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PREFERRED ORIENTATION AND RECRYSTALLIZATION IN  
FABRICATED ZIRCONIUM, URANIUM, AND THEIR ALLOYS

A Literature Survey by W. Uhlmann

November 1961

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FABRICATED ZIRCONIUM, URANIUM, AND THEIR ALLOYS.

A Literature Survey by W. Uhlmann

Preface.

A survey of current publications concerning the subject matter of the title is given, which covers the time from 1957 to the first part of 1961.

Besides publications in current periodicals the following sources have been employed.

Nuclear Science Abstracts vol. 11, 1957 - vol. 15, 1961.

The Review of Metal Literature, vol. 17, 1960 and vol. 18, 1961,

The Atomic Industry Reporter.

The survey does not claim to be exhaustive.

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## GENERAL

1. RELATION BETWEEN POLYGONIZATION AND RECRYSTALLIZATION AND SOME PROBLEMS POSED BY THE THEORIES OF RECRYSTALLIZATION.

Cahn, R.W.

pp. 33-40 in "Symposium on Special Metallurgy held at Saclay June 27-28, 1957" 130 p.

The experimental methods used for the study of the motive force of recrystallization and of the relation between restoration and recrystallization are given, and the results obtained are discussed. The classical methods are the study of the kinetics of recrystallization and the measurement of the energy stored in a cold-worked metal and the release of the energy. The new techniques of studying recrystallization under an applied stress is described, and some results obtained with it are compared with previous data.

2. RELATIONS BETWEEN DEFORMATION STRUCTURE AND RECRYSTALLIZATION STRUCTURE.

Tiedema, T.J.

pp. 21-32 in "Symposium on Special Metallurgy held at Saclay June 27-28, 1957." 130 p.

An attempt is made to define the effect of factors such as metal purity, nature of cold deformation, and size of metal grains on the recrystallization structure by examination of the face-centered cubic structure. The mechanism and others which have been proposed are inadequate for a description of the formation of this structure. The model proposed by Rowland (J. Inst. Metals 83, 1620(1954) is described in detail, and it is shown how it is responsible for the formation of germs of cubic orientation. Studies made with monocrystals of copper are described.

3. ANALYSIS OF THE AMOUNT OF PREFERRED ORIENTATION BY X-RAY DIFFRACTION LINE INTENSITIES.

Vaughan, D.A.

BMI-X-156, June 30, 1960, 11 p.

An analysis of x-ray diffraction theory for the para-focussing diffractometer which involves a correction to the intensity formulas given by other workers for the case of reflection from a thick block of randomly orientated powder is presented. Experimental tests of the theory were made with NaCl both in powder form and as a single crystal and then applied to rolled tungsten specimens.

## ZIRCONIUM AND ZIRCALLOY.

4. HEATING OF ZIRCONIUM FOR ITS TRANSFORMATION WITH HEAT AND WITH COLD. RECRYSTALLIZATION AND RESTORATION OF THE COLD-WORKED METAL.

Whitwham, D., Boghen, J. et al.

pp. 79-88 in "Symposium on Special Metallurgy held at Saclay June 27-28, 1957".

The technology of the transformation of zirconium offers many difficulties. Tests with zirconium and Zircaloy-2 were made to determine a technique of working with zirconium and its alloys in heat or cold. The work, divided into two parts, consisted of following the metal structure during hot forging and rolling and cold rolling with intermediate annealings. The contamination was closely controlled during the operations. Heating for the hot deformation of zirconium was investigated in salt baths and in air. The methods of heating used to soften the metal for cold working and the properties of the metal after various treatments are described.

5. MECHANICAL PROPERTIES AND RECRYSTALLIZATION OF IODIDE ZIRCONIUM.

Savitskii, E.M., Terekhova, V.F. Translated by Lydia Wenters (Argonne Nat. Lab.) from Trudy Inst. Met. im.A.A.Balkova, No. 3, 181-90 (1958) 14p. JCL or LC.

AEC-tr-4093.

