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METALLURGICAL STUDIES OF NIOBIUM-URANIUM ALLOYS

by

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TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	1
INTRODUCTION	1
ALLOY PREPARATION.	2
HOT-HARDNESS SURVEY.	8
FABRICATION	10
Cold and Warm Rolling	13
Hot Rolling and Forging	13
Press Forging	16
Fabrication at 2550 F	22
Fabrication at 3000 F	22
SHORT-TIME TENSILE PROPERTIES	22
CORROSION.	25
In Air at 572, 662, and 752 F	25
In NaK at 1600 F	29
In CO ₂	29
In Water.	29
CONCLUSIONS	41
DISCUSSION.	42
REFERENCES.	42

METALLURGICAL STUDIES OF NIOBIUM-URANIUM ALLOYS

John A. DeMastry, Frederic R. Shober, and Ronald F. Dickerson

In a continuing program, fabrication characteristics, physical and mechanical properties, and corrosion behavior in air, CO₂, NaK, water, and steam were studied for binary niobium fuel alloys containing 10, 20, 30, 40, 50, and 60 w/o uranium. To evaluate the effects of two major impurities of niobium, oxygen and zirconium, three niobium base stocks, differing according to the level of these impurities, were used for each alloy. The impurity combinations employed were 600 ppm oxygen and 0.74 w/o zirconium, 700 ppm oxygen and 0.17 w/o zirconium, and 300 ppm oxygen and 0.02 w/o zirconium.

Representative specimens of these alloys retained their hardness up to 900 C. The 10 and 20 w/o uranium alloys were successfully forged at 2500 F and rolled at 1800 F to sheet. Fabrication characteristics of the remaining alloys are under investigation. The 0.2 per cent offset yield strength of the 10 w/o uranium alloy was 57,200 psi at room temperature and 36,900 psi at 1600 F. For the 20 w/o uranium alloy it was 93,200 psi at room temperature and 71,000 psi at 1600 F.

The corrosion life of all of the alloys in air at 572 F and in CO₂ at 600 F was superior to that of unalloyed niobium. In 1000-hr exposures to 600 F water most of the alloys exhibited corrosion rates only two or three times greater than that of Zircaloy-2. All of the alloys appear compatible with NaK at 1600 F.

The impurity combinations employed in the base niobium appeared to have no effect on the corrosion behavior and mechanical properties of the alloys.

INTRODUCTION

Much current research is directed toward developing reactor fuels capable of high burnups at high temperatures. Such fuels will allow more efficient operation of reactors and will promote more economical fuel cycles. While metallic fuels have very desirable thermal properties, most are not well suited to high-temperature high-burnup service because they have insufficient strength at the operation temperatures of interest (1600 F and above). It is possible, however, that niobium-base fuel alloys may prove to be exceptions to this rule. Recent work has indicated that the high-temperature strength of niobium-rich niobium-uranium alloys should be superior to that of any other metallic fuel material now in use. ⁽¹⁾

Very little information about the fabrication characteristics, mechanical and physical properties, and corrosion behavior in various environments of niobium-rich niobium-uranium alloys can be found in the literature. Most published work has been concerned with uranium-rich alloys. Some of these studies have indicated that the properties of the high-uranium alloys are influenced by impurities, particularly oxygen and zirconium, present in the niobium melting stock. ^(2, 3) No data pertaining to the effects of these impurities in niobium-rich alloys appear to have been published.

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(1) References at end.

The present program was undertaken to obtain information on the fabrication characteristics, mechanical and physical properties, and corrosion behavior of binary niobium alloys containing 10, 20, 30, 40, 50, and 60 w/o uranium. The effect of oxygen and zirconium impurities on the properties of these alloys was also to be studied in the program. This report presents the results of the work so far completed in the investigation.

ALLOY PREPARATION

The niobium used was obtained from two suppliers. Table 1 lists the analysis of the niobium from the Fansteel Metallurgical Corporation which was used as the high-purity base melting stock. This material was in the form of sintered rod, 5/8 in. square. Table 2 lists the analysis for high- and low-zirconium-content niobium which was obtained from Kennametal, Inc. This material was in the form of pressed-powder bars 1 in. square. The uranium used for melting stock was high-quality biscuit which contained 27 ppm oxygen and 2.1 ppm hydrogen.

The uranium was rolled into strip stock and welded (in a helium atmosphere) onto two opposite sides of the niobium electrode for subsequent consumable-electrode arc melting. The uranium strip was rolled to a cross-sectional area which, when the strip was attached to the niobium electrode, would give the exact composition of the alloy being melted. After melting, the ingot was turned on a lathe and then cut into quarters which were welded into an electrode and remelted. The final ingots weighed between 4 and 5 lb each and were 1-3/4 and 2-1/4 in. in diameter, respectively. Table 3 lists spot chemical analyses made to determine average alloy contents.

A section of each ingot was removed for metallographic study and hardness measurements of the as-cast material. Figure 1 shows the as-cast microstructure of the niobium-10, -20, -30, -40, -50, and -60 w/o uranium alloys prepared using niobium containing 700 ppm oxygen and 0.17 w/o zirconium as base melting stock. Figure 2 shows the as-cast structure of niobium-20 w/o uranium prepared from niobium stock containing different amounts of oxygen and zirconium as impurities and Figure 3 shows microstructures of niobium-50 w/o uranium prepared from the same stock. There appears to be no appreciable difference in the structures of either alloy because of impurity content.

The effect of alloy composition and impurity content on the as-cast room-temperature hardness is shown in Figure 4. There is very little difference between room-temperature hardness for alloys containing greater than 20 w/o uranium, indicating that hardnesses do not reflect the effect of impurities on the starting stock after the total amount of alloying elements has exceeded 20 w/o uranium. For alloys containing less than 20 w/o uranium, the room-temperature hardness appears to be a function of the oxygen content more so than of the amount of zirconium present. This is not surprising since oxygen has been shown to be a very potent hardener of unalloyed niobium at room temperature. (4)

TABLE 1. CHEMICAL ANALYSIS OF NIOBIUM MELTING
STOCK HIGH-PURITY GRADE (FANSTEEL
METALLURGICAL CORPORATION)

Element	Amount Present, w/o
Niobium	99.8 minimum
Oxygen	0.009 to 0.030
Nitrogen	0.020 to 0.030
Carbon	0.003 to 0.013
Titanium	0.012 to 0.015
Tantalum	0.075 to 0.082
Nickel	0.001 to 0.005
Tungsten	<0.01
Zirconium	<0.02
Iron	<0.007
Aluminum	<0.0001
Yttrium	<0.01
Lead	<0.001
Chromium	<0.001
Cobalt	<0.001
Molybdenum	<0.020
Silicon	<0.010
Boron	<0.0001
Cadmium	<0.0001
Lithium	<0.0001

TABLE 2. CHEMICAL ANALYSIS OF KENNAMETAL NIOBIUM

Element	Type of Analysis	Amount Present
<u>Low Zirconium Content</u>		
Boron	Spectrographic	<1 ppm
Carbon	Chemical	270 ppm
Nitrogen	Chemical	600 ppm
Oxygen	By ignition	700 ppm
Tantalum	X-ray	700 ppm
Titanium	Spectrographic	<50 ppm
Iron	Spectrographic	200 ppm
Silicon	Spectrographic	100 ppm
Zirconium	Chemical	0.17 w/o
Niobium	Chemical	99.53 w/o
<u>High Zirconium Content</u>		
Boron	Spectrographic	<1 ppm
Carbon	Chemical	280 ppm
Nitrogen	Chemical	580 ppm
Oxygen	By ignition	600 ppm
Tantalum	X-ray	700 ppm
Titanium	Spectrographic	<50 ppm
Iron	Spectrographic	200 ppm
Silicon	Spectrographic	100 ppm
Zirconium	Chemical	0.74 w/o
Niobium	Chemical	99.01 w/o

