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AEC RESEARCH AND DEVELOPMENT REPORT

CONTRIBUTION OF THE JOMINY-TYPE END QUENCH
TO METALLOGRAPHIC PHASE IDENTIFICATION IN URANIUM

L. LEWIS

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DECEMBER 1958
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BRIDGEPORT, CONNECTICUT

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December 8, 1958

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CONTRIBUTION OF THE JOMINY-TYPE END QUENCH TO METALLOGRAPHIC
PHASE IDENTIFICATION IN URANIUM

SUMMARY

Jominy-type end quench tests provide a convenient means of exploring uranium transformation characteristics. Metallographic examinations indicate that macrostructural data provide the best means of phase identification and interpretation.

INTRODUCTION

In extrusion and other fabrication development for uranium, samples are examined metallographically to determine, amongst other factors, the particular heating and cooling the metal has undergone during processing. However, definite determination of the type of heating and cooling has been, at times, difficult. Therefore, the present metallographic investigation was made to help relieve this difficulty.

Uranium, if of a small enough cross-section, transforms from the gamma or beta phases to an alpha-uranium structure characteristic of the cooling treatment given it. Grain size and shape as well as sub-grain and twinning intensity make up the microstructure and are the general characteristics which enable the metallographer to identify the heat treatment of the samples under consideration. However, the similarity of room temperature alpha-uranium microstructures can sometimes make identification and interpretation difficult. The use of macrostructures can be of considerable assistance in identification but supply little, if any, of the desired microstructural characteristics mentioned above.

Additional interpretive difficulties may arise when examining heavier cross-sections. The large difference in cooling undergone by heavy cross-sections can result in a structure varying from that approaching an air cool at the center to a rapid quench nearer the surface. Thus, the metallographer is presented with an additional problem, that of differentiating between a uniformly and non-uniformly cooled structure.

Therefore, the object of this laboratory program is to provide standard uranium macro- and microstructural data with which conclusive determination of uranium heating history can be made.

PROCEDURE

Jominy-type end quench tests were chosen for the experiment as they provide a convenient means of heat treating and then introducing a series of cooling rates through an end quench. The particular end quench was not designed for hardenability tests as are most Jominy tests, but, rather as a means of studying through metallographic techniques the transformation of uranium at various cooling rates to its room temperature structure. The test is advantageous because of the minimum of material and effort required to obtain a maximum of data.

Essentially the technique employed in an end quench of this nature is as follows: heat a specimen to a prescribed temperature, hold at temperature, transfer and vertically position in a quench jig, and then direct a vertical column of water against the bottom face of the specimen for a specified period of time.

Eight slugs from fine-grained, alpha extruded ingot uranium rods machined to a 4-in. length x 1-in. diameter with a flange on one end, comprised the test material for the end quench. They were heated to prescribed gamma and beta phase temperatures for prescribed times in a lithium carbonate (46 per cent) - potassium carbonate (54 per cent) salt bath and then selectively end-quenched by tap water for ten minutes in a manner generally conforming to SAE(1) specifications for a Jominy end quench test. Transfer times from salt bath to quench were made in ten seconds or less.

After heat treatment the samples were cut in half, longitudinally, one half being used for macro- and microstructural analyses, and the other half for Rockwell G hardness profiles. The hardnesses were taken at 1/4-in. intervals along the axis of the sample.

RESULTS AND DISCUSSION

The heat treating schedule is shown in Table I. Representative results of the metallographic study are shown in picture form as Figs. 1a, b, c, d and 4a, b, c, d.

Series 1 is of specimen 6 which was gamma-treated at 1000°C - 1832°F for 15 minutes and then end-quenched. Figure 1a is of the room temperature macrostructure of gamma-cooled uranium. Note how selective quenching leads to preferential columnar grain growth for a distance away from the quenched end and a more random structure in the air-cooled zone. The columnar grains are typical of areas which have undergone rapid cooling

from the gamma phase. If the sample had been totally immersed in water, the growth would have followed the largest temperature gradient, that being in a radial direction towards the center. Figs. 1b, c and d are representative photomicrographs of the quenched end, intermediate and air-cooled zones, respectively. The grain size for the most part increases in coarseness from the quenched to air-cooled end. The structure is relatively free of sub-grains. The shape of the grain boundaries and twinning intensity do not follow a set pattern.

The double and triple gamma-treated macrostructures are quite similar in appearance to the single gamma-treated specimen and do not exhibit any greater grain refinement at the quenched end. They are shown in Figs. 2 and 3.

Series 4 is of specimen 2 which was double beta-treated at 760°C - 1400°F for 15 minutes and end-quenched. Figs. 4a, b, c and d are representative of the typical beta-treated and cooled uranium macro- and microstructures. The macrostructure, Fig. 4a, in comparison to the gamma-cooled macrostructure appears finer and shows less tendency toward columnar growth away from the quenched end. The photomicrographs, Figs. 4b, c and d are typical of the quenched end, intermediate, and air-cooled zones. Note the general coarsening trend. Sub-grains are more prevalent than in the gamma structures. As in the gamma-treated specimens, the amount of twinning and grain boundary shape varies erratically from position to position and are considered of little value in establishing a cooling gradient.

