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SNAP FUEL AND CORE MATERIALS COMPATIBILITY SCREENING TESTS

(Title Unclassified)

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SNAP FUEL AND CORE MATERIALS COMPATIBILITY SCREENING TESTS

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By

L. B. LUNDBERG

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ABSTRACT

Screening tests have been performed to determine the compatibility of many of the materials in contact with each other in the SNAP 10A/2 and SNAP 8 reactor cores. In some cases of known incompatibility, reaction barriers were studied. The tests involved placing small samples of the materials under consideration in intimate contact at temperatures from 1200 to 1450°F for periods up to 1000 hr in environments similar to those encountered in the particular reactor. The mutual compatibility of the sample materials was determined by metallographic examination. Reactions were observed between the following components: fuel and the bare cladding (without hydrogen barrier); fuel cladding and the SNAP 2 beryllium internal radial reflector; and several fuel element thermal bond materials and both the fuel and the enamel hydrogen barrier. Reaction barriers for fuel cladding and the SNAP 2 beryllium internal radial reflector were examined, but none were found to be effective. The majority of the suggested reactor core materials were found compatible in this study.

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I. INTRODUCTION

In the development of nuclear reactors for long term unattended operation at high temperatures, one important property which must be considered is the compatibility of the various materials which are essential to the operation of the system. Three SNAP reactors, under development at Atomics International, will operate in space at temperatures of 1000 to 1500°F for a lifetime of 1 yr. To assure performance reliability, a program of compatibility testing was undertaken to guide the selection of the various core materials for these reactors.

The term "compatibility," as it is used in this report, is defined as the study of the behavior of two or more solid components which are in intimate contact during reactor operation. For the tests described herein, solid materials were considered to be compatible under specific conditions if they could be placed in intimate contact for an extended period of time without the occurrence of a reaction which was observable by standard metallographic techniques.

In analyzing the SNAP reactors for possible compatibility problems, the first consideration was the contact of the materials in question. In a solid-solid system the primary prerequisite for initiation and propagation of a physical or chemical reaction is the intimate contact of the materials. If this condition is met, the environment must then be considered. In addition to high temperatures, all materials in the SNAP reactor cores are exposed to a neutron flux, some components are exposed to hot flowing NaK-78, others are operated in the presence of hydrogen gas, and some must operate in the hard vacuum of space. With the exception of the neutron flux, these parameters were considered in compatibility tests where applicable.

In this report, the tests have been categorized in two groups. The tests on fuel element materials of the SNAP reactors are considered separately from those studies involving other core components, such as internal radial reflectors and the core tank.

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II. COMPATIBILITY OF SNAP FUEL ELEMENT MATERIALS

The fuel elements of the SNAP 8 and 10A/2 reactors consist of a hydrided Zr-10 U alloy fuel-moderator rod contained inside a chromized Hastelloy N envelope. The internal surface of the cladding envelope is coated with a thin ceramic hydrogen barrier. Under normal reactor operating conditions, the fuel and the ceramic are in contact in a hydrogen atmosphere. Should the ceramic barrier be removed from the cladding, the fuel could contact the chromized Hastelloy N cladding.

At various stages in the development of these reactors, special neutron reflector materials were placed inside the fuel elements. In the SNAP 10A/2 reactor, both beryllium and beryllium oxide bodies were considered. Since beryllium was found to be incompatible with the ceramic,[1] various barrier materials were investigated. In the SNAP 8 reactor, liquid metal thermal bond materials were considered to improve the heat flow from the fuel rod to the cladding.

The following paragraphs summarize the experimental studies on the SNAP fuel element materials. In general, the goal of each experiment was to establish whether or not a compatibility problem existed for the particular material under consideration.

A. FUEL AND CLADDING MATERIALS

Tests were performed on several different fuel and cladding materials. Specifically, the ceramic hydrogen barrier coatings included a commercial enamel, Solaramic* S14-35 Sm_2, and an enamel coating developed by Atomics International, AI-8763D. The fuels included several hydride compositions of zirconium-10 wt % uranium alloy, some of which contained additions of carbon (0.5 wt %). Chromized† and bare Hastelloy N were the cladding tube materials used in this study. Table 1 summarizes the compatibility tests performed on these materials.

1. Tests on Enamel Coatings and Fuels

a. Solaramic S14-35 Sm_2 vs Unmodified Zr^{10} wt % U Hydride

Two samples of Solaramic coated chromized Hastelloy N were placed in contact with wafers of zirconium-10 wt % uranium hydride having a hydrogen

*A product of Solar Aircraft Company, San Diego, California

†A chromium diffusion coating applied at elevated temperatures by a proprietary process termed "chromizing" must be used on Hastelloy N to obtain a good enamel coating.

TABLE 1

SUMMARY OF COMPATIBILITY TESTS ON FUEL AND CLADDING MATERIALS

Compatibility Couple	Test Conditions			Observations
	Temperature (°F)	Atmosphere	Time (hr)	
Solaramic (S14-35 Sm_2) vs unmodified Zr-10 wt % U hydride (H/Zr ~1.6)	1450	Hydrogen	513	No reaction, compatible
AI-8763D vs carbon modified Zr-IOU hydride (H/Zr ~1.5)	1450	Hydrogen	320	No reaction, compatible
Chromized Hastelloy N vs Zr-10 wt % U hydride (H/Zr ~1.6)	1450	Hydrogen	513	Reaction zone 0.3 mil thick, incompatible
Bare Hastelloy N vs carbon modified Zr-10 wt % U hydride (H/Zr ~1.5)	1450	Hydrogen	320	Reaction zone 2.5 mil thick, incompatible

zirconium atomic ratio of about 1.6. The samples were placed in a stainless steel holding device designed to maintain intimate contact for the duration of the test.

The mounted samples were sealed in a stainless steel capsule along with some pieces of zirconium hydride which provided a hydrogen atmosphere. The internal surfaces of the stainless capsule were coated with Solaramic to prevent hydrogen loss during the test. The capsule was placed in a muffle furnace and heated at 1450°F for 513 hr. After furnace cooling, the capsule was opened and the samples were prepared for microscopic examination. An examination of the Solaramic-fuel interface did not reveal any significant reaction.

b. AI-8763D vs Carbon Modified Zr-10 wt % U Hydride

The AI-8763D enamel hydrogen barrier was applied to unchromized Hastelloy N, and the enameled side of the sample placed against a piece of carbon modified zirconium-10 wt % uranium hydride with a hydrogen-zirconium ratio of about 1.5. Carbon content of the alloy was 0.5 wt %. The holding device in this test was made of molybdenum, having a coefficient of thermal expansion much lower than stainless steel. This assured that the sample interfaces would be maintained under compression at the test temperature. The samples, mounted in the holder, were sealed in an evacuated Vycor capsule along with some pieces

of zirconium hydride, added to maintain a hydrogen atmosphere over the samples during the test. The capsule was heated in a muffle furnace at 1450°F for 320 hr. The samples were mounted, sectioned, and polished for microscopic examination. As will be noted in Figure 1, there was no observable reaction between either the enamel hydrogen barrier and the fuel material or the enamel and the Hastelloy N.

