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# DEVELOPMENT OF URANIUM NITRIDE-STAINLESS STEEL DISPERSION FUEL ELEMENTS

by

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# DISCLAIMER

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# DEVELOPMENT OF URANIUM-NITRIDE STAINLESS STEEL DISPERSION FUEL ELEMENTS

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*In research in support of the GCRE, procedures for the fabrication of stainless steel-clad flat-plate fuel elements having a core of 28 w/o UN dispersed in an iron-18 w/o chromium-14 w/o nickel-2.5 w/o molybdenum matrix were developed.*

*The preparation of UN and its compatibility with the components of the matrix alloy were studied. The UN for the program was prepared by nitriding uranium metal at 850 C and then dissociating the* $U_2N_3$ *produced to UN in a vacuum at 1300 C. In compatibility studies, UN reacted with nickel alone at 1800 F, but no reaction with nickel was observed when the other matrix components were also present.*

*The effects of fabricational variables were evaluated by metallographic examination, longitudinal and transverse tensile tests, bend tests, and corrosion tests. Studies indicated that minus 200 plus 325-mesh UN dispersed in a minus 325-mesh elemental-powder matrix rolled green with a 30 per cent initial reduction at 2100 F and then annealed 3 hr at 2300 F produced the best fuel core.*

## INTRODUCTION

A previous study at Battelle(1) concerned with the fabrication and compatibility of several uranium compounds (UN, UC, $U_3Si$, $U_6Ni$, and $U_2Ti$) dispersed in either Zircaloy-2 or stainless steel indicated that a dispersion of UN in stainless steel might possess considerable potential as a high-temperature fuel-element material. Because of the high density and uranium content of UN, it can be substituted for $UO_2$ in a dispersion element and occupy considerably less space (approximately 30 per cent) at an equivalent uranium loading. While this in itself should lead to superior mechanical properties, it appeared from the study that UN also had less tendency for particle fragmentation during fabrication as compared with $UO_2$. These data, coupled with the favorable results of a preliminary compatibility study made in the same investigation, prompted the more detailed development study of UN-stainless steel dispersions reported here.

At the conclusion of this present program, more than 100 natural and enriched stainless steel-UN fuel plates were fabricated by the optimum procedures developed to the dimensions approximating the GCRE-I production plates (stainless steel-$UO_2$). These elements were then formed and assembled into concentric-tube subassemblies by Aerojet-General Nucleonics. The subassemblies prepared with natural elements have been subjected to various out-of-pile tests at AGN, while a subassembly made with enriched elements is scheduled to be tested in the BRR in-pile-loop facility.

Concurrently with these studies, ten fuel specimens were prepared, encapsulated, and irradiated at high temperatures at the MTR to burnups of between 2 and 15 per cent of the uranium-235. Results of these tests will be reported by Battelle in the near future.

## PREPARATION OF UN

In general practice, UN is prepared by heating uranium metal in pure nitrogen to form $U_2N_3$ and then decomposing the $U_2N_3$ to form a stable UN compound by heating in vacuum. In the present program, UN was prepared initially in a hydriding apparatus modified to use nitrogen. Clean nibblets or thin plates of uranium metal were placed in a Vycor reaction tube, the tube was sealed to the system, and then the apparatus was evacuated to a pressure of $0.1 \times 10^{-3}$ mm of mercury or less. The uranium was then resistance heated to 850 C, and nitrogen was introduced to form $U_2N_3$. When the material ceased to react with nitrogen, as determined by an open-end monometer, the tube was cooled and removed to an argon-filled dry box. Here the $U_2N_3$ was transferred to a clean BeO crucible.

The crucible containing the $U_2N_3$ was placed in a vacuum-induction furnace and heated to a temperature of 1300 C, which was maintained until a pressure of less than $5 \times 10^{-3}$ mm of mercury was obtained. This operation dissociates the $U_2N_3$ to UN and nitrogen gas, which is evacuated. After cooling, the UN was transferred back to the dry box for storage.

A good-quality UN powder was produced by this process by carefully selecting the starting materials and taking adequate precautions in transferring the powder between operations; therefore, another apparatus (Figure 1) was made to increase the production of UN. This apparatus was designed to produce two batches concurrently or to nitride and dissociate independently. The manifold is made of glass tubing connected to two 7/8 in. ID Mullite reaction tubes, each having a capacity of 175 g of uranium in metal. Heating for nitriding and dissociating is done with two Kanthal-element tube furnaces. The vacuum system consists of a three-stage mercury-diffusion pump, a dry ice-acetone cold trap, and a single-stage vacuum forepump.

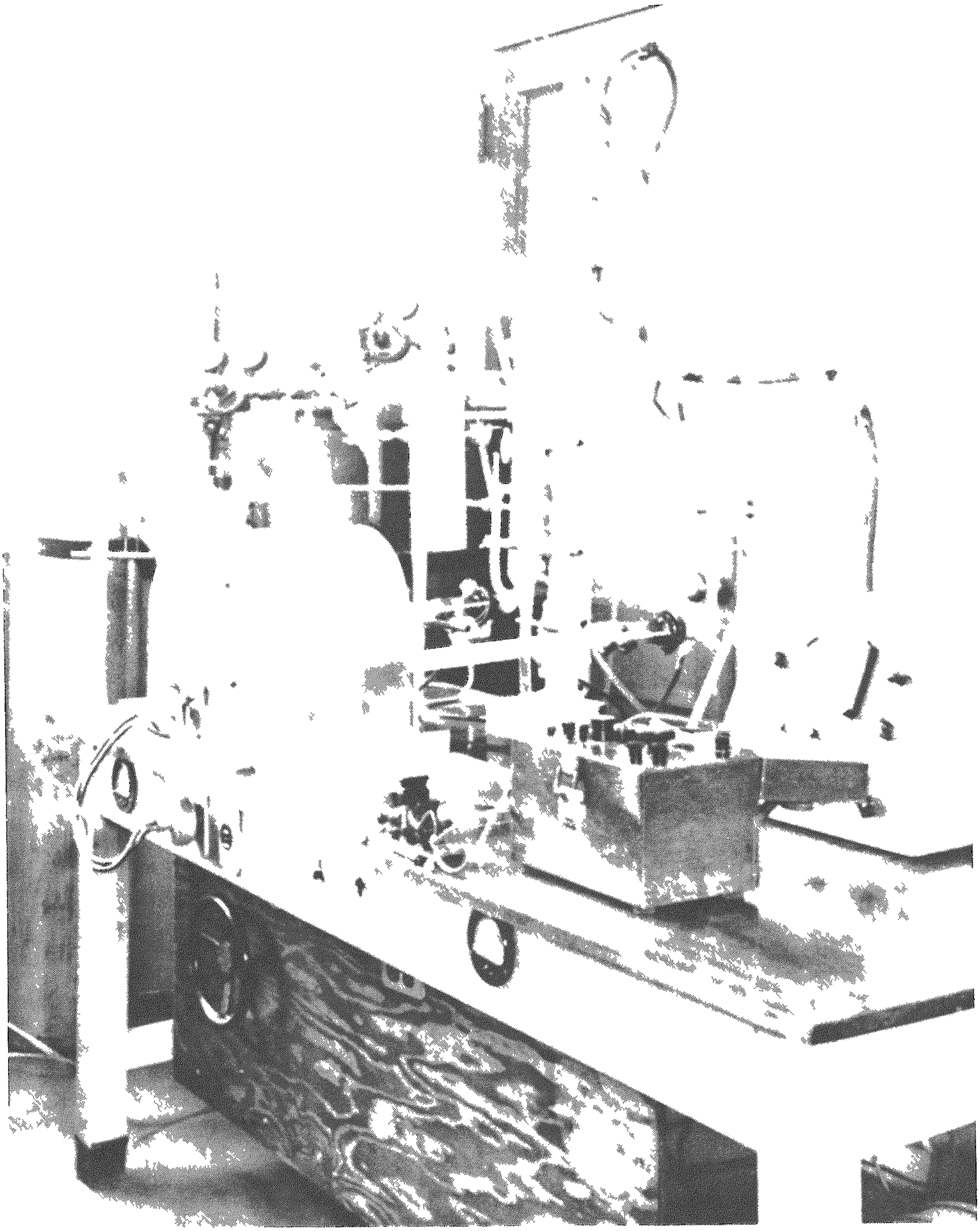
The procedure adopted for producing UN with the nitriding apparatus shown in Figure 1 is as follows:

(1) Nibble uranium metal into small pieces (1/4 by 1/4 by 1/8 in.) or roll to 0.050 in. and shear into small plates.

(2) Clean the metal by pickling in a one-to-one $HNO_2$-$H_2O$ solution, rinse in acetone, and air dry.

(3) Place the clean metal in Mullite reaction tube, evacuate, and then heat to 850 C in vacuum.

(4) Hold the temperature at 850 C and add Matheson prepurified nitrogen to form $U_2N_3$. The reaction may be determined by use of an open-end manometer.



N57461

FIGURE 1. URANIUM NITRIDING APPARATUS

(5) Dissociate the $U_2N_3$ to UN in free nitrogen by heating to 1300 C. Evacuate the nitrogen gas as it dissociates until a pressure of $<5 \times 10^{-3}$ mm of mercury is obtained at 1300 C.

(6) Cool the reaction tube, introduce an argon atmosphere, and transfer the sealed reaction tube to an argon-atmosphere dry box for sizing and storage.

X-ray diffraction analyses of the powder produced by this process showed UN and no remaining traces of $U_2N_3$. The measured UN lattice constant was 4.889 A. This compares very well with the value of 4.880 KX or 4.889 A reported by Rundel(2) and co-workers.

In Table 1, the gas analyses of several batches of UN powder are listed. As experience was gained with the equipment, the oxygen analysis was rather consistently maintained below 0.1 w/o.

TABLE 1. GAS ANALYSES OF UN POWDER PREPARED FOR PROGRAM

| | Analyzed Composition, w/o | |
|---|---|---|
| Batch | Nitrogen | Oxygen |
| | Natural UN | |
| 1 | 5.55 | 0.130 |
| 2 | 5.51 | 0.054 |
| 3 | 5.47 | 0.120 |
| 4 | 5.52 | 0.097 |
| 9 | 5.49 | -- |
| 10 | 5.41 | 0.084 |
| 11 | 5.54 | 0.088 |
| 12 | 5.53 | 0.078 |
| | Enriched UN | |
| E6L | 5.40 | 0.160 |
| E12L | 5.46 | 0.210 |
| E17L | 5.50 | 0.200 |

Although UN is not pyrophoric, it is readily contaminated by oxygen at room temperature. For example, early in the program, a sample of UN powder containing 1070 ppm oxygen exposed to air for a week increased its oxygen content to 2560 ppm. Thus, it is important to handle this material in an inert atmosphere.

The final particle size of the UN may be influenced by varying the processing procedure. A very fine (minus 325 mesh) powder may be produced by initially powdering the metal by the hydriding technique before nitriding. Direct nitriding of uranium metal nibblets results in a somewhat coarser powder (minus 200 plus 325 mesh). Further increases in final particle size may be obtained by special heat treatments of the uranium metal prior to nitriding.

# FABRICATION OF FUEL PLATES

## Materials

Since the primary interest was in stainless steel matrices at the time of this investigation, all UN dispersion fuel elements were made with stainless steel. A flat-plate-type element consisting of a powder-dispersion core rolled in a stainless steel picture-frame pack was used for fabrication studies.

On the basis of the GCRE reference design, the investigations were chiefly concerned with dispersions of 28 w/o UN in Type 318 stainless steel; however, in a previous investigation(3) loadings as high as 43.7 w/o (30 volume per cent) UN were successfully made and Types 302B and 347 stainless steel matrices were also used.

Due to the tendency of UN to pick up oxygen, all storage, screening, weighing, and mixing was done in an argon-filled dry box. Powder mixtures for pressing were removed from the dry box in a sealed jar and not opened until just before pressing. This procedure helped keep oxygen contamination to a minimum.

## Compatibility Studies

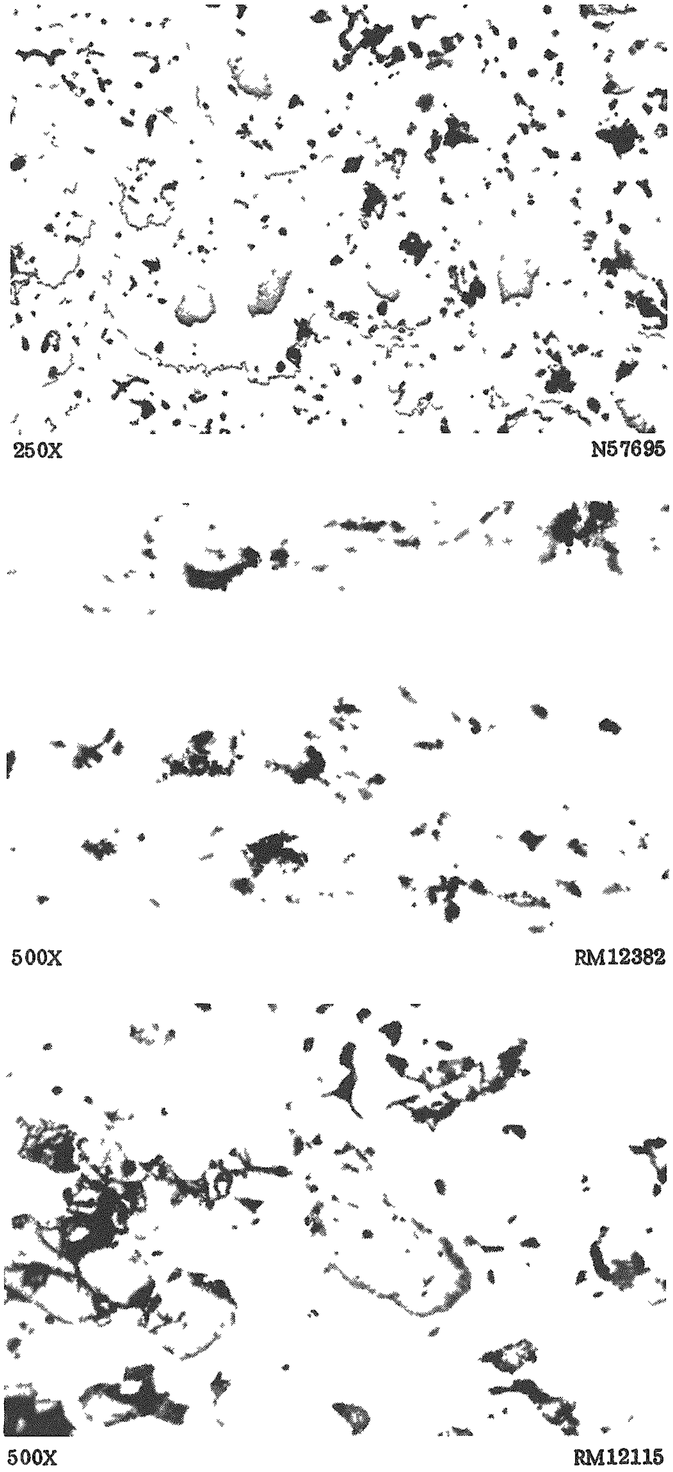
The major constituents of stainless steel (chromium, iron, and nickel) were tested for their compatibility with UN. Cold-pressed compacts were given a series of heat treatments and then a metallographic examination to reveal any evidence of reaction. The results are listed in Table 2.