TABLE 3. TYPICAL CHEMICAL ANALYSIS FOR URANIUM-NIOBIUM ALLOYS

Nominal Composition, w/o			Analyzed Composition, w/o		
Niobium	Uranium	Zirconium	Niobium	Uranium	Zirconium
90	10	0.02	--	10.6	0.02
80	20	0.74	--	19.7	0.64
80	20	0.02	78.8	--	--
70	30	0.17	--	30.1	0.42
60	40	0.02	--	40.0	--
50	50	0.02	45.7	53.3	--
40	60	0.02	39.4	59.8	--

250X

RM11810

a. Niobium-10 w/o Uranium

250X

RM11794

b. Niobium-20 w/o Uranium

250X

RM14437

c. Niobium-30 w/o Uranium

250X

RM11802

d. Niobium-40 w/o Uranium

250X

RM11799

e. Niobium-50 w/o Uranium

250X

RM11796

f. Niobium-60 w/o Uranium

FIGURE 1. TYPICAL AS-CAST STRUCTURE OF NIOBIUM-URANIUM ALLOYS

The niobium used in these alloys contained 700 ppm oxygen and 0.17 w/o zirconium.

250X

RM11807

- a. Niobium Contained 600 PPM Oxygen and
0.74 w/o Zirconium
Hardness: 60 Rockwell A.

250X

RM11794

- b. Niobium Contained 700 PPM Oxygen and
0.17 w/o Zirconium
Hardness: 58 Rockwell A.

250X

RM14436

- c. Niobium Contained 300 PPM Oxygen and
0.02 w/o Zirconium
Hardness: 52 Rockwell A.

FIGURE 2. AS-CAST STRUCTURE AND EFFECT OF DIFFERENT PURITY LEVELS OF NIOBIUM ON THE MICROSTRUCTURE OF NIOBIUM-20 w/o URANIUM

250X

RM11798

- a. Niobium Contained 600 PPM Oxygen and
0.74 w/o Zirconium
Hardness: 64 Rockwell A.

250X

RM11799

- b. Niobium Contained 700 PPM Oxygen and
0.17 w/o Zirconium
Hardness: 64 Rockwell A.

250X

RM11800

- c. Niobium Contained 300 PPM Oxygen and
0.02 w/o Zirconium
Hardness: 65 Rockwell A.

FIGURE 3. AS-CAST STRUCTURE AND EFFECT OF DIFFERENT PURITY LEVELS OF NIOBIUM ON THE MICROSTRUCTURE OF NIOBIUM-50 w/o URANIUM

FIGURE 4. EFFECT OF INCREASING URANIUM CONTENT ON THE AS-CAST HARDNESS OF ALLOYS PREPARED FROM DIFFERENT GRADES OF NIOBIUM MELTING STOCKS

HOT-HARDNESS SURVEY

Hot-hardness measurements were made on as-cast specimens of all alloy compositions investigated. These specimens were arc melted five times and then cast into a slab 1 by 3/8 by 1/8 in. The hot-hardness specimens were then ground through 600-grit paper and polished on a slow wheel with a Forstmann's cloth cover using diamond paste with a kerosene lubricant.

Diamond-pyramid hardnesses of selected alloys were obtained at elevated temperature by means of a vacuum hot-hardness machine. The sapphire indentors, specimens, and stage were resistance heated by a furnace surrounding them. Six 1 by 3/8 by 1/8-in. specimens were placed directly over thermocouples which were fitted into the stage from below. Indentations were made by lowering the indentors until the full weight of the indentors (730 g for 15 sec) rested on the specimens. Then the load was removed, the stage rotated a known distance, and the loading sequence repeated. Four indentations were made at each test temperature between 400 and 600 C, and three indentations were made at temperatures above 600 C. The results obtained at each temperature were then averaged. In this case, the specimens were prepared in triplicate, and the results were again averaged. The resulting hot-hardness numbers are, therefore, the average of from 9 to 12 impressions. Room-temperature hardness measurements were made before and after hot-hardness determinations as a check for contamination of the alloys with oxygen during testing. Table 4 lists the hot-hardness values obtained. The fact that there was no appreciable increase in room-temperature hardness after elevated-temperature measurements were obtained indicates that little, if any, contamination with oxygen had occurred.

TABLE 4. HOT HARDNESS OF NIOBIUM AND NIOBIUM-URANIUM ALLOYS^(a)

Alloy Composition (Balance Niobium), w/o	Impurities		Diamond-Pyramid Hardness at Temperature Indicated									Room Temperature (After Test)
	Oxygen, ppm	Zirconium, w/o	Room Temperature	400 C	500 C	600 C	700 C	800 C	900 C	1000 C	1100 C	
100 niobium	500	0.01	163	130	112	84.4	65.6	58.2	54.8	46.6	42.7	152
100 niobium	700	0.17	246	176	168	143	113	127	167	--	--	266
100 niobium	600	0.74	236	174	159	102	93.3	91.5	89.3	--	--	190
10 uranium	500	0.01	164	143	138	119	113	108	95.1	74.8	63.0	145
20 uranium	500	0.01	213	163	155	145	143	140	133	113	91.6	178
20 uranium	700	0.17	253	183	179	166	173	159	157	--	--	217
20 uranium	600	0.74	243	170	170	169	164	173	138	--	--	184
30 uranium	500	0.01	264	209	200	187	174	179	158	--	--	253
40 uranium	500	0.01	311	216	224	219	200	188	157	118	91.8	316
50 uranium	500	0.01	308	235	228	222	201	191	166	--	--	328
60 uranium	500	0.01	337	228	222	205	203	186	176	--	--	389

(a) Average of three specimens, 9 to 12 impressions.

The impurity content of the stock niobium used as base material was not expected to have a significant effect on the hardness at temperatures above 700 C; therefore, testing was confined to alloys prepared from high-purity niobium melting stock with spot checks of the unalloyed niobium and the niobium-20 w/o uranium alloy. Figure 5 shows the effect of impurities on all three unalloyed niobium base materials. Figure 6 shows the effect on hardness of the niobium-20 w/o uranium alloy produced by the use of these three separate grades of niobium as base melting stock. These graphs show that impurity content does slightly affect the elevated-temperature hardness of unalloyed niobium. This effect is probably due to total impurity content. The hardness of unalloyed niobium is highest at all temperatures for the stock having the greatest oxygen content. In the niobium-20 w/o uranium alloy, the differences in hardness due to impurity content of the melting stock are much less pronounced than with the unalloyed niobium. Apparently, the increase in hardness resulting from the uranium addition tends to mask the effect of impurities. It can be seen in Figure 7 that, as the amount of uranium was increased, the differences among hardness values of the alloys decreased at elevated temperatures.

The effect of increasing alloying additions of uranium to niobium is shown in Figure 7. Addition of more than 40 w/o uranium does not cause a corresponding increase in hardness at elevated temperature. The theoretical maximum hardness above 1000 C should occur at 50 a/o or approximately 72 w/o uranium addition. Generally, the strongest composition of a system having complete solid solubility occurs in the 50 a/o range. Of the alloy compositions studied it does not appear that increases of uranium above 40 w/o result in greatly increased hardnesses at elevated temperatures.