The effect of temperature (-195 to 1200°C) on the mechanical properties of iodide zirconium revealed that the hexagonal  $\alpha$ -zirconium has significant ductility at -196°C.  $\beta$ -Zirconium with a body-centered cubic lattice exhibited a higher ductility than  $\alpha$ -zirconium. The  $\alpha \rightarrow \beta$  polymorphous transition in zirconium was characterized by a break in the shape of the temperature curve for mechanical properties. The dependence of grain size and degree of deformation in cold rolling on the annealing temperature were investigated. The recrystallization diagram is presented. The optimum temperature range for annealing the cold-deformed iodide zirconium was determined to be 700 to 750°C.

6. INFLUENCE DES CONDITIONS DE LAMINAGE ET DE RECUIT SUR LA TEXTURE ET LES PROPRIETES MECANQUES DU ZIRCONIUM.

Orssaud, J.

CEA 1542, June 27, 1958. 72p.

Thesis submitted to the Univ. of Poitiers.

Rolling and annealing textures of zirconium samples at several rolling rates were studied by tracing diagrams of pole figures

vs. the position in the sheet thickness with an automatic recorder. Tensile tests, hardness measurements, and micro-graphic examinations allowed a study of the evolution of the recrystallization and the variation of the mechanical properties after rolling and/or annealing. Annealing textures varied slightly with the annealing temperature. Annealing at 500°C gave several peculiarities. This temperature seemed characteristic in the study of zirconium.

7. PREFERRED ORIENTATION IN URANIUM, THORIUM, ZIRCONIUM AND BERYLLIUM.

McHargue, C.J., Jetter, L.K.

pp. 430-459 in "Progress in Nuclear Energy, Ser. V. Metallurgy and Fuels. Vol. 2".

Preferred orientations developed in uranium, thorium, zirconium, and beryllium by a variety of fabrication processes have been reviewed. The development of the textures was discussed in terms of present theories of texture formation and deformation and annealing mechanisms. Where possible the effect of preferred orientation on physical and mechanical properties was pointed out.

8. QUARTERLY PROGRESS REPORT OF THE TECHNOLOGY SECTION-SPECIAL MATERIALS SECTION FOR THE PERIOD OCTOBER 1 TO DECEMBER 31, 1958.

WAPD-NCE-333-59. Jan. 15, 1959. 30p. OTS.

A cursory study of critical grain growth in Zircaloy-2 showed largest grain growth to occur in specimens cold rolled 9% and annealed at 750°C for 8 hr. The heat treatment behavior of the nickel-free Zircaloy-2 specimens was similar to that of Zircaloy-2.

9. CRITICAL RECRYSTALLIZATION OF ZIRCONIUM.

Bokros, J.C. (General Atomic Div., General Dynamics Corp., San Diego, Calif.)

Trans. Met. Soc. AIME 218, pp. 351-353 (1960) Apr.

At temperatures above 950°F, zirconium which was strained a critical amount will experience critical recrystallization. The large grain size thus formed can result in a reduction in the fatigue life by a factor of 2 to 9 at both high and low temperatures. The critical strains for zirconium vary from 15% at 900°F to about 2% at 1125°F, while those for Zircaloy III vary from 15% at 1050°F to 5% at 1200°F. Zircaloy II experiences no critical recrystallization up to 1200°F.



# 10. DEFORMATION MODES OF ZIRCONIUM AT 77°, 575°, AND 1075°K.

Rapperport, E.J., and Hartley, C.S.

Trans. Met. Soc. AIME 218, pp. 869-876 (1960)

The only slip system observed in zirconium crystals deformed at 77, 575, and 1075°K was  $(10\bar{1}0)$  ( $\bar{1}2\bar{1}0$ ) with a critical resolved shear stress in tension of  $1.0 \text{ kg/mm}^2$  at 77°K;  $0.2 \text{ kg/mm}^2$  at 575°K; and  $0.02 \text{ kg/mm}^2$  at 1075°K. The active twin planes were  $\{10\bar{1}2\}$ ,  $\{11\bar{2}1\}$ ,  $\{11\bar{2}2\}$ , and  $\{11\bar{2}3\}$  with varying temperature dependence. A detailed analysis for the slip direction using Laue spot asterism is appended.

# 11. PREFERRED ORIENTATIONS IN EXTRUDED ZIRCALOY-2 TUBING.

Laidler, J.J.

HW-64815. April 15, 1960.

Texture studies were made on selected specimens of extruded Zircaloy-2 tubing having different fabrication histories.

