TRANSFORMATION KINETICS OF U-5 wt % Fs DRIVER-FUEL PINS
PRODUCED BY AEROJET GENERAL CORPORATION

by

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EBR-II Project

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ABSTRACT

The U-5 wt % Fs fuel pins in driver elements made for EBR-II by Aerojet General Corporation (AGC) shortened during irradiation. Cause of the shortening was stresses on the fuel during the AGC centrifugal bonding, which led to a texture responsible for the axial shortening. This unwanted texture, however, could be removed by subsequent heat-treating; this procedure is being used to salvage the AGC centrifugally bonded fuel. To help determine the most effective annealing cycle, the transformation kinetics of as-cast and of as-bonded AGC pins were studied by relating annealing time to hardness, density, electrical resistivity, microstructure, and diffraction patterns of the fuel. The results showed that: (1) as-cast fuel would be transformed to the $\alpha + \delta + \text{U}_2\text{Ru}$ phases by annealing 40 min at 500°C; (2) as-bonded fuel would be transformed to the $\gamma + \text{U}_2\text{Ru}$ phases by annealing 30 min at 660°C; (3) as-bonded fuel heat-treated at 660°C for 2 hr would be transformed to the $\alpha + \delta + \text{U}_2\text{Ru}$ phases by annealing 40 min at 500°C.

I. INTRODUCTION

For a period extending from the startup of EBR-II until February 1969, all U-5 wt % Fs driver fuel for EBR-II had been produced by ANL. (See Ref. 1 for a description of this fuel.) In February 1969, fuel produced by Aerojet General Corporation (AGC) was inserted into the reactor for the first time as a part of a program to qualify this vendor as a supplier for EBR-II driver fuel. The initial results from this irradiation were

available in April 1969, at which time it was found that the AGC fuel had shortened in length. At a burnup of 0.6 at. %, the maximum shortening was about 0.9 in. A number of mechanisms were postulated as being responsible for the shortening, but preferred orientation induced during the fuel-fabrication process emerged very early as the leading contender. It has since been confirmed that this is indeed the cause and that the AGC centrifugal process for sodium bonding is specifically responsible for a fuel texture that leads to the undesirable irradiation behavior.

AGC centrifugal bonding of as-cast U-5 wt % Fs driver pins is generally carried out in the temperature range of 480-500°C. Time of bonding in that temperature range is usually about 20 min, but the heating and cooling require an additional 85 min. Bonding at 500°C transforms the retained γ phase to the equilibrium phases of $\alpha + \delta + U_2Ru$, with the matrix being α . The AGC centrifugal bonding introduces a stress on the fuel during $\gamma \rightarrow \alpha$ transformation. It is this stress during the transformation that produces the undesirable texture in the transformed orthorhombic α phase. The amount of stress increases from the top end to the bottom (spade) end of the pin. It is 3200 psi at the bottom end. To remove the unwanted texture and avoid irradiation growth in a particular crystallographic direction, a "salvage" heat treatment of the fuel eventually was used to reclaim it for use in the reactor.

The study reported here was part of the effort to determine the best method of heat treatment. Three areas were studied: (1) transformation of as-cast alloy structure to the $\alpha + \delta + U_2Ru$ phases at 500°C; transformation of as-bonded alloy structure to the $\gamma + U_2Ru$ phases at 660°C; (3) transformation of as-bonded, heat-treated (660°C for 2 hr) alloy structure to the $\alpha + \delta + U_2Ru$ phases at 500°C.

II. EXPERIMENTAL PROCEDURES

Property changes under isothermal conditions were investigated to study the kinetics of the phase transformation of the alloy at different temperatures. Annealing heat treatments of the pin samples were carried out in welded Type 304L stainless-steel jackets filled with argon. After each heat treatment, the specimens were quenched in water. A small sample

was examined metallographically for determining grain size and existing phases. Vickers hardnesses were determined using a 500-g load.

Electrical resistivity and density were measured using 5-in.-long samples. Resistivity was measured by establishing a constant current of 100 mA and reading the drop in potential between two razor-blade contacts 10 cm apart. Density was measured by the CCl_4 displacement method.

