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GRAIN REFINEMENT OF URANIUM BY A BETA-QUENCH, ALPHA-ANNEAL PROCESS

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ABSTRACT

A grain size of approximately 0.10 mm may be produced in betatransformed uranium by quenching from a temperature in the beta phase and subsequently annealing in the alpha phase at a temperature above 600° C. This result is in contrast to the grain sizes of 0.35 mm and greater produced in metal that is air cooled after beta transformation. A significant portion of the grain refinement is produced by recrystallization of the beta-quenched structure during the high-alpha anneal.

Preliminary in-pile tests indicate that uranium plate that is heat treated by the "quench-and-anneal" process undergoes no surface roughening, in contrast to air-cooled plate, and is dimensionally stable. Similarly the treated rods also show improved surfaces, but are dimensionally less stable than the plate.

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GRAIN REFINEMENT OF URANIUM BY A BETA-QUENCH, ALPHA-ANNEAL PROCESS

INTRODUCTION

Polycrystalline uranium is subject to several types of dimensional instability during irradiation.⁽¹⁾ In one type, gross changes in dimensions occur due to the preferred orientation induced in the metal during forming operations. This type of instability may be avoided by heating the uranium into the beta phase, which virtually eliminates the preferred orientation and produces a structure that is relatively stable with respect to over-all dimensions. The beta transformation can result, however, in a second type of instability, in which the surface is roughened during irradiation. This form of instability is associated with the relatively large grains that may be produced during beta transformation.

This report presents the description of a heat treatment that produces a relatively small grain size in beta-transformed uranium, and the results of preliminary irradiation tests on the material. Independent studies similar to those described in this report were performed concurrently by Gardner and Riches⁽²⁾ at Hanford.

SUMMARY

Rapid cooling of uranium from beta phase temperatures (662 to 774° C) followed by alpha phase annealing (600 to 650° C) produced a marked refinement of grain size in comparison with metal that was air cooled from beta phase temperatures. Average grain sizes of 0.08 to 0.13 mm were obtained in quenched-and-annealed specimens, compared with an average grain size of 0.35 mm or greater for the air-cooled specimens. Though the beta-quench treatment alone produced a considerable grain refinement, a significant further refinement was produced during the high-alpha anneal, especially in the interior of a specimen. Grain refinement was obtained throughout the cross sections of both 3/16-inch-thick plate and l-inch-diameter rod by the "quench-and-anneal" treatment.

The grain refinement that occurs during annealing depends on a rapid cooling rate through the beta-to-alpha transformation. Samples that were slowly cooled in air exhibited no appreciable grain refinement on subsequent alpha annealing. In delayed quenches from beta temperatures, air cooling times that did not allow the specimen to cool below the beta-alpha transformation before quenching yielded refined structures on subsequent annealing. Longer delay times produced partially or completely unrefined structures, depending upon the extent of transformation at the time of quenching.

The grain size of quenched and fully annealed uranium was not noticeably affected by the annealing temperature. However, the time required for completion of the annealing reaction in plate specimens

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was decreased from 15 to 3 minutes when the temperature was increased from 600 to $650^{\rm o}{\rm C}$.

The grain refinement produced by annealing of the beta-quenched material is apparently a recrystallization process that involves grain boundary movement and the formation of new grains. The recrystallization proceeds until an equiaxed structure with a relatively small grain size is achieved.

Irradiation tests of uranium with the grain size refined by this technique showed that the metal in plate form was essentially dimensionally stable, and that the surface was not roughened as was the surface of metal that was beta transformed and air cooled. However, specimens of l-inch-diameter rod that were similarly heat treated and irradiated, though retaining relatively smooth surfaces, showed significant length increases, up to about 2% per 1000 MWD/T.

DISCUSSION

MATERIALS

The uranium for the experiments was obtained from either 3/16-inch-thick plate or l-inch-diameter rod. Both forms were obtained by hot rolling vacuum-cast ingots at a temperature of about $600^{\circ}C$. The typical microstructure of such material is shown in Figure 1.



