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RECORD COPYSWELLING OF URANIUM ALLOYS AT HIGH EXPOSURES

W. R. McDonnell and W. N. Rankin

The attached paper summarizes the Savannah River Laboratory contribution to the Twenty-fifth High Temperature Fuels Committee Meeting held at Gulf General Atomics, San Diego, California, December 5, 6, and 7, 1967. The paper was transmitted as a passout to members of the Committee. The paper reports results of a program of fundamental studies of swelling-resistant uranium-alloys supported at SRL until termination of funding on June 30, 1967, by the Division of Reactor Development and Technology. A proposal for completion of the work under funding by the Heavy Water Office, Battelle-Northwest, has been submitted.*

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Attachment

* W. R. McDonnell, "Status of Irradiation Tests of Dilute Uranium Alloys in NaK-Containing Stainless Steel Capsules." DPST-67-556; Transmittal letter J. W. Morris to H. Harty, DPST-67-556-TL, October 13, 1967.

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Savannah River Laboratory Contribution
Twenty-fifth High Temperature Fuels Committee Meeting
General Atomics, San Diego, California
December 5, 6, and 7, 1967

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SWELLING OF URANIUM ALLOYS AT HIGH EXPOSURES*

by

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HIGHLIGHTS

1. Irradiation of a group of dilutely alloyed uranium specimens to nominal exposures of 13,000 MWD/T showed that the most swelling resistant compositions were alloys of U - 1.5 to 4.0% Mo - 0.1% Si, that had been solution-treated and quenched in water. Of the very dilute alloys containing Fe, Si, and Al, specimens with high (800 ppm) Al, were more swelling resistant at high exposures than specimens with intermediate (350 ppm) Si, which were more swelling resistant at lower exposures (5000 MWD/T). The reversal in relative swelling resistance of the alloys with increasing exposure is attributed to irradiation-induced dispersion of the aluminum constituents.

* The information contained in this article was developed during the course of work under Contract AT(07-2)-1 with the U. S. Atomic Energy Commission.

SPECIAL REVIEW
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2. The relative swelling resistance of the alloys correlated in a general way with their mechanical properties after heat treatments that simulated the distribution of alloy constituents at the various stages of irradiation. Swelling resistance at intermediate exposures paralleled the hot hardness of specimens in the beta-treated condition, in which Si but not Al constituents were dissolved; swelling resistance at high exposures paralleled the hot hardness of specimens in the gamma-treated condition, in which both Si and Al constituents were dissolved up to maximum concentrations.
3. A mechanism is proposed for the formation of grain boundary and aligned cavities within grains by coalescence of vacancy dislocation loops produced by the recoiling fission fragments. Inhibition of the glide of the loops into coplanar arrays necessary for coalescence to form cavities would account for the stabilizing effects of alloying additions.

IRRADIATION STABILITY OF URANIUM ALLOYS
AT HIGH EXPOSURES (13,000 MWD/T)

Postirradiation examinations were begun of a series of unrestrained dilute uranium alloy specimens irradiated to exposures up to 13,000 MWD/T in NaK-containing stainless steel capsules. This test, part of a program of development of uranium metal fuels for desalination and power reactors sponsored by the Division of Reactor Development and Technology, has the objective of defining the temperature and exposure limits of swelling resistance of the alloyed uranium. The uranium specimens contain small additions of Fe, Si, Al, Cr, Mo, or Zr, and were heat treated by various procedures to determine the stabilizing effects of different microstructural distributions of the alloy constituents. The specimens were irradiated to three exposures (3000, 9000, and 13,000 MWD/T) at calculated central temperatures from 300 to 800°C. The results at 13,000 MWD/T are summarized in this report. Examination of specimens irradiated to 9000 and 3000 MWD/T is incomplete.

