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PROPERTIES OF A FISSION-TYPE ALLOY

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PROPERTIES OF A FISSIUM-TYPE ALLOY

by

Henry A. Saller
Ronald F. Dickerson
Arthur A. Bauer
Norman E. Daniel

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PROPERTIES OF A FISSIUM-TYPE ALLOY

Henry A. Saller, Ronald F. Dickerson, Arthur A. Bauer,
and Normal E. Daniel

The interest in recycled fuels for use in power reactors has led to a study of a fissium-type alloy, which may be defined as an artificial alloy representing fuel containing an equilibrium amount of fission products produced after an infinite number of irradiation and metallurgical reprocessing cycles. The nominal composition of the alloy was as follows: 2.5 w/o molybdenum, 1.5 w/o ruthenium, 0.5 w/o palladium, 0.3 w/o rhodium, 0.1 w/o zirconium, and the balance normal uranium. It was found that the alloy behaved similarly to the binary uranium-5.0 w/o molybdenum alloy during heat treatment and fabrication. The alloy ages relatively fast in the temperature range between 450 and 600 C, but it is relatively soft when quenched from 800 C or above.

Indications are that the alloy is less stable during thermal cycling than binary uranium-molybdenum alloys, and that the tensile properties of the alloy will compare favorably with the uranium-5 w/o molybdenum alloy. However, before any conclusions can be drawn in regard to the possible use of such an alloy in reactor fuel elements, a more exhaustive study of the alloy should be made.

INTRODUCTION

An increased interest in the use of recycled oxide-slugged fuels for use in power reactors has instigated an investigation of the properties of such an alloy. The reference alloy for this program represents a compromise between the high-plutonium fuels such as used in the PBR and the high-uranium fuels used in other reactors. In this report, the alloy referred to as fissium or a fissium-type alloy may be defined as an artificial alloy representing an alloy containing an equilibrium amount of fission products produced as a result of an infinite number of irradiation and pyrometallurgical reprocessing cycles.

Since reactors operate at elevated temperatures, the properties of the alloy at such temperatures should be known as well as the effects of heat treatment on these alloys.

A research program to determine these properties has been conducted at Battelle and the results of this study are presented in this report. It should, however, be emphasized that, as this investigation was designed to be of a general type, an effort has been made to cover many phases briefly

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and no attempt was made to embark on an exhaustive study of any particular phase.

ALLOY PREPARATION

Since the program was limited in scope, it was decided to concentrate the effort upon one synthetically prepared fission-type alloy. The composition of this alloy is given in Table 1.

TABLE 1. NOMINAL AND ANALYZED COMPOSITION OF THE SYNTHETIC FISSION ALLOY

Element	Nominal Composition, w/o	Chemical Analysis, w/o		
		Melt 1	Melt 2	Melt 3
Uranium	95.1	95.12	95.11	94.95
Molybdenum	2.5	2.33	2.48	2.56
Zirconium	0.1	0.10	0.11	0.10
Ruthenium	1.5	1.61	1.45	1.55
Rhodium	0.3	0.27	0.28	0.30
Palladium	0.5	0.57	0.57	0.54

The material was prepared in the form of three 6-kg ingots by induction melting in ZrO_2 crucibles and casting into 2-in. -diameter graphite molds. Extensive porosity and piping present in the castings necessitated the cropping of approximately 1/3 of the top from each ingot prior to fabrication. The chemical analyses of the ingots after cropping also appear in Table 1.

Initial attempts to forge the material at 1200 F (650 C) were unsuccessful so the forging temperature was increased to 1600 F (870 C). The alloy forged readily at this temperature. The material was then rolled from a helium-atmosphere furnace at 1250 F (675 C) to obtain material from which the test specimens were machined to the desired size.