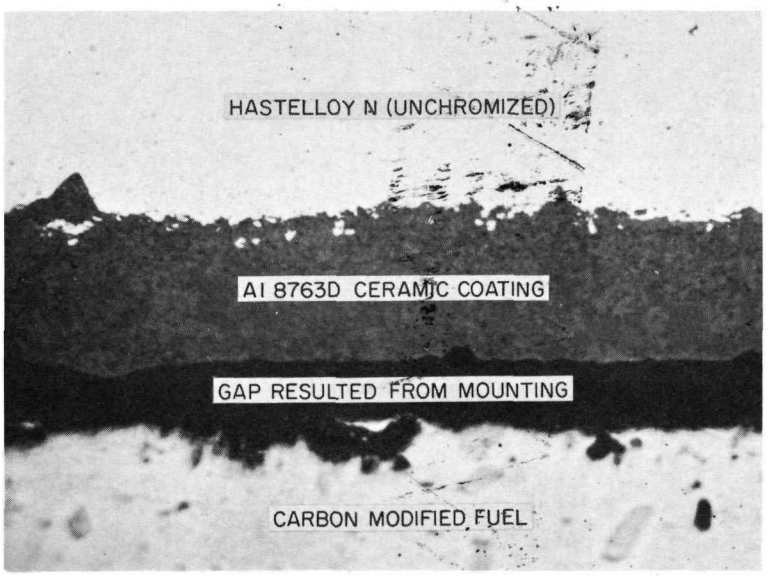

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Figure 1. AI-8763D on Hastelloy N (unchromized) vs Carbon Modified Zy-10 wt % U Hydride (H/Zr ~1.5) Fuel After 320 hr at 1450°F in Hydrogen

c. Discussion

The results of these tests are consistent with those reported by Balkwill[1] in which Solaramic was observed to be compatible with zirconium-10 10 wt % uranium hydride at 1500°F in hydrogen for a period of 190 hr. The inherent stability of these ceramic enamels suggests that compatibility under similar conditions can be expected to last for much longer periods of time.

2. Tests on Fuels and Cladding Chromized Hastelloy N vs Zr-10 wt % U Hydride

Chromized Hastelloy N was placed in contact with zirconium-10 wt % uranium hydride having an H/Zr ratio of ~1.6. Two samples were mounted in a stainless steel holder and sealed in a stainless steel capsule coated internally

with Solaramic. The capsule void volume was packed with pieces of zirconium hydride so that a hydrogen atmosphere would be maintained over the sample during the test. The sealed capsule was heated in a muffle furnace for 513 hr at 1450°F.

After test, the samples were prepared for microscopic examination. Inspection of the interface between the chromized Hastelloy N and the fuel material with a microscope revealed that a reaction had taken place as a result of the test. The resultant diffusion zone measured about 0.3 mil in thickness (Figure 2).

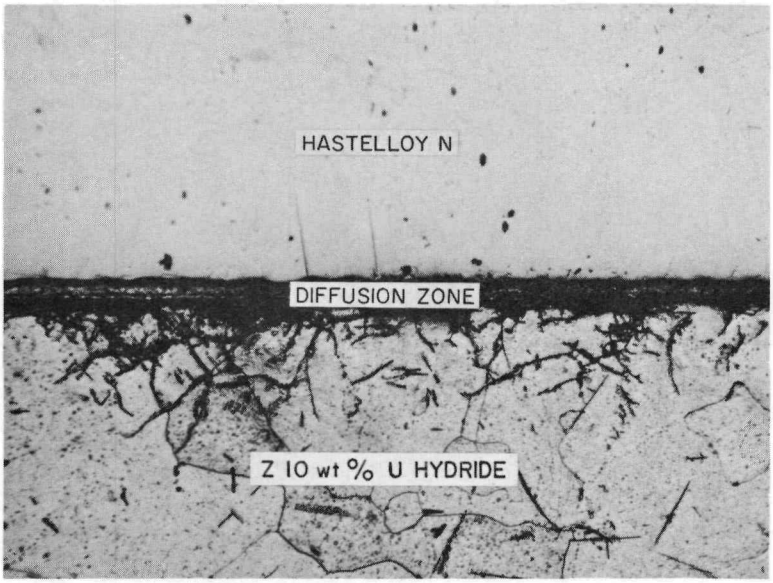

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Figure 2. Chromized Hastelloy N vs Zr-10 wt % U Hydride (H/Zr ~1.6) Fuel After 513 hr at 1450°F in Hydrogen

b. Bare Hastelloy N vs Carbon-Modified Zr-10 wt % U Hydride

Since it was thought, in the early stages of development, that the enamel hydrogen barrier, AI-8763D, could be satisfactorily used on bare Hastelloy N, the compatibility of the modified fuel and bare Hastelloy N was tested at SNAP 8 conditions. Samples of bare Hastelloy N and zirconium-10 wt % uranium hydride having an H/Zr ratio of about 1.5 were bound together in a molybdenum holder and sealed in a Vycor capsule along with some pieces of zirconium hydride used to maintain a hydrogen atmosphere over the samples during the test. The sealed capsule was heated in a muffle furnace at 1450°F for 320 hr.

At the conclusion of the test, the samples were removed from the capsule and prepared for microscopic examination. A study of the interface between the two materials revealed that a reaction had taken place. The diffusion zone measured 2.5 mils in thickness. A photomicrograph of a typical interfacial region is given in Figure 3.

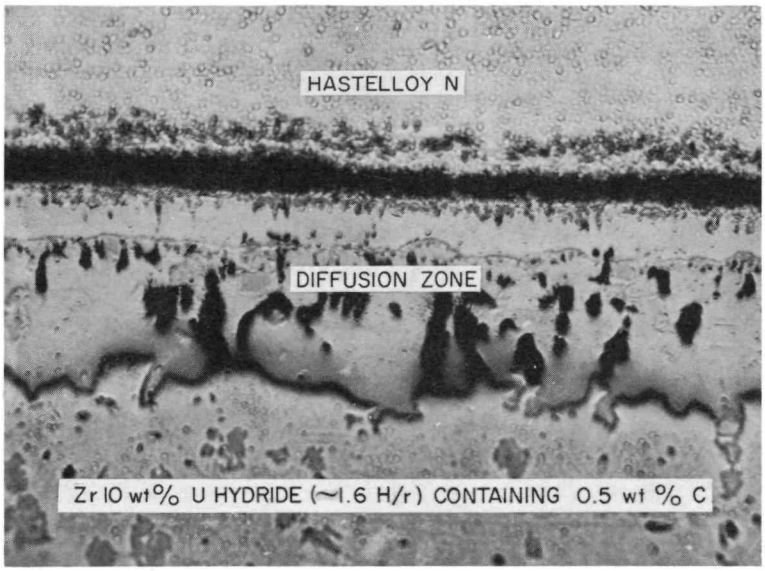

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Figure 3. Hastelloy N vs Carbon Modified Zr-10 wt % U Hydride After 320 hr at 1450°F in Hydrogen

c. Discussion

These tests show quite conclusively that a compatibility problem exists in the SNAP fuel elements if the ceramic hydrogen barrier ceases to separate the fuel and Hastelloy N cladding materials. The type of reaction appears to be solid-state interdiffusion.

In comparing the results of tests on the compatibility of the Hastelloy N cladding material with the SNAP fuels, it can be said that chromized Hastelly N is more compatible with the fuel at 1450°F than is bare Hastelloy N. Carbon-modified fuel (containing about 0.5 wt % carbon) would not be expected to be significantly different in its compatibility behavior from that of noncarbon material, since the carbon exists in the form of an extremely stable carbide (ZrC).

B. REACTION BARRIERS FOR BERYLLIUM FUEL ELEMENT END REFLECTORS

The fuel element of the SNAP 2 experimental reactor (SER) contained beryllium reflector plugs inside each end of the fuel envelope.[2] Since the reflector plug was in contact with the enameled interior of the fuel element, Balkwill[1] performed tests to determine the compatibility of beryllium and Solaramic in hydrogen at 1300°F. A reaction zone about 0.4 mil in thickness was observed between these two materials after testing for 235 hr. As a result of this discovery, a search was made for possible reaction barrier materials. Due to their chemical stability in hydrogen at elevated temperatures and ease of application to solid bodies by plasma arc techniques, the oxides of aluminum, beryllium, thorium, and zirconium were considered as possible barrier materials. Chromium electrodeposited on the beryllium was also considered for use as a reaction barrier.