When the hardness of the niobium-uranium alloys is plotted against increasing temperature on semilog graph paper, a straight line is obtained with a gradual downward slope. This line remains straight up to 900 C. Generally, in pure metals and alloys at some given temperature, the straight line takes a sudden drop and the slope of the curve becomes much steeper. This point is known as the "softening temperature". None of the alloys studied exhibited this softening temperature below 900 C. The niobium-10 and -20 w/o uranium alloys do, however, show evidence of the beginning of such a break between 1000 and 1100 C. An evaluation of the hot hardness indicates that the addition of uranium to niobium greatly increases the hardness of niobium and raises the softening temperature.

FABRICATION

There were two experimental fabrication studies of the niobium-uranium alloys proposed as a part of the program. The first was concerned with cold and warm rolling of the alloys in the as-cast condition. This study was carried out on unclad specimens. The second approach consisted of fabricating specimens which had been canned in stainless steel and molybdenum packs. Alloys were hammer forged, press forged, and rolled while in these cans at temperatures from 2400 to 3000 F.

FIGURE 5. EFFECT OF IMPURITY CONTENT ON ELEVATED-TEMPERATURE HARDNESS OF UNALLOYED NIOBIUM

FIGURE 6. EFFECT OF IMPURITY CONTENT ON ELEVATED-TEMPERATURE HARDNESS OF NIOBIUM-20 w/o URANIUM

FIGURE 7. EFFECT OF INCREASING ALLOYING ADDITIONS OF URANIUM ON HIGH-TEMPERATURE HARDNESS OF NIOBIUM-URANIUM ALLOYS

Cold and Warm Rolling

The cold-fabrication study was conducted on the 1 by 3/8 by 1/8-in. slabs which had been annealed to 900 C during hot-hardness testing and furnace cooling. The uniform thickness and polished surfaces made these specimens especially adapted to cold rolling. Table 5 and Figure 8 indicate the results of cold rolling. The unalloyed niobium was in the as-cast condition, which is unfavorable for cold rolling due to large grain structure. The alloys were reduced 5 per cent per pass in order to find a fabrication technique which would be applicable to production work. When very small reductions were made, the unalloyed niobium could be cold rolled.

TABLE 5. RESULTS OF ROOM-TEMPERATURE ROLLING OF AS-CAST AND ANNEALED HOT-HARDNESS BUTTONS OF NIOBIUM-URANIUM

Alloy Composition (Balance Niobium), w/o	Impurity Content		Thickness, in.		Actual Total Reduction(a), per cent	Remarks
	Oxygen, ppm	Zirconium, w/o	Before Rolling	After Rolling		
100 niobium	500	0.01	0.179	0.098	45	Cracked after 20 per cent reduction
100 niobium	700	0.17	0.190	0.170	10	Cracked immediately
100 niobium	600	0.74	0.180	0.100	44	Cracked after 20 per cent reduction
10 uranium	500	0.01	0.190	0.175	7	Cracked immediately
20 uranium	500	0.01	0.187	0.170	8	Cracked immediately
30 uranium	500	0.01	0.175	0.170	3	Cracked immediately
40 uranium	500	0.01	0.183	0.178	2	Cracked immediately
50 uranium	500	0.01	0.187	0.183	2	Cracked immediately
60 uranium	500	0.01	No rolling attempted			

(a) Nominal reductions were 5 per cent per pass.

After reviewing the results of the room-temperature fabrication of unclad niobium-uranium, attempts to roll these alloys at higher temperatures (575 to 1472 F) were made. After approximately 50 per cent reduction had been obtained with warm rolling, the test specimens were air cooled, and then rolling was continued at room temperature. The results obtained indicate that the cast structure was broken up more successfully by warm working than by cold working and that, when the structure is broken down, the niobium-10, -20, -30 w/o uranium alloys can then be cold rolled without extensive cracking. Table 6 shows the results of the fabrication experiments.

Hot Rolling and Forging

The second phase of the fabrication study was concerned with the working of clad material, generally, at elevated temperatures. The results of the hot-hardness measurements indicated that the niobium-uranium alloys would not fabricate easily at 1852 F. Therefore, it was decided that higher fabrication temperatures would have to be employed, which made it necessary to consider some form of protective cover. Only two materials were found which could be secured in the proper geometries for protective cans; these were stainless steel and molybdenum. In order to prevent the formation of a eutectic with uranium (iron at 1080 C, nickel at 740 C, and chromium at 860 C), specimens to be rolled in stainless steel were wrapped in 0.005-in. molybdenum sheet to prevent contact between the niobium-uranium alloys and the stainless steel. An evacuated stainless steel pack (AISI Type 304) containing 30-g buttons of niobium-10,

2X

RM13262

H90-1: Niobium-10 w/o uranium
 H80-1: Niobium-20 w/o uranium
 H70-2: Niobium-30 w/o uranium

H60-2: Niobium-40 w/o uranium
 H50-1: Niobium-50 w/o uranium

FIGURE 8. EFFECT OF COMPOSITION ON ROOM-TEMPERATURE ROLLING OF NIOBIUM-URANIUM ALLOYS

The niobium used in all alloys contained 300 ppm oxygen and 0.02 w/o zirconium. All alloys were reduced from 2 to 7 per cent.

TABLE 6. RESULTS OF ROLLING OF NIOBIUM-URANIUM ALLOYS IN AIR FROM A HELIUM-ATMOSPHERE FURNACE

Nominal Composition (Balance Niobium), w/o	Temperature, F	Reduction, per cent	Remarks
10 uranium	RT	90	Severe cracking until 60 per cent reduction was obtained; no further cracks noted; material is ductile
10 uranium	572	50	Edge cracking less severe than for above; cold rolled, air cooled
	RT	40	No new cracks; existing cracks opened; sound material obtained
20 uranium	572	50	Severe edge cracking; air cooled
	RT	40	No new cracks; existing cracks opened extensively; material has fair ductility
20 uranium	932	50	Edge cracks not as severe as for above material at 572 F
	RT	40	Some new cracks appeared; fair ductility
30 uranium	932	50	Quite severe cracking
	RT	40	New cracks noted; however, these were not extensive; ductility only fair
40 uranium	1472	50	Extremely severe edge cracking
	RT	20	Many new cracks noted
50 uranium	1472	40	Severe fracture; no cold rolling attempted
60 uranium	1472	20	Severe fracture; no cold rolling attempted

-20, -30, -40, -50, and -60 w/o uranium wrapped in molybdenum foil was rolled at 2400 F. Examination of the pack after rolling showed that the molybdenum sheet had ruptured with a resulting eutectic formation between the stainless steel and the niobium-uranium alloys. No further use of stainless steel as a canning material was made.

Molybdenum was next employed as a protective jacket. Since molybdenum must be protected to prevent oxidation at elevated temperatures, an atmosphere of argon or hydrogen was employed in the furnace. In order to evaluate molybdenum as a canning material, six small disks, 2 in. in diameter and 0.375 in. thick, of the niobium-uranium alloys were prepared for fabrication. Each of these was inserted into an individual can assembly made up of a section of molybdenum tubing closed at the top and bottom by 0.100-in.-thick molybdenum lids. The top and bottom of the can were welded in place in a helium atmosphere. Figure 9 shows the can assembly.

These packs were preheated to 2550 F in an argon atmosphere. The niobium-10 w/o uranium alloy was reduced without difficulty; however, the niobium-20 and -30 w/o uranium alloys did not roll readily and had to be forged before rolling. The niobium-10 and -20 w/o uranium alloys could be reduced with no difficulty; however, the niobium-30 w/o uranium alloys showed what appeared to be radial cracks on the surface. These cracks did not extend entirely through the sheet. Table 7 lists the results of fabrication of these test specimens.