HEAT-TREATMENT STUDIES

During casting and fabrication studies of the fission alloy, wide variations in hardness were observed among different ingots and fabricated

sections. These variations suggested different degrees of transformation and a marked dependence of the degree of transformation on the cooling rate. A brief study was therefore undertaken to determine the heat-treating and transformation characteristics of the alloy.

Experimental Procedures

In order to determine the isothermal-transformation characteristics of the alloy, fabricated-rod specimens, 1/4 in. long by 1/4 in. in diameter, were annealed in a lead bath for 1 hr at 825 C and isothermally transformed for various periods of time in lead or Wood's metal baths at temperatures ranging from 200 to 625 C. Vickers hardness measurements were then made on all specimens.

Selected specimens were prepared for metallographic examination by polishing on Forstmann's cloth, using diamond abrasive. A number of etchants were investigated, but the most satisfactory results were obtained with an electrolytic etchant consisting of phosphoric acid, ethanol, and ethylene glycol in the ratio of 5:4:4.

A few of the metallographic samples were examined by X-ray diffraction techniques. These samples were examined on the metallographically prepared surfaces in a General Electric spectrometer. Etched slivers cut from these samples were also examined in a Debye camera.

An end-quench test was run on a 5/8-in.-diameter bar about 2-1/2 in. long. The bar was sealed in an evacuated Vycor tube and was annealed 1 hr at 825 C. The Vycor tube was broken and one end was quenched in a stream of water, the other end being permitted to cool in air. Flat faces, approximately 1/4 in. wide, were machined on opposite faces of the bar for hardness measurements.

In addition, the effect of a number of furnace-cooling treatments on hardness was determined. Specimens were heat treated in a vacuum furnace under a pressure of 10^{-4} mm of mercury.

Results and Discussion

Hardness curves based on data obtained on specimens isothermally transformed at 200, 300, 400, 450, 500, 550, and 600 C are shown in Figures 1 through 6. No change in hardness was observed after 5000 sec in specimens heat treated at 625 C.

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FIGURE 1. HARDNESS CHANGES IN THE FISSION ALLOY DURING ISOTHERMAL TRANSFORMATION AT 200 AND 300 C

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FIGURE 2. HARDNESS CHANGES IN THE FISSIUM ALLOY DURING ISOTHERMAL TRANSFORMATION AT 400 C

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FIGURE 3. HARDNESS CHANGES IN THE FISSION ALLOY DURING ISOTHERMAL TRANSFORMATION AT 450 C

FIGURE 4. HARDNESS CHANGES IN THE FISSIUM ALLOY DURING ISOTHERMAL TRANSFORMATION AT 500 C

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FIGURE 5. HARDNESS CHANGES IN THE FISSIUM ALLOY DURING ISOTHERMAL TRANSFORMATION AT 550 C

FIGURE 6. HARDNESS CHANGES IN THE FISSIUM ALLOY DURING ISOTHERMAL TRANSFORMATION AT 600 C

At temperatures between 400 and 550 C, where fairly complete hardness curves were obtained, a marked change in hardness is observed with time. While the water-quenched specimen has a hardness of 115 VHN, aged specimens achieve hardnesses of over 600 VHN. X-ray diffraction results show that the structures at these respective stages are that of retained gamma in the soft condition and of alpha uranium in the very hard condition with unidentifiable traces of other phases. At intermediate stages gamma and alpha uranium are identified together.

X-ray diffraction examination of the transformed specimens revealed some evidence of strain in the alpha phase, as indicated by a broadening of back-reflection lines. However, it appears improbable that the marked hardness changes can be attributed to the formation of alpha uranium alone. Metallographic examination indicates that precipitation aging of super-saturated alpha is the more probable cause of the hardening observed.

Figures 7 through 11 illustrate typical microstructural changes which accompany transformation.

Retained gamma, illustrated in Figure 7, shows little evidence of a second phase, indicating essentially complete solution of the alloy constituents at 825 C. Fine twin bands observed within the grains are probably the result of stresses introduced on quenching.