Among the very dilute alloys irradiated, the most stable at high exposures (13,000 MWD/T) contained high Al and Si (800 ppm); in contrast, intermediate Si compositions (350 ppm) were most stable in previous tests at lower exposures (5000 MWD/T).⁽¹⁾

The swelling data for the specimens irradiated to 13,000 MWD/T are arranged in Table I in the order of decreasing stability, as determined by the threshold temperature for cavitation swelling. The threshold temperature for cavitation swelling at this exposure was considered as the temperature at which the total swelling was 6% (2% in excess of the volume increase due to solid fission products). In most cases, density was not measured for those specimens that had obviously swelled a great deal more than 6%.

TABLE I.

Swelling Data for Specimens Irradiated to 13,000 MWD/T

Alloy	Decrease in Density After Irradiation at Indicated Nominal Temperature, % ^(a)					Alloy Composition, ppm ^(b)					Heat Treatment			
	300°C	450°C	550°C	590-660°C	700-830°C	Fe	Si	Al	Cr	Mo	Phase	°C	Time	Cooling
1	2.8	2.7		2.6	7.2					4.0 wt %	γ	1050	24 hr	Water
2	3.3	2.6		4.8	10.3					2.25 wt %	γ	1050	24 hr	Water
3	1.2	3.2	4.6		10.3					1.5 wt %	γ	1050	24 hr	Water
4	3.6	4.1	7.2	S	S	350		800			γ	950	20 min	Oil
5	3.8	3.4	15.6	15.3	18.4	350	350	800		1000	β	725	10 min	Oil
6	3.7	3.9	S	-	-	250	300	800	200		β	725	10 min	Oil
7	3.5	4.8	S	-	S					1.5 wt %	γ	800	20 min	Oil
8	5.1	4.2	25.6	43.5	S	350	350	800			β	725	10 min	Oil
9	4.1	3.9	31.0	38.8	S		800	800			β	725	10 min	Oil
10	4.4	3.7	35.0	-	-		350	800			β	725	10 min	Oil
11	4.5	5.4	38.4	-	-		800				β	725	10 min	Oil
12	4.5	5.0	41.5	-	-	350		800			β	725	10 min	Oil
13	4.6	9.4	S	-	-		350			1000	β	725	10 min	Oil
14	3.4	10.6	S	-	-					1.5 wt %	γ	1050	24 hr	Water
15	5.0	13.7	-	-	S	250	350				β	725	10 min	Oil
16	3.9	17.0	-	-	-					1.5 wt %	γ	800	20 min	Furnace to 500°C, Oil
17	4.4	19.0	-	S	-	250	250	250			β	725	10 min	Oil
18	4.4	26.0	S	-	-	(Unalloyed ingot uranium)					γ	950	20 min	Oil
19	4.9	26.0	-	S	-		350				β	725	10 min	Oil
20	5.7	S	-	S	-	(2 wt % Zr)					γ	800	20 min	Water
21	6.1	S	S	-	-	150	100	100			β	725	10 min	Oil
22	6.1	S	-	-	-	(Unalloyed ingot uranium)					β	725	10 min	Oil
23	6.3	48.0	S	-	-	(2 wt % Zr)					γ	800	20 min	Furnace to 500°C, Oil
24	19.1	21.4	-	-	21.7		0.1 wt %			0.5 wt %	γ	1050	24 hr	Water

(a) Actual temperatures probably 50-150°C lower.

(b) Composition in ppm, except as noted.

S Specimen obviously swelled more than 6%.

- Specimen not examined since threshold had been found at lower temperature.