X-ray diffraction patterns were made with a copper-tube, GE orienter-collimator and a 0.05° detector slit.

III. TRANSFORMATION KINETICS OF AGC AS-CAST FUEL PINS AT 500°C

As-cast U-5 wt % Fs alloy may retain 65-90% of retained γ phase, depending on the cooling rate.¹ According to Mueller,² as-cast γ structure has a distorted tetragonal lattice with a c/a (ratio of length of c axis to that of a axis) slightly less than 1.

Measurements were made of the top and bottom sections of pins from casting batches 302 and 471. Table I shows how the average properties of the sections changed during the isothermal anneals. The data in the table are plotted in Fig. 1 for a pin from batch 302 and in Fig. 2 for a pin from batch 471.

A. Hardness

As-cast U-5 wt % Fs specimens show an average Vickers hardness (500-g load) of 240. As the cast structure, containing mainly the $\gamma + \text{U}_2\text{Ru}$ phases, transforms to $\alpha + \delta + \text{U}_2\text{Ru}$ phases during annealing at 500°C , the hardness increases. Hardness reached an average peak value of 550 for all the specimens after 20 min of annealing time (see Figs. 1 and 2). Continuing the anneal to 80 min did not increase hardness significantly, which indicates that transformation of the $\gamma + \text{U}_2\text{Ru}$ phases to the $\alpha + \delta + \text{U}_2\text{Ru}$ phases probably reached the equilibrium stage after about 20 min. This assumption should be valid, because hardness is mainly contributed to by the discontinuously precipitated, hard δ phase, and

TABLE I. Property Changes of AGC As-cast Fuel Pins during Isothermal Annealing at 500°C

Time of Annealing at 500°C, min	Pin from Casting Batch 302						Pin from Casting Batch 471					
	Top Section			Bottom Section			Top Section			Bottom Section		
	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc
0	242	66.14	17.720	181	66.17	17.799	253	66.05	17.797	236	66.06	17.809
2	256	64.09	17.727	203	65.03	17.815	251	64.61	17.802	226	63.09	17.815
5	241	63.64	17.710	228	65.24	17.791	231	63.37	17.784	218	63.43	17.797
10	311	63.75	17.735	296	63.64	17.810	375	64.04	17.801	258	63.88	17.825
20	530	50.0	17.940	528	49.29	17.988	531	48.80	17.981	533	49.33	17.996
40	542	47.03	17.911	575	48.21	17.996	535	47.14	17.986	560	46.72	17.991
80	540	46.62	17.919	524	46.20	18.006	534	46.03	18.003	561	45.89	18.016

^aWith 500-g load.

Fig. 1. Property Changes of As-cast AGC Pin from Casting Batch 302 during Isothermal Annealing at 500°C

Fig. 2. Property Changes of As-cast AGC Pin from Casting Batch 471 during Isothermal Annealing at 500°C

precipitation of this phase is not involved with strain in the lattice. Prolonged annealing--to 10 hr^1 --at 500°C does not change the hardness values markedly.

B. Resistivity

Measurements by Beck and Fousek³ showed an average electrical resistivity of $69 \mu\Omega/\text{cm}$, at 25°C , for the top, middle, and bottom sections of another as-cast pin (No. 3890). The resistivities of the top and bottom sections of as-cast pins from batches 302 and 471 averaged $66 \mu\Omega/\text{cm}$. With the precipitation of the $\alpha + \delta$ phases, this value drops within 20 min to $49.35 \mu\Omega/\text{cm}$. With prolonged annealing, the rate of change of the resistivity decreases, which indicates that the major part of the transformation has already taken place. The resistivity does, however, continue to decrease slowly, and reaches an equilibrium value of $46 \mu\Omega/\text{cm}$ after 80 min of annealing.

C. Density

Density of the alloy increases as the transformation of $\gamma + \text{U}_2\text{Ru}$ to $\alpha + \delta + \text{U}_2\text{Ru}$ proceeds. The equilibrium value of the density was essentially reached in 20 min at 500°C .

The data show considerably different values of density for the top and bottom sections of the pin from batch 302. The cause of this difference has not been established.