TABLE 2. COMPATIBILITY OF UN WITH STAINLESS STEEL CONSTITUENTS

| Material[a] | Sintering Temperature, F | Sintering Atmosphere | Results |
|---|---|---|---|
| Nickel | 2200 | Vacuum | Melted |
| Nickel | 2000 | Vacuum | Melted |
| Nickel | 1800 | Vacuum | Reaction |
| Chromium | 2300 | Vacuum | No reaction |
| Chromium | 2300 | Hydrogen | No reaction |
| Iron | 2300 | Hydrogen | No reaction |

(a) Carbonyl nickel, and chromium and iron made by the electrolytic process were used in this study.

There was a definite reaction between the UN and nickel (Figure 2a) at 1800 F, while melting occurred at higher temperatures. Both chromium (Figure 2b) and iron (Figure 2c) showed no reaction with UN when heat treated at 2300 F; however, UN did have a tendency to getter oxygen from the metal powders. Oxygen reactions appear (Figure 2c) as dark-gray areas around the edges of the UN particles. The electrolytic iron powder, as received, contained 6000 ppm oxygen.



a. UN Dispersed in Nickel and Vacuum Sintered 4 Hr at 1800 F

Extensive reaction zones (light gray) surround the dark gray particles of UN.

b. UN Dispersed in Chromium and Sintered in Hydrogen 2 Hr at 2300 F

No reaction around the dark-gray particles of UN is evident.

c. UN Dispersed in Iron and Sintered in Hydrogen 2 Hr at 2300 F

Some oxygen reaction at the surface of the dark-gray particles of UN is evident. No reaction was observed when low-oxygen iron was employed as the matrix.

FIGURE 2. TYPICAL MICROSTRUCTURES OBTAINED IN THE STUDY OF THE COMPATIBILITY OF UN WITH NICKEL, CHROMIUM, AND IRON

Although there was some concern about the reaction during high-temperature fabrication of UN with nickel in an elemental powder matrix, no such reaction has been observed in either sintered or hot-rolled materials. Apparently, the initial concentration of nickel is sufficiently low or the solution of nickel progresses rapidly enough at lower temperatures to prevent observable reaction.

## Sintering Studies

Past experience with $UO_2$ dispersions(3) shows that a core having a high per cent of theoretical density prior to rolling tends to produce a superior rolled structure by controlling the stringering effect. Therefore, the cores are generally densified by sintering and coining before roll cladding.

To determine the amount of core densification obtainable by sintering and coining, UN dispersions in both elemental and in prealloyed Type 318 stainless steel powder were prepared. The results in Table 3 show that compacts with an elemental-powder-core matrix pressed to a higher green density than compacts with a prealloyed matrix; however, after sintering and coining the prealloyed compacts may have comparable densities, depending upon the sintering temperature.

TABLE 3. EFFECTS ON DENSITY OF SINTERING AND COINING 30 w/o UN IN TYPE 318 STAINLESS STEEL

| Matrix | Green Density, per cent of theoretical(a) | Sintering Conditions Temperature, F | Time, hr | Density, per cent of theoretical Sintered | Coined(a) |
|---|---|---|---|---|---|
| Elemental(b) | 78.3 | 2000 | 2 | 75.1 | 82.5 |
| Elemental(b) | 78.5 | 2100 | 2 | 75.3 | 82.6 |
| Elemental(b) | 78.5 | 2300 | 2 | 80.3 | 86.0 |
| Prealloyed | 71.5 | 2000 | 2 | 68.2 | 79.0 |
| Prealloyed | 71.7 | 2100 | 2 | 67.0 | 77.2 |
| Prealloyed | 70.5 | 2300 | 2 | 82.0 | 86.1 |

(a) A 50-tsi pressure was used on the die punch.
(b) The elemental powder blend was 18-14-2.5 chromium-nickel-molybdenum.

It was also noted that UN-stainless steel compacts rolled green generally produced a structure comparable with those which were sintered and coined before rolling. This effect is particularly true in the cases of cores prepared with elemental-powder stainless steel matrices. All additional work was therefore restricted to green-rolled compacts to take advantage of the reduction in costs resulting from eliminating the sintering and coining operations.

## Effects of UN Particle Size and Matrix Materials on Core Structure

Initial rolling studies substantiated indications in earlier work with stainless steel dispersions(1) that improved structures could be obtained when UN is substituted for $UO_2$. A comparison of $UO_2$ and UN-stainless steel dispersions shown in Figure 3 plainly indicates the superiority of the UN dispersion structure by its random dispersion, continuous matrix, and the absence of UN particle fracturing and stringering. Hydrothermal $UO_2$ was used in the oxide dispersion.

A series of cores having UN of various particle sizes dispersed in elemental-powder and prealloyed matrices was roll clad to fuel-plate size. Minus 325-mesh prealloyed Type 318 and elemental iron-18 w/o chromium-14 w/o nickel-2.5 w/o molybdenum stainless steels were used as the matrix material. The following procedure was used in making the experimental fuel plates: the powders were screened, weighed, and blended in a dry box and green pressed at 50 tsi. The green cores were placed in clean Type 318 stainless steel picture-frame packs; the packs were welded closed, degassed, and evacuated at 900 F. Then the packs were hot rolled to a 6:1 reduction in thickness at 2000 F, annealed 2 hr at 2300 F, cold rolled to an 18 per cent reduction in thickness, and flat annealed 1 hr at 2050 F.