The listed temperatures are the nominal values and are subject to downward revision by 50-150°C when the swelling of calibration specimens at lower exposures is compared with the results of previous tests. The following principal conclusions were drawn:

- o The highest stability is exhibited by U - 1.5 to 4.0% Mo - 0.1% Si alloys (Alloys 1, 2, and 3) that were solution-treated in the high gamma phase region (1050°C) and quenched in water. However, a U - 0.5% Mo - 0.1% Si (Alloy 24) swelled severely - probably due to internal cracking during the severe water quench.
- o Among the very dilute alloys, the specimens containing high Al or Si additions (800 ppm) (Alloys 5, 6, 8, 9, 10, 11, 12) were more stable at 13,000 MWD/T than those containing intermediate (250-350 ppm) Si without high Al (Alloys 15, 17, 19). This contrasts with a previous test at 5000 MWD/T, in which the intermediate Si alloys were the most stable.
- o U - 350 ppm Si - 1000 ppm Mo (Alloy 13), which was the most stable in the previous test, was the best of the intermediate Si specimens in the present test, but was less stable than those containing 800 ppm Al (Alloys 5, 6, 8, 9, 10, 12).
- o Among the beta-treated, oil-quenched alloys with 800 ppm aluminum, those containing Si, Mo, and possibly Cr, as well as Fe (Alloys 5, 6, 8, 9, 10) were somewhat more stable than the alloy containing only Fe and Al (Alloy 12). As expected, the very dilute alloys of Fe and Si (100-150 ppm) (Alloy 21) and unalloyed ingot uranium (Alloy 22) swelled more than the alloys containing larger amounts of additives.

- o A high-temperature treatment of the dilute alloys in the gamma phase, designed to produce finely dispersed carbide in the metal, effectively increased the stability of U - Fe - Al alloy (Alloy 4) as well as that of unalloyed ingot uranium (500 ppm C) (Alloy 18).
- o The binary U - 1.5% Mo alloy without Si (Alloy 14) swelled more than the similar composition with 0.1 wt % Si (Alloy 3). Oil quenching the U - 1.5% Mo (Alloy 7) from 800°C (low-temperature region of the gamma phase) produced better stability than cooling slowly from the same temperature (Alloy 16). This test demonstrated that, in relatively concentrated alloys, the effect of heat treatment may persist to high exposures, whereas in dilute alloys heat treatment has little persistent effect.
- o The U - 2% Zr binary (Alloy 23) was less stable than U - 1.5% Mo (Alloy 16); little difference was noted between U - 2% Zr (Alloys 20, 23) quenched or cooled slowly from 800°C.

CORRELATION OF MECHANICAL PROPERTIES
WITH IRRADIATION BEHAVIOR

Previously reported measurements were reviewed to establish if the mechanical properties of uranium containing dilute alloying additions could be correlated with irradiation behavior. A general correlation of swelling with mechanical strength can be made, provided the irradiation-induced dissolution of the alloy constituents is considered.

The mechanical properties, established principally by hot hardness measurements,⁽²⁾ depend on alloy content and heat treatment

in a manner which reflects the distribution of alloy constituents in the structure. The dominating strengthening mechanism of the alloys is solid-solution hardening, as produced by solution-quench treatments which retain the low-solubility alloying elements in metastable solid solution. The solid-solution hardening is generally reduced by slow cooling or annealing which precipitate the alloy constituents, except in alloys (principally the Al-containing alloys) in which the alloy constituents are distributed finely enough to produce dispersion hardening.

The dilute alloying elements are soluble in the uranium approximately as follows:

Temp, °C	Concentration, ppm				
	Fe	Si	Al	Cr	Mo
650 (α -phase)	20	150	<80	~1000	~2000
720 (β -phase)	700	1000	350	2000	100% $\beta + \gamma$
800 (γ -phase)	3100	1000	2100	8000	100% γ

Beta treatments, therefore, normally dissolve iron and silicon constituents in the dilute alloys, but not the aluminum constituents, which require gamma treatments. Interaction between the alloying elements may alter these solubilities; for example, molybdenum in silicon-containing alloys forms a complex compound $U_4Mo_5Si_3$ which dissolves above 800°C.