1. Compatibility Tests

A study of the compatibility of barrier materials applied to beryllium and Solaramic applied to chromized Hastelloy N was carried out at 1300°F in hydrogen.[3] Cylindrical pieces of beryllium, were plasma-arc sprayed with the refractory oxides, ground to size and fit inside sections of chromized Hastelloy N SNAP 2 fuel element tubes, which had been coated internally with Solaramic. Similar pieces of beryllium were electro-plated with chromium by standard techniques[4] and fit into fuel element tube sections. Bare beryllium pieces were also tested in a similar arrangement.

The assembled specimens were sealed inside stainless steel capsules along with some pieces of zirconium hydride. As in previous tests of this type, the internal surfaces of the capsules were coated with Solaramic to retain the hydrogen atmosphere over the samples during the test. The sealed capsules were heated in a muffle furnace at 1300°F for 1000 hr; whereupon, they were furnace cooled and opened. The samples were prepared for microscopic examination of the interfaces between the various materials in contact.

Both macroscopic and microscopic observations indicated that all the refractory oxide coatings were compatible with both the Solaramic and the beryllium, to which they had been applied. Macroscopic examination of the Solaramic surfaces that had been in contact with the chrome-plated beryllium revealed

(Figure 4) a wrinkling of the enamel. Microscopic observations on the interfacial regions indicated that the Solaramic coating was still continuous on the surface of the chromized Hastelloy N. A summary of the results of these tests is given in Table 2.

Deformation of the Solaramic was apparently caused by growth of the chrome-plate. This "growth" probably resulted from the mismatch of thermal expansion between the chromium and the other materials in the compatibility

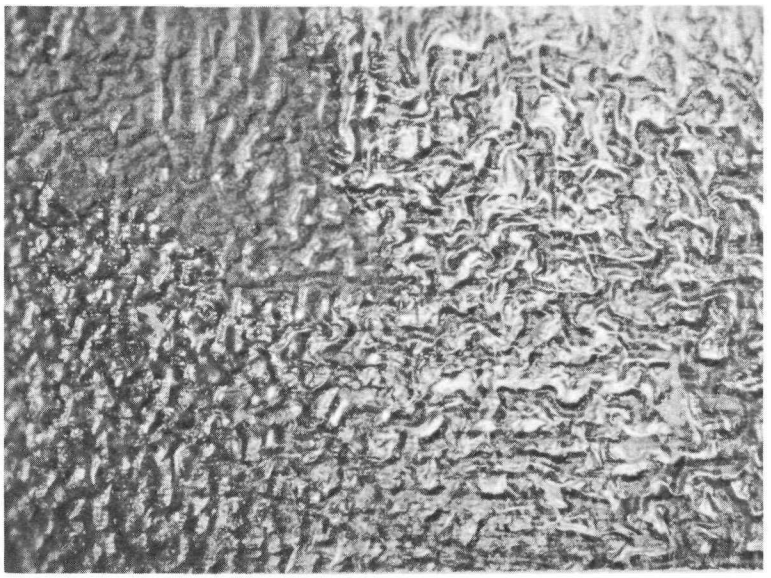

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Figure 4. Wrinkled Surface of Solaramic After Contacting Chrome-Plated Beryllium for 1000 hr at 1300°F in Hydrogen

specimen; i.e., on cooling from 1300°F, the stress-relaxed ring of chromium separated from the beryllium and pushed against the Solaramic which flowed to fit the buckled surface of the chromium ring. The bare beryllium pieces were not observed to react with the Solaramic enamel. A photomicrograph of this material couple after test is shown in Figure 5.

2. Discussion

These tests show that separation of beryllium and Solaramic can be ensured at 1300°F in hydrogen by the use of a refractor oxide reaction barrier. A chromium layer also prevents reaction in this situation, but can cause deformation of the Solaramic barrier. The apparent compatibility of the bare beryllium and the Solaramic which was seen in this study may have been a result of loss of

TABLE 2

SUMMARY OF COMPATIBILITY TESTS ON REACTION BARRIERS FOR BERYLLIUM FUEL ELEMENT END REFLECTORS

Compatibility Couple	Test Conditions			Observations
	Temperature (°F)	Atmosphere	Time (hr)	
Beryllium vs Solaramic	1300	Hydrogen	1000	No reaction, compatible
BeO vs Solaramic	1300	Hydrogen	1000	No reaction, compatible
Al_2O_3 vs Solaramic	1300	Hydrogen	1000	No reaction, compatible
ZrO_2 vs Solaramic	1300	Hydrogen	1000	No reaction, compatible
ThO_2 vs Solaramic	1300	Hydrogen	1000	No reaction, compatible
Chrome-plated Beryllium vs Solaramic	1300	Hydrogen	1000	Solaramic wrinkled, no reaction, essentially compatible

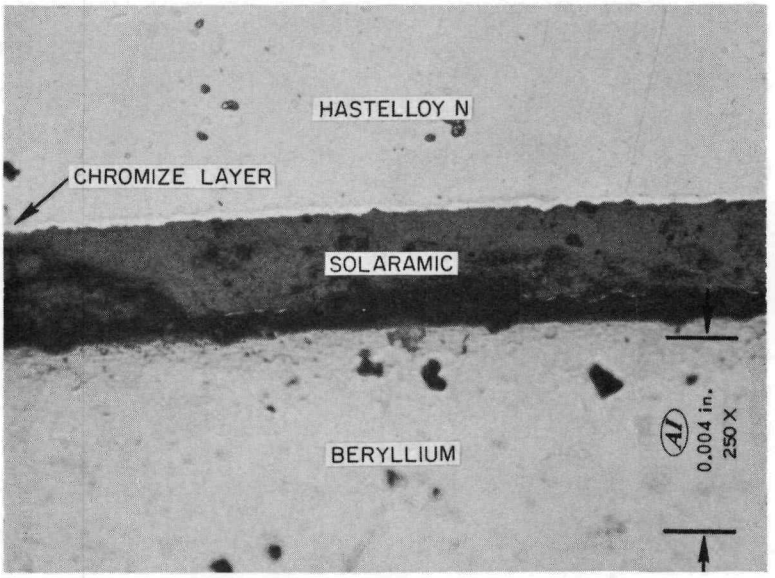

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Figure 5. Beryllium vs Solaramic on Chromized Hastelloy N After 1000 hr at 1300°F in Hydrogen

contact at the interface with time at temperature. Although the initial interfacial pressure was estimated to be 14,000 psi at 1300°F this pressure dropped to a very low value soon after the test temperature was attained because the Solaramic will flow under stress at this temperature. An examination of the enameled surfaces after the test showed that substantial amounts of Solaramic had been squeezed from the regions of contact during the test. Slight furnace temperature fluctuations during the test probably caused intermittent contact of portions of the sample interfaces. This contrasts with the condition maintainted by Balkwill[2] where the contact pressure was reintroduced to the specimen interface continually throughout the test.

C. FUEL ELEMENT THERMAL BOND MATERIALS

In conjunction with the SNAP 8 reactor development, there has been a search for a "thermal bonding" material to replace the hydrogen annulus between the fuel rod and the fuel element cladding. Some metal or alloy, liquid at SNAP 8 operating temperatures, appeared to be the type of material most likely to provide the desired increase in heat transfer from the fuel to the cladding.[5] Besides being liquid at temperature, the candidate materials must:

1) Not boil at operating conditions

2) Should have a negligible volume change during melting

3) Must have a relatively low thermal neutron cross section

4) Must have high thermal conductivity compared with hydrogen gas.

The tests performed on candidate thermal bond materials are discussed in the following sections, and are summarized in Table 3.

