt % uranium (Figure 10): a 3-layer diffusion zone 70 μ wide formed.

Run Number 4 – 65 hr at 1600°F

This run was stopped when a leak developed in the system early in the run. The sample was mounted in the hope of detecting incipient bonding, but the hydride buttons were found to be badly oxidized. No diffusion occurred. Figure 11 shows a typical 2-layer reaction zone on a hydride button.

Run Number 5 – 360 hr at 1600°F

1. Nickel vs hydrided zirconium – 7 wt % uranium (Figure 12): an extremely wide diffusion zone formed on the hydride side of the original interface. Average width of the zone was 225 μ.

2. Inconel-X vs hydrided zirconium – 7 wt % uranium (Figure 13): A diffusion zone 100 μ wide formed on the hydride side of the original interface. The break in cooling came in the brittle hydride.

3. Molybdenum vs hydrided zirconium – 7 wt % uranium (Figure 14): a thin diffusion zone 20 μ wide, not consistently formed, appears on half of the interface.

Run Number 6 – 235 hr at 1300°F

1. Beryllium vs Solaramic (Figure 15): a thin reaction zone 10 μ wide formed between the Solaramic layer and the beryllium button, and remained firmly bonded to the Solaramic when the stainless pulled cleanly away on cooling.

2. Beryllium vs Hastelloy-B (Figure 16): an extremely wide diffusion zone formed, averaging 230 μ in width. The zone cracked along its length on cooling.

3. Beryllium vs Inconel-X (Figure 17): an extremely wide diffusion zone formed here, 280 μ wide. The zone cracked badly internally.

4. Beryllium vs hydrided zirconium – 7 wt % uranium (Figure 18): no reaction or diffusion took place.

5. Inconel-X vs hydrided zirconium – 7 wt % uranium (Figure 19): a diffusion zone 12 μ wide was formed.

Run Number 7 – 494 hr at 1300°F

This run was an attempt to confirm the results of the previous run and establish whether the Solaramic-beryllium reaction proceeds linearly with time.

However, in assembling the test the top pressure rod was not made concentric with the sample sheath, and the load was carried on one side of the sheath. In Run Number 5, when the same thing happened, the test temperature was so high that the sheath crushed, keeping the buttons in contact. In this run at the lower temperature the sheath bent, springing the buttons apart. A diffusion zone was found only at one interface, beryllium *vs* Hastelloy-B. Lack of reaction at other interfaces was attributed to the separation of the buttons.

The top pressure rod and end-cap were modified after this run to prevent eccentric loading.

Run Number 8 – 334 hr at 1300°F

This was the first run made with the applied pressure held constant automatically; and the effects of this constant pressure, and improved contact, are seen in the beryllium reactions.

1. Beryllium *vs* Hastelloy-B (Figure 20): a very wide diffusion zone, 24 mils thick at its widest, was formed. The zone remained firmly bonded to the Hastelloy and pulled away from the beryllium. About four-fifths of the zone was on the Hastelloy side of the original interface.

2. Beryllium *vs* Inconel-X (Figure 21): another very wide diffusion zone, 16 mils thick at its widest, was found; it was brittle, and broke up severely.

3. Beryllium oxide *vs* Solaramic (Figure 22): no diffusion or reaction took place. The BeO buttons cracked under the load, and Solaramic extruded into these cracks (Figure 23); but no reaction was found even with this intimate contact.

4. Beryllium oxide *vs* Hastelloy-B (Figure 24), Inconel-X, and Type 347 stainless: there was no diffusion or reaction.

IV. CONCLUSIONS

From the test runs made, we have drawn the five following conclusions.

The fuel alloy will form a diffusion zone with any of the proposed SNAP 2 cladding materials at SER operating temperatures, unless separated from the cladding by a layer of Solaramic or similar nonreactive ceramic material. The use of such a layer for the primary purpose of hydrogen retention in the present reactor design solves this fuel-cladding alloying problem.

The beryllium reflector plugs used in the present SNAP 2 fuel-element designs will react with the Solaramic hydrogen barrier if the two are in contact. With the excellent contact provided in these capsule tests, the reaction destroyed 0.4 mil of the coating in 235 hr. If the reaction continues at this rate, the standard 2.5 mil Solaramic coating used on the SER would be destroyed in 1500 hr.

Beryllium oxide, because it does not react with Solaramic, could be used as a reflector in place of beryllium metal to solve the Solaramic destruction problem, although use of the oxide might include a weight and reactivity penalty.

Although beryllium oxide does not react with Solaramic, the reaction shown in Figure 15 between beryllium and Solaramic did not stop as soon as the metal and ceramic were separated by a film of reaction product, that is undoubtedly BeO. Thus, we conclude that there is a transport mechanism operating to carry beryllium through the porous reaction film to the Solaramic; or to carry oxygen from the Solaramic to the beryllium. The second mechanism is believed less likely, because Solaramic decomposition in the absence of beryllium was not noted in Run Number 6, or in other runs where zirconium could have served to getter the oxygen. The first mechanism is postulated as the formation of beryllium hydride, which could have a real vapor pressure at this temperature, followed by transport of the hydride vapor through the reaction film to the Solaramic where it reduces the ceramic oxides.

If this conclusion is correct, plain beryllium used in the fuel element would reduce Solaramic even if the reflector were sized to leave an appreciable gap between it and the Solaramic.

Beryllium will form a diffusion zone with any of the proposed SNAP 2 cladding materials at SER operating temperatures. Beryllium oxide will not. If the beryllium-Solaramic reaction does continue to the complete destruction of the Solaramic as postulated above, a similar beryllium-cladding reaction may then begin; but we do not now have evidence to confirm or deny this supposition.

V. RECOMMENDATIONS

On the basis of the evidence presented, the five following recommendations are hereby made.

Continue beryllium-Solaramic compatibility tests at constant temperature to determine how this reaction proceeds with time.

Repeat the original beryllium-Solaramic test at varying temperatures.

React Solaramic with beryllium powder in hydrogen, and analyze the reaction product to determine if the beryllium is reducing all or only a certain few of the various oxides that make up the Solaramic formula.

Conduct stability studies of Solaramic in hydrogen at varying temperatures.

Continue fuel-metal-beryllium compatibility studies, with a view to finding a foil to separate the Solaramic and beryllium.