Tubing extruded with a 14:1 extrusion ratio has a principle texture with the basal planes perpendicular to the surface of the tube and parallel to the tube length. In the nominal cold-working texture, the  $\langle 10\bar{1}0 \rangle$  direction is parallel to the tube axis. Annealing or extruding at high temperature results in a rotation of the basal plane about its pole so that the  $\langle 11\bar{2}0 \rangle$  direction tends to become aligned parallel to the tube axis.

Tubing extruded at 37:1 extrusion ratio and sheath material coextruded at an effective 33:1 ratio have two primary orientations of the basal planes: (1) inclined at about 50° to the surface of the tube and parallel to the tube axis; and (2) perpendicular to the surface of the tube and parallel to the tube axis. The direction orientations are similar to those for the tubing with a 14:1 extrusion ratio.

The marked difference in orientation as a function of extrusion ratio is probably an effect of prior texture. Separate work on extruded Zircaloy-2 sheaths has shown that the textures produced with a 15:1 extrusion ratio are quite similar to those for a 35:1 extrusion ratio.

It is concluded that only  $\{10\bar{1}0\} \langle 11\bar{2}0 \rangle$  slip was operative during extrusion and cold-working. It is doubtful that basal slip occurred, even at the higher extrusion temperatures. Twinning is prevalent during cold-working.

12. A COMPARATIVE STUDY OF TITANIUM AND ZIRCONIUM BY THE DILATOMETRIC METHOD.

Cizeron, G., Lacombe, P.

Mém. sci. rev. mét., Vol.57, pp.179-193 (1960) (In French)

The metals Zr and Ti from different sources (with different contents in impurities) were compared by the dilatometric method. Their behavior was studied during the passage from the cold-worked state to the recrystallized one by annealing in the  $\alpha$ -field or during the  $\alpha \rightleftharpoons \beta$  transformation. The presence of small contents of Fe in Zr and Si in Ti, if these contents are superior to the limit of solubility, can be revealed by some dilatometric anomaly. This anomaly corresponds to an eutectoid reaction in the phase diagram of each system Zr-Fe and Ti-Si. The allotropic transformation itself occurs in a smaller temperature range if the metal is purer. If during the cooling the metal undergoes a small stress of compression, the sense of the  $\beta \rightarrow \alpha$  dilatometric anomaly is inversed. A crystallographic mechanism, similar to that which has been proposed by W. Burgers, is described in order to explain this anomalous behavior.

13. A STUDY OF PREFERRED ORIENTATION IN EXTRUDED ZIRCALOY-2 PRESSURE TUBES.

Tuxworth, R.H.

CRMet-901, Nov. 1960. 28p.

The preferred orientations present in large-diameter extruded Zircaloy-2 pressure tubes and, for comparison, in a relatively thick-walled tube, was investigated. The most consistent feature observed for the pressure is the occurrence of the basal planes parallel to the working direction. Textures of (10 $\bar{1}$ 0) and (11 $\bar{2}$ 0) types, as well as randomness about the transverse direction, are found. The thick-walled extruded tube also exhibits a basal-plane texture. An attempt is made to explain the higher transverse yield strength as compared with the longitudinal yield strength in terms of the preferred orientation present. This treatment suggests that the preferred slip system at room temperatures is of the form (10 $\bar{1}$ 0)(12 $\bar{1}$ 0).

14. TEXTURE OF EXTRUDED ZIRCONIUM AND ZIRCALOY-2 TUBING.

Nerses, V.

NMI-1222, July 11, 1960. 35p.

The textures developed in extruded thin-wall zirconium and Zircaloy-2 tubes were studied for materials with two different fabrication histories. The two primary fabrication methods used were extrusion cupping and rolling. In all cases, the textures were similar with the (10.0) poles parallel to the

extrusion direction. The results of burst-strength tests, conducted on the extruded thin - wall tubes, and coextrusion experiments were also independent of the primary fabrication history.

15. MONTHLY PROGRESS REPORT FOR APRIL - MAY 1956.

BRB - 31. June 29, 1956. Decl. Mar. 30, 1960. 39p.