The alloys which were reduced 75 per cent were sectioned and used for further fabrication study. It has been postulated that when the cast structure is broken up, these alloys can be fabricated at room temperature or slightly higher. The results of further fabrication of the three alloys shown in Table 7 are shown in Table 8. Approximately 0.30-in. sheet was obtained from all of the test specimens rolled. This material was sound as can be seen in Figures 10, 11, and 12.

Sections of the niobium-10, -20, and -30 w/o uranium alloy, which had been fabricated at 2550 F, were vacuum annealed at 1832 F for 24 hr and furnace cooled. Figures 10, 11, and 12 show preannealing and postannealing microstructures of these alloys. The niobium-30 w/o uranium alloy showed evidence of recrystallization during working and after annealing it appeared to be approximately 80 per cent recrystallized (Figure 12). Hardness measurements support this. Figure 11 shows the microstructure of the niobium-20 w/o uranium alloy. This alloy was hot worked at 2550 F. The microstructure of the niobium-10 w/o uranium alloy (Figure 10) also appears to have been hot worked at this temperature.

Press Forging

A separate study of press forging was conducted simultaneously with the hammer-forging study. Four evacuated molybdenum packs were prepared containing niobium-60, -50, -40, and -30 w/o uranium, respectively. The niobium used contained 300 ppm oxygen and 0.02 w/o zirconium. These packs were heated to 2400 F in an argon furnace and then placed on an asbestos-covered die and pressed at 700 tsi. Examination of the microstructure and hardness measurements indicate that fabrication at 2400 F resulted in cold working of all alloys fabricated. Recrystallization was approximately 50 per cent complete after annealing for 24 hr at 1832 F. This method of fabrication does not

1X

RM13261

FIGURE 9. VIEW OF MOLYBDENUM CAN ASSEMBLY

Left side shows molybdenum lids 0.100 in. thick which were welded onto center tube to produce finished can on right.

TABLE 7. FABRICATION OF MOLYBDENUM PACKS AT 2550 F FROM AN ARGON ATMOSPHERE

Nominal Composition (Balance Niobium), w/o	Impurity Content		Fabrication Method	Total Reduction of Alloy, per cent	Remarks
	Oxygen, ppm	Zirconium, w/o			
10 uranium	700	0.17	Rolling	75	Sound 0.095-in. sheet obtained
10 uranium	600	0.74	Rolling	53	Molybdenum pack broke and specimen was removed from test and quenched when oxidation became apparent
20 uranium	700	0.17	Rolling	48	Ditto
20 uranium	600	0.74	Forging and rolling	78	Pack forged from 3/4 to 1/4-in. thickness and then rolled; sound sheet was obtained
30 uranium	700	0.17	Forging and rolling	75	Packs forged from 3/4 to 1/4-in. thickness and then rolled; cracks present in sheet
30 uranium	600	0.17	Forging and rolling	77	Ditto

TABLE 8. FABRICATION OF PREVIOUSLY WORKED NIOBIUM-URANIUM ALLOYS REDUCED 75 PER CENT AT 2550 F^(a)

Nominal Composition (Balance Niobium), w/o	Impurity Content		Fabrication Temperature, F	Reduction, per cent	Remarks
	Oxygen, ppm	Zirconium, w/o			
10 uranium	700	0.17	RT	78	Slight edge cracking
10 uranium	700	0.17	1292	72	Very little edge cracking, sound sheet
10 uranium	700	0.17	1832	75	Ditto
20 uranium	600	0.74	RT	75	Some edge cracking
20 uranium	600	0.74	1292	72	No edge cracking
20 uranium	600	0.74	1832	75	Ditto
30 uranium	700	0.17	1292	70	Slight edge cracking
30 uranium	700	0.17	1832	70	Ditto

(a) Materials used in this fabrication were obtained by shearing strips from the sheet described in Table 7.

1X

RM11669

- a. Rolled at 2550 F, Reduced 75 Per Cent
As-cast hardness: 49 Rockwell A.

- b. Rolled at 2550 F
Hardness: 51 Rockwell A.

- c. Annealed at 1000 C, 24 Hr, Furnace Cooled
Hardness: 49 Rockwell A.

FIGURE 10. NIOBIUM-10 w/o URANIUM ALLOY SHOWING PREANNEALING AND POSTANNEALING MICROSTRUCTURES

The niobium used contained 700 ppm oxygen and 0.17 w/o zirconium.

1X

RM11668

- a. Forged and Rolled at 2550 F, Reduced 78 Per Cent
As-cast hardness: 60 Rockwell A.

100X

RM11692

100X

RM12010

- b. Forged and Rolled at 2550 F
Hardness: 52 Rockwell A.

- c. Annealed at 1000 C, 24 Hr, Furnace Cooled
Hardness: 52 Rockwell A.

FIGURE 11. NIOBIUM-20 w/o URANIUM ALLOY SHOWING PREANNEALING AND POSTANNEALING MICROSTRUCTURES

The niobium used contained 600 ppm oxygen and 0.74 w/o zirconium

1X

RM11667

- a. Forged and Rolled at 2550 F, Reduced 75 Per Cent
As-cast hardness: 57 Rockwell A.

250X

RM11689

250X

RM12008

- b. Forged and Rolled at 2550 F
Hardness: 61 Rockwell A.

- c. Annealed at 1000 C
Hardness: 57 Rockwell A.

FIGURE 12. NIOBIUM-30 w/o URANIUM ALLOY SHOWING PREANNEALING AND POSTANNEALING MICROSTRUCTURES

The niobium used contained 700 ppm oxygen and 0.17 w/o zirconium.

appear to be feasible for these alloys. At the moment of fabrication, the temperature of the ingot was probably considerably below 2400 F due to heat losses to the large die surface.

Fabrication at 2550 F

The niobium-10 and -20 w/o uranium alloys were successfully fabricated at 2550 F by forging, so a full-size ingot (3 to 5 lb) of each of the six alloys in this composition group was prepared for fabrication. The results of fabrication of these six ingots and a test-size ingot (1/2-in. -thick wafer) of the niobium-30 w/o niobium are shown in Table 9. Figures 13 through 15 show pictures of the forged slabs after removal from the canning material. The above-mentioned technique was successful except for the crack in the niobium-30 w/o uranium alloy. These alloys were warm rolled at 1800 F to sheet material. No effect due to use of the different niobium melting stocks was noted.

Fabrication at 3000 F

It was thought that a higher working temperature would permit fabrication of the niobium-30 through -60 w/o uranium alloys. Large ingots (3 to 5 lb) of the niobium-30, -40, -50, and -60 w/o uranium were prepared. These alloys were upset forged at 3000 F from a hydrogen atmosphere, but only three of the ingots could be fabricated. It was noted that all alloys exhibited the same sort of failure, cracks which were radial in nature. These cracks probably occurred during the initial portion of fabrication, perhaps in the first 5 to 10 per cent of reduction. Examination of the microstructure of the niobium-30 w/o uranium alloy and hardness measurements indicated that this alloy was hot worked at 3000 F. Table 10 lists the results of fabrication of 30 and 50 w/o uranium alloys.

A study of the fabrication characteristics of the alloys of interest yielded valuable information. The niobium-10 and -20 w/o uranium alloys can be easily fabricated at 2500 F by forging and subsequent warm rolling at 1800 F. After warm rolling, cold rolling will yield sound sheet material. Fabrication of the niobium-30 w/o uranium by forging between 2500 and 3000 F appears to be marginal but should be feasible. No successful fabrication of alloys containing more than 30 w/o uranium was obtained. The outlook for fabrication of these alloys is optimistic since it has been demonstrated that after the as-cast structure is broken down further, fabrication proceeds with little difficulty. Further studies of methods of initial breakdown of cast structure are in progress.