D. Structure

Metallographic examinations were made after each heat treatment. With the etching techniques available, the transformations to different structures could not be easily identified. Figure 3 shows the microstructures of the top and bottom sections of an as-cast pin from batch 471. It is evident that there is a considerable difference in grain size between the two sections. This difference is attributed to the difference in the cooling rates for the sections during pin casting. Some differences

133B3-2

500X

TOP

133B4-1

500X

BOTTOM

Fig. 3. Microstructures of As-cast Pin from Casting Batch 471

in composition might even exist because of the difference in cooling rate. Measurements of grain size by the Heyn intercept method gave the following results:

<u>Batch No. and (Pin Section)</u>	<u>Average Intercept, mm</u>	<u>ASTM Grain Size</u>
302 (Top)	0.0141	9.0
302 (Bottom)	0.0323	6.5-7.0
471 (Top)	0.0135	9.0-9.5
471 (Bottom)	0.0391	6.0-6.5

The grain sizes were almost the same after the annealings at 500°C as they were before the annealings. The photomicrograph of the bottom section of pin 471 in Fig. 3 shows γ phase and light, globular U_2Ru .

Figure 4 shows the transformed structure of a pin from batch 302 after it had been annealed at 500°C for 40 min. The bottom portion of the pin shows the $\alpha + \delta + U_2Ru$ phases and is similar to the structure when equilibrium at 500°C is reached.¹

E. Diffraction Patterns

Figure 5 shows the diffraction pattern of the γ -tetragonal structure of an as-cast pin from batch 302. The pattern peaks at 36.8°. The slight amount of U_2Ru that was observed by the microstructural examination cannot be detected here. When the same pin is heated at 500°C for 40 min, the diffraction pattern (Fig. 6) undergoes a significant change, which shows the transformation from γ to α . The " γ " designation used in Fig. 6 is not the same as that in Fig. 5. We have not been able to observe sufficient x-ray lines to positively identify the γ phase in the annealed specimen. In Fig. 6, the " γ " designation indicates intensity between the αU triplet and the αU (111) reflection; in other words, the intensity near 37.2°. The intensities of the αU triplet (021) > (110) > (002) indicate near-random orientation and complete transformation.

151B1-1

500X

TOP

151B2-1

500X

BOTTOM

Fig. 4. Microstructures of As-cast Pin from Casting
Batch 302 after 40-min Anneal at 500°C

Fig. 5. Diffraction Pattern of As-cast Pin from Casting Batch 302

Fig. 6. Diffraction Pattern of As-cast Pin from Casting Batch 302 after 40-min Anneal at 500°C

IV. TRANSFORMATION KINETICS OF AGC AS-BONDED FUEL PINS AT 660°C

AGC centrifugal bonding of as-cast U-5 wt % Fs driver pins was done in the temperature range of 480-500°C. The total time of exposure of the fuel in the $\alpha + \delta + U_2Ru$ temperature region was sufficient to achieve a major amount of transformation of as-cast phases to the $\alpha + \delta + U_2Ru$ phases.⁴ The stresses imposed during bonding caused the orthorhombic α uranium to develop a texture which, in turn, caused axial shortening and radial growth of the pins during irradiation. Heat treatment at 660°C ensures formation of γ nuclei; subsequent growth of the nuclei essentially eliminates the textured α grains and produces random orientation.

Property changes under isothermal conditions were investigated to study the kinetics of phase transformation of the as-bonded alloy at 660°C. Table II shows the data obtained during isothermal annealing, at 660°C, of two pins (4618 and 4627) from casting batch 132. Figure 7 plots these data for pin 4618, and Fig. 8 for pin 4627.

A. Hardness

The average Vickers hardness of U-5 wt % Fs alloy having the $\alpha + \delta + U_2Ru$ phases in equilibrium is 540. The samples of AGC as-bonded pins 4618 and 4627 exhibited an average hardness of 500. When the samples were annealed at 660°C, the hard α and δ phases disappeared and the soft γ phase formed. The hardness values, after showing slight increases at the start of the anneal, decreased rapidly to a value of approximately 200 after 10 min and remained fairly constant at that value even after an annealing of 80 min. These results indicate that most of the transformation has probably taken place within 10 min.

