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PHYSICAL PROPERTY CHANGES
IN THORIUM POSTIRRADIATION ANNEALED TO 950 C

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PHYSICAL PROPERTY CHANGES
IN THORIUM POSTIRRADIATION ANNEALED TO 950 C

By

R. D. Leggett

Metallurgy Research Section
Reactor and Materials Technology Department

January 1965

PACIFIC NORTHWEST LABORATORY
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INTRODUCTION

This report describes the mechanical properties of irradiated thorium tested in tension at 590 and 815 C and the swelling characteristics of certain selected specimens subjected to postirradiation annealing. The latter specimens were the grip ends of broken tensile specimens. Tensile properties determined for both unirradiated and irradiated specimens tested at room temperature have been reported previously.⁽¹⁾

This investigation was initiated to provide knowledge of the degree of damage induced by fast neutrons and the fission event in a material possessing an isotropic crystal structure (fcc below 1400 C). This information is not only directly useful in evaluating the potential of thorium as a fuel but indirectly useful in evaluating the cause and effects of damage in the anisotropic structure of uranium. Thorium is notably resistant to damage (growth and swelling) by neutron irradiation. The reasons for this are not well understood. Its stability is assumed to be associated with the high melting point (~ 1750 C) and the favorable crystal structure (fcc below 1400 C). Postirradiation annealing provides a convenient means of establishing the swelling characteristics of thorium and at the same time provides basic data concerning the behavior of inert gases in metals. These data should provide very realistic first estimates of the swelling that might be expected in thorium were it irradiated at a temperature equal to the postirradiation annealing temperature. Because thorium is face centered cubic, the severe anisotropic growth effects which account for irradiation swelling in uranium⁽²⁾ would be absent, and only the inert gas behavior would have to be considered.

SUMMARY

Tensile specimens of two lots of thorium were irradiated to ~ 0.06 at. % burnup, and tested at 590 and 815 C, respectively. The strength of all specimens was reduced at the elevated temperature, but the differences in yield and ultimate strengths revealed that a large amount of work hardenability

remained in the unirradiated specimens. The irradiation doubled the yield strength, greatly reduced the strain-hardenability, and significantly reduced the percent total elongation. In the irradiated specimens extensive reduction in area occurred at 590 C, whereas little occurred at 815 C. This may be due to segregation of fission products to the grain boundaries at 815 C. The effects were much smaller than have been observed with uranium and are attributed to the isotropic crystal structure of thorium.

The grip ends of broken tensile specimens from this and a previous study were examined metallographically before and after several postirradiation anneals, and hardness and density measurements were made. The only metallographic evidence of damage in the as-irradiated state was the severe fragmentation of oxide inclusions in a very high burnup (~ 1 at.%) specimen. Irradiation caused a pronounced increase in hardness; much of which was recoverable during annealing. Very little density change occurred in specimens with burnups as high as 0.18 at.% upon annealing up to 950 C for 100 hr, and even a high burnup (~ 1 at.%) specimen swelled only about 20% with this treatment. Severe grain boundary porosity existed in the latter specimen, and very tiny ($< 0.1 \mu$) pores existed throughout the matrix.

It is suggested that the high melting point of thorium results in its remarkable swelling resistance rather than its isotropic crystal structure. A secondary attribute, stemming of course from its isotropic crystal structure, is its excellent mechanical properties after irradiation.

MECHANICAL TESTING

A description of the fabrication and irradiation history and of the testing procedures was reported in detail by Bement⁽¹⁾ and, hence, will be only briefly outlined.

The room temperature tests were performed with a 60,000 lb Tinius Olsen hydraulic testing machine, and the specimens were pulled at a constant loading rate of approximately 90 lb/min. The high temperature tests were performed in a similar manner and in a dynamic vacuum. Because cross-head motion was taken as a measure of deformation, some uncertainty exists in the values derived for the yield stress. Based on reconstructed plots, the error in yield strength is estimated to be less than ± 1000 psi.

Two lots of thorium were tested. The BMI lot contained 70 ppm iron, 370 ppm carbon, and 1125 ppm oxygen. It had a grain size of 3 grains/cm² at 100X. The HAPO lot contained 155 to 315 ppm iron, 295 to 730 ppm carbon, and 850 ppm oxygen. It had a grain size of 23 grains/cm² at 100X.