SHORT-TIME TENSILE PROPERTIES

Room-temperature measurements were made on both longitudinal sheet specimens and on ASTM standard 3/16-in. -diameter tensile bars using SR-4 Type A7 strain gages. The strain measurements with these gages were sensitive to $\pm 1 \mu\text{in.}$ and were accurate

1X

RM11839

FIGURE 13. NIOBIUM-10 w/o URANIUM ALLOY FORGED AT 2500 F

The niobium used contained 300 ppm oxygen and 0.02 w/o zirconium

1X

RM11836

FIGURE 14. NIOBIUM-20 w/o URANIUM ALLOY FORGED AT 2500 F

The niobium used contained 700 ppm oxygen and 0.17 w/o zirconium.

1X

RM12036

FIGURE 15. NIOBIUM-30 w/o URANIUM ALLOY FORGED AT 2500 F

The niobium used contained 600 ppm oxygen and 0.74 w/o zirconium

TABLE 9. RESULTS OF HAMMER FORGING 2-IN. -DIAMETER NIOBIUM-URANIUM INGOTS IN MOLYBDENUM PACKS AT 2500 F

Nominal Composition (Balance Niobium), w/o	Impurity Content		Fabrication Temperature, F	Thickness, in.		Reduction, per cent	Remarks
	Oxygen, ppm	Zirconium, w/o		Before Fabrication	After Fabrication		
10 uranium	300	0.02	2500	2.875	0.570	80	Sound material
10 uranium	700	0.17	2500	1.625	0.460	71	Ditto
10 uranium	600	0.74	2500	1.8125	0.420	76	Ditto
20 uranium	300	0.02	2500	2.9375	0.640	78	Ditto
20 uranium ^(a)	700	0.17	2500	2.625	0.625	76	Ditto
20 uranium	600	0.74	2500	2.000	0.550	72	Slight crack in center
30 uranium	700	0.17	2500	0.5625	0.185	67	One crack from center to edge

(a) Ingot was 1.750 in. in diameter.

TABLE 10. RESULTS OF HAMMER FORGING 2-IN. -DIAMETER NIOBIUM-URANIUM INGOTS IN MOLYBDENUM PACKS AT 3000 F FROM A HYDROGEN ATMOSPHERE

Alloy Composition (Balance Niobium), w/o	Impurity Content		Remarks
	Oxygen, ppm	Zirconium, w/o	
30 uranium	300	0.02	Pack ruptured; it appears that alloy was not reducing; cracks present in alloy
30 uranium	600	0.74	Alloy reduced 70 per cent; however, alloy was badly cracked
50 uranium	300	0.02	Alloy reduced 70 per cent; alloy fractured radically, many cracks present

to $\pm 4 \mu\text{in.}$ The testing strain rate was 0.002 in. per min. Table 11 shows the results of this testing. There were not sufficient data to determine if the impurity level of the niobium used had any effect on the mechanical properties.

The elevated-temperature tensile tests were performed on both sheet specimens and ASTM standard 3/16-in. -diameter tensile bars using a mechanical extensometer. The strain rate for all specimens in the elastic region was 0.002 in. per in. per min. The specimens were tested in a vacuum, and they showed no visual signs of oxygen contamination after testing. Results of testing are shown in Table 12.

It appears that uranium has a pronounced strengthening effect on niobium at both room and elevated temperature. The most remarkable feature of the niobium-uranium alloys is their retention of strength from room temperature to 1600 F. At room temperature, both the niobium-10 and -20 w/o uranium alloys exhibit, for a fuel, acceptable strength, but at 1600 F the tensile strength is in the superalloy class. As a comparison, commercial stainless⁽⁵⁾ steel, such as Type 304, has an ultimate tensile strength of 21,000 psi at 1500 F, whereas the niobium-10 and -20 w/o uranium alloys have ultimate tensile strengths of 51,000 psi and 71,000 psi, respectively. Hastelloy⁽⁶⁾, which is highly regarded as a high-strength elevated-temperature alloy, has an ultimate tensile strength of 40,000 psi at 1650 F in the wrought, fully annealed condition. This comparison indicates that the strength of the niobium-uranium alloys is superior to most structural materials and far superior to other metallic fuels now in use.

CORROSION

Corrosion testing in the prospective coolant is necessary to evaluate the worth of alloys for reactor use. The media used here were selected on the basis of their applicability as reactor coolants.

In Air at 572, 662, and 752 F

All alloys were checked for their oxidation resistance in air at 572, 662, and 752 F. The tests at 572 F were run for 500 hr with intermediate weighings at 20-hr intervals. The corrosion blanks were approximately 1/2 by 1/2 by 1/8 in. Accurate measurements were made of each specimen prior to exposure. The specimens were placed in ceramic containers to prevent loss of any oxide which might spall from the surface of the blanks during testing. All tests were run in duplicate. All alloys formed a black adherent oxide film at 572 F. Weight gains were moderate and far less than weight gains of unalloyed niobium at the same temperature. Results of testing at 572 F in air are shown in Table 13. At both 662 and 752 F, a brown spalling oxide was formed after 40 hr of testing. In many cases, this oxide was formed in as little as 20 hr of testing. For many alloys, at 752 F, complete oxidation occurred between 20 and 40 hr of testing. A summary of oxidation at 662 and 752 F is shown in Table 14.

TABLE 11. SHORT-TIME ROOM-TEMPERATURE TENSILE PROPERTIES OF NIOBIUM-URANIUM ALLOYS^(a) AS FORGED AT 2550 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Type	0.1 Per Cent Offset Yield Strength, psi	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Modulus of Elasticity, 10 ⁶ psi
	Oxygen, ppm	Zirconium, w/o							
10 uranium	600	0.74	3/16-in. -diameter bar	55,900	57,200	72,900	30	53	18.4
	700	0.17	0.030-in. sheet	69,300	--(b)	70,000	3	11	18.6
	300	0.02	0.030-in. sheet	76,800	--(b)	77,900	3	16	16.0
20 uranium	600	0.74	0.030-in. sheet	81,000	84,100	95,500	6	19	18.1
	700	0.17	3/16-in. -diameter bar	70,500	73,200	90,200	7	9	18.4
	300	0.02	0.030-in. sheet	90,000	93,200	102,000	7	21	20.3

(a) Average of two specimens.

(b) Strain gages failed before 0.2 per cent offset yield strength was reached.

TABLE 12. SHORT-TIME TENSILE PROPERTIES AT 1600 F OF NIOBIUM-URANIUM ALLOYS^(a) AT 2550 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Type	0.1 Per Cent Offset Yield Strength, psi	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Modulus of Elasticity, 10 ⁶ psi
	Oxygen, ppm	Zirconium, w/o							
10 uranium	600	0.74	3/16-in. -diameter bar		36,900	49,500	13	31	13.2
	700	0.17	0.025-in. sheet	51,700	--(b)	53,100	1	12	12.7
20 uranium	600	0.74	0.025-in. sheet	52,500	58,600	71,000	2	16	14.0
	700	0.07	3/16-in. -diameter bar	54,000	60,000	71,600	10	37	14.3
	300	0.02	0.025-in. sheet	52,300	58,100	68,400	2	7	12.4
30 uranium	600	0.74	1.8-in. -diameter bar	--(c)	--(b)	71,200	2	--	--

(a) Average of two specimens.

(b) Extensometer slipped before 0.2 per cent offset yield strength was reached.

(c) Extensometer slipped before 0.1 per cent offset yield strength was reached.