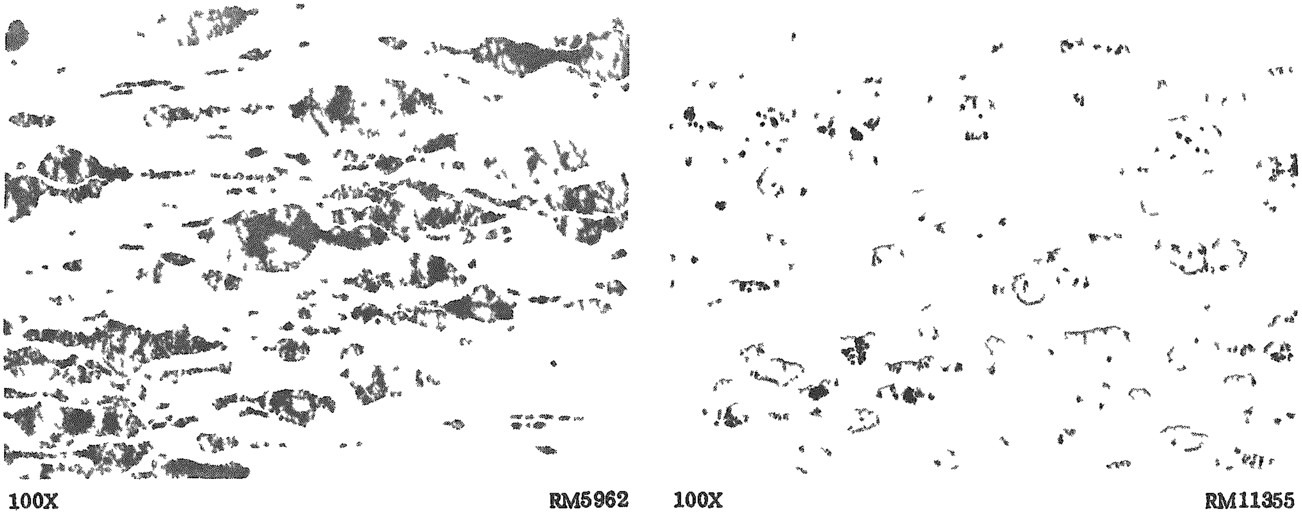
Longitudinal core sections were cut from these fuel plates and mounted and polished for metallographic examination. Typical photomicrographs are shown in Figure 4. It can be seen in the photomicrographs that elemental powder is superior to prealloyed stainless steel as a matrix material in that it results in less stringering of the UN particles. Of the UN particle sizes used, minus 200 plus 270-mesh UN powder gave the best core structure. However, as shown in Figure 4f, utilization of minus 200 plus 325-mesh UN powder produced a satisfactory structure, and, since from the material economics standpoint this was a more practical size range, minus 200 plus 325-mesh UN was employed in subsequent studies. ✓

## Effects of Rolling Temperature and Initial Reduction on Core Structure

The investigation of rolling temperatures was restricted to a range of 2000 to 2200 F. Previous experience has shown that rolling at temperatures below 2000 F reduces the prospects of a good metallurgical bond between the core and cladding, and, in addition, because of the lower ductility of the matrix at the lower temperatures, increases the possibility of UN fracture and stringering during rolling. As rolling temperatures are increased above 2200 F the oxidation of the thin stainless steel cladding becomes excessive even though heating is done in a hydrogen-atmosphere muffle.

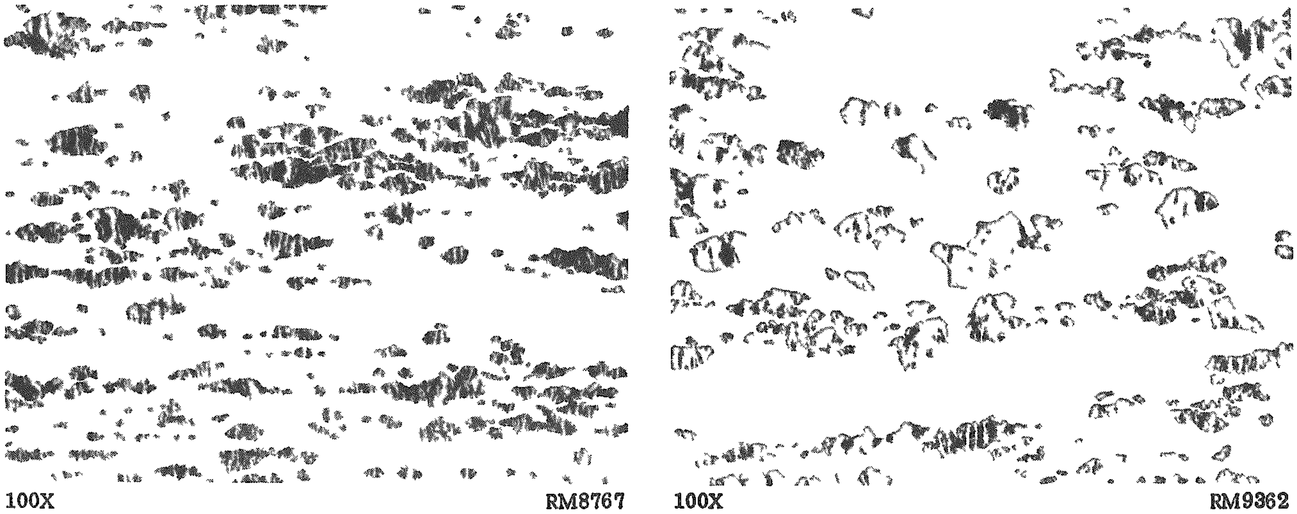
A rolling temperature of 2100 F was selected after roll cladding a series of cores at 2000, 2100, and 2200 F. Higher temperatures result in a slightly improved core structure, but leave a bad fishtail effect at the end of the core. Lower rolling temperatures reduce the end effects, but increase the breakup of the UN.

To determine the most favorable initial reduction at the 2100 F rolling temperature, fuel plates were made with initial hot-rolling passes of 30, 35, 40, and 45 per cent



25 w/o $UO_2$ Dispersed in Elemental Powder and Rolled Green

28 w/o UN Dispersed in Elemental Powder and Rolled Green

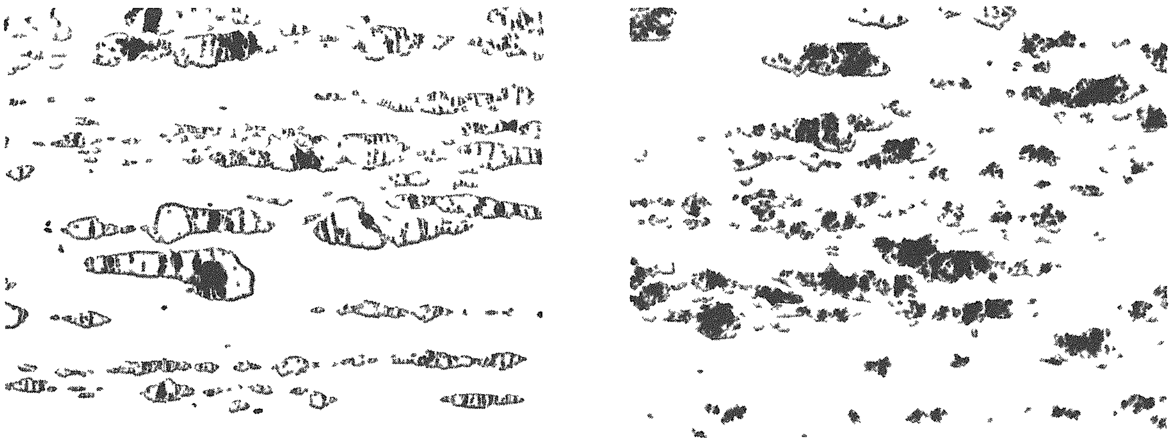


25 w/o $UO_2$ Dispersed in Prealloyed Powder and Sintered and Coined Before Rolling

25 w/o UN Dispersed in Prealloyed Powder and Sintered and Coined Before Rolling

FIGURE 3. TYPICAL LONGITUDINAL SECTIONS OF ROLLED CORES OF $UO_2$ AND UN DISPERSED IN TYPE 318 STAINLESS STEEL

The random dispersion, continuous matrix, and minimum fuel-particle fracturing and stringering of the UN-stainless steel dispersions is clearly evident in the photomicrographs.