FIGURE 1 - MICROSTRUCTURE OF AS-ROLLED URANIUM (Polarized Light, Mag. 50 X)

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TO: RECIPIENTS OF <u>DP-328</u>, <u>GRAIN REFINEMENT OF URANIUM BY A</u> <u>BETA-QUENCH, ALPHA-ANNEAL PROCESS</u>, BY C. L. ANGERMAN, ET AL. (CONFIDENTIAL)

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The chemical analysis of the uranium is shown in the table below. Since many lots of metal were used, only the extremes in the concentrations of the major impurities are listed.

Chemical Analysis of Uranium

ElementConcentration Range, ppmCarbon380-725Iron135 maximumNitrogen100 maximumNickel80 maximumChromium35 maximum

EQUIPMENT AND PROCEDURES

The heat treatments were carried out in a salt bath that was composed of a eutectic mixture of lithium and potassium carbonates. The temperature of the bath was measured with a chromel-alumel thermocouple located as near the sample as possible. The bath temperature was controlled within a range of about $\pm 3^{\circ}$ C.

Various procedures were used to quench the specimens from the beta and gamma phase temperatures. Where no delay time was specified, the specimens were removed from the salt bath and immersed in the quenching bath in less than 1 sec. In many of the experiments the specimens were held in air for a period of time before immersion in the quenching bath; this delay in quenching is referred to as the air cooling time. The quenching media were brine containing 15 wt %sodium chloride, tap water, vacuum pump oil, and air. The agitation in the liquid quenching media was moderate; the temperature of the bath was 20 to 25° C.

Samples were cooled from the alpha phase annealing temperatures by immersion in water.

In the investigation of the mechanism of the annealing reaction, the change in the microstructure of a specified area of a quenched sample was observed by a special technique. The sample was heated for five minutes at 720° C and quenched in water. A surface of the sample was prepared for microscopic examination, indexed, and photographed. The sample was then wrapped in tantalum foil and placed in a "Vycor" tube. A number of zirconium chips were also inserted in the tube. The tube was outgassed at about 300° C until a pressure of less than 5 x 10^{-5} mm of mercury was attained, and then sealed. The zirconium chips inside the tube were heated to remove the last traces of oxygen and nitrogen.

The evacuation tube was heated for the annealing treatment in the salt bath for 3 min at 650° C. The time for the specimen to reach the bath temperature was found to be 1.0 to 1.5 min, depending upon the area of

contact between the specimen and the tube wall.

Samples that were sealed in this manner could be re-examined under the microscope after a brief electrolytic polish. The total amount of uranium lost from the surface after each annealing treatment was normally less than 0.005 mm. This technique was repeated until no further changes in the microstructure were observed. As many as 8 to 10 reheatings were required to complete the annealing reaction.

DESCRIPTION OF THE "QUENCH-AND-ANNEAL" PROCESS

When uranium is slowly cooled through the beta-to-alpha transformation, the typical structure contains grains of widely varying size, many of them large and irregularly shaped with many twins and subgrains. After annealing for 2 hours at 650° C, the grains grow in size and become more regular in shape by the normal process of grain growth in which the larger grains consume the smaller ones. The appearance of these structures is illustrated in Figure 2.



a. As cooled



b. After reheating for 120 min at 650°C

FIGURE 2 - MICROSTRUCTURE OF AIR-COOLED URANIUM (Polarized Light, Mag. 50 X) Samples were air cooled after 5 min at 720° C. However, if a uranium specimen is rapidly cooled through the transformation by quenching in water, the resulting structure consists of fine grains on the surface of the specimen, and coarser grains with ragged boundaries in the interior. When the quenched sample is annealed, its structure changes by a recrystallization mechanism in which the smaller grains in the structure act as nuclei and grow. As shown in Figure 3, the resulting structure is equiaxed and has a much smaller grain size than uranium that has been air cooled.



a. As quenched



b. After annealing for 120 min at 650⁰ C

FIGURE 3 - MICROSTRUCTURE OF QUENCH-AND-ANNEALED URANIUM (Polarized Light, Mag. 50 X) Rod samples 1 inch in diameter were transformed for 5 min at 720° C, air cooled for 15 sec and then guenched in water. The structure at the center of the rod is shown in as guenched and as-annealed conditions.

