LA 69

March 15, 1944

This document contains 18 pages

URANIUM ALLOY DEVELOPMENT

Part V

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ABSTRACT

The uranium alloys of intended composition two atomic per cent titanium, zirconium, chromium, columbium, nickel and platinum have not shown interesting properties. Of these six alloy types, only the columbium system appears to offer promise on the basis of earlier work.

The results of vacuum remelting one gram uranium reductions are given.

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URANIUM ALLOY DEVELOPMENT - PART V

HEAT TREATMENT OF URANIUM ALLOYS CONTAINING TITANIUM, ZIRCONIUM, CHROMIUM, COLUMBIUM, NICKEL AND PLATINUM

In LA report 68, the microstructure and hardness of uranium alloys of intended composition two atomic per cent of titanium, zirconium, chromium, columbium, nickel and platinum were described for the 900° C. quenched condition. The type of X-ray diffraction pattern was described either as the Alpha (room temperature type) uranium or "structure X," the latter structure being possibly due to the intermediate high temperature or beta form of uranium.

Effect of Heat Treatment at 600° C.

The next step consisted of taking other samples from the same cast bars, heating them to 900° C., holding for two hours, and then cooling them in an argon atmosphere at room temperature. This relatively slow cooling was used in an effort to prevent cracking of some of the alloys when quenched in water. The chromium, platinum and nickel alloys were susceptible to quench cracking, presumably because of volume changes on cooling. However, it was found that even gas cooling did not prevent cracking. The chromium alloy was much the worst, with the nickel and platinum alloys far less troublesome in this respect. After cooling from 900° C., the samples were held for 24 hours at 600° C. and water quenched

to be examined for hardness, microstructure and X-ray structure.

The purpose of the 600° C. heat treatment was to use as high a temperature as possible and yet remain in the alpha uranium region. Some of the alloys which had previously shown the "structure X" might now be expected to exhibit the alpha uranium structure. If so, there could hardly be any doubt that the "structure X" was due to a high temperature form of uranium. The hardness and X-ray diffraction results are shown in Table I, and the microstructures are shown in Figs. 1-6. Figs. 1, 2 and 4 show the familiar alpha uranium structure with particles of carbide and possibly bits of a second phase. The columbium alloy shows a rather copious scattering of a second phase. Fig. 3, the chromium alloy, shows two cracks and a two-phase matrix. The nickel alloy, Fig. 5, has a fine grained matrix with a few rather large carbide dendrites, while Fig. 6, the platinum alloy is chiefly distinguished by a second phase in the grain boundaries. There is also a suggestion of a fine precipitate within the grains; the black particles are probably carbides.

Hardness

Eberbach micro hardness was determined as well as Rockwell A hardness because some of the specimens had numerous cracks, and it was considered that the Rockwell A values would be too low in such samples.

Fig 1. 0.40-0.23 Ti-U Alloy
24 hours at 600° C.
Etched electrolytically in 10%
Oxalic Acid

2277-2-0 x 250

Fig. 2. 2.7-0.1 Zr-U Alloy
25 hours at 600° C.
Etched electrolytically in 10%
Oxalic Acid

2281-2-0 x 250

Fig. 3. 0.45 Cr-U Alloy
24 hours at 600° C.
Etched electrolytically in 10%
Oxalic Acid

2282-2-1 x 250

Fig. 4. 1.39-0.25 Cb-U Alloy
24 hours at 600° C.
Etched electrolytically in 10%
Oxalic Acid

2283-2-0 x 250

[redacted]

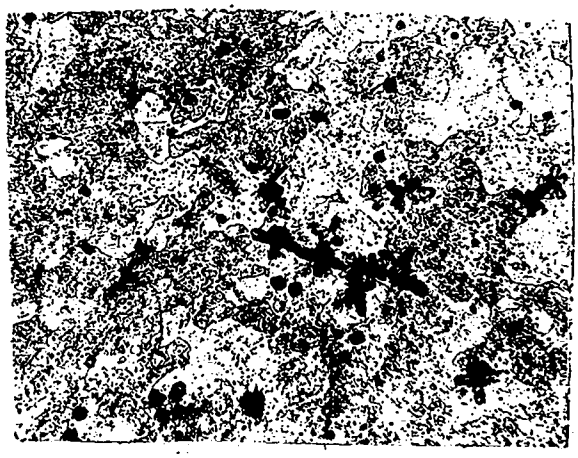

Fig. 5. 0.42 Ni-U Alloy
24 hours at 600° C.
Etched electrolytically in 10%
Oxalic Acid

2292-2-1 x 250

Fig. 6. Intended 1.66 Pt-U Alloy
24 hours at 600° C.
Etched electrolytically in 10%
Oxalic Acid

2293-2-1 x 250

Table I. Hardness and X-ray Diffraction Structure of the 600° C. Heat Treated Uranium Alloys

Sample No.	Composition analysis	Rockwell A Hardness Measured	Eberbach Micro Hardness D.P.N.*	Rockwell A Hardness as converted from Micro Hardness	X-ray Structure
2277-2	0.40-0.23 Ti	54	188	49	------
2281-2	2.7-0.1 Zr	49	222	56	------
2282-2	0.45 Cr	59	308	64	alpha uranium
2283-2	1.39-0.25 Cb	58	202	54	------
2292-2	0.42-0.48 Ni	58	256	59	------
2293-2	intended 1.66 Pt	54	358	67	alpha uranium

* Impressions made on chief constituent or matrix.

It will be seen from the table that in general the Rockwell A hardness values converted from the Eberbach micro hardness values (equivalent to Vickers diamond pyramid numbers) are higher than those observed directly.