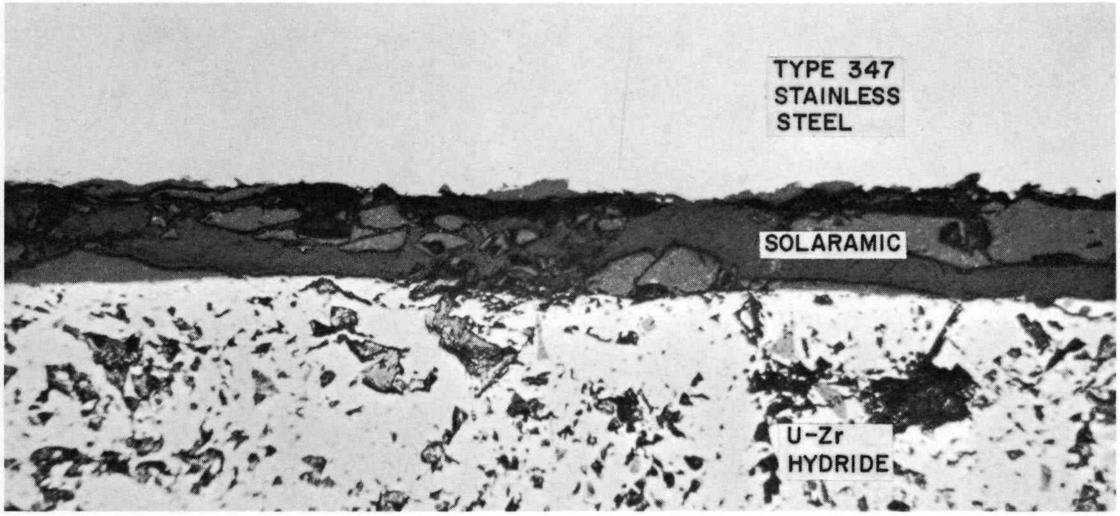


Figure 2. Solaramic vs U-Zr Hydride — 216 Hr at 1200°F

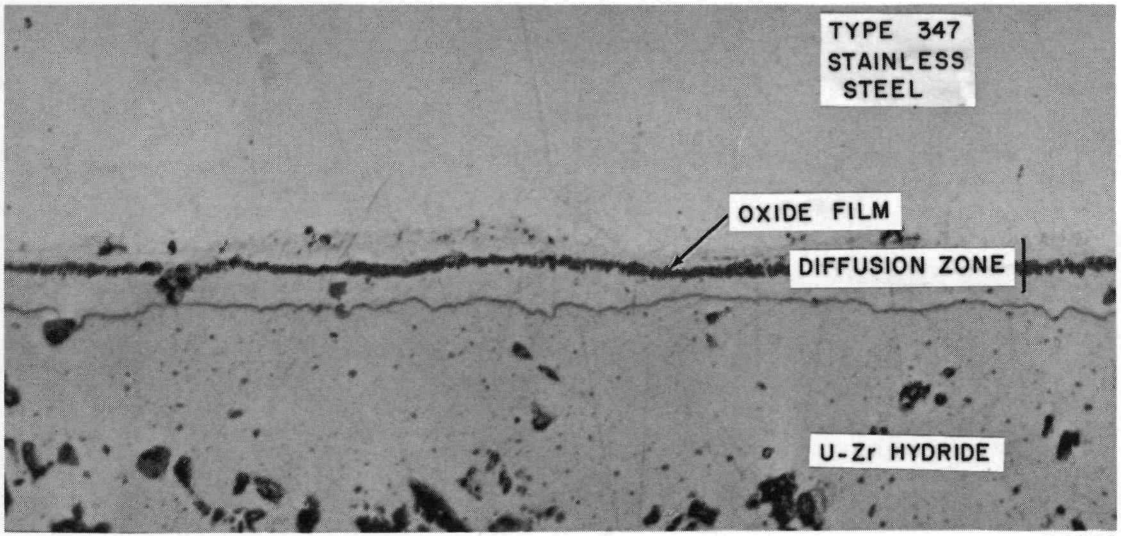


Figure 3. Oxidized Stainless Steel vs U-Zr Hydride — 216 Hr at 1200°F

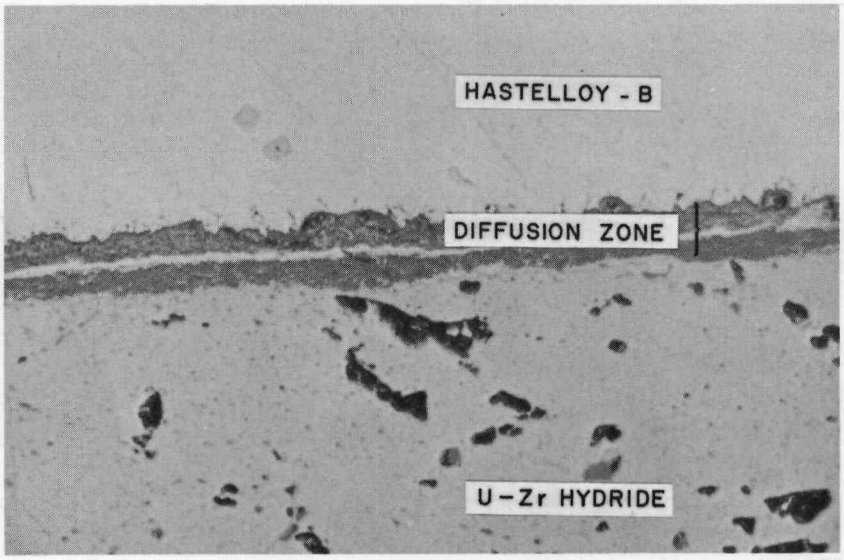


Figure 4. Oxidized Hastelloy-B vs U-Zr Hydride — 216 Hr at 1200°F