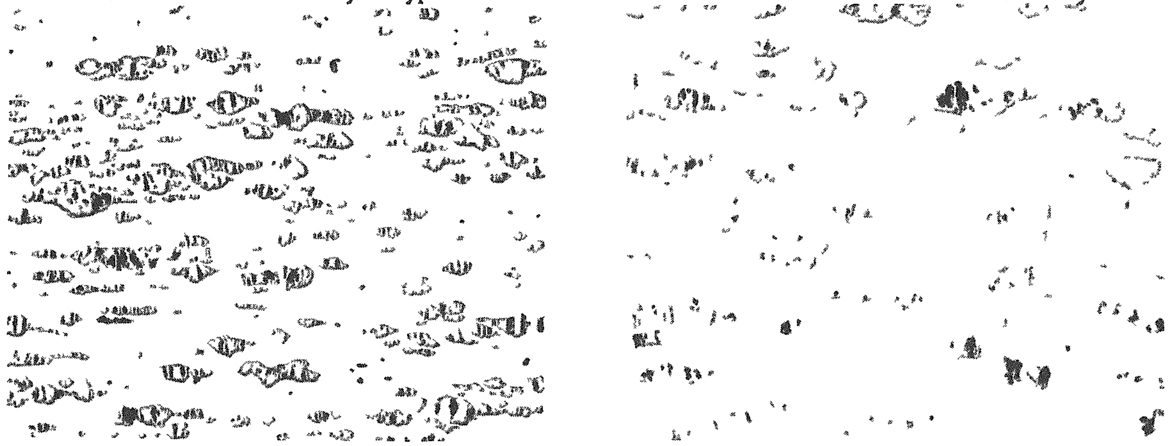


100X RM11091

a. 30 w/o Minus 100 Plus 140-Mesh UN in Minus 325-Mesh Prealloyed Type 318

100X RM11398

b. 28 w/o Minus 100 Plus 200-Mesh UN in Minus 325-Mesh Elemental 18-14-2.5 Alloy

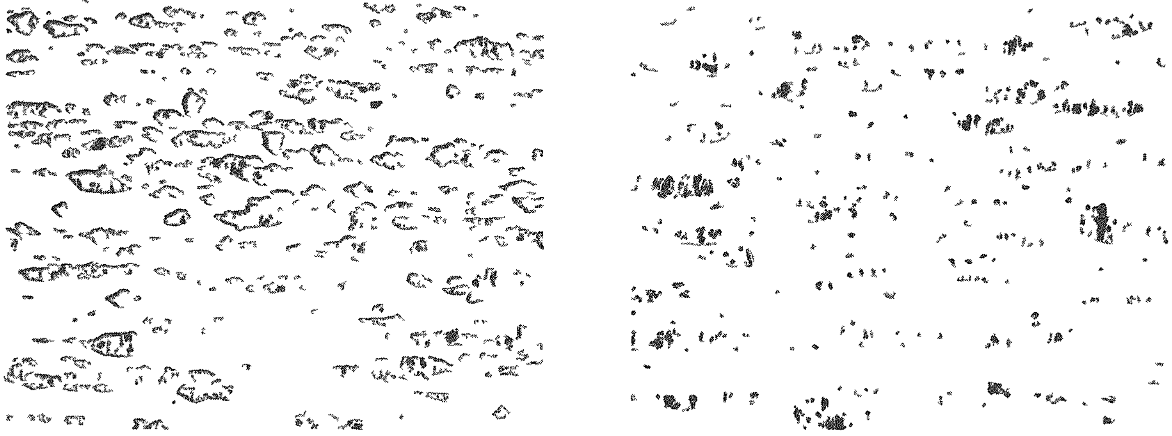


100X RM11103

c. 30 w/o Minus 200 Plus 270-Mesh UN in Minus 325-Mesh Prealloyed Type 318

100X RM11323

d. 28 w/o Minus 200 Plus 270-Mesh UN in Minus 325-Mesh Elemental 18-14-2.5 Alloy



100X RM11087

e. 30 w/o Minus 270 Plus 325-Mesh UN in Minus 325-Mesh Prealloyed Type 318

100X RM11324

f. 28 w/o Minus 200 Plus 325-Mesh UN in Minus 325-Mesh Elemental 18-14-2.5 Alloy

FIGURE 4. ROLLED CORE STRUCTURES RESULTING FROM VARYING UN PARTICLE SIZE AND MATRIX MATERIAL

Minus 200 plus 270-mesh UN dispersed in the elemental matrix showed the least stringering and fracturing. Regardless of the particle size, more alignment and stringering occurred in cores prepared with prealloyed matrices. All specimens were pressed green at 50 tsi, rolled 6 to 1 at 2000 F, annealed 2 hr at 2300 F, cold rolled 18 per cent, and flat annealed 1 hr at 2050 F.

reduction in thickness in one pass followed by reductions of 20 per cent per pass. The plates were then cold rolled a total of 18 per cent reduction in thickness after annealing at 2300 F. This cold reduction is used to give a good surface finish and dimensional control.

Metallographic examinations of longitudinal core sections cut from elemental-matrix fuel plates given initial reductions of 30, 35, 40, and 45 per cent showed that there was an increasing tendency for stringers to form as the initial reduction was increased. This effect can be seen in the photomicrographs in Figure 5. On the basis of this study an initial reduction of 30 per cent appeared to produce the best rolled structure.

### Annealing

A 3-hr anneal at 2300 F between hot rolling and cold rolling was found necessary to assure solution of the elemental-matrix constituents. After cold rolling, a 1-hr anneal at 2050 F between massive plates was used to stress relieve and flatten the fuel plates. Both anneals were given in a dry-hydrogen atmosphere.