On aging at 500 C, precipitation is first observed at the gamma grain boundaries, as evidenced by a thickening of these boundaries shown in Figure 8. With increased aging times, illustrated by Figures 9 and 10, precipitation within the grains becomes evident by a gradual darkening of the matrix. This precipitation of very fine particles that are unresolvable at low magnifications is accompanied by increased grain-boundary precipitate. Figure 11 shows a structure in which coalescence of these particles and consequent overaging has begun.

The time-temperature relationships for the hardness changes accompanying the retained-gamma decomposition have been estimated from the hardness curves and a plot of these relationships is constructed in Figure 12. Apparently, if hardness changes and transformation are directly related, the maximum transformation rate in the alloy occurs at about 475 C.

In addition to the aging mechanism just described, a low-temperature pretransformation aging phenomenon is observed in the fissium alloy. The consequences of this are illustrated in Figure 13 by the hardness changes obtained on an alloy bar which was end quenched. The hardness changes are representative of those obtained at cooling rates varying from water quenching to air cooling. A distinct hardness plateau of about 207 VHN separates two hardness increases.

250X

RM5008

N31538

Polarized Light

FIGURE 7. FISSIUM ALLOY HEAT TREATED FOR 1 HR
AT 825 C AND WATER QUENCHED

250X

RM5010

N31539

FIGURE 8. FISSIUM ALLOY HEAT TREATED FOR 1 HR AT 800 C AND
ISOTHERMALLY TRANSFORMED FOR 100 SEC AT 500 C
AND WATER QUENCHED

250X

RM5010

N31540

FIGURE 9. FISSIUM ALLOY HEAT TREATED FOR 1 HR AT 825 C AND ISOTHERMALLY TRANSFORMED FOR 400 SEC AT 500 C AND WATER QUENCHED

250X

N31541

FIGURE 10. FISSIUM ALLOY HEAT TREATED FOR 1 HR AT 825 C AND ISOTHERMALLY TRANSFORMED FOR 2000 SEC AT 500 C AND WATER QUENCHED

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500X

N31542

FIGURE 11. FISSIUM ALLOY HEAT TREATED FOR 1 HR AT 825 C AND
ISOTHERMALLY TRANSFORMED FOR 26 HR AT 500 C
AND WATER QUENCHED

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FIGURE 12. TIME-TEMPERATURE RELATIONSHIPS FOR THE BEGINNING AND END OF HARDNESS INCREASE IN AN ISOTHERMALLY TRANSFORMED FISSIUM ALLOY

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FIGURE 13. HARDNESS CHANGES IN A FISSIUM-ALLOY BAR END QUENCHED AFTER 1 HR AT 825 C

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Examination of the hardness curves obtained from isothermally transformed specimens shows that this same hardness change occurs at 200, 300, and 400 C. X-ray diffraction examination of a specimen transformed for 3 hr at 300 C revealed a retained-gamma structure with a trace of alpha uranium, indicating that the hardness increase represents an aging of the gamma phase.

It, therefore, appears that, at low temperatures, precipitation aging of the gamma phase precedes the transformation of gamma to alpha uranium. However, whether the absence of this aging phenomena at 450 C and above indicates that this pretransformation precipitation does not proceed at higher temperatures, or that the aging effects are annealed out as a result of the higher temperatures, is uncertain.

The hardness changes produced by various furnace-cooling treatments are listed below:

<u>Heat Treatment</u>	<u>Hardness, VHN</u>
1 hr at 800 C and furnace cool	468
1 hr at 800 C and furnace cool to 700 C, hold 24 hr and furnace cool	268
1 hr at 800 C and furnace cool to 625 C, hold for 24 hr and furnace cool	296

Comparison of the hardness values for the various treatments indicates that any precipitation occurring on cooling from 800 C to 700 C has no effect on the hardening mechanism in the alloy and that the most effective means of reducing the hardness in the alloy is an overaging treatment of the alpha phase at a temperature of at least 625 C.