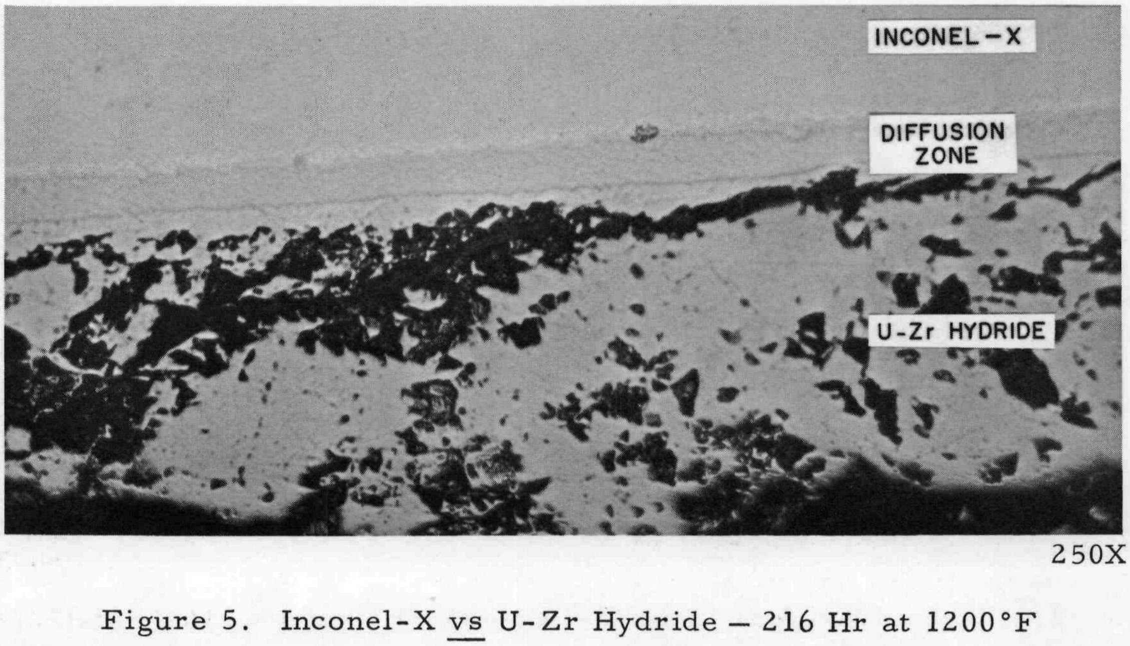
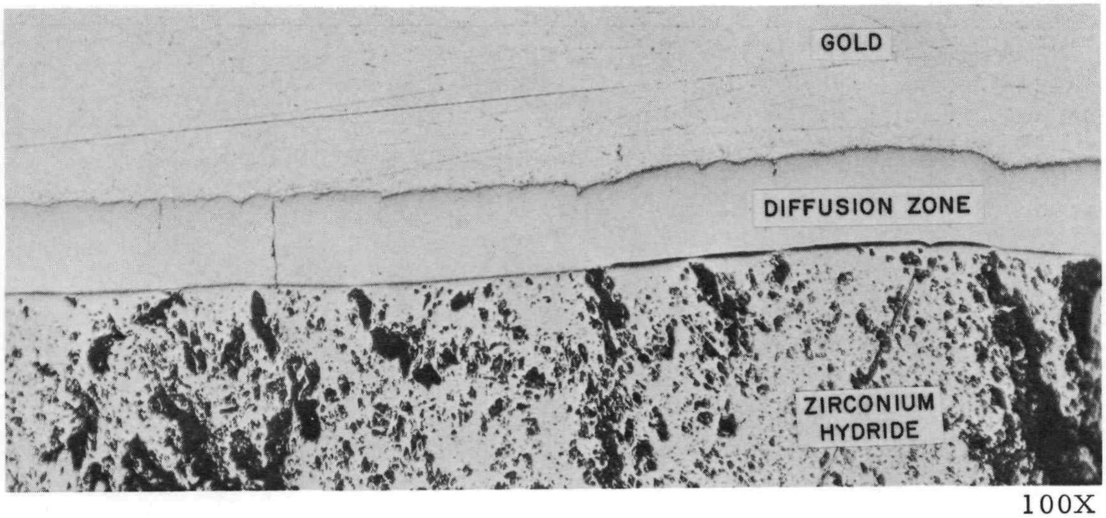


Figure 5. Inconel-X vs U-Zr Hydride — 216 Hr at 1200°F



100X

Figure 6. Gold vs Zirconium Hydride — 210 Hr at 1200°F

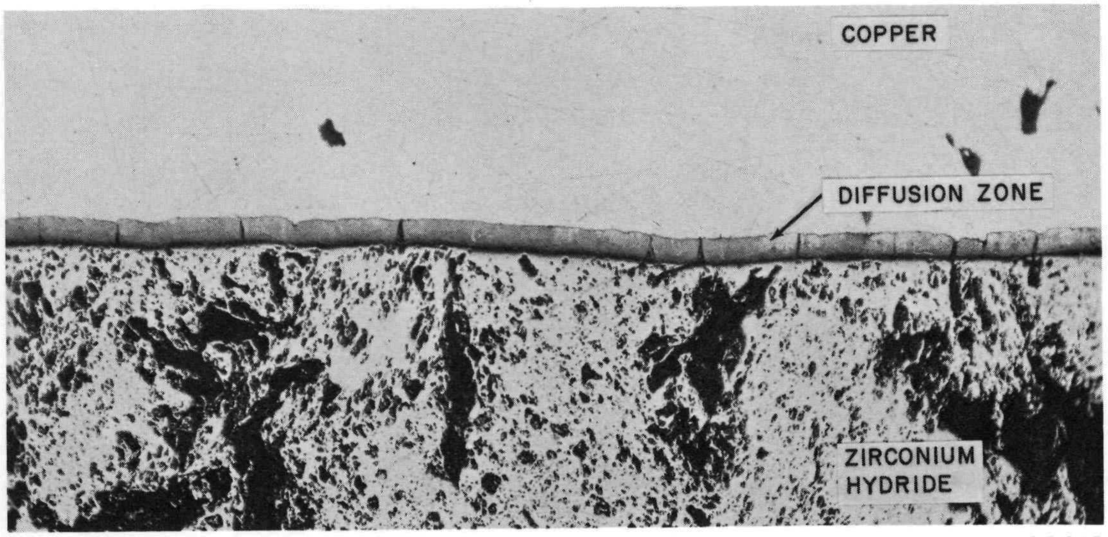


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Figure 7. Copper vs Zirconium Hydride — 210 Hr at 1200°F