1. Compatibility Studies on a Lead-Bismuth Thermal Bond Material

The lead-bismuth eutectic (55.5 wt % Bi) fits the above requirements fairly well, since it is molten above 257°F, boils at 3038°F, has a thermal conductivity of 9 Btu/hr-ft-°F, and has a nuclear cross section of 0.093 barns. A study of the compatibility of liquid Pb-Bi eutectic with zirconium 7 wt % uranium hydride having an H/Zr ratio of 1.7 was performed at 1500° F under static conditions.[6]

The compatibility test involved heating pieces of the zirconium-uranium-hydride fuel material in an evacuated Vycor capsule filled with prehomogenized

TABLE 3

SUMMARY OF COMPATIBILITY TESTS ON FUEL ELEMENT THERMAL BOND MATERIALS

Compatibility Couple	Test Conditions		Observations
	Temperature (°F)	Time (hr)	
Pb-Bi Eutectic vs Zr-7 wt % U Hydride (H/Zr ~1.7)	1500	100	Reaction zone, incompatible
Pb-Bi Eutectic vs Zr-7 wt % U Hydride (H/Zr ~1.7)	1500	250	Reaction zone, incompatible
Pb-Bi Eutectic vs Zr-7 wt % U Hydride (H/Zr ~1.7)	1500	500	Reaction zone, incompatible
Pb-Bi Eutectic vs Zr-7 wt % Hydride (H/Zr ~1.7)	1500	1000	Reaction zone, incompatible
Pb vs Zr-7 wt % U Hydride (H/Zr ~1.7)	1350	110	No reaction, compatible
Bi vs Zr-7 wt % U Hydride (H/Zr ~1.7)	1350	110	Reaction zone, incompatible
Pb vs Zr-7 wt % U Hydride (H/Zr~1.7)	1500	336	Reaction zone, incompatible
Sb-23 wt % Tl vs Zr-7 wt % U Hydride (H/Zr~1.7)	1500	336	Reaction zone, incompatible
Sb-23 wt % Tl vs Solaramic	1500	336	Some enamel removed from Type 347 stainless steel substrate
Sb-21 wt % Pb vs Zr-7 wt % U Hydride (H/Zr ~1.7)	1500	336	Reaction zone, incompatible
Sb-21 wt % Pb vs Solaramic	1500	336	Reaction zone, incompatible
Sb-21 wt % Sn vs Zr-7 wt % U Hydride (H/Zr ~ 1.7)	1500	336	Reaction zone, incompatible
Sb-21 wt % Sn vs Solaramic	1500	336	Reaction zone, incompatible

lead-bismuth eutectic. The test temperature was 1500°F, and the test durations for separate samples were 100, 250, 500, and 1000 hr. Visual examination of transverse sections of each of the samples revealed that a reaction had taken place between the molten metal and the fuel during even the shortest test. Control experiments were carried out at 1350°F to determine the compatibility of the pure materials, bismuth and lead, with this fuel material. The bismuth was found to be incompatible with the fuel in a test lasting 110 hr, but the lead was found to be compatible with the fuel in a test of the same duration. Another test of lead and this fuel material was performed at 1500°F for 336 hr, and here the two were found to be incompatible. This lack of compatibility between the fuel and lead at 1500°F was not unexpected. Previous studies,[7] where an alloy of 80% zirconium, 15% yttrium, and 5% uranium, hydrided to 1.64% weight gain, was heated in lead at 1500°F for 45 hr indicated the lead and fuel alloy to be incompatible.

2. Compatibility Studies on Antimony-Base Alloy Thermal Bond Materials

In another study, antimony-base alloys were considered for use as a fuel element thermal bond material. Binary antimony base alloys containing 23 wt % thallium (m.p. 1040°F), 21 wt % lead (m.p. 1095°F), and 21 wt % tin (m.p. 1005°F) were tested for compatibility with Solaramic, which was applied to Type 347 stainless steel, and zirconium-7 wt % uranium hydride having an H/Zr 1.7, at 1500°F. In a static test lasting 336 hr, all of the molten antimony-base binary alloys were found to be incompatible with the fuel material, and although no diffusion type reaction was noted to have occurred between these alloys and the Solaramic, some removal of the enamel was observed to have resulted from the test.

The results of these tests demonstrate that, if thermal bonding of the SNAP 8 fuel element is necessary, further studies will be required to find a suitable heat transfer material.

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III. COMPATIBILITY OF OTHER SNAP CORE MATERIALS

Exterior to the fuel elements of the SNAP reactors, there are several core components made of dissimilar materials which are in intimate contact during reactor operation. The core tank is in contact with the interior and exterior radial reflectors, the top and bottom grid plates, and probably some of the fuel elements. The internal radial reflectors also come in contact with a few of the fuel elements and the grid plates.

Compatibility studies were performed on many of the materials proposed for and used in the above-mentioned components. A summary of the tests is presented in Table 4. In some cases where incompatibility was found to exist, reaction barriers were considered and tested as a means of solving the problem. These tests are summarized in Table 5. As in the compatibility studies on the fuel element materials, the tests were only intended to indicate whether or not a compatibility problem existed.

A. COMPATIBILITY STUDIES FOR CORE COMPONENTS EXTERIOR TO THE FUEL ELEMENTS

1. Core Component Materials in Contact With the Core Tank Materials

Several different materials have been considered for use in the core tanks of the SNAP reactors. Of these, Hastelloy C and Type 316 stainless steel have been tested to determine their compatibility with the materials considered for some of the other core components. The "other" core components included the internal and external radial reflectors, the top and bottom grid plates, and the fuel element cladding. The tests involving materials used for these various components are described below.

a. Internal and External Radial Reflector Materials

The radial reflectors for the SNAP reactors have been constructed from both beryllium and beryllium oxide. The SNAP 10A/2 reactor presently uses unclad beryllium for both the internal and external reflectors,[9] while the SNAP 8 reactor uses unclad beryllium for the external reflector and beryllium oxide clad with electroplated nickel,[10] a Hastelloy alloy or a stainless steel[11] for the internal reflectors. SNAP 8 Experimental Reactor (S8ER) internal

TABLE 4

SUMMARY OF COMPATIBILITY TESTS ON CORE COMPONENT MATERIALS EXTERIOR TO THE FUEL ELEMENTS

Compatibility Couple	Test Conditions			Observation
	Temperature (°F)	Atmosphere	Time (hr)	
Beryllium vs chromized Hastelloy C	1300	Flowing NaK-78	1000	No reaction, compatible
Hastelloy N vs Hastelloy C	1300	Argon-Helium	1000	No reaction, compatible
Nickel vs Type 316 stainless steel	1300	Argon-Helium	1000	Reaction zone, incompatible
Beryllium oxide vs nickel	1300	Argon-Helium	1000	No reaction, compatible
Beryllium oxide vs Type 347 stainless steel	1300	Argon-Helium	1000	No reaction, compatible
Hastelloy C vs Type 316 stainless steel	1300	Argon-Helium	1000	No reaction, compatible
Hastelloy N vs beryllium	1200	NaK-78	1000	Reaction zone, 1 to 2 mil thick, incompatible
Hastelloy N vs beryllium	1300	NaK-78	1000	Reaction zone, 1 to 2 mil thick, incompatible
Hastelloy N vs beryllium	1300	Argon-Helium	1003	Reaction zone, 15 mil thick, incompatible
Chromized Hastelloy N vs beryllium	1300	Argon-Helium	1003	Reaction zone, 10 mil thick, incompatible
Hastelloy N vs Type 347 stainless steel	1300	Argon-Helium	1000	Reaction zone, 1 mil thick, incompatible
Hastelloy N vs nickel	1300	Argon-Helium	1000	Reaction zone, 1 mil thick, incompatible

radial reflectors are clad with Hastelloy N.[12] Tests were performed to determine the compatibility of the beryllium reflector and chromized Hastelloy C core tank material as follows:

1) Hastelloy N internal radial reflector cladding and Hastelloy C core tank material
2) Nickel radial reflector cladding and Type 316 stainless steel core tank material
3) Beryllium oxide and nickel radial reflector cladding material
4) Beryllium oxide and Type 347 stainless steel radial reflector cladding material.