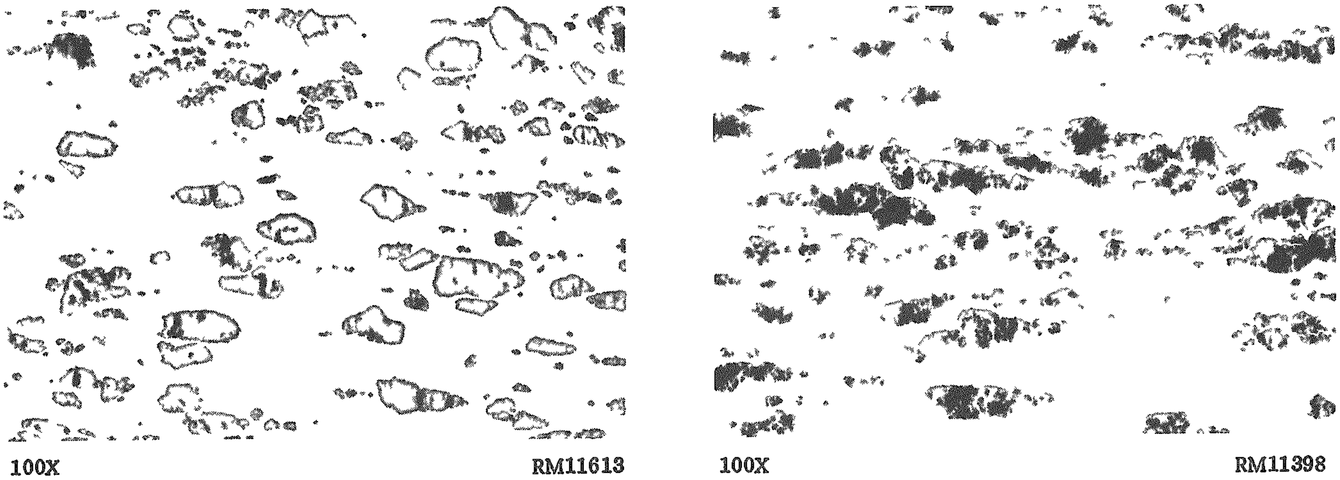
## MECHANICAL PROPERTIES OF FUEL PLATES

A parallel program(4) of mechanical-property studies was in progress at the same time the experimental fuel plates were being examined metallographically for core structure properties. Plates were checked for bend ductility and transverse and longitudinal tensile strengths.

### Bend Tests

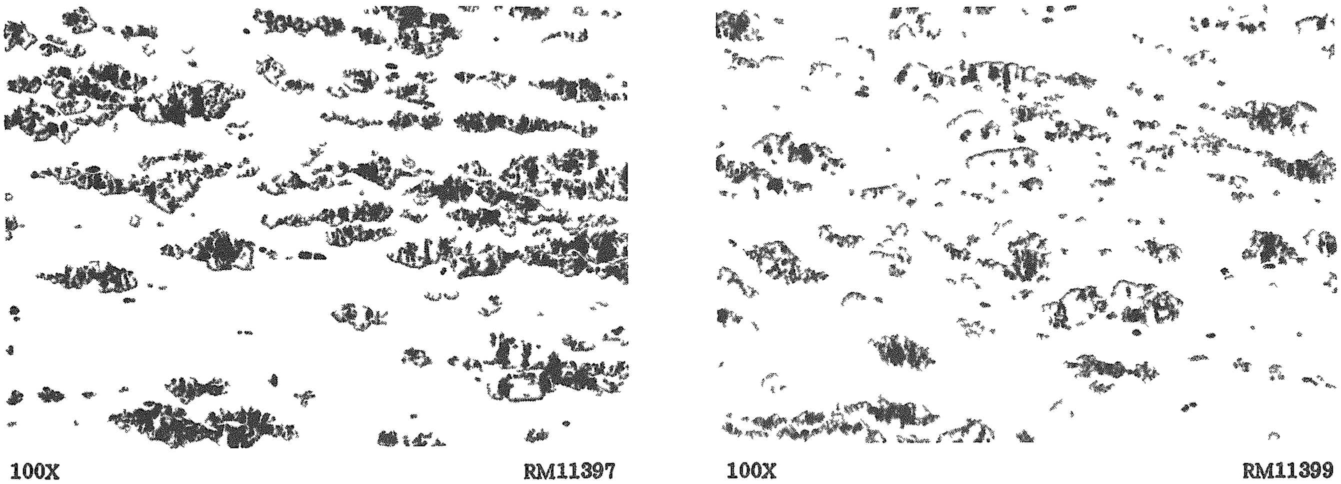
A 90-deg V-die with matching punches was used for determining bend ductility. Tests were made by pressing 1/2-in. -wide flat specimens between the die and punches having progressively smaller end radii until a crack was detected visually in the cladding. A T-bend rating consisting of the ratio of minimum bend radius before cracking to plate thickness was used in the evaluation. The fuel plates used for the tests measured 0.045 in. thick and had a cladding-core-cladding ratio of 6-33-6.

Variations in UN particle size and reduction schedules had very little effect on the ratings. Mean T-bend ratings of 1.4 for 30 w/o UN in Type 318 prealloyed stainless steel and 2.8 for 28 w/o UN in elemental 18-14-2.5 alloy were obtained. The probable reason for the poorer T-bend values of the elemental plates was found on metallographic examination of the matrix structure. Heterogeneous areas in the microstructure were found, indicating an incomplete solution of the elemental-matrix components. By increasing the 2300 F anneal from 2 hr to 3 hr, the T-bend value of the elemental plates approached the values for prealloyed plates.



a. 30 Per Cent Reduction First Pass, 20 Per Cent Remaining Passes

b. 35 Per Cent Reduction First Pass, 20 Per Cent Remaining Passes



c. 40 Per Cent Reduction First Pass, 20 Per Cent Remaining Passes

d. 45 Per Cent Reduction First Pass, 20 Per Cent Remaining Passes

FIGURE 5. EFFECTS OF INITIAL HOT REDUCTION ON ROLLED CORE STRUCTURES

The tendency for stringering to increase with an increase in initial reduction can be seen in the photomicrographs. All cores had 28 w/o UN (minus 200 plus 325 mesh) dispersed in elemental 18-14-2.5 powder. The specimens were pressed green at 50 tsi, rolled 6 to 1 at 2100 F, annealed 2 hr at 2300 F, cold rolled 18 per cent, and flat annealed 1 hr at 2050 F.

## Longitudinal Tensile Tests

Sheet tensile specimens of conventional shape were cut from fuel plates and tensile tested at room temperature and at 1350, 1500, and 1650 F. A gage section 0.5 in. wide by 0.045 in. thick was used. Cores were exposed along the edges with only one exception. All tests were performed in air.

Table 4 lists the results of individual tests which were performed on fuel plates containing 28 w/o UN dispersed in elemental-powder 18-14-2.5 alloy. The tensile strength appeared to be relatively insensitive to variations in UN particle size over the range tested. The 0.020-in. cladding on the core edges of the single specimen tested in this condition did not materially affect the results.

TABLE 4. EFFECT OF UN PARTICLE SIZE ON TENSILE PROPERTIES OF UN-STAINLESS STEEL FUEL PLATES(a)

| UN Particle Size | Test Temperature, F | Ultimate Tensile Strength, psi | Elongation in 2 In., per cent |
|---|---|---|---|
| -100 +200 | Room | 59,700 | 14 |
| -100 +200 | Room | 59,800(b) | 16 |
| -200 +270 | Room | 59,000 | 13 |
| -200 +270 | Room | 61,000 | 15 |
| -200 +325 | Room | 60,200 | 16 |
| -200 +325 | Room | 62,200 | 14 |
| -100 +200 | 1350 | 31,500 | 13 |
| -100 +200 | 1350 | 32,500 | 6 |
| -200 +325 | 1350 | 32,600 | 9 |
| -100 +200 | 1500 | 20,900 | 13 |
| -100 +200 | 1500 | 22,700 (b) | 11 |
| -200 +325 | 1500 | 22,500 | 13 |
| -100 +200 | 1650 | 15,100 | 8 |
| -200 +325 | 1650 | 14,100 | 14 |
| -200 +325 | 1650 | 14,800(b) | 11 |

(a) 28 w/o UN dispersed in 18-14-2.5 elemental alloy core 0.033 in. thick clad with 0.006 in. of Type 318 stainless steel.
(b) 20-mil cladding left on core edges.