A difference in the cupping characteristics between two lots of Zircaloy-2 deep drawing strip led to the investigation of strip orientation. A  $\gamma$ -phase extrusion was initiated to investigate the feasibility of producing solid U fuel element for Hanford by extrusion and swaging. Laboratory evaluation was completed on the extrusion of hollow U fuel element and results are presented. The crystal orientation of a hollow slug tube  $\alpha$ -extruded at 1175°F with an area reduction of 24:1 was investigated. Three Zircaloy-2 billets (Z-C series) of 8 in. O.D. x 20 in. length were successfully extruded as ribbed tube. (For preceding period see BRB-30.)

16. METALLURGY OF ZIRCALOY-2. PART I. THE EFFECTS OF FABRICATION VARIABLES ON THE ANISOTROPY OF MECHANICAL PROPERTIES.

Rittenhouse, P.L., Picklesimer, M.L.

ORNL-2944, Nov. 15, 1960, 108 p.

The anisotropy of mechanical properties of Zircaloy-2 was studied as a function of fabrication variables. The variation in tensile and impact properties with specimen orientation was taken as the measure of the anisotropy of mechanical properties for each material. A qualitative separation of the effects of the fabrication variables on the resulting anisotropy of mechanical properties is made, but it is valid only on the rolling plane of the plate. A contractile strain ratio, a ratio of the natural contractile strain in the rolling plane to that in the direction normal to the rolling plane (measured on the round tensile specimen after testing), is introduced to aid in the interpretation of the tensile data. A Zircaloy-2 fabrication schedule (consisting of, in succession, ingot breakdown at a temperature of 1800 to 1900°F, major reduction at a temperature of 1800 to 1900 or 1350 to 1450°F, a  $\beta$  heat treatment of 1800 to 1850°F for 30 min., followed by either a water-quench or a rapid air-cool to below 1200°F, a final reduction of 25 to 40% at 1000°F, and an anneal at 1400 to 1425°F for 30 min) was found to produce a much more nearly isotropic material than any of the schedules investigated. This material is anisotropic in strain behavior and tensile properties in comparison to the common cubic materials. The elimination of the intermediate  $\beta$  heat treatment from the fabrication schedule resulted in the production of a material with tensile properties for all directions in the plane of rolling essentially the same, but

which allowed little contractile strain to occur in the thickness direction of the plate. This indicated that a high degree of three-dimensional anisotropy existed in the material. The effect of cross rolling on the anisotropy of mechanical properties of Zircaloy-2 was found to be a function of the temperature and stage of fabrication at which it was performed, the position of the ingot axis relative to the final fabrication directions, and the type of cross rolling, whether unidirectional or rotational. It was concluded that the use of other methods of examination and interpretation was necessary to satisfactorily evaluate the effects of variation of the fabrication variables on the anisotropy of Zircaloy-2.

17. A STUDY OF THE TENSILE PROPERTIES OF ZIRCALOY-2 AT VARIOUS STAGE OF FABRICATION.

Goodwin, J.G.

WAPD-BT-20 (p.39-51)

This work was performed to determine changes which occur in the tensile properties of Zircaloy-2 various stages of fabrication from ingot to cold-rolled strip. Material size, test direction, and the effect of round and flat specimens on the tensile properties were studied at both room temperature and 600°F. It was found that the type of specimen had little effect on the strength properties but did influence ductility; both size of material and test direction influenced the results of strength measurements.

18. EFFECT OF HEAT TREATMENT OF THE STRUCTURE AND HARDNESS OF GAMMA EXTRUDED URANIUM.  
Final Report, Problem Assignment 2-M.

Padden, R.M.

HW-8358(Rev.) Dec. 24. 1947. Decl. Dec. 15, 1959. 13p.

Tests were conducted to determine the optimum time-temperature treatment for producing of recrystallized grain structure in gamma extruded uranium. Study of approximately 160 samples revealed no definite time-temperature treatment that would consistently produce a complete or nearly complete recrystallized structure, and no definite correlation between hardness values and recrystallization was established.

19. INFLUENCE OF THE MODE OF PLASTIC DEFORMATION ON THE RECRYSTALLIZATION, AFTER COLD WORKING, OF IMPERFECT SINGLE CRYSTALS OF URANIUM PRODUCED BY  $\beta \rightarrow \alpha$  TRANSFORMATION.

Calais, D., Lacombe, P. et al.