In accord with these relationships, specific effects of the various alloying additions on hot hardness are as follows:

- o In beta-quenched alloys containing Fe, Si, and Al, hot hardness increases with increasing iron and silicon content (Fig. 1) but is nearly independent of aluminum content, in agreement with relative quantities of the alloying elements dissolved at

beta-phase temperatures. In gamma-quenched alloys, however, hot hardness increases with increasing aluminum, iron, and silicon, up to maximum alloying element concentration (Fig. 2).

- o Incremental additions (1000 ppm) of molybdenum to the Fe, Si, and Al alloys produce somewhat higher hot hardness after beta quench, though not after gamma quench, than alloys without molybdenum. The strengthening effects of the high-solubility molybdenum additions are thus apparent only in the absence of the more pronounced effects of the low-solubility aluminum additions.
- o Annealing (aging) treatments which precipitate alloy constituents from the solution-quenched specimens generally soften the metal, except for the aluminum-containing alloys for which particle distributions are fine enough (<1 micron spacing) to produce dispersion hardening. Typical interparticle spacing in alloy specimens annealed after gamma quenching is shown in the following table.

Interparticle Spacing of Dilute Uranium Alloys
(Gamma treated 800°C, 1 hr; annealed 600°C, 24 hr)

<u>Nominal Composition, ppm</u>	<u>Interparticle Spacing, microns</u>
U - 350 Si	6.0
U - 250 Fe - 350 Si	3.0
U - 250 Fe - 250 Si - 250 Al	1.0
U - 350 Si - 800 Al	0.3
U - 350 Si - 1000 Mo	5.0

- o Extended-time tests of mechanical properties, such as creep penetration* or stress rupture⁽³⁾ at high alpha temperatures reflect the effect of alloying additions only up to the limit of their alpha-phase solubility, except for the aluminum-containing alloys for which some dispersion hardening occurs.

Correlation of these results with the swelling behavior of the alloys is complicated by emerging evidence that the relative resistance of the alloys to swelling (e.g., the temperature threshold for cavity formation) depends on exposure, as previously described. The changes in the relative swelling resistance of the alloys may be accounted for by irradiation-induced changes in the distribution of alloy constituents in the metal, which are progressively dissolved (or are otherwise dispersed to below the limits of resolution by replica electron microscopy) during irradiation. The differing solubilities, as well as the differing initial distributions of the alloying elements, can account for the relative rates at which the alloying elements are dissolved or dispersed in the metal during irradiation. Thus the aluminum constituent, which was initially in a relatively coarse dispersion, tends to require longer exposures to dissolve under irradiation than the silicon constituents, which were initially in metastable solid solution or a very fine dispersion.

These differences in the behavior of the various alloy constituents during irradiation parallel the dependence of the mechanical properties of the alloys on heat treatment. For intermediate irradiation exposures at which silicon is in solid solution but aluminum may be incompletely dispersed, the relative swelling

* In the creep-penetration test, a load is applied to a hardness indenter over an extended time at elevated temperature to produce an indentation indicative of the hardness of the specimen under these conditions.