## Transverse Tensile Tests

In order to get a better idea of the actual core strengths, a transverse tensile test was devised. The test consisted of a tensile pull in a plane perpendicular to the fuel-plate surface so that the core structure would support all the applied stress.

TABLE 5. EFFECTS OF PARTICLE SIZE AND FABRICATION PROCEDURE ON TRANSVERSE TENSILE STRENGTH OF UN-STAINLESS STEEL FUEL PLATES(a)

| UN Particle Size | Rolling Temperature, F | Reduction First Pass(b), per cent | Annealing Temperature, F | Cold Work, per cent | Test Temperature, F | Ultimate Tensile Strength(c), psi |
|---|---|---|---|---|---|---|
| 28 w/o UN in Elemental 18-14-2.5 Alloy Core | | | | | | |
| -100 +200 | 2100 | 30 | 2300 | 18 | Room | 24,100 |
| -200 +270 | 2100 | 30 | 2300 | 18 | Room | 33,600 |
| -200 +270 | 2000 | 30 | 2300 | 18 | Room | 23,700 |
| -200 +325 | 2100 | 30 | 2300 | 18 | Room | 29,200 |
| -200 +325 | 2100 | 30 | 2300 | 18 | 1300 | 15,100(d) |
| 30 w/o UN in Prealloyed Type 318 Stainless Steel Core | | | | | | |
| -100 +140 | 2000 | 30 | 2050 | 18 | Room | 16,800 |
| -140 +200 | 2000 | 30 | 2050 | 18 | Room | 17,500 |
| -270 +325 | 2000 | 30 | 2050 | 18 | Room | 17,300 |
| -325 | 2000 | 30 | 2050 | 18 | Room | 21,400 |
| 200 -270 | 1900 | 30 | 2050 | 18 | Room | 22,000 |
| 200 -270 | 2100 | 30 | 2050 | 18 | Room | 27,200 |
| 200 -270 | 2200 | 30 | 2050 | 18 | Room | 22,200 |
| 200 -270 | 2000 | 20 | 2050 | 18 | Room | 22,600 |
| 200 -270 | 2000 | 30 | 2050 | 18 | Room | 22,900 |

(a) All cores were 0.033 in. thick and were clad with 0.006 in. of Type 318 stainless steel.

(b) Reduction was 20 per cent on remaining passes; packs were flipped and reversed after each pass.

(c) Average of two or more tests.

(d) The tensile strength of the braze was reached at this temperature.

To make a transverse tensile bar, a 5/8-in.-diameter disk was punched from the core area of a fuel plate and copper brazed between two 3/4-in.-diameter stainless steel pins in a hydrogen furnace. This composite was then machined to 3/8 in. in diameter, and the ends were threaded to fit the grips of the tensile-testing machine. This 2-in.-long specimen was then pulled in a normal manner.

All testing was done in air at room temperature and, for one series of specimens, at 1300 F. Heat-resistant aluminum paint was applied to the exposed core section before making elevated-temperature tests to reduce oxidation of the UN in the core.

Table 5 shows the results of transverse tensile tests on 28 w/o UN dispersed in elemental 18-14-2.5 alloy cores and 30 w/o UN dispersed in Type 318 stainless steel cores. Effects of variations of UN particle size, rolling temperatures, and initial reductions are shown.

Although a direct comparison cannot be made between the 28 w/o UN-elemental cores and the 30 w/o UN-prealloyed cores, several trends are evident: (1) elements rolled at 2100 F give higher strengths than those rolled at lower temperatures, (2) elements with UN particles of minus 200 plus 270-mesh size possess higher strength, and (3) elements prepared with elemental powder possess a higher strength, particularly when coarse UN particles are used. These trends are in general agreement with what might be predicted from an observation of the core structures.

## CORROSION TESTS

### Corrosion in 680 F Degassed Water

Type 318 stainless steel-clad dispersions of 30 w/o UN in Type 318 prealloyed stainless steel and in 18-14-2.5 elemental stainless steel alloy were corrosion tested in water. Two types of specimens were used: one fully clad with an 0.40-in. hole drilled into the core center and the other with two core edges exposed.

The specimens with the edges exposed were placed in 680 F degassed water for 155 days. Examination of both the prealloyed- and elemental-type cores after testing showed small cracks but no bulging or swelling.

The defected specimens were held in 680 F degassed water for 3 weeks. No evidence of attack was observed on these specimens.

### Corrosion in 700 F NaK

Defected and edge-exposed specimens of the type used for water corrosion were sealed in capsules filled with NaK and heated at 700 F for 500 hr. The outward appearance of the specimens after exposure showed little or no change. Metallographic examination of the exposed core surfaces showed that slight attack occurred on the UN particles. The NaK penetrated the prealloyed matrix 0.005 in. and the elemental-alloy matrix to a slightly greater extent.

## Corrosion in 1800 F Air

To investigate the effects of high-temperature oxidation on exposed cores, specimens were heated to 1800 F in flowing air for 100 hr. A specimen of UN dispersed in prealloyed Type 302B stainless steel and clad with Type 347 stainless steel was compared with a specimin of $UO_2$ dispersed in prealloyed Type 347 stainless steel and clad with Type 347 stainless steel.

Metallographic examination of the two specimens (Figure 6) showed oxide penetration into both cores at the exposed edges. The UN specimen exhibited the least penetration (under 0.030 in.). The deeper oxide penetration of the $UO_2$ specimen illustrates the effects of continuous stringering in a rolled structure. The UN specimens prepared in this study in general exhibited less continuous stringering than comparable $UO_2$ specimens.

## DISCUSSION OF RESULTS

The results of development studies on the fabrication of UN dispersion-type flat-plate fuel elements were presented in the preceding sections.

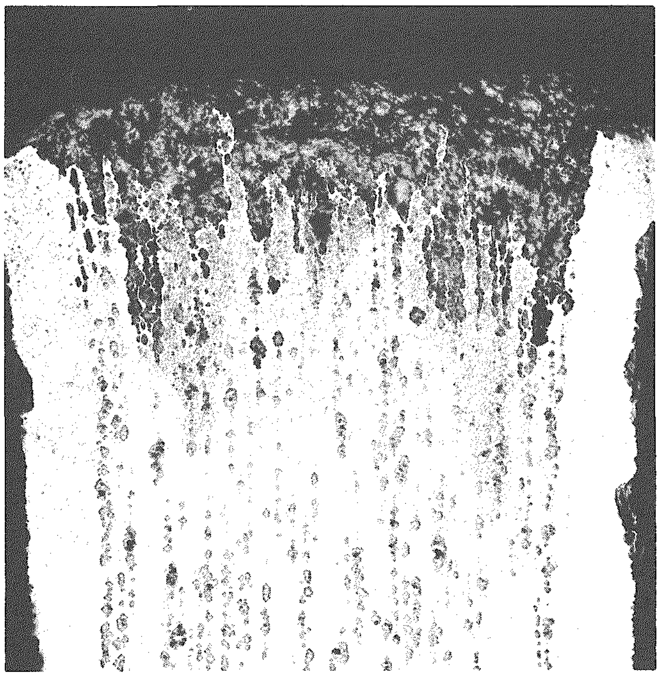
In the compatibility studies it was noted that, while UN did not react with chromium or iron up to 2300 F, reaction with nickel occurred at 1800 F. However, examination of UN dispersions in stainless steel made from elemental powders showed no UN-nickel reaction, probably because of the early solution of nickel in the iron.