The effect of the heat treatment variables on the quenched-andannealed structure is described in the following paragraphs.

EFFECT OF QUENCHING CONDITIONS

The rate of cooling through the beta-to-alpha transformation is the variable that exerts the greatest effect on the final structure. The ultimate grain size after annealing is smaller when the cooling through the transformation temperature is more rapid. Negligible grain refinement was observed in specimens that were air cooled after beta transformation. The effect of cooling rate on the grain size at the surface and center of 1-inch-diameter rods that have been quenched in salt brine, water, oil, and air, and subsequently annealed, is shown in the following table. Microstructures of the rod surfaces are shown in Figure 5, page 11.

Effect of Cooling Rate on Structure of 1-Inch-Diameter Rod

Specimens were quenched from 720°C and annealed at 650°C for 2 hours

Quenching Medium	Calculated Cooling Rate at Center, ^o C/sec at 660 ^o C	Grain Size at Surface, mm	Grain Size at Center, mm	
Brine	60	0.08	0.130	
Water	40	0.09	0.130	
011	20	0.13	0.200	
Air	5	0.30	0.300(no refinement	
			on annealing)	

As illustrated in the above table the effect of cooling rate was also demonstrated by the marked variation of grain size within a given specimen. In every case where refinement was observed after annealing a quenched specimen, the average diameter of surface grains was about two-thirds the average diameter of the center grains; an example of this variation is shown in Figures 4a and 4b.





a. Surface structure grain size- 0.08 mm b. Center structure grain size-0.13 mm

FIGURE 4 - VARIATION OF GRAIN SIZE WITH LOCATION IN THE SPECIMEN (Polarized Light, Mag. 50 X) Rod samples 1 inch in digmeter were heated for 5 min at 720° C, air cooled for 10 s

Rod samples 1 inch in diameter were heated for 5 min at 720° C, air cooled for 10 sec and guenched in water. The samples were then annealed for 15 min at 650° C.



h. Water quench grain size-0.09 mm



a. Brine quench grain size-0.08mm



d. Air cool grain size-0.30mm



c. Oil quench grain size-0,13mm

FIGURE 5 - EFFECT OF QUENCHING MEDIA ON FINAL GRAIN SIZE Samples 1 inch in diameter were transformed for 5 min at 720° C, quenched

Samples 1 inch in diameter were transformed for 5 min at 720° C, quenched as shown, and, except for sample (d), were annealed for 60 min at 625° C. Sample (d) was annealed for 120 min at 650° C. All specimens were taken near the surface of the sample with polarized light at 50 X. No consistent correlation was observed between the final structure and time and temperature in the beta phase.

EFFECT OF DELAYED QUENCH

The effect of air cooling before quenching and subsequent alpha annealing is summarized in Figure 6 for specimens of 3/16-inch plate; quenching from both beta and gamma phase temperatures is shown. The effect of air cooling before quenching was studied for the practical reason that a finite time is required to transfer a large uranium piece from a salt bath to a quench tank.

For quenching from the beta phase in the range 680 to 760° C, the results were predictable. The grains were refined throughout the plate on subsequent alpha annealing if the sample was completely in the beta phase when quenched; this condition is shown in Area I of the beta phase region of Figure 6. For a typical beta treating temperature, 720° C, delay times of 10 seconds or less yielded a refined structure. At somewhat longer delay times, when the surface portions of the samples had cooled into the alpha phase, refinement was observed only in the center sections, as shown in Area III of the beta phase region. The typical air-cooled structure was observed when the specimens were quenched after still longer times, as outlined by Area IV of the beta phase region.

The behavior of plate specimens that had been heated into the gamma phase before quenching and subsequent alpha annealing was anomalous. When the air cooling time was sufficient (15 sec) to allow the uranium to cool to below the gamma range, the results were substantially the same as for samples that were heated originally into the beta phase; structures corresponding to those in Areas I, III, and IV were obtained as designated in Figure 6. If the sample was quenched when it was completely in the gamma range (air cooled for less than 5 sec), refinement was also observed throughout its thickness. However, when specimens contained both gamma and beta phases (air cooled 5 to 10 sec), grain refinement was observed only in the beta-phase portions contained in the surface of the sample. The center of the sample, which was in the gamma phase, contained large grains that were similar in appearance to grains produced by air cooling. The structures that were obtained by annealing uranium that had been quenched from the gamma phase after air cooling for 5, 10, and 15 sec are shown in Figure 7 on page 14.