PHYSICAL PROPERTIES

Melting Point

The melting-point determinations on the alloy were made utilizing small slivers cut from the rolled material. A Chromel-Alumel thermocouple was welded between two of the slivers and the resulting sandwich was clad in a platinum envelope. The clad envelope was then inserted into a hot furnace while a time-temperature record was obtained on a high-speed recorder. The data are given on the following page.

<u>Beginning of Melting, F</u>	<u>End of Melting, F</u>
1830	1973
1832	1980
1845	1978
Average 1836 (1002 C)	1977 (1081 C)

Fluidity

Fluidity tests were conducted in a vacuum induction-type furnace utilizing a foundry-type spiral graphite mold. The superheats used and results of these tests are listed below:

<u>Pouring Temperature, F</u>	<u>Superheat, F</u>	<u>Amount of Flow, in.</u>
2101 (1150 C)	125 (69 C)	3/16
2277 (1245 C)	300 (167 C)	1/2
2402 (1317 C)	425 (236 C)	1

Thermal Conductivity

Thermal-conductivity measurements were made by the steady-heat-flow method. During the test, the specimen was protected by a vacuum of approximately 1×10^{-5} mm of mercury. Five thermocouples, equally spaced along the length of the specimen, were used to measure the temperature drops along the specimen. Four thermal-equilibrium tests were run, yielding a total of 20 values. These values were plotted on a graph of thermal conductivity versus temperature. The data shown in Table 2 are taken from a mean curve drawn through the experimental points. The absolute accuracy of the reported values is estimated to be ± 5 per cent.

The thermal-conductivity values of the fissium alloy as well as similar values obtained on pure uranium and on a uranium-10 w/o molybdenum alloy are shown in Figure 14. From the graph it can be seen that the fissium alloy possesses greater thermal conductivity than does the uranium-10 w/o molybdenum alloy at temperatures exceeding 100 C. As the temperature is increased, this difference in thermal conductivity becomes more pronounced and at 800 C it increased approximately tenfold over its value at 100 C.

Pure uranium possesses greater thermal conductivity than either of the alloys. The greatest differences occur at the lower temperatures. However, as the temperature increases the differences in the thermal conductivities decrease and if the values for the uranium are extrapolated,

TABLE 2. THERMAL CONDUCTIVITY OF THE
FISSIUM ALLOY

Temperature, F	Thermal Conductivity, w/(cm ²)(cm)(C)
20	0.110
100	0.143
200	0.183
300	0.221
400	0.260
500	0.298
600	0.334
700	0.370
800	0.407
900	0.441

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FIGURE 14. COMPARISON OF THE THERMAL CONDUCTIVITY OF THE FISSIUM ALLOY WITH UNALLOYED URANIUM AND URANIUM-10 w/o MOLYBDENUM

it can be seen that the thermal conductivity values for the fissium and the uranium will be equal at approximately 840 C. However, it would be necessary to extrapolate the uranium and the uranium-10 w/o molybdenum curves to temperatures in excess of 1200 C before they would intersect. From these data it can be said that at temperatures within the normal operating range of a reactor the thermal conductivity of the fissium-type alloy compares more favorably with unalloyed uranium than it does to the uranium-10 w/o molybdenum alloy.

Linear Thermal Expansion

Linear-thermal-expansion measurements were made in a recording quartz-tube dilatometer. The specimen was protected by a vacuum of approximately 5×10^{-5} mm of mercury during the test. Table 3 shows the mean linear coefficients of thermal expansion over the temperature ranges shown. Figure 15 shows the curve for linear thermal expansion versus temperature for the alloy. Figure 16 shows the linear thermal expansion of a uranium-5 w/o molybdenum alloy, which is representative of uranium-molybdenum alloys containing up to 10 w/o molybdenum. Upon inspection of these graphs, it can be seen that the fissium alloy in the gamma-quenched condition exhibits a much greater difference in linear expansion between the heating and cooling cycles than do the uranium-molybdenum alloys in this composition range. There is also evidence of contraction taking place in the fissium alloy during the heating cycle at approximately 475 C. This is no doubt the result of transformation of the gamma during heating.