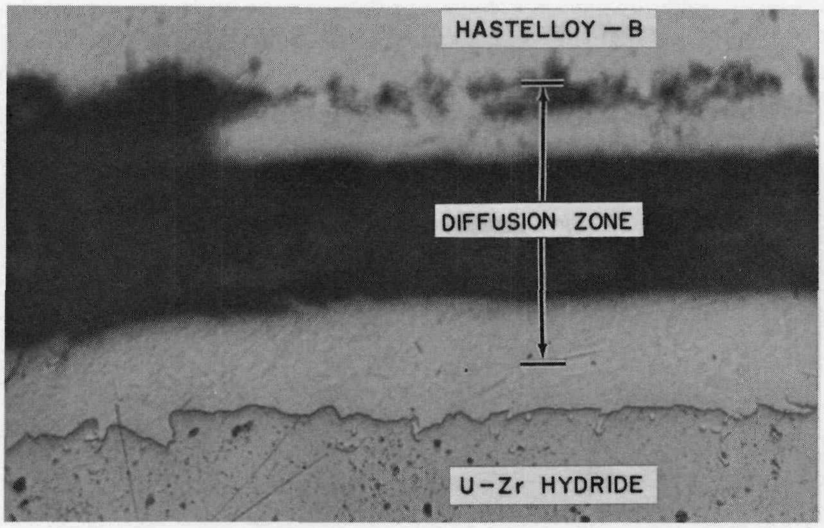


Figure 8. Hastelloy-B vs U-Zr Hydride – 190 Hr at 1500°F

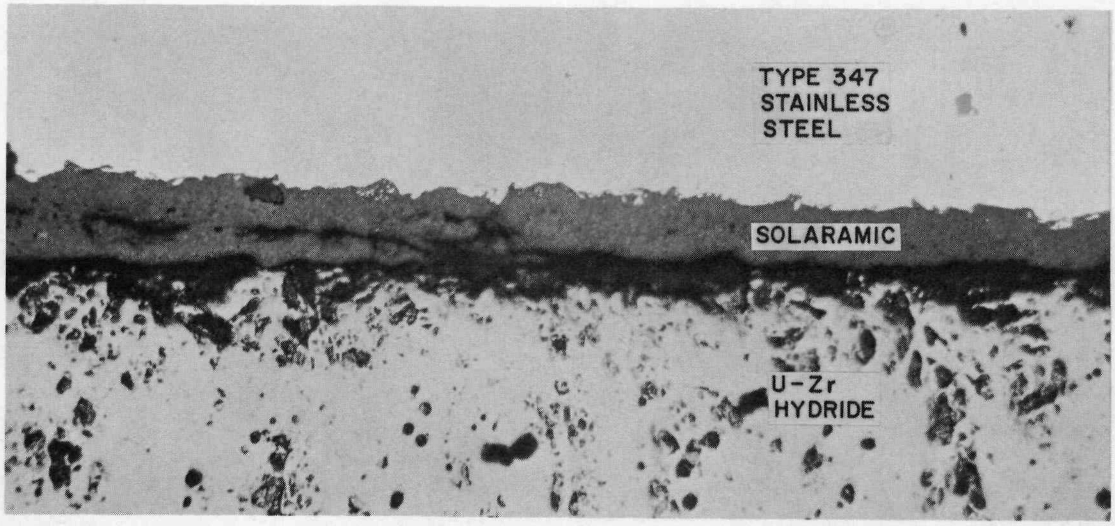


Figure 9. Solaramic vs U-Zr Hydride – 190 Hr at 1500°F

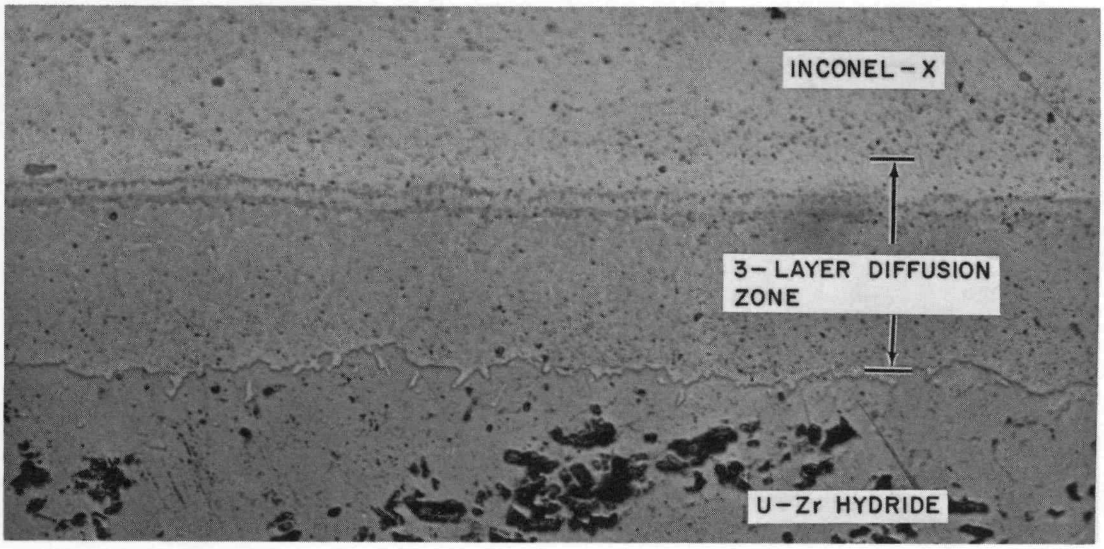


Figure 10. Inconel-X vs U-Zr Hydride — 190 Hr at 1500°F

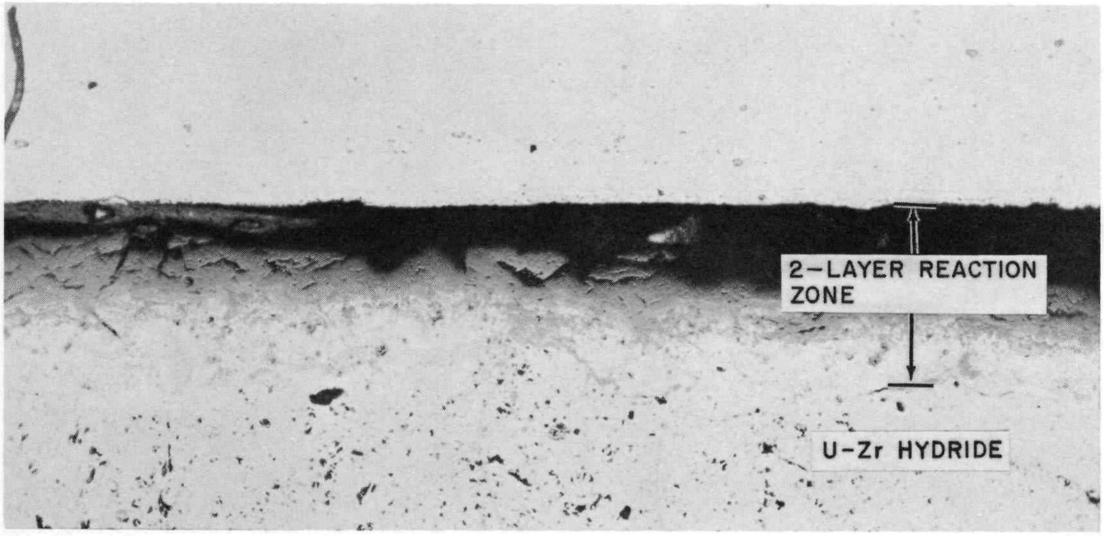
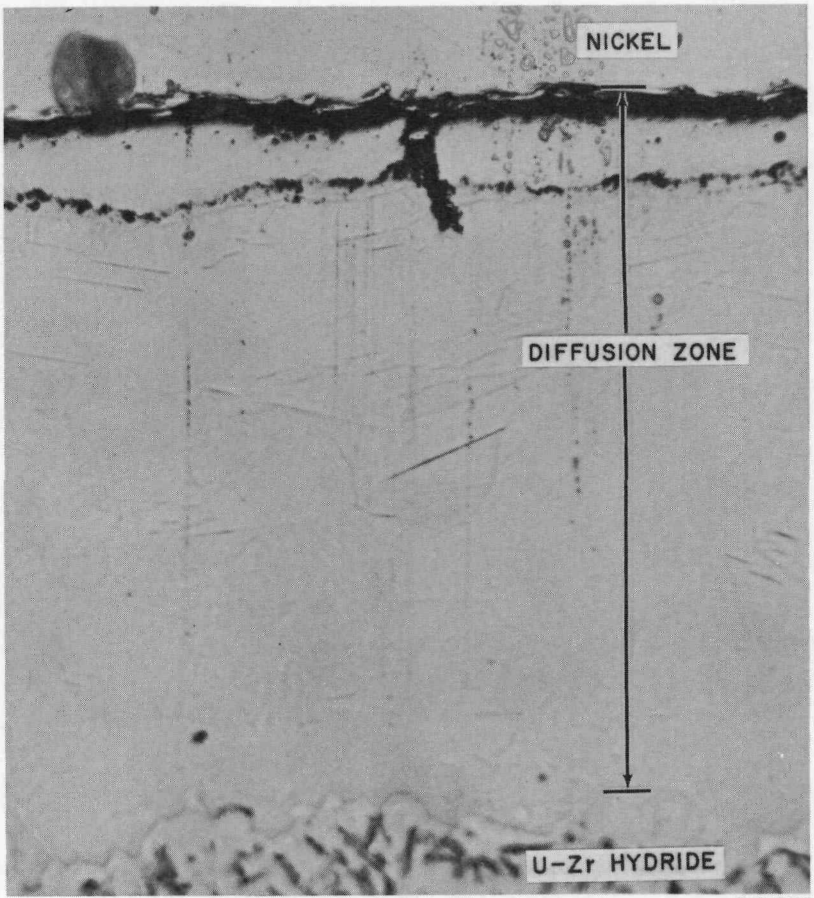