B. Resistivity

Like hardness, the resistivities of the as-bonded samples are comparatively lower than those of the samples annealed and brought to equilibrium at 500°C. The resistivities of as-bonded pins 4618 and 4627 agree

TABLE II. Property Changes of AGC As-bonded Fuel Pins during Isothermal Annealing at 660°C

Time of Annealing at 660°C, min	Pin 4618 from Casting Batch 132						Pin 4627 from Casting Batch 132					
	Top Section			Bottom Section			Top Section			Bottom Section		
	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc	Vickers Hardness Number ^a	Resistivity, $\mu\Omega/\text{cm}$	Density, g/cc
0	458	42.67	18.027	473	45.74	18.019	484	42.01	18.101	565	43.88	18.104
2	488	43.01	18.026	527	45.37	18.022	465	42.15	18.107	550	44.26	18.099
5	376	44.99	17.979	324	48.18	17.953	481	40.24	18.058	570	42.01	18.059
10	217	52.13	17.908	222	52.73	17.830	206	53.25	17.981	207	50.33	18.015
20	380	56.57	17.876	493	56.67	17.881	470	48.88	18.026	434	52.34	18.006
40	195	65.34	17.787	172	64.99	17.797	203	64.40	17.888	195	64.61	17.886
80	176	64.89	17.784	198	64.92	17.802	214	65.17	17.870	187	64.40	17.853

^aWith a 500-g load.

Fig. 7. Property Changes of As-bonded Pin 4618 from Casting
Batch 132 during Isothermal Annealing at 660°C

Fig. 8. Property Changes of As-bonded Pin 4627 from Casting
Batch 132 during Isothermal Annealing at 660°C

very well with the Beck and Fousek³ resistivity measurement of 42 $\mu\Omega/\text{cm}$ for as-bonded AGC pin 01834 from batch 112. The resistivity of the alloy increased as a function of the annealing time and attained a maximum value of 65 $\mu\Omega/\text{cm}$ after 40 min. This value is close to the average resistivity of 66 $\mu\Omega/\text{cm}$ measured for as-cast specimens (which contain retained γ phase and U_2Ru).

C. Density

Density of the alloy decreased as the less-dense γ phase formed during the anneal. As with resistivity, the densities of the two pins approached a plateau after 40 min of annealing time and did not change significantly as the annealing continued.

D. Structure

Measurements of grain size of the as-bonded pins by the Heyn intercept method were as follows:

<u>Pin No. and (Section)</u>	<u>Average Intercept, mm</u>	<u>ASTM Grain Size</u>
4618 (Top)	0.0109	9.5-10.0
4618 (Bottom)	0.0270	7.0-7.5
4627 (Top)	0.0109	9.5-10.0
4627 (Bottom)	0.0270	7.0-7.5

Comparison of these grain sizes with those of the as-cast alloy (see Sect. III.D) indicates that grain size is not changed markedly by bonding. As with the as-cast pins, the bottom sections of the as-bonded pins exhibited larger grain sizes than the top sections.

Figure 9 shows photomicrographs of the top and bottom sections of as-bonded pin 4627. The microstructure of the top section is grossly heterogeneous. It consists of $\alpha\text{-U}$ of low Fs content (light particles) and shows segregation of secondary phases (dark particles) at parent γ -grain boundaries. The microstructure of the bottom section of the pin, in contrast, has a coarser grain, but also is characterized by some homogeneous segregation of secondary phases at grain boundaries.

133B7-1

500X

TOP

133B8-1

500X

BOTTOM

Fig. 9. Microstructures of As-bonded Pin 4627 from Casting Batch 132

Figure 10 shows the microstructure of the top and bottom sections of the same pin after 80 min of annealing. The top section of the pin has a uniform structure that consists of a matrix of retained γ phase and coalesced particles of U_2Ru ¹ distributed at the grain boundaries. The microstructure of the bottom section of the pin is essentially the same, except for intragranular precipitate probably formed as a result of low quenching rate. The matrix is a mixture of retained γ and α phases.