TABLE 3. MEAN COEFFICIENT OF LINEAR THERMAL EXPANSION OF THE FISSIUM ALLOY OVER THE TEMPERATURE RANGES SHOWN

Temperature Range, C	Mean Coefficient of Linear Thermal Expansion, per C $\times 10^6$	
	Heating	Cooling
20-100	11.0	13.7
20-200	12.5	14.4
20-300	13.6	15.1
20-400	14.0	15.7
20-500	--	--
20-650	16.6	--
20-700	16.6	23.3
20-800	16.8	23.4

FIGURE 15. LINEAR THERMAL EXPANSION VERSUS TEMPERATURE OF THE FISSIUM ALLOY WATER QUENCHED FROM 675 C

A-19363

- (A) 1 hr at 800 C, water quenched.
(C) 1 hr at 800 C, furnace cooled to 500 C, held for 24 hr, air cooled.

FIGURE 16. LINEAR THERMAL EXPANSION VERSUS TEMPERATURE FOR THE URANIUM-5 w/o MOLYBDENUM ALLOY

Density

Table 4 shows the density values of the alloy at the temperatures indicated. The room-temperature value was obtained from the measured volume and weight of the specimen. The density values at elevated temperatures were calculated from the heating thermal-expansion data. It was assumed that the alloy was isotropic and that the volume-thermal-expansion coefficient was equal to three times the linear-thermal-expansion coefficient.

TABLE 4. DENSITY OF THE FISSIUM ALLOY

Temperature, C	Density, g per cm ³
20	17.95
650	17.4
700	17.4
800	17.3

Thermal Stability

Thermal-stability tests were conducted on specimens of the alloy approximately 1/2 in. in diameter by 1 in. long. Four specimens which were hot rolled at 675 C were sealed in evacuated Vycor tubes and cycled 250 times in the temperature range from 150 to 725 C. Two of these specimens exhibited very little visual deformation as shown in Figure 17, but they grew approximately 3.1 per cent in length. The other two specimens were deformed to a greater extent and grew more than 6 per cent in length with a corresponding decrease in diameter. These two specimens are shown in Figure 18. The growth exhibited by the specimens in both cases was much greater than that exhibited by uranium-molybdenum alloys, containing 3.5 to 10 w/o molybdenum, which were cycled in the same manner. The variations in deformation and growth of these samples may be the result of minor differences in fabrication, since the rolling temperature was just above the temperature where alpha occurs.

Hot Hardness

Hot-hardness values are often an indication of the relative high-temperature strength of an alloy. The ease with which values may be

2.5X

N31543

FIGURE 17. FISSIUM-ALLOY SPECIMENS NOT EXHIBITING VISUAL DEFORMATION AFTER THERMAL-STABILITY TESTS

These specimens elongated 3.1 per cent when cycled 250 times between 150 and 725 C.

1

2

2.5X

N31544

FIGURE 18. FISSIUM-ALLOY SPECIMENS SHOWING VISUAL DEFORMATION AFTER THERMAL-STABILITY TESTS

These specimens elongated over 6 per cent when cycled 250 times between 150 and 725 C.