Figure 11. Oxidized U-Zr Hydride — 65 Hr at 1600°F



250X

Figure 12. Nickel vs U-Zr Hydride — 360 Hr at 1600°F

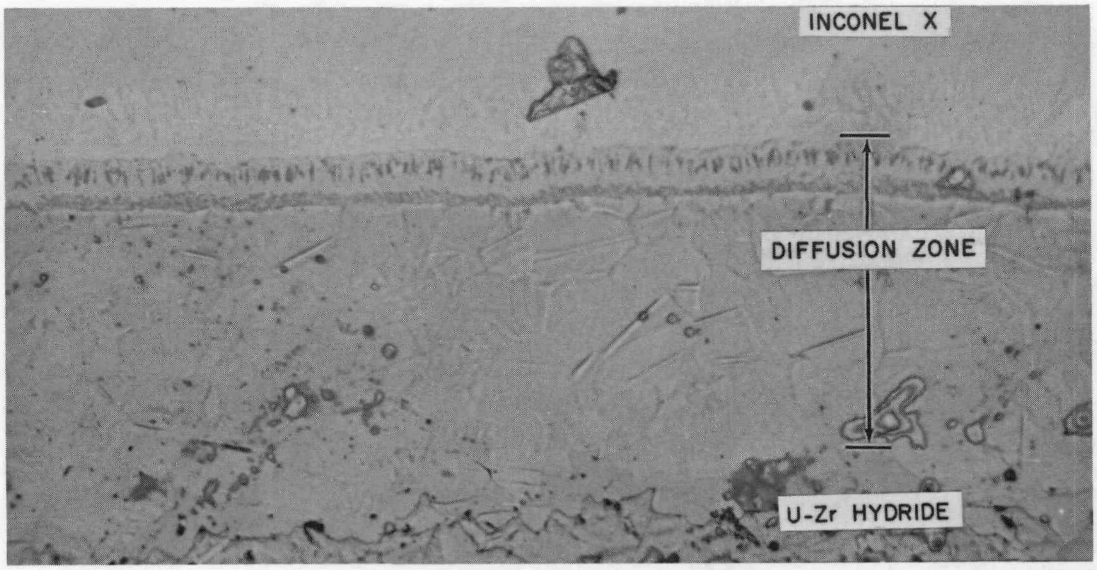


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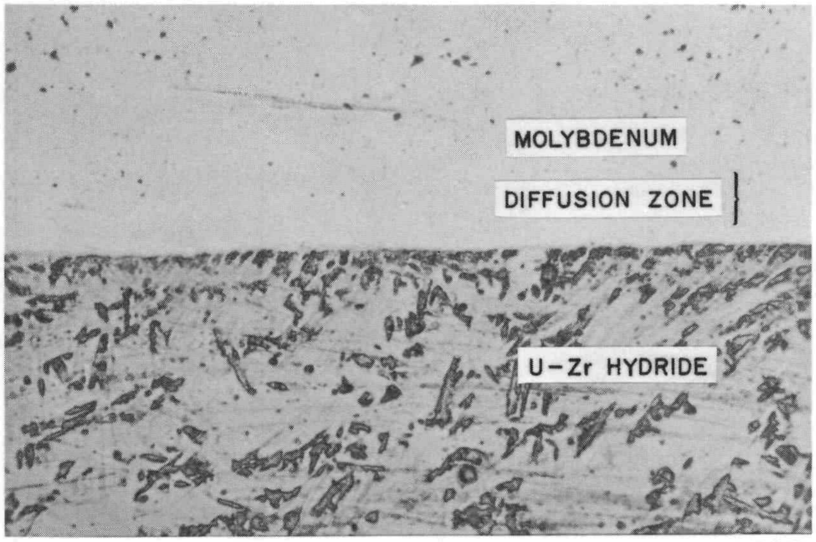
Figure 13. Inconel-X vs U-Zr Hydride — 360 Hr at 1600°F



250X

Figure 14. Molybdenum vs U-Zr Hydride —
360 Hr at 1600°F

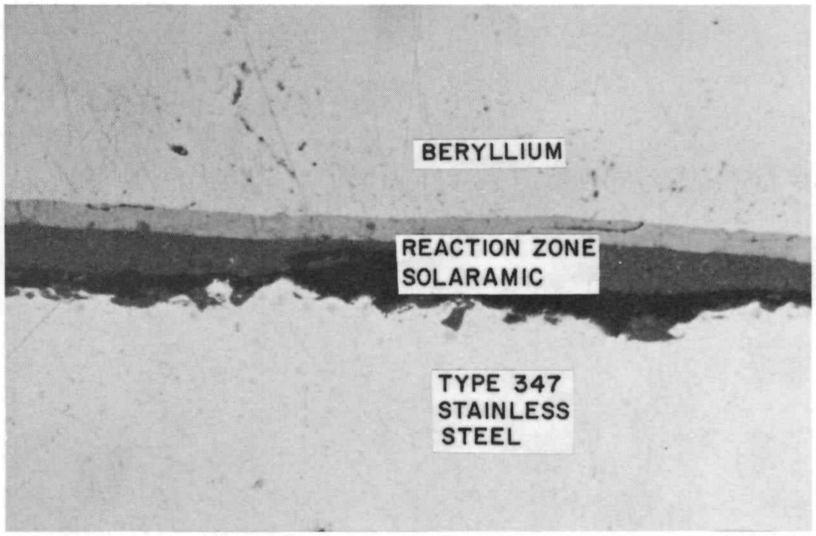


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Figure 15. Solaramic vs Beryllium —
235 Hr at 1300°F