Mem. sci. rev. mét., Vol. 56, pp. 261-272 (1959) (In French)

A new method for the preparation of perfect crystals of uranium was developed. It is based on a stress deformation of the imperfect crystals from the  $\beta \rightarrow \alpha$  phase transformation, followed by a high temperature anneal above the  $\alpha$  phase. Depending on the orientation of the original crystal, deformation occurs by slip, twinning, or deformation bands. Annealing gives perfect recrystallization crystals, whose orientation has a direct relationship with that of the original crystals. During the recrystallization there are some crystallographic characteristics of the deformation mechanism. The (0 0 1), which is the rotation axis allowing the formation of the deformation bands, is preserved during recrystallization. In the case of deformation by twinning, the recrystallized grains show less clearly marked orientation relationships, but the growth of larger crystals is facilitated.

20. SECONDARY RECRYSTALLIZATION AND CRITICAL COLD-WORK RECRYSTALLIZATION OF HIGH-PURITY URANIUM.

De Libanati, N. et al.

After 150 to 400% cold-working of high-purity U, a reheat to 650°C caused a primary recrystallization followed by exaggerated growth. Cold-working of 2 to 6% applied to the primary texture caused growth of large crystals of a different texture from that of secondary crystals. These effects constitute criteria for the purity of the metal.

21. PREFERRED ORIENTATION IN ROLLED AND IN RECRYSTALLIZED HIGH-PURITY URANIUM ROD.  
Final Report of Metallurgy Program 4.1.17.

Mueller, M.H., Knott, H.W.

ANL-5887, Apr. 1959. 16p.

The preferred orientation of a relatively small piece of high-purity uranium rod, rolled to an 85% reduction at 300°C, has been determined in the as-rolled and in the recrystallized conditions. The 12 different charts obtained indicated that the as-rolled texture could be described as a duplex (041) and (352) with the (041) being the major component and with considerable spread about each component. The recrystallized rod showed approximate (041) and (392) components with considerable spread. These texture components for both the as-rolled and the recrystallized rods are not too different from those previously reported for reactor-grade uranium rod. However, it was noted that the texture appeared to be quite sharp for the reduction used, and the maximum intensity on an inverse pole figure was considerably displaced from the periphery of an (001) standard projection for both the rolled and recrystallized rods.

22. METALLURGY DIVISION QUARTERLY PROGRESS REPORT FOR PERIOD ENDING JULY 31, 1951.

Miller, E.C., Bridges, W.H.

ORNL-1108, Feb. 6, 1952. Decl. Oct. 9, 1959. 92p.

..... In order to determine whether or not the type of preferred orientation developed in  $\alpha$ -extruded uranium rod was dependent on the actual extrusion temperature, x-ray-diffraction studies were made of completely recrystallized and unrecrystallized extruded uranium. The results show that the texture developed upon deformation is not always retained upon recrystallization. ....

23. EXTRUSION OF URANIUM; URANIUM ALLOYS, AND URANIUM COMPACTS.

Davis, T.F.

TID-3563, 1961, 33p.

A literature search.

24. RECRYSTALLIZATION IN ROLLED URANIUM SHEET.

Lloyd, L.T., Mueller, M.H.

ANL-6359(p.86-8).

An investigation of the occurrence of recrystallization without an apparent preferred orientation change was undertaken. The work was conducted with high-purity uranium. The individual results are not much different from those of the earlier work. A plot for the change in mean coefficient of expansion of transverse samples as a function of volume per cent of recrystallization is included. The samples annealed at lower temperatures did not show significant changes in expansion coefficient, whereas those annealed at higher temperatures did. The material from the first work seemed to recrystallize at a lower temperature than that used currently. This is probably associated with the greater heterogeneity of deformation in the former. Studies show that recrystallization, at low and moderate temperatures, of high-purity uranium sheet reduced approximately 80% in thickness at room temperature is characterized by the formation of extremely fine recrystallized grains which are later absorbed by growth of larger grains.

25. NEUTRON STUDY OF THE CRYSTALLINE TEXTURE OF URANIUM BARS.

Laniesz, J. et al.