FIGURE 6 - THE EFFECT OF INITIAL TEMPERATURE AND TIME OF AIR COOLING BEFORE QUENCHING ON THE CHARACTER OF GRAIN REFINEMENT IN PLATES (Plate thickness-3/16 inch)



a. Surface structure grain size-0.09mm



c. Surface structure grain size-0,11mm



e. Surface structure grain size-0.08mm Air cooled 5 sec

> Air cooled 10 sec



b. Center structure grain size-0,12mm



d. Center structure grain size-0.25mm



f. Center structure grain size - 0.13 mm

FIGURE 7 - MICROSTRUCTURES OF GAMMA-TRANSFORMED PLATE (Polarized Light, Mag. 50 X) Samples 3/16-inch thick were heated for 5 min at 800° C, air cooled as shown, and quenched in water. The samples were then reheated for 30 min at 650° C.



Air cooled

No explanation for the anomalous behavior of the material quenched from the gamma phase has been developed, but it is believed that the difference is caused by a change in the stresses that are induced by quenching when the gamma and beta phases are both present at the moment the samples were quenched.

The assumptions concerning the phases that were present at the instant of quenching were confirmed by direct measurement of the time required for samples to cool through the transformation temperatures from 680, 720, 750, and 800° C. The measurements were made with chromel-alumel thermocouples that were located near the centers of the samples. These results are compared with the metallographic results of Figure 6 in the following table.

Initial Temp, ^o C	Cooling Rate in Air, ^o C/sec	Cooling Rate During Quench, ^O C/sec	Time for Beginning of Transformation During Air Cooling, 	Maximum Air Cooling Time for Complete Refinement, sec*
680	5.0	460	4	4-5
720	5.0-5.5	398	11	10-12
750	5.7	400	15	13-15
800	5.1-6.1	-	2(gamma to be 32(beta to alp)	ta) 5-10 na) 20-25

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		Compai	rison	of	Data	
from	Cooling	Curves	with	Me	tallographic	Results

* These limits were obtained by metallographic examination and represent the longest time at which complete refinement and the shortest time at which partial refinement, respectively, were observed.

A study of the effect of the delay time in air before quenching on the structures of subsequently annealed rod specimens showed that the geometry of the specimen markedly influenced the results. The relative effects of beta temperature and time of air cooling before quenching on the grain refinement produced in rods by subsequent annealing is shown in Figure 8, on page 16, for comparison with that of plate specimens in Figure 6. After beta transformation at 720°C, a refined structure was obtained throughout the specimen for delay times before quenching of 30 sec or less. As the delay time was increased, coarse grains were formed at the center of the rod, with fine grains in regions near the surface. After delay times of 60 to 90 sec, a coarse-grained structure was evident throughout the rod. Similar structures were noted for beta transformation at 680°C, with delay times permissible for refinement correspondingly shorter.



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FIGURE 8 - THE EFFECT OF TEMPERATURE AND TIME OF AIR COOLING BEFORE QUENCHING ON THE CHARACTER OF GRAIN REFINEMENT IN RODS