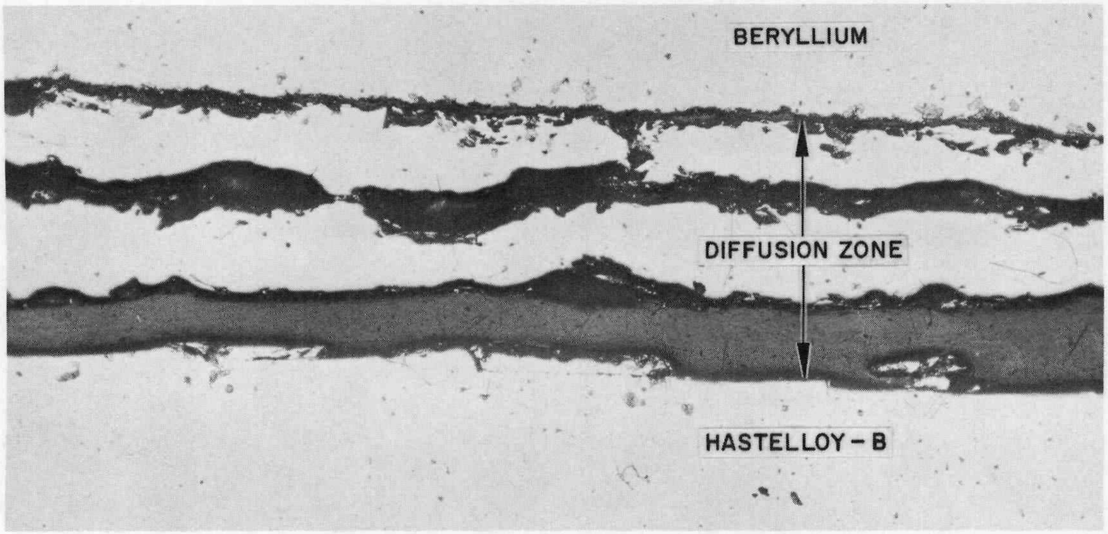

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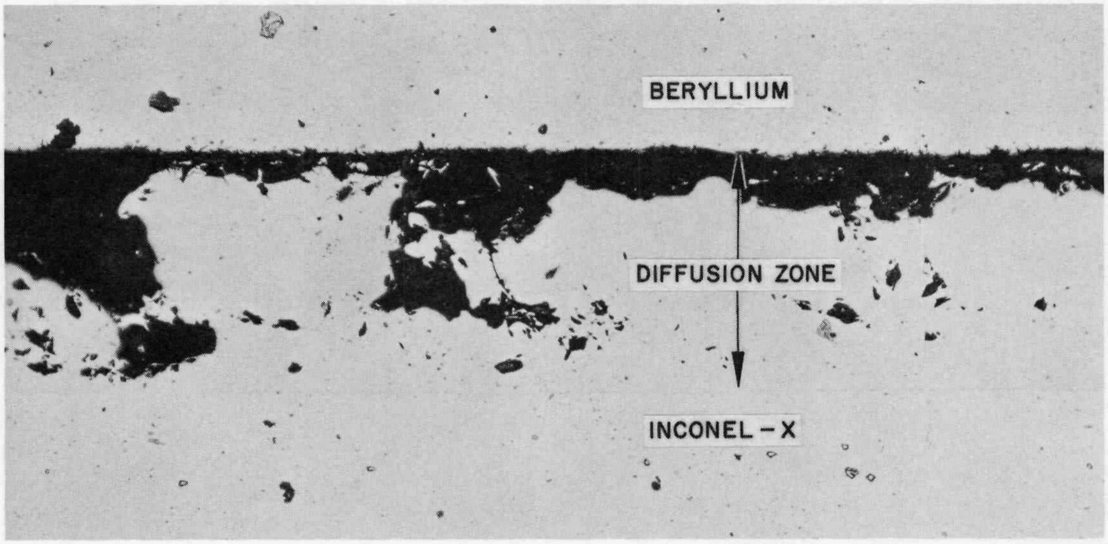
Figure 16. Beryllium vs Hastelloy-B — 235 Hr at 1300°F