The data for tests conducted at 590 and 815 C are summarized in Table I. Unirradiated control data and room temperature data are included for comparison. Specimens from the HAPO lot are stronger than those from the BMI lot when tested in the unirradiated condition and at room temperature. At elevated temperatures the strength of both unirradiated lots is greatly reduced. The differences in yield and ultimate strength reveal a large amount of work hardenability remaining. Neutron irradiation to

TABLE I
ROOM TEMPERATURE AND ELEVATED TEMPERATURE
TENSILE PROPERTIES OF IRRADIATED THORIUM

Sample Number	Testing Temperature, C	Thermal nvt x 10 ⁻²⁰	Burnup, at. %	Strength x 10 ⁻³ , psi		Total Elongation, % in 1 in.
				0.2% Yield	Ultimate	
<u>BMI Material</u>						
CT-1	RT	0	--	16.7	24.4	41
CT-2	RT	0	--	16.4	24.6	50
CT-6	RT	0	--	17.5	26.5	57
CT-8	815	0	--	~7	~17	~43
EX	RT	8.1	0.077	44.2	44.6	24
GX	RT	7.1	0.058	44.5	44.9	29
AX	590	7.3	0.060	~24	~27	~22
CX	815	7.6	0.065	~20	~21	~9
<u>HAPO Material</u>						
CT-9	RT	0	--	21.0	31.7	55
CT-12	RT	0	--	22.3	29.9	(a)
CT-10	590	0	--	~9	~20	~92
CT-4	815	0	--	~8	~16	~26
DX	RT	7.7	0.070	50.5 ^(b)	51.1	(a)
FX	RT	8.4	0.085	48.6 ^(b)	49.5	(a)
9X	590	7.3	0.060	~22	~23	~24
BX	815	7.5	0.065	~19	~19	~16

(a) Sample broke outside of 1 in. gage length

(b) Lower yield strength

about 7.5×10^{20} nvt (< 1 MeV) more than doubles the yield strength, greatly reduces strain hardenability, and significantly reduces the percent total elongation at room temperature. At testing temperatures of 590 and 815 C, the ultimate strength is only slightly greater than the corresponding unirradiated values. However, the yield strengths are about 20,000 psi compared to about 10,000 psi in the unirradiated state. This reveals that a marked impedance to plastic flow, due to either neutron damage or fission products or both, is active at the high testing temperatures.

Examination of the tensile failures of the irradiated specimens tested at 590 and 815 C showed that extensive reduction in area had occurred at 590 C; whereas, essentially no reduction in area had occurred at 815 C. This result is illustrated in Figure 1. It is compatible with the comparatively low values obtained for total elongation during the 815 C tests (Table I). Because the room temperature tests also showed extensive reduction in area for an exposure of 7×10^{20} nvt, the apparent brittleness at 815 C may be related to a change in the mode of deformation. Deformation should be intragranular below and intergranular above the equicohesive temperature. Available data for the unirradiated state places the equicohesive temperature for thorium at about 400 C. The amount of shift that may occur in this temperature with irradiation is unknown. However, the present testing temperatures are well above the value for unirradiated thorium. The data reveal that grain boundaries play an important role in the deformation, particularly at 815 C. If significant segregation of fission products occurs at the grain boundaries at these testing temperatures, brittle fracture will be promoted. This is currently accepted as the explanation for the reduced ductility displayed at 815 C for both the HAPO and BMI lots.

SWELLING DURING POSTIRRADIATION ANNEALING

Some of the grip ends of broken tensile specimens were examined metallographically for evidence of swelling, and certain of these were subjected to additional vacuum annealing treatments. Data from the hardness and density measurements are summarized in Table II.