An explanation of these results may be tentatively given by reference to schematic cooling curves for the several regions of the rod. as shown in Figure 9 on page 18. On quenching of a massive piece of metal, the interior of the specimen requires a longer time to achieve a high cooling rate than the surface requires. If the specimen temperature at the time of quenching was high enough that the surface and interior cooled at a high rate through the transformation, then subsequent annealing would produce refinement of both surface and interior of the slug, as illustrated in Figure 9a. This condition is designated by Area I of Figure 8. A delay in air before quenching the rod could, however, lower the temperature to a point at which the interior was cooling at a rate not great enough to produce grain refinement, although the surface regions did cool sufficiently rapidly, as indicated in Figure 9b. Thus a structure of large grains at the center of the rod and refined grains near the surface would result at the intermediate times, designated by Area II in Figure 8. An even more complex structure would result if the immediate surface of the slug had been air cooled below the transformation point but the intermediate and center regions remained above the transformation at time of quenching, as indicated in Figure 9c. Such a structure would show a region of somewhat coarsened grains at the surface (similar to Region III of the plate specimens at intermediate delay times) adjacent to an intermediate layer of refined grains, which in turn enclosed the coarse-grained center of the slug. Instances of such a complex structure were observed in the rod specimens, one of which is shown for the intermediate delay times designated as Area III! in Figure 8. Finally, the very long delay times indicated in Figure 9d would yield a coarse-grained structure throughout the specimen, designated by Area IV in Figure 8.

Complex and incompletely refined structures were produced when rods quenched from gamma-phase temperatures were subsequently annealed. When the air cooling time was short (2 sec) a pronounced columnar structure was produced throughout the rod as shown in Figure 10a on page 19. No grain refinement was evident in such specimens, in contrast to the complete refinement achieved in plate specimens that were rapidly quenched from the same temperatures. With longer delay times (20 sec) grain refinement was produced at the surface of the rod, as shown in Figure 10b on page 19, but the interior of these specimens showed very large grains, becoming somewhat smaller at the extreme center of the rod. The refinement only at the surface was similar to that obtained in plates that were air cooled until both the gamma and beta phases were present, and may again represent a condition which is obtained on quenching from a two-phase condition.

EFFECT OF ANNEALING VARIABLES

Annealing of beta-quenched uranium caused the microstructure to change from irregularly shaped grains of a wide range of diameters to equiaxed grains of nearly uniform size. The characteristics of the

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FIGURE 9 - CORRELATION BETWEEN QUENCH CONDITIONS AND GRAIN STRUCTURE In the above diagrams, curve s represents the cooling behavior of the surface of either plate or rod, curve i represents the cooling behavior of the interior of the plate, or the equivalent intermediate region between surface and center of the rod, curve c represents the center of the rod.



a. Air cooled 2 sec



b. Air cooled 20 sec

FIGURE 10 - MICROSTRUCTURES OF GAMMA-TRANSFORMED ROD

Cross sections from rod 1 inch in diameter were transformed for 5 min at 800°C, air cooled as shown, and quenched in water. The samples were then annealed 30 min at 650°C. All photographs were taken of macroetched samples at 4X. annealed structure were not appreciably affected by changes in the annealing temperature in the range of 600 to 650° C. The grain size of the annealed structure was invariably 0.080 to 0.120 mm at the surface and 0.130 to 0.180 mm at the center of both rod and plate. However, as shown in Figure 11, the time required to obtain the fully annealed structure was dependent on the temperature; for example, when the annealing temperature was increased from 600 to 650° C, the time for completion of annealing decreased from about 15 to 3 minutes. One sample was found to be incompletely annealed after 20 hours at 550° C.

Detailed studies of the kinetics of the annealing reaction have been carried out at Hanford.⁽²⁾



FIGURE 11 - TIME FOR COMPLETION OF THE ANNEALING REACTION

MECHANISM

The grain refinement that occurs during annealing of the beta-quenched structure is apparently due to a recrystallization process. The microstructural features of the recrystallization during annealing may be summarized as follows:

- 1. As-quenched uranium is characterized by grains with ragged boundaries and a complex substructure. Large single grains contain subgrains with mismatched orientations and diffuse boundaries. Heavily twinned grains, and occasionally, small isolated recrystallization nuclei can also be observed.
- 2. Upon annealing, some of the large grains appear to break up into regions of smaller, well-defined grains, and the diffuse subgrain boundaries become discrete. The new grains thus formed and the recrystallization nuclei existing in the quenched structure grow at the expense of the remaining large grains. The ragged grain boundaries of the as-quenched structure become smooth and the grains become more equiaxed, which indicates a general reduction of grain boundary energy.
- 3. On continued annealing, a general coarsening of the grain structure occurs by continued adjustment of grain boundaries and by elimination of smaller grains in the structure.