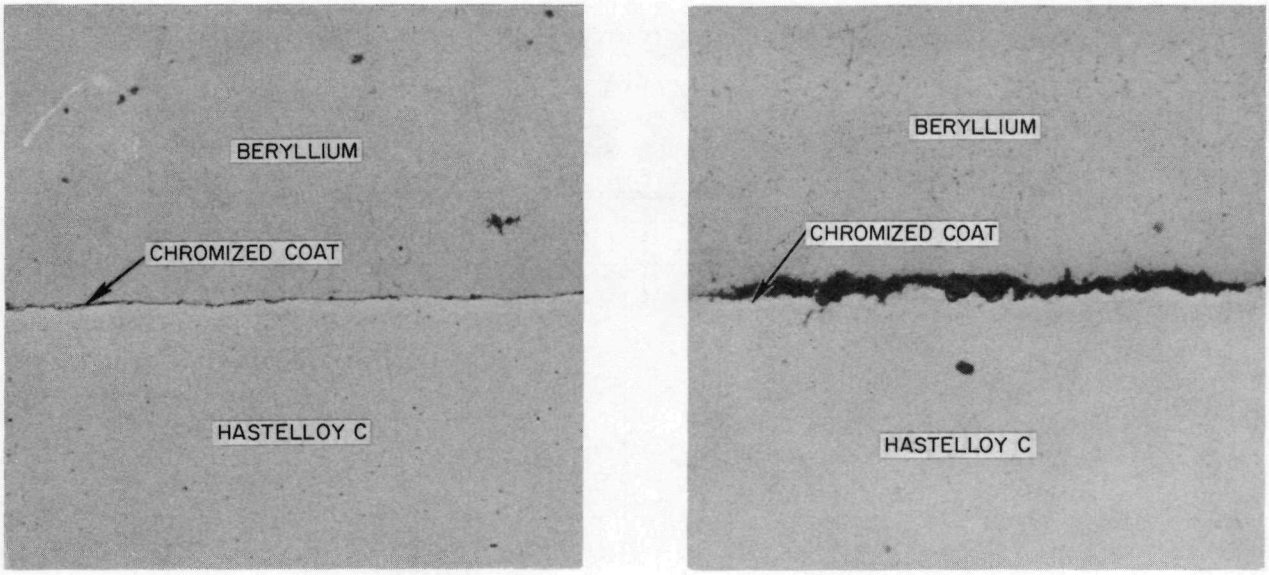
TABLE 5

RESULTS OF A 1000 HR COMPATIBILITY TEST IN 1300°F NaK INVOLVING REACTION BARRIER MATERIALS FOR BERYLLIUM INTERNAL RADIAL REFLECTORS

Compatibility Couple	Observations
Chromium-plated beryllium vs Hastelloy N	0.8 mil diffusion zone. Plate separated from beryllium.
Chromium-plated beryllium vs Hastelloy C	0.5 mil diffusion zone. Plate separated from beryllium
Chromium-plated Hastelloy N vs beryllium	5.2 mil diffusion zone.
Chromium-plated Hastelloy C vs beryllium	6.3 mil diffusion zone.
Titanium vs beryllium	0.3 mil diffusion zone.
Titanium vs Hastelloy N	0.6 mil diffusion zone.
Titanium vs Hastelloy C	2.2 mil diffusion zone
Chromized Hastelloy C vs beryllium	No reaction

(1) Beryllium vs Chromized Hastelloy C

This compatibility couple was tested in flowing NaK-78 at 1300°F to simulate the normal operating environment encountered by the internal radial reflector and the inside of the core tank of the SNAP 10A/2 reactor. Pieces of beryllium and chromized Hastelloy C were bound together with a stainless steel machine screw. The sample hung, along with several other compatibility samples involving several different materials, in the hot leg of an Inconel NaK thermal convection loop.[13] The hot leg of the loop was maintained at 1300°F for the test, and the NaK was continuously cold trapped at about 70°F. After the test had operated for 1000 hr, the samples were removed and washed with butyl alcohol. Microscopic examination of the interface beryllium and chromized Hastelloy C did not reveal (Figure 6) any evidence of a reaction.



UNETCHED 300X 1966-3A

a. Control Specimen

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b. Specimens Run for 1000 hr in 1300°F NaK

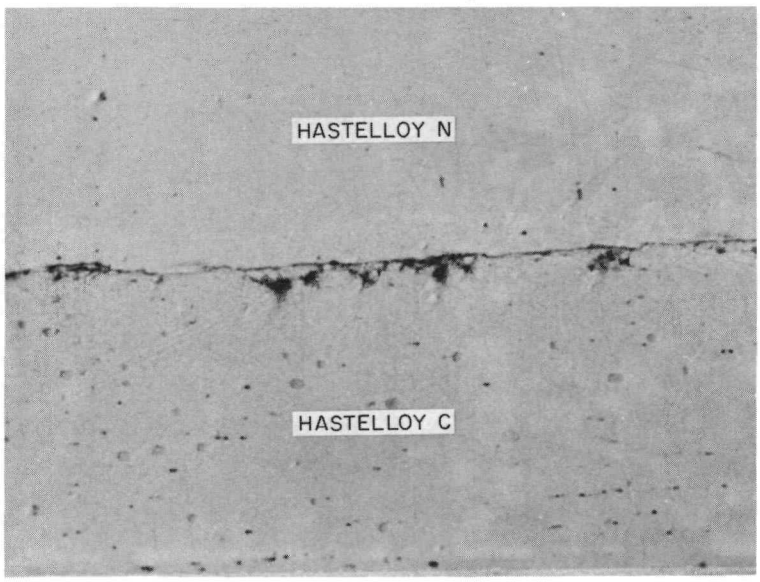
Figure 6. Chromized Hastelloy C – Beryllium Interface

(2) Hastelloy N vs Hastelloy C

In this test, samples of the two materials were placed in intimate contact and mounted in a stainless steel holder. The samples were then sealed in a stainless steel capsule containing an argon-helium atmosphere. The capsule was heated at 1300°F for 1000 hr in a muffle furnace. After the test was concluded, the samples were examined using metallographic techniques, and it was found that no observable reaction had taken place (Figure 7). These component materials operate in NaK during the reactor operation, but this test was performed in an inert atmosphere because it was desired to know the compatibility of these materials only as a function of temperature and intimate contact.

(3) Nickel vs Type 316 Stainless Steel

Since it was desired to determine the compatibility of these two materials, samples mounted in a stainless steel holder were heated in an argon-helium atmosphere at 1300°F for 1000 hr. As is shown in Figure 8, a reaction between the samples had occurred.