AX

9X

BX

CX

AX: BMI Thorium Tested at 590 C BX: HAPO Thorium Tested at 815 C
9X: HAPO Thorium Tested at 590 C CX: BMI Thorium Tested at 815 C

FIGURE 1

Broken Thorium Tensile Specimens Irradiated to 0.06 at. % Burnup
and Tested at Elevated Temperatures

Neg. A-1076, A-1112

Irradiation increases the hardness of thorium, and recovery occurs on annealing at 750 C. Specimen S-3 was significantly softer in the center than on the edge. This sample was irradiated in a somewhat higher flux than other specimens of this series and may have operated at a temperature sufficiently high to permit some recovery of irradiation damage, particularly in the center. Both areas recovered in hardness on annealing.

The density measurements indicate that thorium undergoes a slight amount of densification (0.5%) at about 0.1 at.% burnup; but, at burnups beyond this, density decreases again as the effect of the lighter fission products overcomes the effect of the heavy transmutation products. Specimen S-3 (0.92 at.% burnup) exhibited a density that is almost 1.5% less than the original density in the as-irradiated condition. Annealing at 750 C caused a slight volume increase in the two higher burnup specimens, B-2 and S-3, but none was detected in the lower burnup specimen JX. After the 950 C anneal, specimen S-3 showed pronounced swelling (21%), and B-2 showed a significant increase (0.6%). Both B-2 and S-3 were processed for metallography in each of the three conditions indicated in Table II; the other specimens were only processed for metallography in the one condition listed in Table II. After the 950 C anneal, B-2 and S-3 had become quite thin and difficult to handle.

The as-polished and lightly etched appearance of samples CT-9, FX, and JX are indicated in Figure 2. The grains of these specimens are all small,

TABLE II
HARDNESS AND DENSITY OF IRRADIATED THORIUM

Sample	Condition	Burnup, at. %	Hardness, R _H	Density, g/cm ³	% Swelling $\frac{\rho_1 - \rho_f}{\rho_f} \times 100$	"R", % Swelling at. % Burnup
CT-9	Nonirradiated	0	62 ^(a)	11.67	--	--
FX	As-irradiated at ~200 C	0.085	64	11.73	(-) 0.5	(-) 5.9
JX	Irradiated at ~200 C + 100 hr annealed at 750 C	0.095	33	11.74	(-) 0.6	(-) 6.3
B-2 ^(b)	As-irradiated at ~200 C	0.18	78	11.67	0	0
B-2 ^(b)	Annealed at 750 C; 100 hr		46	11.65	0.2	1.1
B-2 ^(b)	Annealed at 950 C; 100 hr		No data	11.60	0.6	3.3
S-3	As-irradiated at 300 C	0.92	69	11.50	1.5	1.5
S-3	Annealed at 750 C; 100 hr		48	11.45	1.9	2.1
S-3	Annealed at 950 C; 100 hr		No data	9.62	21.3	23.2

(a) R_F SCALE. This sample was too soft for R_B scale.

(b) BMI material, 70 ppm Fe; 370 C; 1125 ppm O; grain size of 3 grains/cm² at 100X. Other samples from IIAPO lot: 155 to 315 ppm Fe, 295 to 730 C; 850 ppm O; grain size of 23 grains/cm² at 100X.

The as-polished and lightly etched appearance of samples CT-9, FX, and JX are indicated in Figure 2. The grains of these specimens are all small, equiaxed, and uniform in size, 40 μ in diameter. There is no observable distortion of either the matrix material or the grain boundaries. The thorium contains large oxide inclusions (dark angular particles in the as-polished condition) as well as a light gray spheroidized phase and a lighter grain boundary network precipitate. The identity of these phases is uncertain; the light gray material is assumed to be thorium carbide and the grain boundary network Th_7Fe_3 . Additional study will be necessary to positively identify the phases. It is interesting to note that the grain boundary network precipitate is not at the final boundaries but at some prior set of boundaries. This network precipitate had grown in the irradiated specimen JX during the 100 hr anneal at 750 C.

Figure 3 illustrates the structure of an etched surface of the unirradiated thorium control material as viewed in the electron microscope. The tentative identification of the second phase constituents is indicated on the electron micrograph of Figure 3. Samples FX and JX presented a similar appearance.