J. Nuclear Materials, Vol.2, pp. 69-74 (1960) (In French)

The method of studying textures by neutron diffraction was applied to a uranium bar extruded in the  $\alpha$  range at 600°C. By means of a single series of experiments, a good statistical image of the texture was obtained. The specimen was a cylinder of about 5 cm<sup>3</sup>, the axis of which, normal to the direction of drawing, coincided with the axis of a beam of monochromatic neutrons ( $\lambda = 1.143 \text{ \AA}$ ). The neutron detector was BF<sub>3</sub> counter which describes a circle in a horizontal plane, centered on the specimen. The diffraction spectrum obtained from a piece of uranium bar, extruded in the  $\alpha$ -range and treated in the  $\beta$ -range, revealed that this specimen has only a weak degree of preferred orientation, whereas an as-extruded specimen shows a marked (1 1 0) texture in the direction of extrusion. The results obtained are in agreement with those determined by x-ray diffraction by various authors with uranium bars extruded or rolled in the  $\alpha$ -range.

26. PROPERTIES OF COLD DRAWN URANIUM ROD. I. ON THE THERMAL EXPANSION COEFFICIENT AND THE THERMAL CYCLING GROWTH OF RECRYSTALLIZED SPECIMENS.

Soeno, K. (Hitachi Ltd., Japan)

J. Atomic Energy Soc. Japan, Vol. 2, pp. 89-95 (1960)  
(In Japanese).

$\beta$ -treated, cold-drawn uranium rods were studied to determine the relation between the thermal expansion coefficient in

the drawing direction and the working degree (2 to 30% reduction in area); the change of the coefficient due to grain growth after recrystallization; and the thermal cycling growth of recrystallized specimens. The preferred orientation was obtained by only two percent work and the dimensional instability was apparent on thermal cycling. The (010) plane as one component of preferred orientation was arranged parallel to the rod axis regardless of the degree of working. The grain growth after recrystallization was found to affect the formation of the preferred orientation.

## 27. TEXTURES IN EXTRUDED URANIUM.

Russel, R.B.

Paper from "Advances in X-Ray Analysis", v. 3, Plenum Press, Inc., New York, 1960, pp. 315-330.

Effect of extrusion variables such as prior texture, billet and liner temperatures, reduction in area and ram speed on the texture of extruded uranium rods and tubes is examined by crystallographic (inverse) pole figures. 2 ref.

## 28. REKRISTALLISATION KALTGEWALZTEN URANS.

Sergeev, G.J. et al.

Kernenergie, Vol. 4, pp. 239-243, (1961)  
(Atomn. Energ. 9, 104-9(1960) Aug.)

Es werden Untersuchungsergebnisse ueber den Einfluss des Walzens und Gluehens im  $\alpha$ -Gebiet auf Struktur und mechanische Eigenschaften des Urans betrachtet. Als Ausgangsmaterial diente gegossenes, im  $\gamma$ -Gebiet gewalztes und aus dem  $\beta$ -Gebiet abgeschrecktes Uran. Feinkörniges rekristallisiertes Uran besitzt eine bedeutend höhere Festigkeit als das urspruengliche grobkörnige. Es werden die angenäherten Rekristallisationsdiagramme fuer Verformungsgrade von 5-40% und Gluehen bei Temperaturen von 350-650°C mit 10-stuendigem Anlassen aufgestellt. Die Rekristallisation ist praktisch nicht von dem untersuchten Ausgangszustand des Materials abhängig. Es werden Daten ueber die Kinetik des Rekristallisationsprozesses angegeben. Ein Zusatz von 0,1 Gew.-% Molybdän verlangsamt den Rekristallisationsprozess beträchtlich und erhöht die Festigkeit des feinkörnigen Urans um 20%.

## 29. THE EFFECT OF THE $\alpha - \beta - \alpha$ PHASE TRANSFORMATION ON THE PREFERRED ORIENTATION OF A URANIUM.

Butscher and Baverstock



AERE-R-3217, J. Nuclear Materials, 3: 30-40, Jan. 1961.

Dilatometric, metallographic, and x-ray observations were made on specimens of rolled uranium rod subjected to varying amounts of transformation into the  $\beta$  phase in a temperature gradient. The results show that the phase change is accomplished by a transformation band of mixed amount of the loss of preferred orientation is a function of the degree of transformation. A mechanism to account for this is discussed.

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