100X

Figure 17. Beryllium vs Inconel-X — 235 Hr at 1300°F

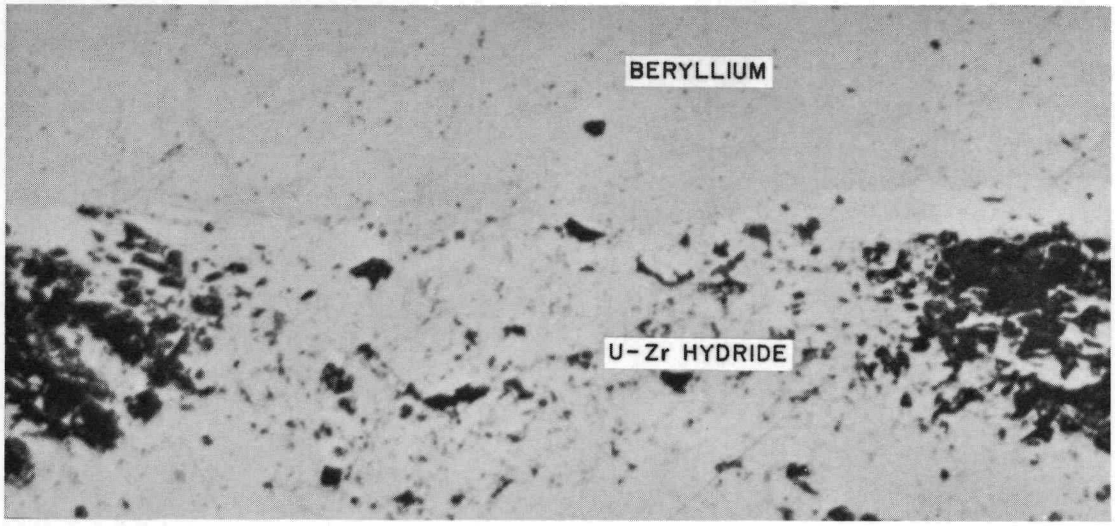


Figure 18. Beryllium vs U-Zr Hydride — 235 Hr at 1300°F

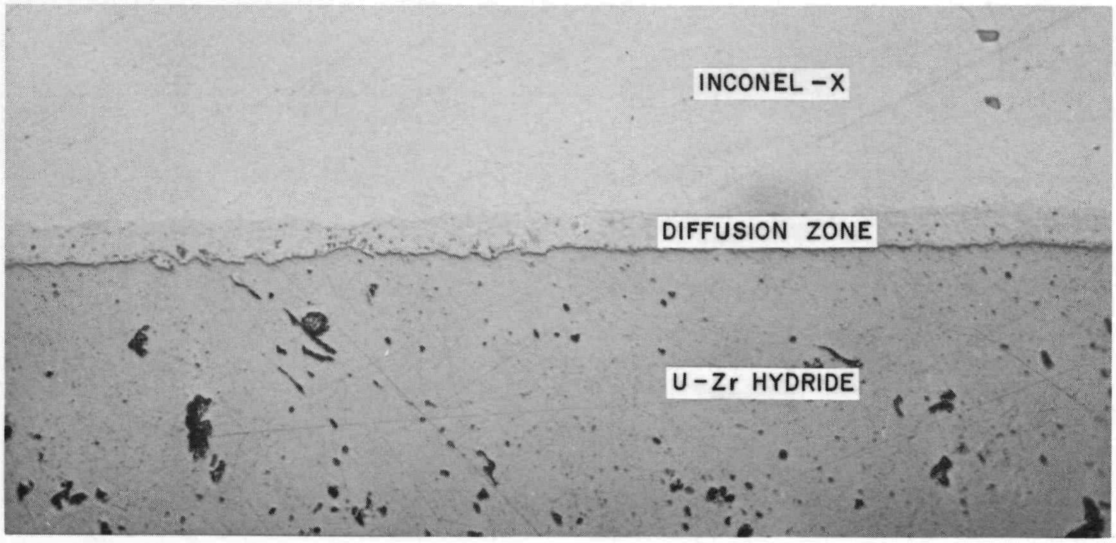
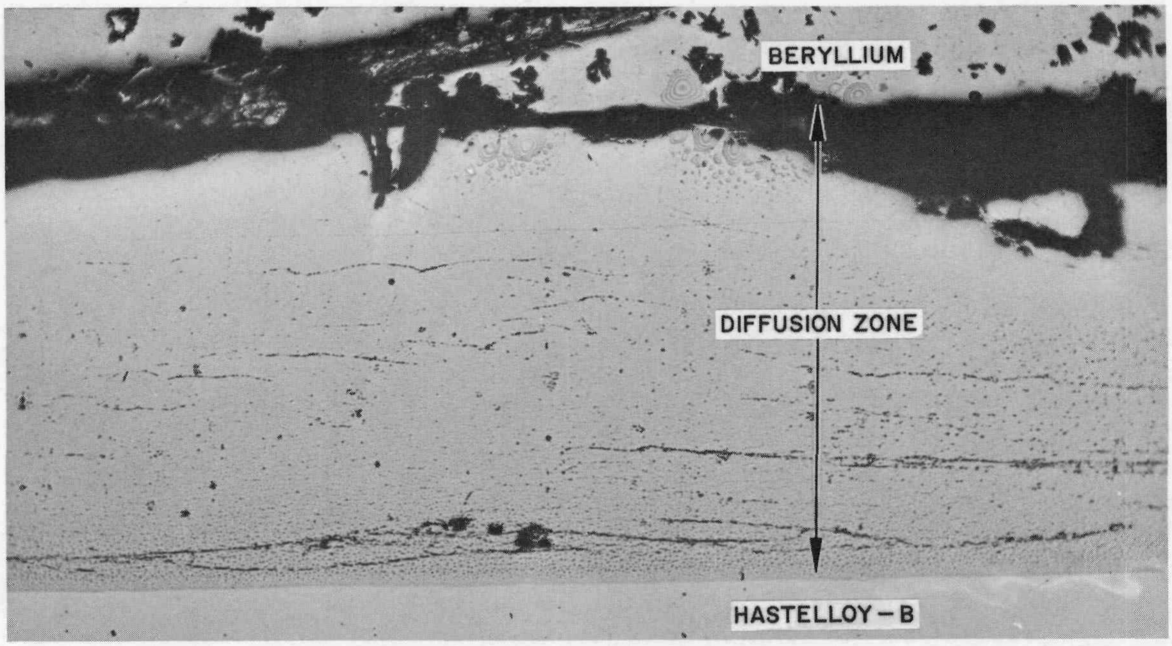


Figure 19. Inconel-X vs U-Zr Hydride — 235 Hr at 1300°F



100X

Figure 20. Beryllium vs Hastelloy-B – 334 Hr at 1300°F

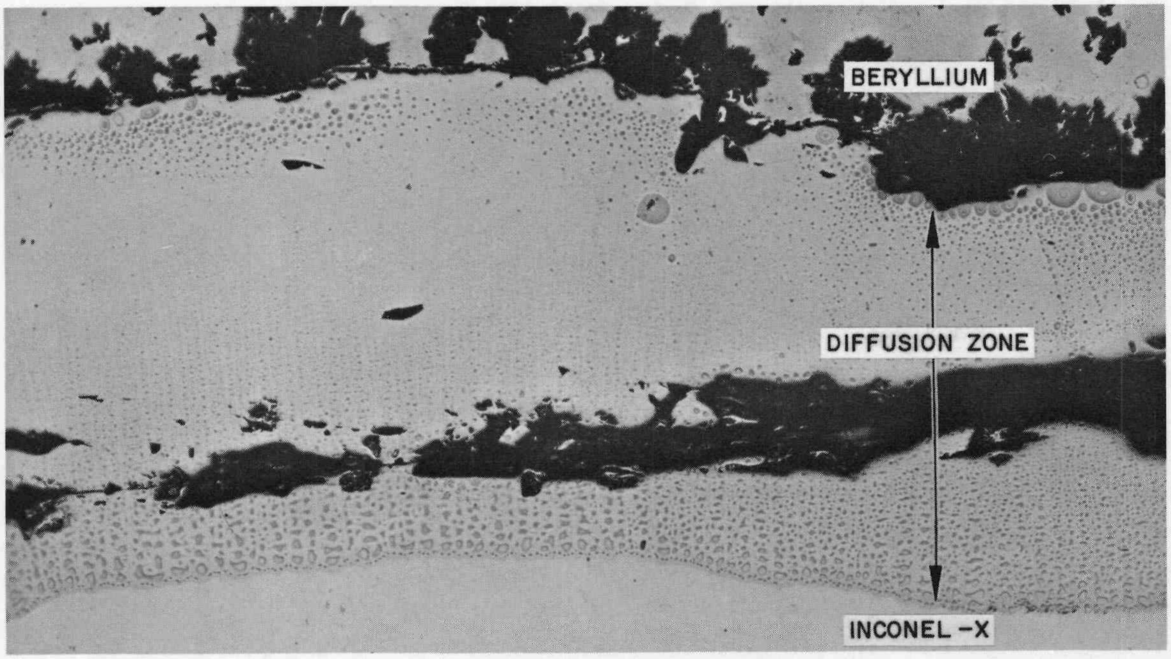


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Figure 21. Beryllium vs Inconel-X – 334 Hr at 1300°F