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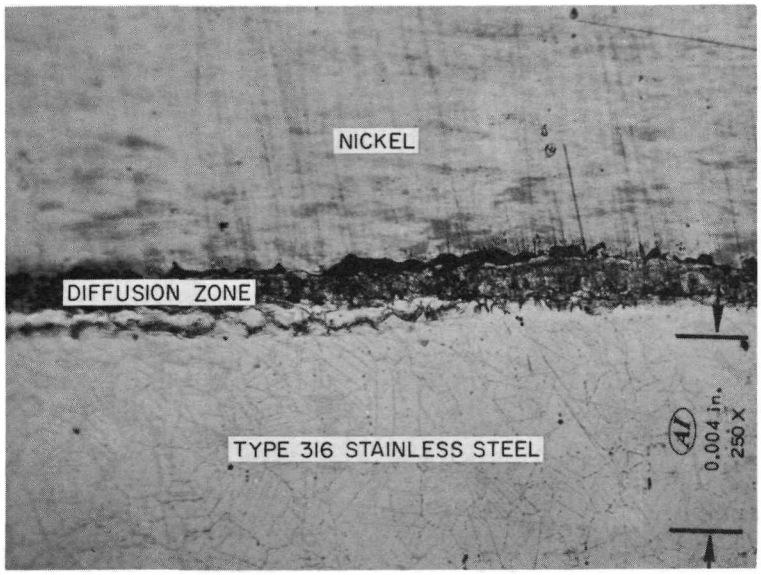
2701-3-1

Figure 7. Hastelloy N vs Hastelloy C
After 1000 hr at 1300°F



ETCH (5% H_2SO_4) 250X
2111-1B

Figure 8. Nickel vs Type 316 Stainless Steel
After 1000 hr at 1300°F

(4) Beryllium Oxide vs Nickel

These two materials were placed in intimate contact in a stainless steel holder and sealed in a stainless steel capsule containing an argon-helium atmosphere. The capsule was heated for 1000 hr at 1300°F in a muffle furnace. Microscopic examination of the interface between the beryllium oxide and nickel samples revealed (Figure 9) that a reaction had not taken place as a result of the test.

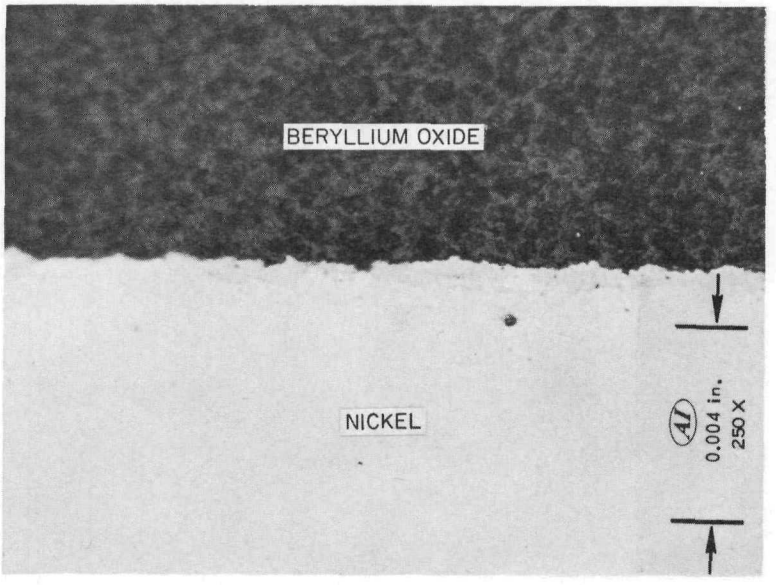


Figure 9. Beryllium Oxide vs Nickel
After 1000 hr at 1300°F

(5) Beryllium Oxide vs Type 347 Stainless Steel

The test on these two materials was performed identically to the previously described test, and the samples were found to be compatible. (Figure 10).

b. Grid Plate Materials

Various grid plate materials have been suggested for use in the SNAP reactors with Hastelloy C and nickel receiving the most serious consideration. Compatibility tests were performed to determine whether these materials would react with the Type 316 stainless steel core tank material. After 1000 hr at 1300°F in a sealed capsule containing an argon-helium atmosphere, samples of

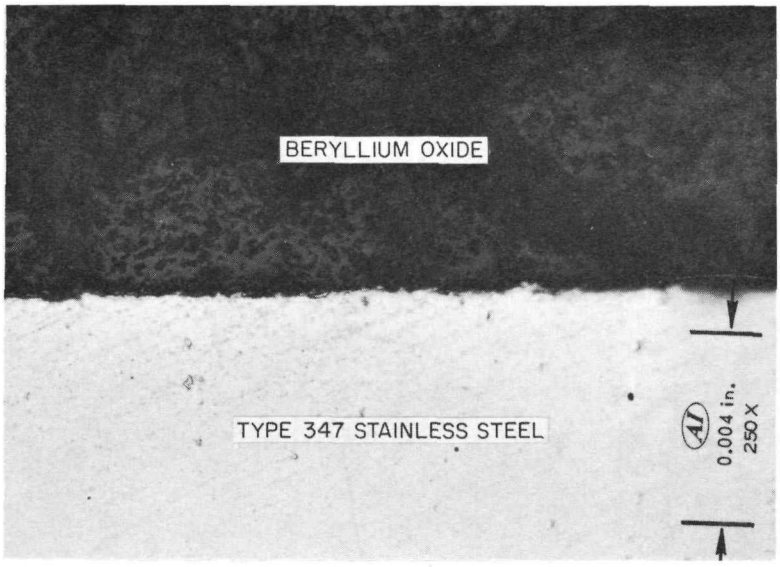


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Figure 10. Beryllium Oxide vs Type 347 Stainless Steel After 1000 hr at 1300°F

nickel vs Type 316 stainless steel were found to have reacted, as indicated previously, while similar samples of Hastelloy C vs Type 316 stainless steel were unreacted (Figures 8 and 11).

c. Fuel Element Cladding Material

Hastelloy N, the fuel element cladding material for both the SNAP reactors, was tested in contact with the Hastelloy C core tank material for 1000 hr at 1300°F. The test of Hastelloy N vs Hastelloy C has been described previously in this section.

d. Discussion

The results of the tests indicate there is a compatibility problem associated with contact of the nickel grid plate and the Type 316 stainless steel core tank of the SNAP reactors. A realistic assessment of the magnitude of this problem would require further study.

2. Other Core Component Materials in Contact With the Internal Radial Reflector

As previously stated, the internal radial reflectors of the SNAP reactors come in contact with the grid plates and a few of the fuel elements. Tests were performed to determine the compatibility of several of the materials considered

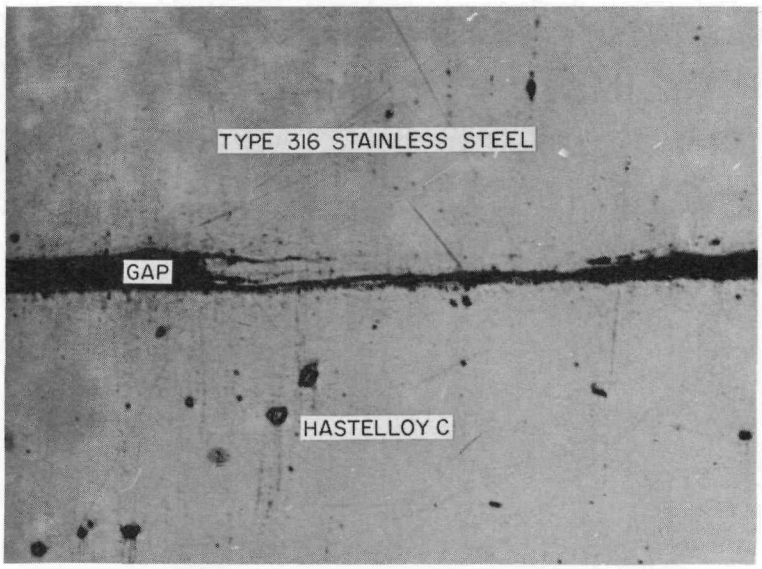

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Figure 11. Hastelloy C vs Type 316 Stainless Steel After 1000 hr at 1300°F

for these two components. These materials will all operate in a hot NaK environment in the SNAP reactors, but not all the tests were performed in this medium, since it was, in some cases, desired to know only the effects of elevated temperature and intimate contact.