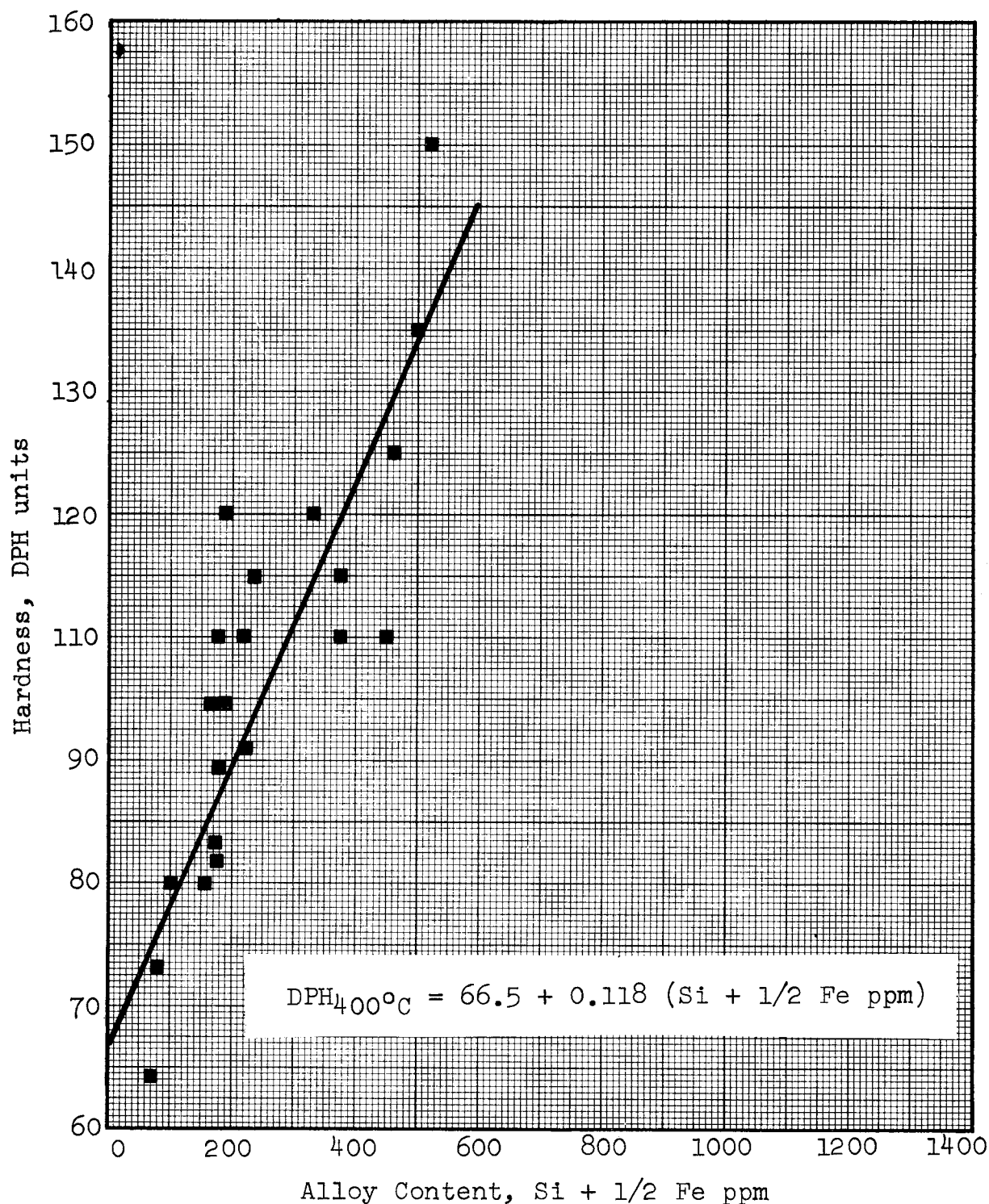


Fig. 1. Effect of Fe and Si Content on Hot Hardness (400°C) of Beta-Treated, Oil-Quenched Uranium Alloys (NMI-7097-1)