The various beta-heat treatments given the slugs did not seem to influence the characteristics to any noticeable degree. For example, slug 4 which was beta-treated for 1 hr. at 760°C - 1400°F conformed to the macro- and microstructural characteristics exhibited by slugs 1, 2, and 3 which were single, double and triple beta-treated, respectively at 760°C - 1400°F for 15 minutes. In addition, slug 5 transformed from the beta-gamma transformation temperature conformed in structural appearance to the beta-cooled rather than gamma-cooled slugs. Photomicrographs of these slugs are shown in Figs. 5-8.

Rockwell G hardness data taken along the axis of the Jominy end quench specimens and shown in Table II reveal the susceptibility of uranium to hardness reversals along a temperature gradient resulting from a beta and gamma heat treatment. Fig. 9 shows the behavior for slugs 3 and 5. Gamma-treated hardnesses range from a maximum of Rockwell G 76.0 at the quenched end to 63.0 at the air-cooled end as compared to beta-treated hardnesses of Rockwell G 81.0 and 65.0, respectively. The softening correlates with gamma and beta-cooled structures which exhibit a gradual coarsening of grains in the air-cooled direction. However, in many instances the coarse grained areas upon further exam-

ination were found to exhibit isolated fine grained areas. The sporadic increases in hardness may be attributed to the isolated fine grained and/or sub-grained areas, or perhaps to areas of large hydride and other non-metallic inclusions.

Reference is made to hydride inclusion, although, it was not investigated in this program, because it perhaps provides an explanation for part of the hardness reversal. Work carried out by Riches and Gardner⁽²⁾ indicates that cooling rates of .002 to 300°C/sec. are conducive to the formation through precipitation of uranium hydride as discrete particles at grain boundaries. The size of the particles increases with decreased cooling rates and has been reported to reach a length as great as .3 mm. Therefore, it is not difficult to presuppose that hardness readings taken in an area highly concentrated with hydrides or other inclusions could lead to hardness reversals, particularly when the greatest concentration of hydrides can be expected along the axis of the specimen, that being the part of the sample that would undergo the slowest cooling from the high temperature phases.

Upon sectioning the end-quench specimens, it was noticed that gamma treated slugs 7 and 8 in the unetched condition, Figs. 10 and 11, exhibited small voids in the air-cooled zone. A brief literature⁽³⁾ search indicated that voids, but much larger, were encountered in uranium slugs that had undergone a multiple cycling across the alpha-beta transformation temperature. Other sites have also reported this phenomenon. However, it was not determined at which cycle the voids were initiated. High internal stresses set up as a result of preferential transformation and restraint of adjacent metal were cited as major causes for the void formation. Therefore, upon completion of the present examination all the samples were quartered in a longitudinal direction and examined for further evidence of voids. Only one additional specimen exhibited voids, that being gamma-treated slug 6 which contained very small voids. It is interesting to note that of the three samples exhibiting voids all were gamma-treated, with the magnitude of the voids increasing from the single gamma on through the triple gamma-heat treatment and end-quench. Therefore, it appears gamma-heat treatment may be severe enough to cause void formation after one cycle whereas beta-heat treatment requires many cycles.

CONCLUSIONS

1. The identification of phases and interpretation of phase transformation through microstructural analysis are still difficult even with metallographic standards as a guide.

2. Macrostructures provide a better guide to determination of the origin of the quenched phase and the degree of quenching.
3. Grain sizes are of some assistance in determining the cooling conditions employed.
4. Twinning concentration and grain boundary shape are inconclusive characteristics to use in determining the origin of the quenched phase and the degree of quenching.
5. Sub-grains are more prevalent in beta-treated uranium.
6. Hardness profiles provide highly irregular data and are of little assistance in interpretation of heat treating conditions.
7. Single and multiple gamma-beta-alpha cycles appear severe enough to cause the formation of small voids in the air-cooled zones of end quench samples.

REFERENCES

- (1) 1947 SAE Handbook, Pg. 303.
 - (2) H.R. Gardner and J.W. Riches, "The Effect of Cooling Rate on the Nucleation and Growth of Beta-Uranium Hydride in Metallic Uranium," Paper No. 57, 2nd Nuclear Engineering and Science Conference.
 - (3) B.R. Hayward, "Dimensional Changes Resulting from Alpha-Beta Thermal Cycling of Uranium and Uranium Alloys," NAA-SR-1434, Unclassified.
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TABLE I