The microstructures of Specimens B-2 and S-3 are compared in Figure 4. Sample B-2 (from the BMI lot of thorium) has slightly larger grains, somewhat fewer carbides, and vary little of the prior grain boundary network phase. Th_7Fe_3 , that was observed in the specimens described in Figure 2. This correlates with the lower iron content and the tentative identification of the network phase. Specimen S-3, irradiated to 0.92 at.% burnup, is distinctly different from the other samples. The oxide inclusions are no longer single particles dispersed throughout the matrix but appear to be clusters of many particles. This appearance of the oxides persisted throughout the two anneals that this sample received. Thus, it is concluded that this fragmentation of the oxides is a real irradiation effect. Figure 5 shows one of these fragmented oxide particles as it appeared in the electron microscope. Other than this no twisting of grain boundaries, formation of twins or other microstructural evidence of irradiation damage was observed. This, of course, is in sharp contrast to highly anisotropic uranium metal which exhibits such pronounced changes in microstructure with irradiation.

As-Polished

Lightly Cathodic Vacuum Etched

B3762

a. CT-9 Unirradiated Control

B3966

B4104

b. FX Irradiated to 0.085 at.% Burnup at <200 C

B3975

B3774

c. JX Irradiated to 0.095 at.% Burnup at <200 C
and then Vacuum Annealed at 750 C for 100 hr

B3988

FIGURE 2

Microappearance of Irradiated and Nonirradiated Thorium
(500X)

FIGURE 3

Microstructure of Nonirradiated Thorium (CT-9)
Showing Oxides, Carbides, and Th₇Fe₃ Second Phase
(7500X)

Neg. 1527C

As-Polished

B-5034

(250X)

As-Cathodic Vacuum Etched

B-5040

(500X)

a. B-2 Irradiated to 0.18 at.% Burnup at ~200 C

B-5053

(250X)

B-5062

(500X)

b. S-3 Irradiated to 0.92 at.% Burnup at ~300 C

B-5054

(250X)

B-5069

(250X)

c. Crack in Specimen S-3

FIGURE 4

Microappearance of Irradiated Thorium

FIGURE 5

Thorium Oxide Particle in Thorium Specimen S-3
Irradiated to 0.92 at. % Burnup at ~ 300 C
Showing Fragmentation That Occurred During Irradiation
(7000X)

Neg. 1644C

Sample S-3 also contained a large crack that branched into several smaller cracks which followed grain boundaries. The origin of the crack is uncertain and may have formed independent of the irradiation. A crack may have been present in the original material which propagated during tensile testing, or it may have formed during the test. The fact that this sample exhibited limited ductility during the tensile test and broke in the shoulder is possibly evidence that the crack existed in the original material. The electron micrographs of Specimen S-3 (Figure 6) indicate some grain boundary porosity which may be either fission gas pores or voids formed during the tensile test. Faceting during cathodic vacuum etching was also appreciably less with Specimens B-2 and S-3. This etching effect may be real and the result of irradiation. Additional study of this effect is necessary.

The vacuum anneal at 750 C for 100 hr did not alter the microstructure of either B-2 or S-3. After reannealing at 950 C for 100 hr, Specimen B-2 (0.18 at.% burnup) had still changed very little, but Specimen S-3 developed gross porosity at the grain boundaries. The optical metallographic appearance of these specimens is shown in Figure 7 at 250 and 1000X. Some grains in both specimens showed a pronounced pitting attack that was apparently sensitive to grain orientation with respect to the sectioned surface. Etching the specimens a second time did not eliminate the effect although the severity was somewhat reduced. It is difficult to assess whether this phenomenon is associated with a basic metallurgical change in the metal or is due to etching conditions.

In Specimen S-3 the grain boundary porosity has coalesced into grain boundary cracks. Examination of replicas of these surfaces in the electron microscope revealed large pores at the grain boundary in Sample S-3 and many very tiny ($<0.1 \mu$) pores in the general matrix. Figures 8 and 9 indicate the appearance of one of the cleaner areas in Specimen S-3. It was not possible to photograph the boundaries that contained the bulk of the porosity as the replica broke in these regions. The segregation of large pores at grain boundaries can be clearly seen in Figures 8 and 9 along with the zones depleted in tiny pores adjacent to the boundaries. Very careful examination

FIGURE 6

Microappearance of Thorium Specimen S-3
Irradiated to 0.92 at.% Burnup at ~ 300 C
(Note fine porosity at the grain boundaries)
(7500X)