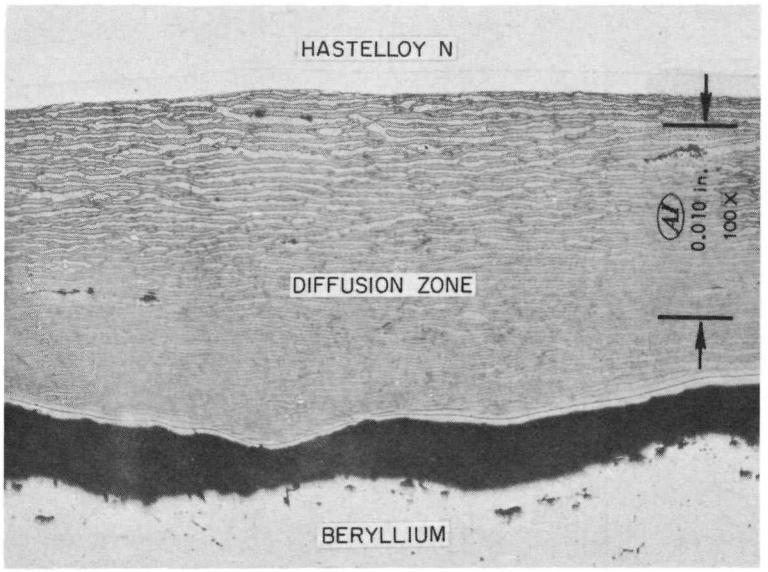
a. Fuel Element Cladding Material

Compatibility tests were performed to determine whether the fuel element cladding material, Hastelloy N, would react with the SNAP 10A/2 beryllium internal radial reflector material, and the cladding materials for the SNAP 8 beryllium oxide internal radial reflector, Type 347 stainless steel or nickel. Both chromized and bare Hastelloy N were used in the tests, and the tests were performed both in flowing NaK and in an inert atmosphere. The tests are briefly described below.

(1) Hastelloy N vs Beryllium

Samples of Hastelloy N and beryllium were bound together with stainless steel wire and tested for 1000 hr at 1200 and 1300°F in a NaK thermal convection loop.[14] Metallographic examination of the interface after test revealed that a reaction had taken place. The reaction zone was measured and found to be from 0.001 to 0.002 in. in thickness.

Further tests were performed on this material couple at 1300°F in an argon-helium atmosphere. In this test, the samples were placed in intimate contact in a molybdenum holder and sealed in a capsule containing about one-third atmosphere of the inert gas mixture. The test was concluded after 1003 hr, and the samples were examined using metallographic techniques. A very large diffusion zone was observed to have resulted from the test, and its thickness was measured to be about 0.015-in. A photomicrograph of the interface, after testing, is shown in Figure 12. As is shown, the diffusion zone is multiphased and probably contains several intermetallic compounds such as NiBe and Ni_5Be_{21}.

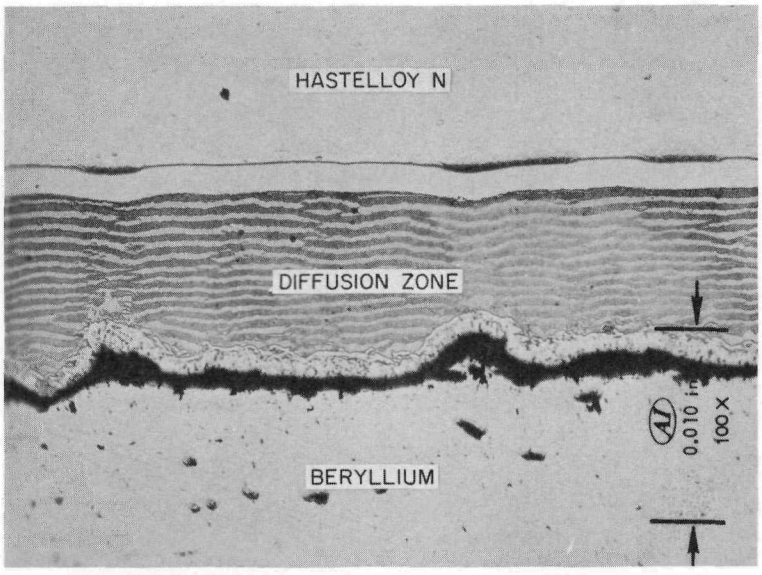


ETCH (5% Oxalic Acid Polish) 100X
2701-51

Figure 12. Beryllium vs Hastelloy N
After 1003 hr at 1300°F

Chromized Hastelloy N was tested in contact with beryllium using the same procedure as that for the test just mentioned. The diffusion zone which resulted from this test was about 0.010-in. thick (Figure 13). This suggests that the chromized layer tends to reduce somewhat the extent of reaction between Hastelloy N and beryllium under these particular conditions.

A fair comparison cannot be made between the tests in NaK and the argon-helium atmosphere because the two different binding devices provided different degrees of contact. The molybdenum holder applied a significant initial pressure to the specimen interfaces at temperature, due to differential thermal expansion, while the stainless steel wire did not.



ETCH (5% Oxalic Acid Polish) 100X
2701-52

Figure 13. Beryllium vs Chromized Hastelloy N After 1003 hr at 1300°F

(2) Hastelloy N vs Type 347 Stainless Steel

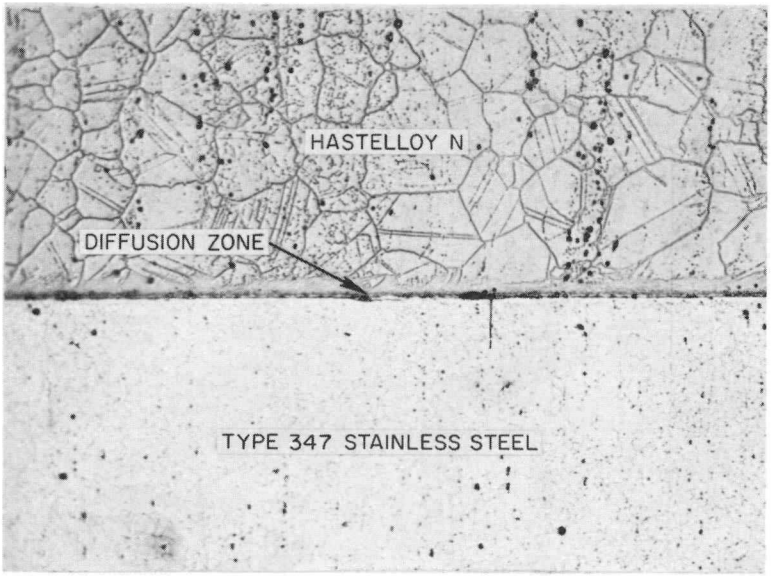
Pieces of Hastelloy N and Type 347 stainless steel bound together in a stainless steel holder were heated at 1300°F for 1000 hr in a sealed capsule containing an argon-helium atmosphere. Metallographic examination of the interface between the two materials after testing revealed (Figure 14) the presence of a diffusion zone about 0.001 in. thick.

(3) Hastelloy N vs Nickel

Two samples involving this material couple were tested using the same test procedures described in the previous section. Metallographic examination showed that a diffusion-type reaction had taken place during the test. The photomicrograph of the interface showing the 0.001 in. thick diffusion zone, is shown in Figure 15.