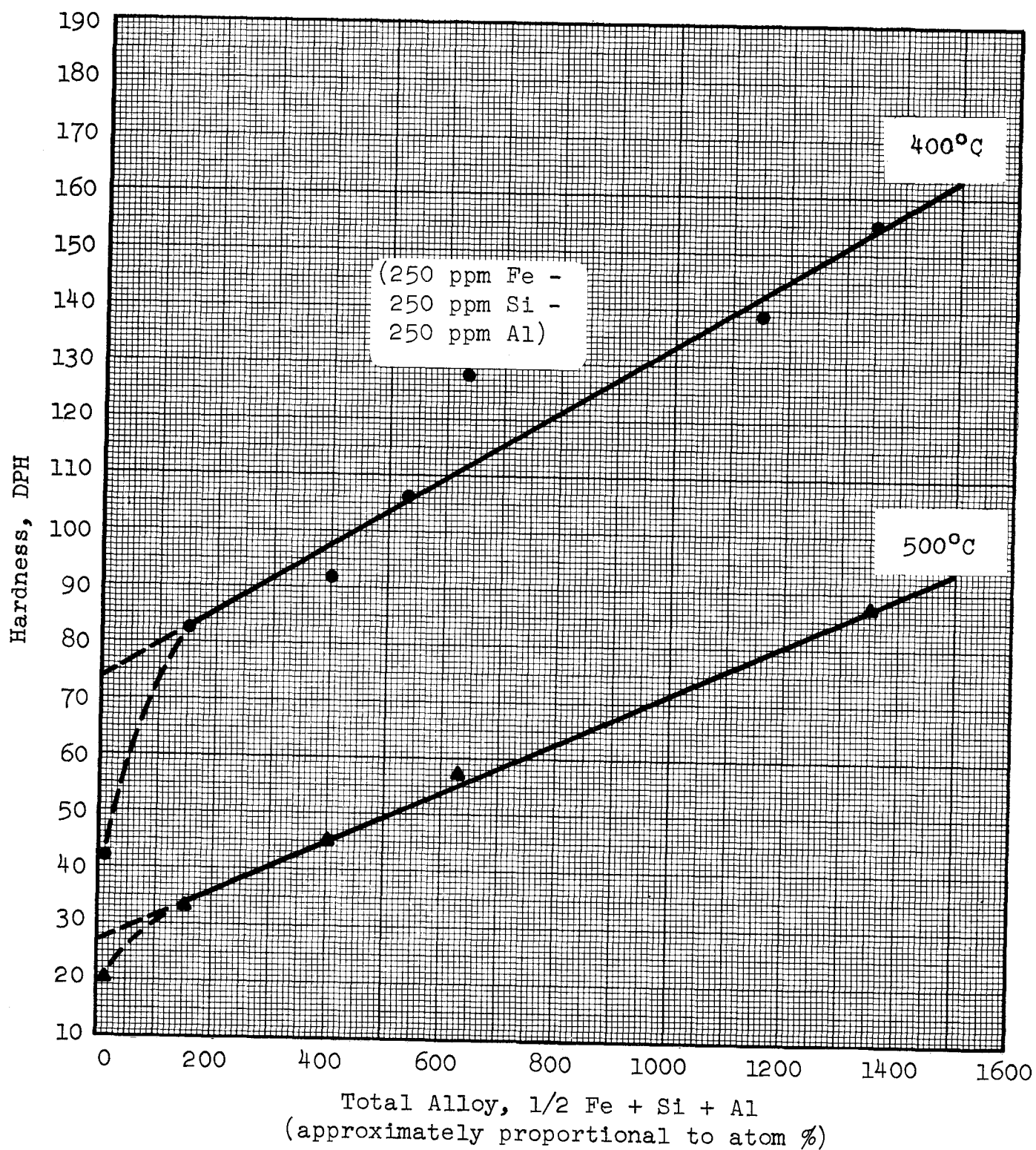


Fig. 2. Effect of Fe, Si, and Al Content on Hot Hardness (400 and 500°C) of Gamma-Treated (800°C, 1 hr), Oil-Quenched Uranium Alloys (NMI-7097-1)

resistance of the various alloys correlates with their hot hardness after beta treatment. The silicon-containing alloys, which are harder following beta treatment than the aluminum-containing alloys, are also more swelling resistant; dilute molybdenum additions (1000 ppm) to the alloys increase both hardness and swelling resistance.

For long irradiation exposures in which the aluminum as well as the silicon is dissolved, the relative swelling resistance of the various alloys correlates with their hot hardness after gamma treatment. The aluminum-containing alloys, which are harder following gamma treatment than the silicon-containing alloys, are more swelling resistant, and the incremental molybdenum addition is less effective than the aluminum.