The core structures of UN-stainless steel dispersions rolled with sintered cores were somewhat better than those rolled with green cores; however, the improvement in core structure was not believed to be sufficient to justify economically a sintering treatment prior to rolling.

It is interesting to note the correlation between the appearance of the various core structures produced and their transverse tensile properties. In almost every case the rolled core structures having discrete particles and minimum stringering exhibited the highest transverse tensile strengths.

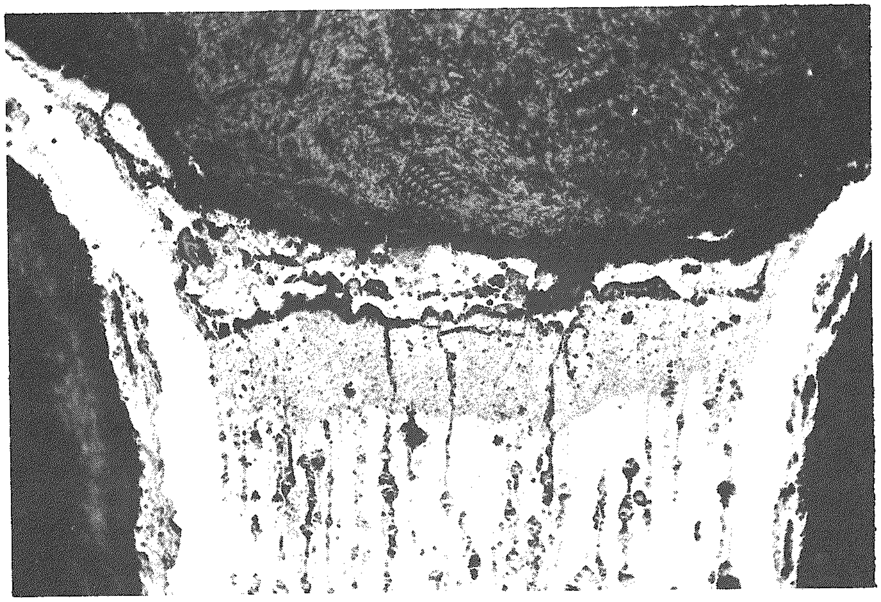
These optimum properties were best obtained with dispersions of minus 200 plus 270- or minus 200 plus 325-mesh UN dispersed in minus 325-mesh elemental 18-14-2.5 alloy powder. Hot rolling at 2100 F with an initial reduction in thickness of 30 per cent and remaining reductions of 20 per cent proved to be the most favorable rolling procedure.

The final fabrication procedure is listed in Table 6. More than 100 stainless steel dispersion-type flat fuel plates were made using this procedure.



50X RM10803

a. UN Dispersed in Type 302B Stainless Steel, Clad With Type 347 Stainless Steel



50X RM12402

b. $UO_2$ Dispersed in Type 347 Stainless Steel, Clad With Type 347 Stainless Steel

FIGURE 6. EFFECT OF HEATING DISPERSION SPECIMENS FOR 100 HR AT 1800 F IN FLOWING AIR

TABLE 6. OPTIMUM PROCEDURE FOR FABRICATION OF STAINLESS STEEL-CLAD UN-STAINLESS STEEL DISPERSION FUEL ELEMENTS HAVING 28 w/o UN DISPERSED IN AN ELEMENTAL 18-14-2.5 ALLOY CORE

1. Blend a minus 325-mesh elemental stainless steel powder mix containing 65.5 w/o electrolytic iron, 18 w/o electrolytic chromium, 14 w/o carbonyl nickel, and 2.5 w/o molybdenum, in a V-type mixer.
2. Prepare a batch of minus 200 plus 325-mesh UN powder.
3. Prepare a mixture of 28 w/o UN in elemental-powder stainless steel, and blend in a V-type mixture for 2 hr dry, then add 1/2 w/o camphor-alcohol binder, and mix for an additional 2 hr in a dry box.
4. Remove blend from dry box in sealed jars to minimize oxygen contamination prior to pressing.
5. Press green at 50 tsi.
6. Machine stainless steel picture-frame packs to size.
7. Clean packs by scrubbing with steel wool and alcohol, rinsing in cold water, washing in 180 F Alconox-water solution, rinsing alternately in hot and cold water, and drying with a lint-free absorbent material.
8. Place compact in a clean pack, and weld cover plates and stem onto the pack frame in an inert-atmosphere dry box.
9. Check pack for leaks.
10. Degas pack at 900 F to a vacuum of less than $1 \times 10^{-3}$ mm of mercury and seal stem closed by hot forging.
11. Hot roll from a hydrogen-atmosphere muffle at 2100 F, giving a 30 per cent reduction in thickness in the first pass, and 20 per cent reductions in thickness in the remaining passes down to 0.055 in.
12. Descale in a hot solution of 10 volume per cent $HNO_3$-2 volume per cent HF-88 volume per cent $H_2O$.
13. Anneal in a dry-hydrogen atmosphere for 3 hr at 2300 F.
14. Cold roll to 0.045 in.
15. Flat anneal in a stainless steel flattening jig for 1 hr at 2050 F in a dry-hydrogen atmosphere.
16. Radiograph and inspect.

## CONCLUSIONS

A series of development studies was made on fabrication variables in an effort to reduce each variable to a favored process which in conjunction with the other steps would produce an optimum fabrication procedure for UN-stainless steel dispersion fuel elements.

The Type 318 stainless steel cladding and matrix material and 28 w/o fuel loading were fixed in accordance with GCRE specifications; however, the elemental powder 18-14-2.5 alloy was chosen as the matrix material in preference to the prealloyed Type 318 stainless steel because of its superior structure in the fuel element. A reaction was found to occur between UN and a matrix of 100 per cent nickel; however, when the nickel was diluted to the specified 14 per cent in the matrix no evidence of a reaction was found.

A UN particle size of minus 200 plus 325 mesh was selected as being the most favorable from the standpoint of a combination of core structure, strength, and utilization of the largest particle size range of fuel. The elemental powders were all minus 325 mesh.

Sintering studies indicated that the core compact can be roll bonded in the green-pressed condition without a detrimental effect on the final structure. A high-temperature anneal of 3 hr at 2300 F after hot rolling is employed to assure the solution of the elemental matrix constituents.

A rolling temperature of 2100 F with a 30 per cent initial reduction in thickness followed by 20 per cent reduction and a final 18 per cent cold roll reduction followed by a 1 hr anneal at 2050 F in hydrogen was found to produce an optimum UN-stainless steel dispersion-type fuel element.

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