Neg. 1643A

(250X)

(1000X)

B-2 (0.18 at. % Burnup)

(250X)

(1000X)

S-3 (0.92 at. % Burnup)

FIGURE 7

Thorium Specimens Postirradiation Annealed
at 750 C for 100 hr Followed by 100 hr at 950 C

FIGURE 8

Thorium Specimen S-3 (0.92 at.% burnup) Annealed at 750 C for 100 hr
Followed by an Anneal at 950 C for 100 hr
(7500X)

Neg. 1963D

Grain
Boundary

(15,000X)

(30,000X)

FIGURE 9

Thorium Specimen S-3 (0.92 at.% burnup) Annealed at 750 C for 100 hr
Followed by an Anneal at 100 hr at 950 C

(Note denuding of tiny matrix pores at the grain boundary)

Neg. 1976B, 1976C

of replicas prepared from Specimen B-2 disclosed only an occasional small pore at grain boundaries. The microscopy is in general agreement with the density measurement.

RESULTS AND CONCLUSIONS

The relative effects of irradiation on the mechanical properties of thorium and uranium have been extensively discussed previously.⁽¹⁾ In general, the marked superior performance of thorium over that of uranium has been explained on the basis of its isotropic, face centered cubic, crystal structure (as opposed to the highly anisotropic, orthorhombic crystal structure of uranium). The high temperature tensile test data reported here essentially corroborate this concept.

Available high temperature tensile data for uranium irradiated at 0.07 at.% burnup reveal a marked structural dependency. Whereas comparable ductility (about 36% elongation) occurs in the unirradiated state for the alpha, beta, and gamma phases, the gamma phase exhibits about 7 to 11% elongation after 0.07 at.% burnup compared to less than 1% in the alpha and beta phases. At this burnup, the gamma phase of uranium, which is body centered cubic, exhibits at 800 C comparable ductility to face centered cubic thorium at 815 C. However, it must be noted that under these conditions⁽¹⁾ the total exposure to neutrons and, hence, the fast neutron damage to thorium is much the greater, and⁽²⁾ the homologous temperature (based on absolute melting temperature) is much less for thorium than for uranium. For a testing temperature of 590 C and a burnup of 0.07 at.%, thorium is still reasonably ductile (25% elongation), while uranium is quite brittle. This again emphasizes the effect of crystal structure, which at this temperature is complex orthorhombic, for uranium and is face centered cubic for thorium.

The marked resistance to swelling during postirradiation annealing exhibited by the thorium specimens examined in this study may also be related to its isotropic crystal structure. Uranium, of course, has been observed to swell many times more when subjected to the same postirradiation heat treatments employed here. Care must be taken, however, that the

comparisons are made in the appropriate frame of reference. Thorium certainly resists volume change to a much higher temperature than does uranium. If, however, comparisons are made at the same homologous temperature (i.e., fraction of the absolute melting point) and if, further, the swelling values are divided by the burnup ("B" in Table II) to normalize to roughly the same amount of gas in the lattice, one is lead to the conclusion that both metals exhibit the same fission gas-induced volume increase.

The 950 C annealing temperature employed here with thorium is the same homologous temperature as is 575 C for uranium. If the data at the same homologous temperature are normalized to unit burnup, respective "R" values for thorium and uranium are 24 and 21. Thus it may well be the high melting point of thorium (approximately 1700 C) rather than the isotropic crystal structure that is responsible for its apparent superior swelling resistance. A complication arising with uranium, of course, is the fact that the volume changes which occur in-reactor in the alpha phase are not caused by fission gas porosity but rather by mechanical processes brought about by the fission event itself.⁽²⁾ Thorium, being isotropic, does not exhibit this behavior and is thus less prone to damage at equivalent fuel temperatures. This comparison was based on thorium specimen S-3 which had a very high burnup (1 at.%). Specimen B-2 which had only a 0.18 at.% burnup showed an appreciably smaller "R" value, indicating perhaps that there may be a burnup threshold above which uranium and thorium may behave similarly and below which thorium is far superior.