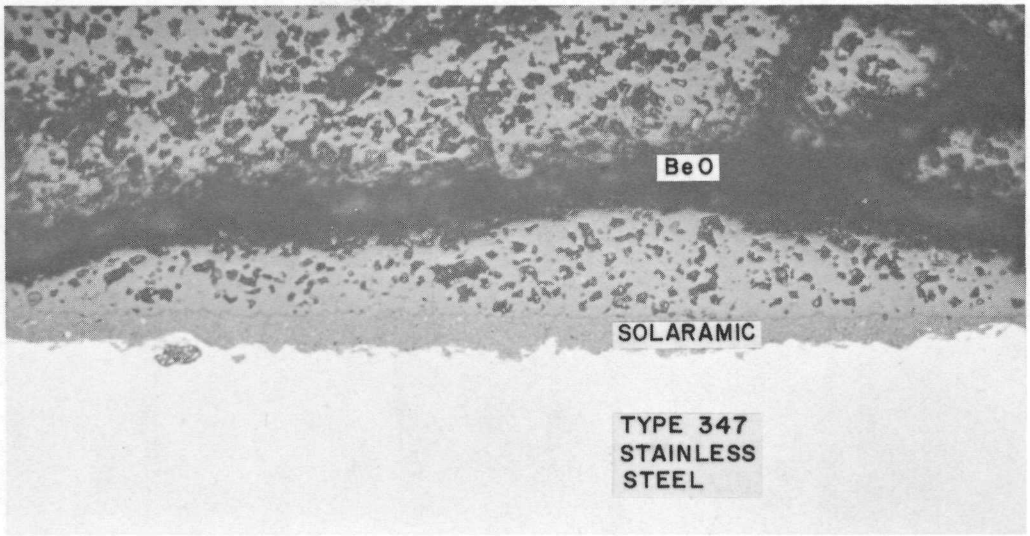

Figure 22. Solaramic vs Beryllium Oxide – 334 Hr at 1300°F

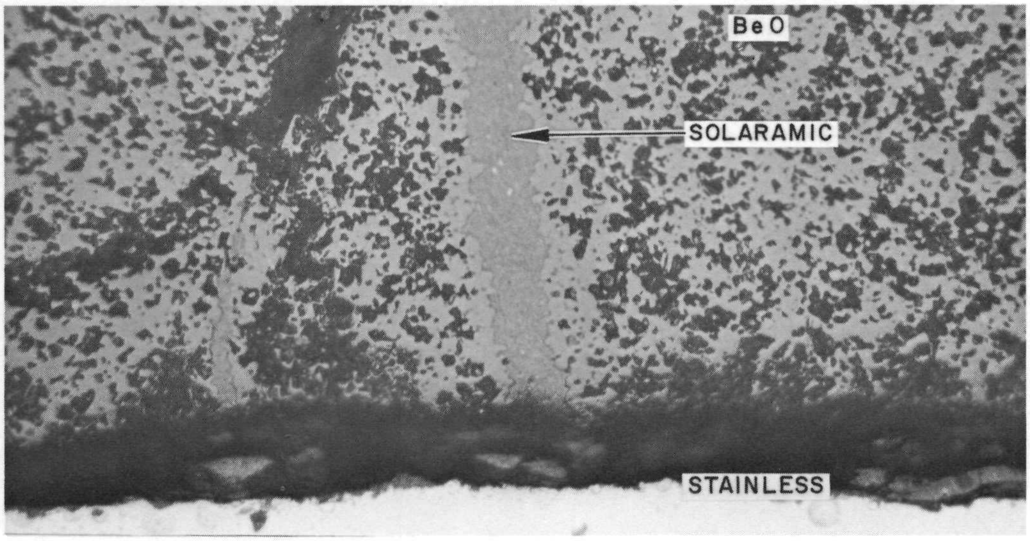

Figure 23. Solaramic vs Beryllium Oxide – 334 Hr at 1300°F

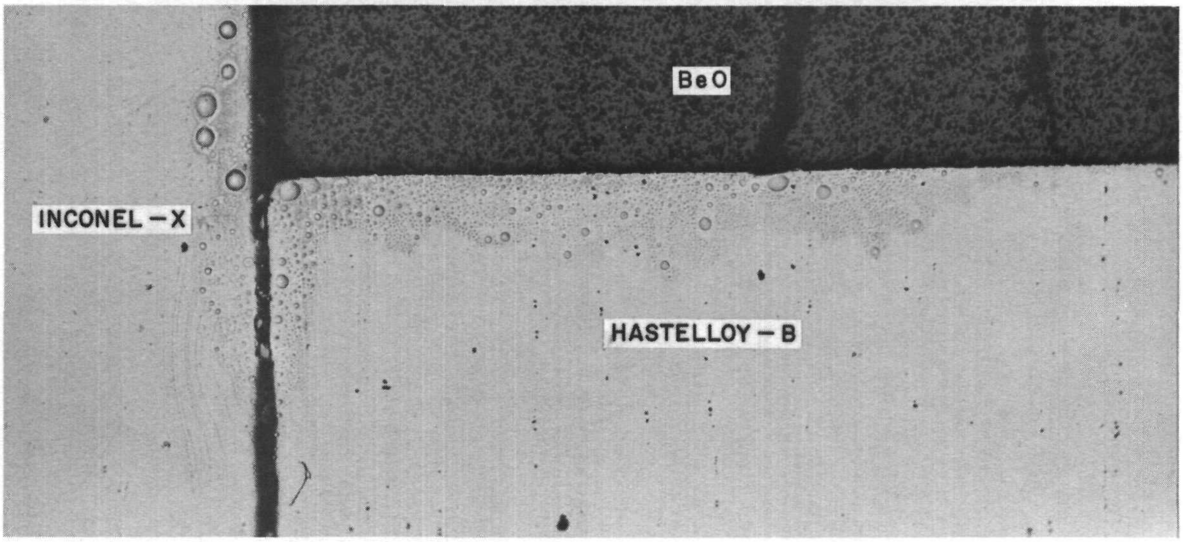

Figure 24. Beryllium Oxide vs Hastelloy-B and Inconel-X — 334 Hr at 1300°F