The swelling resistance of the alloys thus is correlated with the mechanical properties of the alloys to the extent that the alloy phase distributions at various stages of irradiation can be simulated by an appropriate heat treatment. Measurement of mechanical properties during irradiation would be required to confirm this correlation in detail.

MECHANISMS FOR CAVITATIONAL SWELLING OF URANIUM AND ITS CONTROL BY DILUTE ALLOYING ADDITIONS

Swelling in uranium at temperatures between 400 and 600°C is caused by the formation of cavities in the metal. Between 400 and 500°C, the cavities are large and irregular and are located at grain boundaries and twin interfaces of a highly ~~distributed~~ ^{distorted} uranium structure; ⁽⁴⁾ between 500 and 600°C, the cavities are smaller and crystallographically aligned into rows within relatively undistorted grains. ⁽⁵⁾

Two mechanisms for cavitation swelling have been proposed, differing principally in the mode of nucleation of the cavities. In both cases, the irradiation growth (change of shape) of the uranium single crystal during irradiation is regarded as the basic driving force for cavity formation. In the first case,^(4,5) the cavities are presumed to be mechanically nucleated as the result of intergranular stresses arising from interactions between individual grains undergoing anisotropic growth. These interactions produce a grain-boundary shear, which in the given temperature range may result in cavities in the same manner as during creep deformation. Cavities produced in uranium by thermal cycling give evidence that such a mode of cavity formation is possible.

In the second case, cavities are assumed to be nucleated by fission gas bubbles formed in the uranium.⁽⁶⁾ Under stress caused by intergranular interaction, gas bubbles that exceed a critical size in metal above a given temperature will increase in size spontaneously to form a large cavity. The validity of this mechanism has been demonstrated by mechanical stressing of irradiated beryllium containing small gas bubbles at grain boundaries.⁽⁷⁾

In each case, cavity formation will occur only after a threshold burnup is exceeded, which accounts for an important kinetic feature of cavitation swelling.⁽¹⁾ Other details of the cavitation swelling mechanism are not well explained — for example, the formation of small aligned cavities within grains at higher temperatures in the swelling range.

Consideration of the high-temperature mode of swelling leads to a third mechanism for the cavitation swelling of uranium: the cavities are nucleated by the agglomeration of the vacancy dislocation loops that are formed during irradiation and cause

anisotropic irradiation growth of the crystal.^(8,9) By this mechanism the cavities result as a direct consequence of the disappearance of given lattice planes in the crystal, at grain boundaries and other crystal interfaces at lower temperatures, and within the grains at higher temperatures in the cavitation swelling range.

The anisotropic growth of the uranium crystal results from the formation of vacancy and interstitial dislocation loops, which result from displacement of lattice atoms by recoiling fission fragments.⁽⁸⁾ The vacancy and interstitial dislocation loops are generated from aggregates of vacancy and interstitial atoms, respectively. The loops are formed on different planes of the crystal due to anisotropic thermal expansion of the metal in the fission spike. This expansion favors agglomeration of vacancies on planes perpendicular to the a-axis direction to relieve compressive stresses, and agglomeration of interstitials on planes perpendicular to the b-axis direction to relieve tensile stresses.

The resulting dislocation loops can glide into approximately coplanar arrays and coalesce to remove planes of atoms perpendicular to the a-axis direction and to add planes of atoms perpendicular to the b-axis direction.⁽⁹⁾ Loops formed on atom planes perpendicular to the c-axis direction contain a stacking fault and are thus sessile; no significant dimensional change can occur in this direction.

The approximately coplanar arrays of dislocation loops require some climb of individual atoms to coalesce into complete atom planes. The actual location within the crystal at which this occurs will depend on specimen temperature. At low temperature, vacancy loops might glide to grain boundaries (or other crystal

interface) before coalescence, where, if not accommodated by distortion of the neighboring grain they would form an intergranular cavity. Such a cavity would tend to be located at crystal boundaries aligned perpendicular to the a-axis of the crystal. Similar coalescence of the interstitial loops would produce the pronounced grain distortions observed under these conditions.