E. Diffraction Patterns

X-ray examination of as-bonded pin 4618 showed α -U, with peak intensities $(110) > (021) > (002)$. Figure 11 shows the diffraction pattern of the same pin after it had been annealed at 660°C for 10 min. It indicates a γ phase which appears near 36.82° and confirms the transformation to γ phase. After the pin was annealed for 80 min, its diffraction pattern was the same (see Fig. 12). Because it is present in small quantities, the U_2Ru phase was undetected in the diffraction analysis. The diffraction pattern of Figs. 11 and 12 is similar to that obtained from samples heat treated for 70 hr at 850°C and then water-quenched.⁵

V. TRANSFORMATION KINETICS OF BONDED AND HEAT-TREATED

U-5 wt % Fs FUEL PINS AT 500°C

As-cast (and as-bonded) U-5 wt % Fs pins contain a major portion of retained γ phase; the amount depends generally on the cooling rate. This subsequently should influence the time of transformation of $\gamma + U_2Ru$ to $\alpha + \delta + U_2Ru$ when heating to 500°C. Another factor which may also influence the transformation kinetics is the presence of tetragonal " γ " having a distorted lattice and a c/a ratio slightly less than 1.² The tetragonal " γ " phase is formed at a lower temperature, but the amount of the phase present in the as-cast alloy has not been investigated. The purpose of this part of the study was to gain information concerning the uncertainties with the nonequilibrium as-cast structure of U-5 wt % Fs alloy and to determine the kinetics of conversion of homogenized $\gamma + U_2Ru$ to $\alpha + \delta + U_2Ru$.

152B7-1

500X

TOP

152B8-1

500X

BOTTOM

Fig. 10. Microstructures of Bonded Pin 4627 from Casting Batch 132 after 80-min Anneal at 660°C

Fig. 11. Diffraction Pattern of Bonded Pin 4618 from Casting
Batch 132 after 30-min Anneal at 660°F

Fig. 12. Diffraction Pattern of Bonded Pin 4618 from Casting
Batch 132 after 80-min Anneal at 660°C

To achieve homogenization, bonded U-5 wt % Fs alloy pins were first heat treated at 660°C for 2 hr and then water quenched. Microscopical observations and x-ray diffraction patterns were used to confirm that the heat treatment was effective for bringing $\alpha + \delta$ to a solid γ solution.

Property changes under isothermal conditions were investigated to study the kinetics of phase transformation at 500°C. Table III shows the data obtained during annealing of two pins (4618 and 4627, both from casting batch 132) at 500°C. Figure 13 plots these data for pin 4618, and Fig. 14 for pin 4627.

A. Hardness

The heat-treated and water-quenched U-5 wt % Fs specimens show an average hardness (500-g load) of 190. This is significantly less than the hardness of the as-cast structure.¹ With further annealing at 500°C, however, the hard $\alpha + \delta$ phases start precipitating, resulting in an increase in hardness. Hardness reaches its peak after about 20 min, when an average value of about 525 is attained. Prolonged annealing causes coalescence in the alloy, resulting in a slight drop in hardness. These data indicate that transformation of the $\gamma + U_2Ru$ phases to the $\alpha + \delta + U_2Ru$ phases probably reaches the equilibrium stage in about 20 min.

B. Resistivity

Resistivities of the heat-treated pins average about 66 $\mu\Omega/cm$, just about the same as the average resistivity of as-cast pins. With the precipitation of the $\alpha + \delta$ phases, the resistivity decreases within 20 min to an average value of about 46 $\mu\Omega/cm$. After 20 min, the resistivity decreases much more slowly with increase in annealing time, again probably because of slow coalescence of the precipitated particles.