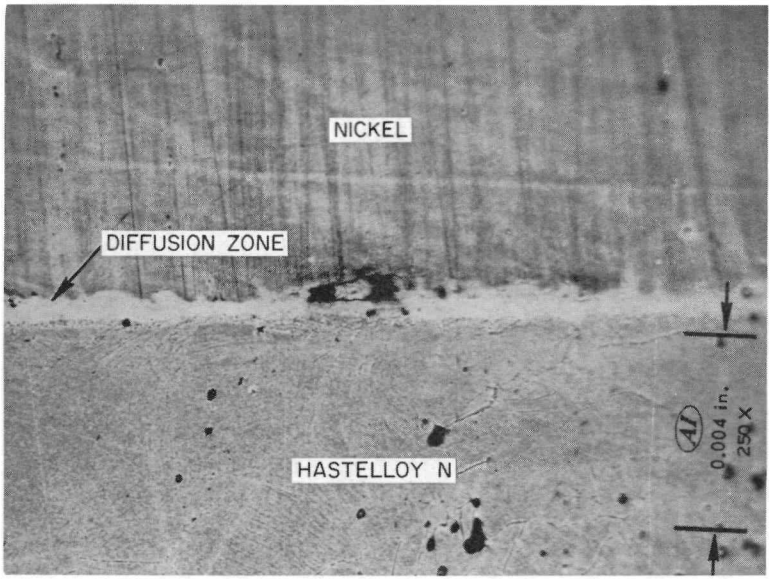
b. Grid Plate Material

Tests were performed to determine the compatibility of Hastelloy N, an internal radial reflector cladding material, and Hastelloy C, a grid plate material, at 1300°F for 1000 hr, and no reaction was observed. The test was previously described in this section.



ETCH (5% H_2SO_4) 75X
2154-1-1

Figure 14. Hastelloy N vs Type 347 Stainless Steel After 1000 hr at 1300°F



ETCH (5% H_2SO_4) 250X
2111-1A

Figure 15. Nickel vs Hastelloy N After 1000 hr at 1300°F

c. Discussion

The incompatibility observed between the Hastelloy N fuel element cladding material and the bare beryllium internal radial reflector may be indicative of a serious compatibility problem in the SNAP 2 reactor. If these components are maintained in intimate contact for long periods of time, perhaps hundreds or thousands of hours, the properties of the component materials will probably undergo significant physical and metallurgical changes which might result in a premature reactor shutdown. Further study and analysis will be required to obtain a realistic picture of the problem. A similar, but less severe situation prevails in the Hastelloy N fuel cladding, Type 347 stainless steel or nickel reflector cladding compatibility problems.

B. REACTION BARRIER MATERIALS FOR USE BETWEEN FUEL ELEMENT CLADDING, BERYLLIUM INTERNAL RADIAL REFLECTOR, AND CORE TANK

Due to the previously mentioned reaction between the Hastelloy N fuel cladding material and the beryllium internal radial reflector material, investigations were made to obtain a reaction barrier material that could be placed between these two component materials and render them compatible. Hastelloy C core tank material was also considered to be incompatible with beryllium, since it is also a nickel base alloy* and barrier materials were considered for use between the core tank and the internal radial reflectors. Studies[13] were performed to determine the effectiveness of an electroplated chromium layer† and titanium metal as reaction barriers. Also, chromizing the surfaces of Hastelloy C was investigated as a means of preventing reaction. The chromium electroplate was applied to the beryllium and both the Hastelloys. A single compatibility test was performed in 1300°F flowing NaK for 1000 hr to determine whether these materials could be considered for use as reaction barriers inside the SNAP 10A/2 reactors.

One sample each, having the following interfaces, were tested:

1) Chromium electroplated on beryllium and bare Hastelloy C

*Hastelloy C is nominally 54-Ni, 17-Mo, 15-Cr, 5-Fe, and 4-W while Hastelloy N is 70-Ni, 17-Mo, 7-Cr, and 5-Fe.

†This series of tests was performed before it was established that chromium is not an effective reaction barrier between Hastelloy N and beryllium, as described in this section.

2) Chromium electroplated on beryllium and bare Hastelloy N

3) Titanium metal, beryllium, and chromized Hastelloy C

4) Bare Hastelloy C, titanium, bare Hastelloy N

5) Chromium electroplated on Hastelloy N and bare beryllium chromium electroplated on Hastelloy C.

Metallographic examination of tested and untested interfaces revealed that all the couples were incompatible except chromized Hastelloy C and beryllium couple. It is suspected that the apparent compatibility of these materials resulted from loss of interfacial contact during the test. A summary of the observations made on the tested samples is given in Table 6.

TABLE 6

RESULTS OF COMPATIBILITY TESTS
ON REACTION BARRIER MATERIALS

Interface	Observation
Chromium-plated beryllium vs Hastelloy N	0.8-mil diffusion zone. Plate separated from beryllium
Chromium-plated beryllium vs Hastelloy C	0.5-mil diffusion zone. Plate separated from beryllium.
Chromium-plated Hastelloy N vs beryllium	5.2-mil diffusion zone.
Chromium-plated Hastelloy C vs beryllium	6.3-mil diffusion zone.
Titanium vs beryllium	0.3-mil diffusion zone.
Titanium vs Hastelloy N	0.6-mil diffusion zone.
Titanium vs Hastelloy C	2.2-mil diffusion zone.
Chromized Hastelloy C vs beryllium	No reaction.

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IV. CONCLUSION

Although the tests described in this report are somewhat cursory in nature, their results can be used to indicate the mutual compatibility of the several contacting component materials, used in the SNAP 10A/2 and SNAP 8 reactors. More exact assessments of compatibility can only be obtained by further testing.

Since true operational environments are very difficult to simulate, one of the most fruitful studies for confirming laboratory tests on the compatibility of the core materials, is a detailed examination of the various components of previously operated experimental or developmental SNAP reactors. Studies of this type have been initiated on the SNAP 2 Developmental Reactor core.[15] Two of the fuel elements which operated in this reactor, showed definite signs of reaction where they had been in contact with the beryllium internal reflectors, confirming one of the findings of this report. Further work of this type is strongly recommended.

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REFERENCES

1. J. K. Balkwill, "Compatibility of SNAP Fuel and Clad Materials," NAA-SR-5413 (October 15, 1960)

2. M. W. Hulin and J. Beall, Ed., "Preliminary Results of the SNAP 2 Experimental Reactor," NAA-SR-5991 (April 1, 1961)

3. L. B. Lundberg, "Compatibility of Some SNAP 2/10A Fuel Element Materials," NAA-SR-MEMO-7217 (March 1962)

4. J. G. Beach and C. L. Faust, "Electroplating on Beryllium," BMI-732 (April 1, 1952)

5. C. E. Johnson, "Liquid Metal Thermal Bond Between Fuel and Cladding," NAA-SR-MEMO-4176 (July 24, 1959)

6. J. A. Curley and A. W. Sommer, "A compatibility Study Between the Lead-Bismuth Eutectic Alloy and (Zr, U)Hx at 1500°F," NAA-SR-MEMO-5170 (April 12, 1960)

7. W. K. McCarty, "Compatibility of Fuel With Lead," NAA-SR-MEMO-2315 (November 20, 1957)

8. J. A. Curley, unpublished data (April, 1960)

9. J. K. Balkwill, Personal Communication (February 27, 1963)

10. H. N. Rosenberg, "SNAP 8 Experimental Reactor Preliminary Charter" (unpublished Inter-Office Letter to D. J. Cockeram, November 23, 1960)

11. C. E. Johnson, "SNAP 8 Design Data Sheet," NAA-SR-MEMO-5566 (August 5, 1960)

12. W. E. Seroy, "S8ER Core Mechanical Design," NAA-SR-MEMO-6678 (October 10, 1961)

13. L. B. Lundbert, "An Evaluation of the Compatibility of Some of the SNAP 2 Core Materials," Atomics International Technical Data Record No. 5952 (December 9, 1960)

14. W. F. Dennison, "Evaluation of a Hastelloy N-Beryllium-347 Stainless Steel-NaK Compatibility Test," Atomics International Technical Data Record No. 5708 (September 23, 1960)

15. J. Susnir and T. Harman, Ed., "SNAP 2/10A Reactor Quarterly Progress Report, January-March 1963," NAA-SR-8394 (June 7, 1963)