This mechanism is illustrated in Figure 12 on page 22, which shows the change in appearance of a quenched sample during the annealing process. The quenched structure is shown in Figure 12a; each successive photograph shows the appearance of the same area at progressively longer annealing times.

The arrow in Figure 12a points out a small grain that becomes the nucleus of the larger grain shown in the other photographs. Careful comparison of the photomicrographs in this sequence will reveal many such nuclei.

Thus, the annealing reaction is a recrystallization process, with many features in common with the recrystallization that follows cold work. $^{(4)}$ The significant distinction lies in the means of imparting energy to the structure; in the quench-annealing reaction the quench supplies strain energy, possibly by a martensitic-type transformation, $^{(5)}$ while in a conventional recrystallization process, the strain energy is supplied by cold work. In both cases, the stored energy is probably interfacial energy of a fine substructure. In quenched uranium, the substructure appears to be composed of subgrains with diffuse boundaries produced by the very rapid transformation, while in cold-worked uranium, the substructure is assumed to be mechanically fragmented crystallites and twins. $^{(4)}$



a. As - quenched



b. After approximately 1 min



c. After approximately 3 min



d. After approximately 5 min



e. After approximately 7 min

i



f. After approximately 9 min

FIGURE 12 - CHANGES IN MICROSTRUCTURE DURING ANNEALING (Polarized Light, Mag. 50 X) Sample was transformed for 5 min at 720° C and quenched in water. The microstructure is shown as-quenched and after progressive periods of annealing at 650° C. All photographs show the same area of the sample. Quench-induced strains, as evidenced by twinned grains, may also contribute to the recrystallization of the quenched structure.

Apparent differences in the recrystallization of quenched and coldworked structures can be ascribed to the microstructural differences of the initial state. When quenched metal is recrystallized, there apparently is less nucleation of new grains than occurs when coldworked metal is recrystallized. The migration of grain boundaries during the growth of grains from a few nuclei and the sharpening of diffuse boundaries of subgrains appear to be the prevalent mechanisms for recrystallization of quenched uranium.

IRRADIATION BEHAVIOR

PLATES

Several two-foot segments of quenched-and-annealed uranium plate, along with control segments of air-cooled uranium plate, were clad in aluminum sheaths and irradiated to a maximum exposure of 1100 MWD/T.

The quenched-and-annealed samples were transformed for 3 minutes at 720° C, quenched in water with a 8-second delay, and annealed for 15 minutes at 625° C, whereas the controls were cooled in air after 5 minutes at 720 to 730° C.

The effect of the quench-anneal treatment on surface stability during irradiation is illustrated in Figure 13 on page 24. It can be seen that the "bumps" that are characteristic of air-cooled uranium are absent in the quench-annealed samples.

Measurement of the changes in length of the segments, shown in Figure 14 on page 25, demonstrated that both the quench-annealed and air-cooled structures cause only small increases in length of about 0.24 and 0.32% per 1000 MWD/T exposure, respectively. Dimensional changes in the width and thickness of the plate were too small for measurement.



a. Core was transformed for 3 min at 720⁰ C, air cooled for 8 sec, quenched in water, and then annealed for 15 min at 625⁰ C. Note the relative absence of surface roughening. (Horizontal marks are spacer ribs)



b. Core was transformed for 5 min at 720°C and air cooled. Note the roughened surface. (Horizontal marks due to abrasion of ribs on adjacent plate a)

FIGURE 13 - SURFACE APPEARANCE OF AI-CLAD URANIUM PLATES AFTER IRRADIATION Flat surfaces of the 3-inch-wide plate are shown. The "quenched-and-annealed" plate, shown in a, was irradiated adjacent to the "air-cooled" plate, shown in b.

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RODS

Preliminary results on the irradiation of aluminum-clad rod specimens indicate that both beta quench and beta quench-anneal heat treatments promote a relatively smooth surface as compared to air cooling. However, the results also indicate that considerably more dimensional instability is induced by the beta-quenching treatments in rods than in plates. Length increases up to about 2% per 1000 MWD/T were observed in both beta-quenched and quench-annealed rod specimens. The irradiation results obtained on rod specimens will be described in detail in a subsequent report.

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