HEAT TREATING SCHEDULE

<u>SPECIMEN NO.</u>	<u>DESCRIPTION</u>
1	Heated @ 760°C - 15 minutes Transfer Time - 6-1/2 seconds End-Quench Time - 10 minutes
2	Double beta-treated @ 760°C - 15 minutes Transfer Times - 7-1/2 and 9 seconds End-Quench Times - 10 minutes each
3	Triple beta-treated @ 760°C - 15 minutes Transfer Times - 8-1/2, 6-1/2, and 7 seconds End-Quench Times - 10 minutes each
4	Heated @ 760°C - 1 hour Transfer Time - 9-1/2 seconds End-Quench Time - 10 minutes
5	Heated @ 785°C - 15 minutes Transfer Time - 6-1/2 seconds End-Quench Time - 10 minutes
6	Heated @ 1000°C - 15 minutes Transfer Time - 8 seconds End-Quench Time - 10 minutes
7	Double gamma-treated @ 1000°C - 15 minutes Transfer Times - 5-1/2 and 6 seconds End-Quench Times - 10 minutes each
8	Triple gamma-treated @ 1000°C - 15 minutes Transfer Times - 7, 5 and 7 seconds End-Quench Times - 10 minutes each

Alpha-Beta Transformation Temperature - 668°C - 1234°F

Beta-Gamma Transformation Temperature - 774°C - 1426°F

760°C - 1400°F

785°C - 1445°F

1000°C - 1832°F

TABLE IIAVERAGE ROCKWELL G HARDNESS

DISTANCE FROM QUENCHED END <u>1/4-in.</u>	<u>S P E C I M E N N O .</u>							
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
1	76	76	77	81	77	76	76	74
2	75	76	75	79	75	76	76	75
3	75	77	75	79	77	77	73	78
4	72	75	75	75	72	73	74	76
5	72	76	73	79	74	75	74	78
6	72	73	75	76	73	77	74	75
7	72	72	73	80	74	75	78	74
8	71	73	74	77	74	75	75	78
9	70	75	70	75	73	74	76	76
10	69	74	73	76	73	71	74	73
11	69	73	73	74	70	72	72	65
12	67	76	74	76	73	73	67	69
13	67	73	74	70	69	73	69	68
14	66	74	78	77	72	66	70	78
15	65	69	67	75	70	72	63	69

2X

1X

Fig. 1a - Typical gamma-cooled macrostructure. Specimen 6. Gamma-treated at 1000°C - 15 min. and end-quenched. HCl and HNO₃ Etch.

Fig. 1(b) - Typical gamma-quenched microstructure.
Specimen 6 (Quenched End). Quenched from 1000°C.
Electrolytic Etch.

Mag. 100X Lab. Sample No. U604-J-3A Pd. Lt.

Fig. 1(c) - Typical gamma-cooled microstructure.
Specimen 6 (Intermediate zone). Cooled from
1000°C. Electrolytic Etch.

Mag. 100X Lab. Sample No. U604-J-3B Pd. Lt.

Fig. 1(d) - Typical room temperature microstructure of slowly cooled gamma phase uranium. Electrolytic Etch.

Mag. 100X

Lab. Sample No. U604-J-3C

Pd. Lt.

Fig. 2 - Specimen 7. Double gamma-treated at 1000°C - 15 min. and end-quenched.

Mag. 1X

Lab. Sample No. U604-H-4

Fig. 3 - Specimen 8. Triple gamma-treated at 1000°C - 15 min. and end-quenched.

Mag. 1X

Lab. Sample No. U604-J-4

2X

1X

Fig. 4a - Typical beta-cooled macrostructure. Specimen 2. Double beta-treated at 760°C - 15 min. and end-quenched. HCl and HNO₃ Etch.

Fig. 4(b) - Typical beta-quenched microstructure. Specimen 2 (Quenched End). Double beta-treated and quenched from 760°C. Electrolytic Etch.

Mag. 100X Lab. Sample No. U604-H-2A Pd. Lt.

Fig. 4(c) - Typical beta-cooled microstructure. Specimen 2 (Intermediate zone). Double beta-treated and cooled from 760°C. Electrolytic Etch.

Mag. 100X Lab. Sample No. U604-H-2B Pd. Lt.

Fig. 4(d) - Typical room temperature structure of slowly cooled beta phase uranium. Electrolytic Etch.

Mag. 100X Lab. Sample No. U604-H-2C Pd. Lt.

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Fig. 5 - Specimen 1. Single beta-treated
at 760°C - 15 min. and end-quenched.

Mag. 1X

Lab. Sample No. U604-J-1

Fig. 6 - Specimen 4. Single beta-treated
at 760°C - 1 hr. and end-quenched.

Mag. 1X

Lab. Sample No. U604-H-1

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Fig. 7 - Specimen 3. Triple beta-treated at 760°C - 15 min. and end-quenched.

Mag. 1X

Lab. Sample No. U604-H-3

Fig. 8 - Specimen 5. End-quenched from beta-gamma transformation temperature, 785°C - 15 min.

Mag. 1X

Lab. Sample No. U604-J-2

Fig. 10 - Voids found in the air-cooled region of double gamma-treated uranium. Slug 7.

Mag. 1X

Lab. Sample No. U604-H-4

Fig. 11 - Voids found in the air-cooled region of triple gamma-treated uranium. Slug 8.

Mag. 1X

Lab. Sample No. U604-J-4