TABLE 13. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS^(a) IN AIR AT 572 F FOR 500 HR

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change, mg per cm ²
	Oxygen, ppm	Zirconium, w/o		
10 uranium	600	0.74	Fabricated	0.53
10 uranium	700	0.17	Fabricated	0.35
10 uranium	300	0.02	Fabricated	0.80
20 uranium	600	0.74	Fabricated	0.40
20 uranium	700	0.17	Fabricated	1.01
20 uranium	300	0.02	Fabricated	0.45
30 uranium	600	0.74	As cast	0.17
30 uranium	700	0.17	Fabricated	0.29
30 uranium	300	0.02	As cast	0.28
40 uranium	600	0.74	As cast	0.08
40 uranium	700	0.17	As cast	0.20
40 uranium	300	0.02	As cast	0.47
50 uranium	600	0.74	As cast	0.28
50 uranium	700	0.17	As cast	0.31
50 uranium	300	0.02	As cast	0.18
60 uranium	600	0.74	As cast	1.36
60 uranium	700	0.17	As cast	0.43
60 uranium	300	0.02	As cast	0.40

(a) Results of duplicate tests.

TABLE 14. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS^(a) IN AIR AT 662 AND 752 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change in 100 Hr at Indicated Temperature, mg per cm ²	
	Oxygen, ppm	Zirconium, w/o		662 F	752 F
10 uranium	600	0.74	Fabricated	4.5	32.4
10 uranium	700	0.17	Fabricated	1.6	25.7
10 uranium	300	0.02	Fabricated	1.07	2.34
20 uranium	600	0.74	Fabricated	2.60	44.0
20 uranium	700	0.17	Fabricated	1.44	18.2
20 uranium	300	0.02	Fabricated	4.19	20.0
30 uranium	600	0.74	Fabricated	25.2	164.0
30 uranium	700	0.17	Fabricated	20.0	27.6
30 uranium	300	0.02	Fabricated	160.0	288.0
40 uranium	600	0.74	As cast	9.0	30.2
40 uranium	700	0.17	As cast	10.0	27.6
40 uranium	300	0.02	As cast	8.9	37.1
50 uranium	600	0.74	As cast	3.74	26.7
50 uranium	700	0.17	As cast	11.0	24.0
50 uranium	300	0.02	As cast	13.1	29.6
60 uranium	600	0.74	As cast	1.82	22.2
60 uranium	700	0.17	As cast	2.31	26.2
60 uranium	300	0.02	As cast	2.81	30.4

(a) Result of duplicate tests.

In NaK at 1600 F

Fabricated niobium-10 and -20 w/o uranium alloys and niobium-30, -40, -50, and -60 w/o uranium cast alloys were exposed to NaK at 1600 F. The NaK was used in preparation for irradiation studies, as it is a common heat-transfer medium. After testing in NaK is completed, testing in sodium will be conducted. The as-cast alloys tested were made from the niobium containing 300 ppm oxygen and 0.02 w/o zirconium. The specimens were exposed in capsules prepared from stainless steel pipe. The end caps were welded into place in a helium-filled dry box. The two specimens in each capsule were completely immersed in the NaK (potassium-44 w/o sodium). The capsules were placed in a stainless steel can which had a thermocouple well fixed so that measurements from the center of the shell could be made. This center shell was held at 1600 F for 156 hr. Tests were run in duplicate.

Table 15 shows the results of the exposure to NaK. Figures 16 and 17 show representative specimens before and after exposure. The surfaces of these specimens were left rough deliberately so that the worst possible conditions for corrosion would be present. These macrographs indicate that no attack by the NaK at 1600 F should be expected. Exposure to NaK at 1600 F will be continued for a total of 1000 hr.

In CO₂

In preliminary testing, specimens were placed in a flowing CO₂ atmosphere at 700, 800, 1000, and 1200 F. At all three upper temperatures, a brown spalling product, similar in appearance to that formed in air at 752 F, was formed in 24 hr, and tests were discontinued. All niobium-uranium alloys were then exposed to a CO₂ flow of 1 liter per min for 50 hr at 600 F. The test specimens were cut from as-cast ingots for alloys of niobium-30, -40, -50, and -60 w/o uranium and from the fabricated sheet of the niobium-10 and -20 w/o uranium alloys. All specimens were run in duplicate. Testing was done in small crucibles so that any corrosion by-products present which might spall from the surface of the specimen would be retained. No spalling was noted, and a black adherent film, similar to that formed in air at 572 F, was formed. In general, the results (Table 16) compare with those of corrosion in air at 572 F. These results indicate that if a failure in the cladding occurred in a CO₂ atmosphere and the temperature was held below 600 F, no catastrophic failure would occur for short-time exposure.

In Water

The niobium-uranium alloys were also tested in 600 and 680 F water and 750 F steam. The specimens were prepared by machining approximately 0.0007 in. from each surface and pickling in a solution of nitric acid, water, and HF to remove an additional 0.0002 in. from the specimen surfaces. The specimens were then weighed and placed in autoclaves for testing. All tests were run in duplicate.

TABLE 15. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS(a) IN NaK AT 1600 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change After 156 Hr in 1600 F NaK, mg per cm ²
	Oxygen, ppm	Zirconium, w/o		
10 uranium	600	0.74	Fabricated	None
10 uranium	700	0.17	Fabricated	-0.66
10 uranium	300	0.02	Fabricated	-0.33
20 uranium	600	0.74	Fabricated	0.92
20 uranium	700	0.17	Fabricated	0.67
20 uranium	300	0.02	Fabricated	0.31
30 uranium	300	0.02	As cast	0.93
40 uranium	300	0.02	As cast	1.48
50 uranium	300	0.02	As cast	0.96
60 uranium	300	0.02	As cast	1.36

(a) Results of duplicate tests.

2X

RM12978

a. Before Exposure to NaK

2X

RM13271

b. After Exposure to NaK at 1600 F for 156 Hr

FIGURE 16. NIOBIUM-10 AND -20 w/o URANIUM ALLOYS BEFORE AND AFTER EXPOSURE TO NaK AT 1600 F

Specimens 16-1 and 16-2 are niobium-10 w/o uranium alloys prepared from niobium containing 600 ppm oxygen and 0.74 w/o zirconium. Specimens 15-1 and 15-2 are niobium-20 w/o uranium alloys prepared from niobium containing 300 ppm oxygen and 0.02 w/o zirconium.

2X

RM12974

a. Before Exposure to NaK

2X

RM13270

b. After Exposure to NaK at 1600 F for 156 Hr

FIGURE 17. NIOBIUM-50 AND -60 w/o URANIUM ALLOYS BEFORE AND AFTER EXPOSURE TO NaK AT 1600 F

Specimens 6-1 and 6-2 are niobium-50 w/o uranium alloys prepared from niobium containing 300 ppm oxygen and 0.02 w/o zirconium. Specimens 3-1 and 3-2 are niobium-60 w/o uranium alloys prepared from niobium containing 300 ppm oxygen and 0.02 w/o zirconium.

TABLE 16. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS^(a) IN CO₂ AT 600 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change After 50 Hr in 600 F CO ₂ , mg per cm ²
	Oxygen, ppm	Zirconium, w/o		
10 uranium	600	0.74	Fabricated	0.17
10 uranium	700	0.17	Fabricated	0.28
10 uranium	300	0.02	Fabricated	0.23
20 uranium	600	0.74	Fabricated	0.43
20 uranium	700	0.17	Fabricated	0.12
20 uranium	300	0.02	Fabricated	0.13
30 uranium	600	0.74	As cast	0.92
30 uranium	700	0.17	Fabricated	0.65
30 uranium	300	0.02	As cast	0.51
40 uranium	600	0.74	As cast	0.44
40 uranium	700	0.17	As cast	0.36
40 uranium	300	0.02	As cast	0.22
50 uranium	600	0.74	As cast	0.32
50 uranium	700	0.17	As cast	0.28
50 uranium	300	0.02	As cast	0.33
60 uranium	600	0.74	As cast	0.34
60 uranium	700	0.17	As cast	0.39
60 uranium	300	0.02	As cast	0.33

(a) Results of duplicate tests.