At higher temperatures, the vacancy loops might coalesce within the grains to form aligned cavities with the same crystal orientation as the vacancy loops from which they derived. Less pronounced growth at the higher temperature would reduce the grain distortion observed.⁽⁸⁾

EFFECT OF ALLOY ADDITIONS

The control of cavitation swelling by alloying additions can be accounted for by any one of the three mechanisms for cavity formation. The evident correlation of swelling resistance with the strength of the various alloys detailed in the previous section supports in an immediate way the hypothesis that the cavities are mechanically nucleated. However, the correlation can serve as well for the other two mechanisms.

In the gas bubble mechanism, achievement of a critical bubble size by agglomeration of smaller bubbles is necessary for subsequent growth into a large cavity by capture of vacancies under a given intergranular stress. Gas bubbles tend to attach to dislocations in the metal; their agglomeration will be promoted by dislocation movement. The inhibition of dislocation movement by the alloy addition, e.g., strengthening of the metal, would therefore prevent agglomeration of the gas bubbles to the critical size

required to nucleate a large cavity. Thus the mechanical properties of the alloy should correlate with their swelling resistance, as previously described.

In a similar way, alloying additions that inhibit the movement of the dislocation loops that cause anisotropic growth would diminish cavity formation by the third mechanism, which then also accounts for the beneficial effect of alloying additions on swelling resistance. Such inhibition of the fundamental irradiation growth process by alloying additions should be readily detected experimentally by irradiation of textured specimens. Preliminary evidence for such an effect has been obtained for beta-treated uranium specimens with minor amounts of texture generated during heat treatment.⁽¹⁰⁾ More positive confirmation is being sought by irradiation tests of textured uranium alloy specimens in the as-worked (not heat treated) condition.

ANISOTROPIC GROWTH OF URANIUM ALLOYS

To establish the basic mechanism for improved swelling behavior of the uranium alloys, specimens of various compositions having measured amounts of texture, as determined by X-ray techniques, have been irradiated. As previously indicated, the irradiation-induced anisotropic growth (change of shape) of the uranium crystal is the basic driving force for the formation of large cavities that cause swelling at intermediate temperatures. The effect of alloying on the growth process may be an important factor in the control of swelling.

The test specimens were dilute alloys of Fe, Si, Al, Cr, and Mo (Table II), in the as-extruded (not heat treated) condition. The specimens were irradiated to exposures of 500, 1000, 1500, and

2000 MWD/T, mostly at low temperature (below the cavitation swelling range) to establish the magnitude of anisotropic growth. A few specimens were irradiated at intermediate temperatures (in the cavitation swelling range) to establish the orientations of cavities that cause swelling in the textured metal. The specimens are awaiting postirradiation examination.

TABLE II.

Specimens for Irradiation Test
of Textured Uranium Alloys

<u>Code(a)</u>	<u>Fe</u>	<u>Si</u>	<u>Al</u>	<u>Cr</u>	<u>Mo</u>	<u>C</u>
DA77X	350	-	800	-	-	50
IA77X	350	-	800	-	-	500
IB77X	350	350	800	-	-	500
DC77X	250	350	-	-	-	50
IC77X	250	350	-	-	-	500
DD77X	-	350	800	-	-	50
ID77X	-	350	800	-	-	500
DE77X	250	-	-	-	-	50
IE77X	250	-	-	-	-	500
IF01X	-	800	800	-	-	500
IG77X	-	350	-	-	-	500
IH77X	-	800	-	-	-	500
IU77X	250	300	800	200	-	500
DW77X	-	350	-	-	1000	50
IW77X	-	350	-	-	1000	500
IY77X	350	350	800	-	1000	500
IV77X	150	100	-	-	-	500
IO77X	-	-	-	-	-	500

(a) 77X As-extruded.

01X β -treated 725°C, 10 min, oil quenched.

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