Insofar as the application of thorium as a fertile material is concerned, these results indicate that it would behave very well at temperatures at least as high as 750 C and in all probability a great deal higher. Obviously, more data than those reported here will be necessary to evaluate and understand fully the irradiation behavior of thorium. Other studies^(3, 4) have shown that the excellent stability of thorium is retained in fuels made by alloying with uranium. This is to be expected so long as the additive remains in solution or precipitates as discrete particles in a predominantly thorium matrix.

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2	BROOKHAVEN NATIONAL LABORATORY	1	MARTIN-MARIETTA CORPORATION
1	BUREAU OF MINES, ALBANY	1	MINNESOTA MINING AND MANUFACTURING COMPANY (BRANDT)

TID-4500
(39th Ed.)

UC-25
METALS, CERAMICS, AND MATERIALS

Ptd.	Standard Distribution	Ptd.	Standard Distribution
1	MOUND LABORATORY	1	SAN FRANCISCO OPERATIONS OFFICE
		2	SANDIA CORPORATION, ALBUQUERQUE
1	NASA LEWIS RESEARCH CENTER	1	SANDIA CORPORATION, LIVERMORE
		1	SOUTHWEST RESEARCH INSTITUTE
1	NASA MANNED SPACECRAFT CENTER	1	STANFORD UNIVERSITY (SLAC)
2	NASA SCIENTIFIC AND TECHNICAL INFORMATION FACILITY	1	SYLVANIA ELECTRIC PRODUCTS, INC.
		1	TENNESSEE VALLEY AUTHORITY
2	NATIONAL BUREAU OF STANDARDS	1	TRW SPACE TECHNOLOGY LABORATORIES (NASA)
1	NATIONAL BUREAU OF STANDARDS (LIBRARY)	1	UNION CARBIDE CORPORATION, CLEVELAND
2	NATIONAL LEAD COMPANY OF OHIO	2	UNION CARBIDE CORPORATION (ORGDP)
4	*NATIONAL REACTOR TESTING STATION (PPCO)	5	UNION CARBIDE CORPORATION (ORNL)
1	NAVAL POSTGRADUATE SCHOOL		
3	NAVAL RESEARCH LABORATORY	1	UNION CARBIDE CORPORATION (PADUCAH PLANT)
		2	UNITED NUCLEAR CORPORATION (NDA)
1	NRA, INC.	1	U. S. GEOLOGICAL SURVEY, DENVER
1	NUCLEAR MATERIALS AND EQUIPMENT CORPORATION	1	U. S. GEOLOGICAL SURVEY, MENLO PARK
1	NUCLEAR METALS, INC.	1	U. S. GEOLOGICAL SURVEY, WASHINGTON
1	NUCLEAR UTILITY SERVICES, INC.	1	U. S. PATENT OFFICE
1	OFFICE OF ASSISTANT GENERAL COUNSEL FOR PATENTS (AEC)	2	UNIVERSITY OF CALIFORNIA, BERKELEY
2	OFFICE OF NAVAL RESEARCH	2	UNIVERSITY OF CALIFORNIA, LIVERMORE
1	OFFICE OF NAVAL RESEARCH (CODE 422)	1	UNIVERSITY OF PUERTO RICO
1	OHIO STATE UNIVERSITY	1	WESTERN RESERVE UNIVERSITY (MAJOR)
1	PETROLEUM CONSULTANTS	4	WESTINGHOUSE BETTIS ATOMIC POWER LABORATORY
1	PHYSICS INTERNATIONAL, INC.	1	WESTINGHOUSE ELECTRIC CORPORATION
1	PICATINNY ARSENAL	1	WESTINGHOUSE ELECTRIC CORPORATION (NASA)
1	POWER REACTOR DEVELOPMENT COMPANY	285	DIVISION OF TECHNICAL INFORMATION EXTENSION
3	PRATT AND WHITNEY AIRCRAFT DIVISION		
1	PURDUE UNIVERSITY		
1	RADIOPTICS, INC.		
1	RAND CORPORATION		
1	REACTIVE METALS, INC.		
1	REACTIVE METALS, INC., ASHTABULA		
1	RENSSELAER POLYTECHNIC INSTITUTE		

*New listing or change in old listing.