At 600 F, the weight gains are comparable to those of Zircaloy-2. They are of a magnitude of two to three times worse than Zircaloy-2. The results obtained are much better than those obtained with the unalloyed niobium. In Table 17, the constants K and n are given. These constants are from the equation

$$\Delta m = Kt^n ,$$

which is used in its logarithmic form

$$\Delta m = \log K + n \log t ,$$

where m is weight gain and t is time, K is obtained by setting $t = 1$, and n is the slope of the straight line obtained when weight gain versus time is plotted on logarithmic scale. When n is equal to 1, the rate law is linear. When it is equal to 1/2, the rate law is parabolic. As can be seen in Table 17, the rate law is parabolic for the alloys studied here. Figures 18 and 19 are examples of plots of the niobium-20 w/o uranium and niobium-50 w/o uranium alloys, respectively. Figure 20 shows plots of each composition of alloys prepared using niobium which contained 300 ppm oxygen and 0.02 w/o zirconium. No evidence of breakaway corrosion in any alloys at 600 F has been found as yet. The niobium-60 w/o uranium alloys have begun to lose weight after 6 weeks of exposure. All specimens have a black adherent film which appears to be protective for the 1000 hr of testing.

At 680 F, the results indicate that niobium-uranium alloys are more resistant to water corrosion than is unalloyed niobium. All of the alloys tested are losing weight after 6 weeks on test. This is an indication that a protective film is forming which is penetrated and flakes off, and a new film is then formed, and the cycle repeats. This is not a breakaway type of corrosion; however, if a point is reached where the rate of penetration becomes faster than the rate of healing, breakaway corrosion could result. Figure 21 shows photomicrographs of a niobium-uranium specimen which was removed from test because it cracked. The specimen surface was examined in order to determine the mechanism of corrosion. When examined in an unetched condition the type of corrosion found was intergranular with oxide concentrating in the grain boundary. The etched subsurface of the same specimen, shown in Figure 21, indicates that the corrosion mechanism apparently shifted to a transgranular type in which cracks may have been formed by stresses built up by the oxide. At the present time, it has not been determined which of the two mechanisms of corrosion is more prevalent. Test results are shown in Table 18. In 750 F steam, much the same process is occurring as at 680 F, only more severely, as would be expected. A black oxide is present; however, all alloys are experiencing losses in weight indicating spalling. Table 19 shows test results. Testing will not be continued past 1000 hr.

The corrosion testing in water indicates that the niobium-uranium alloys will perform with little difficulty for 1000 hr at 600 and 680 F if a cladding failure occurs. In 750 F steam, the exposure time can be as high as 1000 hr, but severe loss of weight will be experienced beyond 300 hr for most alloys.

TABLE 17. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS(a) IN WATER AT 600 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change in 600 F Water After Indicated Time, mg per cm ²				Corrosion Constants	
	Oxygen, ppm	Zirconium, w/o		168 Hr	336 Hr	672 Hr	1000 Hr	K	n
10 uranium	600	0.74	Fabricated	0.12	0.21	0.28	0.31	1.108	0.42
10 uranium	700	0.17	Fabricated	0.25	0.31	0.48	0.54	1.034	0.43
10 uranium	300	0.02	Fabricated	0.29	0.35	0.53	0.63	0.663	0.51
20 uranium	600	0.74	Fabricated	0.18	0.25	0.42	0.49	0.914	0.42
20 uranium	700	0.17	Fabricated	0.25	0.39	0.49	0.64	0.950	0.50
20 uranium	300	0.02	Fabricated	0.18	0.25	0.33	0.39	0.800	0.55
30 uranium	600	0.74	As cast	0.31	0.43	0.53	0.63	1.060	0.50
30 uranium	700	0.17	Fabricated	0.27	0.33	0.42	0.50	1.113	0.38
30 uranium	300	0.02	As cast	0.31	0.45	0.64	0.76	1.147	0.38
40 uranium	600	0.74	As cast	0.52	0.64	0.83	0.99	0.964	0.45
40 uranium	700	0.17	As cast	0.29	0.33	0.50	0.51	1.191	0.30
40 uranium	300	0.02	As cast	0.23	0.28	0.40	0.51	1.410	0.35
50 uranium	600	0.74	As cast	0.24	0.31	0.47	0.51	1.030	0.43
50 uranium	700	0.17	As cast	0.25	0.33	0.46	0.48	1.040	0.43
50 uranium	300	0.02	As cast	0.26	0.31	0.46	0.47	1.034	0.43
60 uranium	600	0.74	As cast	0.27	0.35	0.42	0.15	0.954	0.40
60 uranium	700	0.17	As cast	0.31	0.36	0.48	0.45	1.26	0.31
60 uranium	300	0.02	As cast	0.20	0.24	0.36	0.30	1.152	0.31
100 niobium	131	0.05	Fabricated	--	1.73	2.30	2.08	--	--
100 niobium	500	0.03	Fabricated	--	1.95	3.50	3.84	--	--
Zircaloy-2				0.09	0.12	0.14	0.16	--	--

(a) Results of duplicate tests.

FIGURE 18. EFFECT OF IMPURITIES ON THE WEIGHT GAIN OF UNALLOYED NIOBIUM AND NIOBIUM-20 w/o URANIUM ALLOYS IN 600 F WATER

FIGURE 19. EFFECT OF IMPURITIES ON THE WEIGHT GAIN OF NIOBIUM-50 w/o URANIUM ALLOYS IN 650 F WATER

FIGURE 20. WEIGHT GAINS OF VARIOUS NIOBIUM-URANIUM ALLOYS IN 600 F WATER

The niobium base for all alloys contained 300 ppm oxygen and 0.02 w/o zirconium.

a. Unetched Surface

b. Etched Subsurface

FIGURE 21 TYPICAL MICROSTRUCTURES OF NIOBIUM-50 w/o URANIUM ALLOY WHICH CRACKED AFTER EXPOSURE TO 680 F WATER FOR 1 WEEK

The alloy was prepared from niobium containing 600 ppm oxygen and 0.74 w/o zirconium.

TABLE 18. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS^(a) IN WATER AT 680 F

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change in 680 F Water After Indicated Time, mg per cm ²			
	Oxygen, ppm	Zirconium, w/o		168 Hr	336 Hr	672 Hr	1000 Hr
10 uranium	600	0.74	Fabricated	0.28	0.32	0.28	0.23
10 uranium	700	0.17	Fabricated	0.09	-1.52	-3.66	-5.66
10 uranium	300	0.02	Fabricated	-4.22	-14.3	-23.8	-31.8
20 uranium	600	0.74	Fabricated	0.30	0.48	0.61	0.74
20 uranium	700	0.17	Fabricated	0.43	0.68	1.05	1.20
20 uranium	300	0.02	Fabricated	0.22	0.38	0.55	0.66
30 uranium	600	0.74	As cast	1.00	1.19	0.38	0.21
30 uranium	700	0.17	Fabricated	0.97	0.88	0.43	-0.43
30 uranium	300	0.02	As cast	0.50	0.70	0.68	-0.82
40 uranium	600	0.74	As cast	0.66	0.75	1.00	1.08
40 uranium	700	0.17	As cast	0.46	0.43	0.30	0.29
40 uranium	300	0.02	As cast	0.46	0.65	0.80	1.05
50 uranium	600	0.74	As cast	0.83	0.47	0.29	0.29
50 uranium	700	0.17	As cast	0.44	0.39	0.40	0.23
50 uranium	300	0.02	As cast	0.38	0.27	None	0.12
60 uranium	600	0.74	As cast	1.86	-24.0	-31.5	-39.0
60 uranium	700	0.17	As cast	0.57	-0.26	-2.4	-4.29
60 uranium	300	0.02	As cast	0.31	-0.02	-1.15	-2.41
100 niobium	131	0.05		--	0.79	1.00	-1.07
100 niobium	500	0.03		--	0.41	-1.36	-46.8
Zircaloy-2				0.12	0.15	0.20	0.23

(a) Results of duplicate tests.