TABLE 5. HOT HARDNESS OF URANIUM, THE FISSIUM ALLOY, AND OF TWO URANIUM-MOLYBDENUM ALLOYS

Temperature, C	Hot Hardness, DPH kg per mm ²			
	Uranium	Fissium	U-5 w/o Mo	U-9 w/o Mo
R. T.	208	125	119	276
100	162	--	--	--
200	119	--	--	--
300	84	264	--	--
400	52	394	152	136
450	--	324	--	--
500	28	179	104	120
550	--	89	65.6	111
600	16	22.5	26	89
650	11, 30	10.6	19	69
700	33	7.1	13.2	51
750	26, 1.5	4.8	9.3	38
800	--	3.7	7.5	30

FIGURE 19. COMPARISON OF HOT-HARDNESS VALUES FOR URANIUM-MOLYBDENUM ALLOYS AND THE FISSIUM ALLOY

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TABLE 6. TENSILE PROPERTIES OF THE FISSION ALLOY, URANIUM-5 w/o MOLYBDENUM ALLOY, AND URANIUM AT SEVERAL TEMPERATURES

Test Temperature, C	Material Composition	0.2 Per Cent Offset				
		Young's Modulus, psi	Yield Strength, psi	Ultimate Strength, psi	Elongation in 2 In., per cent	Reduction in Area, per cent
290	Fission	9.9×10^6	(a)	--	--	--
300	Pure U	--	17,500	35,200	49	--
500	Pure U	--	5,450	10,450	57	--
540	Fission	8.6×10^6	12,640	32,630	13.3	25
540	Fission	9.6×10^6	15,000	43,300	8.6	17
540	U-5 w/o Mo	--	14,000	31,850	--	--
690	Fission	--	(b)	2,120	100(c)	--
690	Fission	--	(b)	2,330	100(c)	--
690	U-5 w/o Mo	--	2,810	4,070	100(c)	--

(a) Broke in grip before reaching 0.2 per cent offset. Stress in reduced section at maximum load was 77,500 psi.

(b) Temperature too high for satisfactory yield-strength determination.

(c) Specimens elongated to limit of tank but did not fracture.

Note: Tensile properties of pure uranium were taken from AECD-3647. Those of uranium-molybdenum alloys were taken from unpublished information.

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obtained over a wide temperature range makes this method practical in some cases for determining temperature ranges in which transformation of the alloy occurs. The values listed in Table 5 show a very decided decrease in hardness in the temperature range between 450 and 500 C. This is probably due to overaging or recrystallization of the alloy.

The hardness data obtained on the fabricated and water-quenched alloy and similar data obtained on pure uranium and on two uranium-molybdenum alloys, listed in Table 5, are plotted in Figure 19 on a hardness versus temperature graph. The curves exhibited by the fissium alloy and the 5 w/o molybdenum alloy are similar. The increase in hardness occurring from 300 to 450 C may be compared to the blue-brittleness phenomena in steels. At temperatures above 600 C, the fissium alloy softens faster than does either the 5 or 9 w/o molybdenum alloys. The effects of age hardening are not exhibited by the 9 w/o alloy because the time interval during which the specimen is at the elevated temperatures is not sufficient to allow the more sluggish reaction to occur. Although it would appear from the hardness readings that the fissium alloy could be fabricated at temperatures as low as 600 C, this did not prove to be true in actual practice.

Tensile Properties

The tensile properties of the alloy were investigated at 290, 540, and 690 C in vacuum with a strain rate above yield of 0.02 in. per min crosshead speed. Flat-plate-type specimens in the as-rolled condition were used for these tests. Comparative data were also obtained on pure uranium and on a uranium-5 w/o molybdenum alloy. The results of these tests are shown in Table 6. No attempt can be made to compare the tensile properties of these alloys due to the paucity of available information.

CONCLUSIONS

It is significant that the low fluidity of the alloy and its tendency to be very porous when cast under a partial pressure of an inert gas will offer some problems to those intending to utilize the alloy for fuel elements. However, once a sound casting is obtained, it can be hot fabricated readily to almost any desirable shape and size. The tensile properties of the alloy compare favorably with the uranium-5 w/o molybdenum alloy at 540 and 690 C, but unpublished data indicate that the 9 w/o molybdenum alloy is superior in this temperature range. Another factor that may be detrimental to the use of such an alloy in reactors is the extreme hardening and embrittling reaction that occurs during heat treatment.

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