TABLE III. Property Changes of AGC Bonded and Heat-treated^a Fuel Pins during Isothermal Annealing at 500°C

Time of Annealing at 500°C, min	Pin 4618 from Casting Batch 132						Pin 4627 from Casting Batch 132					
	Top Section			Bottom Section			Top Section			Bottom Section		
	Vickers Hardness Number ^b	Resistivity, μΩ/cm	Density, g/cc	Vickers Hardness Number ^b	Resistivity, μΩ/cm	Density, g/cc	Vickers Hardness Number ^b	Resistivity, μΩ/cm	Density, g/cc	Vickers Hardness Number ^b	Resistivity, μΩ/cm	Density, g/cc
0	191	67.94	17.783	188	67.91	17.782	214	65.58	17.790	186	65.62	17.801
2	191	65.03	17.769	201	65.27	17.795	220	65.27	17.755	212	63.47	17.768
5	194	65.30	17.755	213	65.38	17.768	425	68.00	17.778	200	67.32	17.776
10	504	54.21	17.949	490	53.69	17.940	537	48.29	17.960	549	53.97	17.965
20	536	47.56	17.984	504	49.29	17.983	510	44.02	17.992	557	43.46	18.005
40	508	44.4	17.990	521	46.90	17.986	515	45.44	17.983	487	43.26	18.003
80	465	41.28	18.017	507	42.90	18.006	511	41.70	18.000	484	39.34	18.013

^aHeat treated for 2 hr at 660°C and then water quenched.

^bWith a 500-g load.

Fig. 13. Property Changes of Bonded and Heat-treated Pin 4618 from Casting Batch 132 during Isothermal Annealing at 500°C

Fig. 14. Property Changes of Bonded and Heat-treated Pin 4627 from Casting Batch 132 during Isothermal Annealing at 500°C

C. Density

Density increases rapidly with increase in annealing time until it reaches a plateau at about 20 min. Further annealing results in a very slow increase in the density.

D. Structure

Figure 15 shows the microstructures of the top and bottom sections of pin 4627 after the 2-hr heat treatment at 660°C followed by the water quench. The top section has a uniform structure consisting of retained γ phase and coalesced particles of U_2Ru along the grain boundaries. The structure of the bottom section is the same except for intragranular precipitate distribution.

Microscopical inspection showed that transformation was progressing within the first 10 min of the subsequent annealing at 500°C. Figure 16 shows the microstructures of pin 4618 after it had been annealed 40 min at that temperature. Both photomicrographs in the figure show the presence of three phases: α (light), γ (gray), and precipitated U_2Ru (black).

E. Diffraction Patterns

Isothermal annealing at 500°C changes the cubic γ pattern, which has a peak intensity at 36.8°. A 10-min anneal at 500°C produces very diffused α uranium with peak intensities $(002) > (110) > (021)$, thereby indicating nonrandom orientation of the metal crystals (see Fig. 17). The intensity ratio of the peaks also indicates that the transformation is incomplete. Prolonged annealing increases the intensity of the (021) peak and appears to create random orientation in the alloy¹ (see Fig. 18).

VI. CONCLUSIONS

The following conclusions are made on the basis of the results of this study:

1. The transformation of the as-cast $\gamma + U_2Ru$ to $\alpha + \delta + U_2Ru$ at 500°C should be complete if an anneal of 40 min is used.

Fig. 15. Microstructures of Bonded Pin 4627 from Casting Batch 132
after 2-hr Heat Treatment at 660°C Followed by Water Quench

171B1-1

500X

TOP

171B2-1

500X

BOTTOM

Fig. 16. Microstructures of Bonded and Heat-treated Pin 4618 from Casting Batch 132 after 40-min Anneal at 500°C

Fig. 17. Diffraction Pattern of Bonded and Heat-treated Pin 4627
from Casting Batch 132 after 10-min Anneal at 500°C

Fig. 18. Diffraction Pattern of Bonded and Heat-treated Pin 4627
from Casting Batch 132 after 80-min Anneal at 500°C

2. The transformation of as-bonded $\alpha + \delta + \text{U}_2\text{Ru}$ to $\gamma + \text{U}_2\text{Ru}$ at 660°C should be essentially complete if an anneal of 30-40 min is used.
3. The transformation of $\gamma + \text{U}_2\text{Ru}$ to $\alpha + \delta + \text{U}_2\text{Ru}$ in heat-treated (at 660°C) and water-quenched U-5 wt % Fe alloy should be complete if a subsequent anneal of 40 min is used.

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