TABLE 19. CORROSION DATA FOR NIOBIUM-URANIUM ALLOYS^(a) IN 750 F STEAM

Alloy Content (Balance Niobium), w/o	Impurity Content		Specimen Condition	Total Weight Change in Indicated Time, mg per cm ²								
	Oxygen, ppm	Zirconium, w/o		24 Hr	72 Hr	168 Hr	336 Hr	504 Hr	672 Hr	840 Hr	1000 Hr	
10 uranium	600	0.74	Fabricated	0.25	0.36	0.51	0.49	0.24	-0.09	-0.42	-0.94	
10 uranium	700	0.17	Fabricated	0.32	0.38	-1.45	-5.59	-7.59	-9.34	-11.6	-13.3	
10 uranium	300	0.02	Fabricated	0.43	0.57	-0.43	-13.9	-18.5	-21.7	-24.3	-26.3	
20 uranium	600	0.74	Fabricated	0.30	0.45	0.56	0.63	-0.43	-5.06	-12.12	-17.00	
20 uranium	700	0.17	Fabricated	0.36	0.62	0.77	1.28	-3.87	-8.53	-15.0	-18.2	
20 uranium	300	0.02	Fabricated	0.25	0.40	0.57	0.76	0.27	-0.91	-1.60	-2.45	
30 uranium	600	0.74	As cast	0.43	0.14	-0.59	-1.88	-2.24	-2.27	-2.41	-2.80	
30 uranium	700	0.17	Fabricated	0.61	0.46	-1.65	-2.79	-3.14	-3.34	-3.31	-3.75	
30 uranium	300	0.02	As cast	0.44	0.28	-1.80	-4.20	-4.98	-5.29	-5.90	-6.00	
40 uranium	600	0.74	As cast	0.33	0.29	-0.02	-0.03	-0.28	-0.50	-0.76	-1.16	
40 uranium	700	0.17	As cast	0.38	0.24	0.14	0.00	-0.33	-0.57	-1.02	-1.67	
40 uranium	300	0.02	As cast	0.29	0.47	0.49	0.73	0.81	0.68	0.37	-0.71	
50 uranium	600	0.74	As cast	0.56	0.23	-0.31	-3.84	-6.90	-8.70	-10.8	-13.2	
50 uranium	700	0.17	As cast	0.28	0.14	-0.35	-1.54	-3.52	-5.45	-12.6	-29.7	
50 uranium	300	0.02	As cast	0.28	-0.07	-1.30	-3.95	-6.47	-7.37	-8.70	-9.7	
60 uranium	600	0.74	As cast	0.76	-0.29	-3.92	-8.22	-11.1	-16.0	-19.8	-23.1	
60 uranium	700	0.17	As cast	0.28	-0.26	-2.83	-0.49	-8.60	-11.1	-13.6	-16.1	
60 uranium	300	0.02	As cast	0.16	-0.70	-2.10	-4.32	-6.87	-9.0	-11.6	-13.7	
100 niobium	131	0.05		--	--	--	--	-3.22	-3.33			
100 niobium	500	0.03		--	--	--	--	-8.05	(b)	--	--	
Zircaloy-2				0.12	0.16	--	0.22	0.30	--	0.42	0.55	

(a) Results of duplicate tests.

(b) Completely oxidized.

CONCLUSIONS

Although the information presented in the preceding paragraphs is only interim in nature a number of interesting and important conclusions can be derived. These conclusions are as follows:

- (1) Oxygen in quantities up to 700 ppm and zirconium in amounts up to 0.74 w/o or combinations thereof in binary niobium-uranium alloys containing from 10 to 60 w/o uranium have little or no effect on the fabrication characteristics, the tensile strength, or the corrosion life in NaK, water, air, and CO₂. It is interesting to note that oxygen does have an effect on the room-temperature and hot hardness of unalloyed niobium. However, as uranium is added, the effects of the uranium addition begin to greatly override any effect of oxygen.
- (2) Alloys containing 10 and 20 w/o uranium can be readily forged in molybdenum cans at 2500 F. The resulting forgings can then be warm rolled at 1800 F to sheet. The warm-rolled sheet with the cast structure completely broken up can be cold rolled.
- (3) Difficulty has been encountered in the fabrication of alloys containing 30 w/o or more of uranium. These stem primarily from an inability to break up the cast structure satisfactorily. Once this is accomplished, warm and cold-rolling can be achieved. Studies devoted to understanding the techniques required to satisfactorily accomplish the initial breakdown are indicated.
- (4) The niobium-10 w/o uranium alloys exhibit tensile properties comparable to 300 series stainless steel at room temperature but are two to three times stronger at 1600 F. The niobium-20 w/o uranium alloys also compare favorably with the 300 series stainless steels at room temperature but are from three to five times stronger at 1600 F. Tensile properties of the rest of the alloy series will be determined as a part of the continuing program.
- (5) All alloys have sufficient resistance to oxidation in 572 F air so that exposures up to 500 hr would not induce failure. An adherent oxide film forms at this temperature; however, temperatures above 572 F produce a spalling oxide.
- (6) The resistance of all alloys to corrosion by 600 F water is comparable to that of Zircaloy-2 and superior to that of unalloyed niobium. All of the alloys have sufficient resistance to corrosion by 680 F water and 750 F steam if cladding defects should occur to prevent catastrophic failure. On the basis of available data, it is estimated that a cladding failure would not lead to serious corrosion for at least 300 hr in 750 F steam and 680 F water. No serious difficulty should occur if cladding failures were to go undetected for periods up to 1000 hr.
- (7) Tests to date indicate that the alloys do not react with NaK at 1600 F. Longer exposures in both NaK and sodium are in process.

- (8) Although exposure of the alloys to flowing CO₂ at 600 F resulted in the formation of an adherent oxide surface film, it is not recommended that these alloys be exposed to flowing CO₂ for any length of time.

DISCUSSION

The data obtained from this research to date indicates strongly that the niobium-uranium alloys have great potential as high-temperature fuel materials. These data can be further supplemented by the excellent behavior of a niobium-20 w/o uranium in radiation⁽⁷⁾. This alloy was irradiated to a uranium burnup of about 12 a/o at temperatures in the vicinity of 1200 F with no apparent damage. Postirradiation anneals up to 1800 F produced only minor dimensional changes.

Although the preponderance of mechanical-property data currently available is for the 10 and 20 w/o uranium alloys, it is probable that the strength of the niobium-30 to -50 w/o uranium alloys is higher. This, coupled with the higher uranium density of these alloys, makes them particularly attractive. As has been previously indicated, research on these alloys is continuing. Emphasis is being placed on improvement of fabrication techniques, determination of physical properties, such as thermal expansion, thermal conductivity, and electrical resistivity, and completion of mechanical-property determinations, which will include long-time creep.

This program should then culminate with a well-planned irradiation test which has been designed not only to determine behavior characteristics but also to study the correlation between tensile properties, creep in particular, and irradiation behavior.

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