X-ray Structure

X-ray diffraction results on the two samples (chronium and platinum) in this series which previously showed "structure X" as quenched from 900° C., showed the alpha uranium structure as heat treated at 600° C. This means that "structure X" is almost certainly due to beta uranium. The brittleness of the alloys with "structure X" is the main reason for eliminating the possibility of this structure belonging to the gamma modification.

Effect of Successive Heat Treatment from 300° C. to 600° C.

New samples of the same alloy bars were given the usual 900° C. heat treatment (two hours at temperature, followed by quenching in water) and were then heat treated successively in the vacuum quenching furnace for two hours at 300° C., 400° C., 500° C., and 600° C. Rockwell A hardness results are shown in Table II.

It will be noted from the table that none of the alloys show more than seven points increase in hardness on reheating from the 900° C. quenched condition, and most show changes of the order of 2-3 points. There is some question about the seven point increase of the nickel alloy, 2292-3, because previously a different sample from the same cast bar showed a hardness of 67 as cast instead of 60 as indicated here. More significant,

perhaps, is the observation that there is no hardness increase between 300° C. and 400° C., and at 600° C. the hardness is at the same level as in the lower temperature range.

While because of segregation there is a possibility of having very low alloy contents in the case of the alloys of titanium, zirconium, and columbium, it will be recalled (Cf. IA 42) that these three alloys were previously made with higher alloy contents without showing very interesting properties except in the case of columbium.

To sum up, it appears that none of the alloy types shown in Table II are worth further investigation except possibly uranium-columbium alloys.

Table II. Rockwell A Hardness Values on Reheating 900° C. Quenched Samples 300° C. to 600° C.

Sample No.	Composition (analysis)	Rockwell A Hardness				
		900° C. Quenched	300° C.	400° C.	500° C.	600° C.
2277-3	0.40-0.23 Ti	59	58	57	57	56
2281-3	2.7-0.1 Zr	57	57	58	57	55
2282-3	0.45 Cr	66	66	68	64	57
2283-3	1.39-0.25 Cb	58	60	59	63	59
2292-3	0.42-0.48 Ni	60	63	62	67	63
2293-3	Intended 1.66 Pt	65	66	64	62	56

VACUUM MELTING ONE GRAM URANIUM REDUCTIONS

In order to find out how one gram uranium reductions would behave in vacuum melting several one gram uranium reductions made by the stationary bomb method and the centrifugal bomb method were vacuum melted.

These one gram reductions behaved quite differently from one gram melts of Westinghouse uranium wire because of the impurities present, particularly iron and calcium. The latter boils out during melting and tends to cause splattering of the molten metal on the sides of the furnace walls and usually a black deposit, presumably largely calcium, is left on the inside of the furnace. The iron, when present in amounts of the order of 0.2 per cent or more, causes extreme brittleness.

The stationary bomb reductions were in general more easily handled than the centrifuged reductions because less volatile matter was present and also because of the shape of the reduced buttons.

Remelting the one Gram Centrifugal Reductions

The centrifuged buttons were cone shaped and were too large in area to be readily melted down in the BeO crucibles inside the tantalum heater crucible. It was found that by first centrifuging the cones into a BeO crucible to obtain a more convenient shape, some of the volatile matter was removed during the process. Hence in remelting the centrifuged cones, less difficulty in spattering was observed. The as-reduced cones

were placed on top of a BeO crucible recessed to fit the cone and sealed off under vacuum (2-3 microns) in a pyrex tube. The pyrex tube containing the BeO crucible and cone was placed in a high frequency induction furnace coil, and the cone heated to 1200-1400° C. When maximum temperature was attained, the tube was quickly transferred to a centrifuge and most of the metal forced down into the cylindrical part of the crucible, about 0.15 inches in diameter. The centrifuged metal was then removed, weighed, and inserted into a BeO sleeve which was seated inside a tantalum crucible and the metal again melted in a two micron vacuum. This second melting always caused more volatile impurities to be removed.

After the second melting, the ingots were weighed again, and used for microscopic examination and for analysis.

Remelting the one Gram Stationary Bomb Reductions

The stationary bomb reductions could be vacuum melted in the furnace directly without prior centrifuging because much less volatile impurities were present and because the reduced button could be fitted into the BeO sleeve. The procedure from this point was the same as for the vacuum centrifuged cones.

Ductility of the One Gram Reductions

The one gram stationary bomb buttons were always quite malleable, but the centrifuged uranium cones made by calcium reduction were usually brittle, probably because of greater iron content. However, the lithium

reduced centrifuged cones were comparable in ductility to the stationary bomb calcium reduced metal.

Loss in Weight During Remelting

Several stationary bomb and centrifugal bomb reduced melts of uranium have been vacuum melted according to the procedure outlined above. The changes in weight accompanying centrifuged remelting and vacuum furnace remelting are shown in Table III. Most of the loss of the centrifuged cones occurred during the centrifugal remelting operation, because not all of the uranium could be forced down inside of the centrifuging crucible. A crust of metal and oxide, etc., the bottom of the cone, was usually left behind.

Microstructures

Many of the one gram uranium reductions were examined metallographically as reduced, as centrifuged, and mostly after vacuum remelting. The various structures observed are shown in Figs. 7-21. The iron content and the amount of reductant (Ca or Li) in the samples is given where this information is available.

Table III. Change in Weight of One Gram Uranium Reductions during Vacuum Remelting

Centrifugal Bomb Reduction Vacuum Remelts

Specimen No.	Original Weight	Weight after Centrifuging grams	Final Weight grams	Per Cent Recovery
1827 A	1.0196	0.7380	0.7372	72
2374	1.0076	0.6531	0.6523	65
2375	1.0185	0.9163	0.8681	85
2373	0.9893	0.8483	0.8477	86
2376	0.8315	Very little centrifuged into crucible		
2377	0.9250	0.5990	0.5922	64
2378	0.9478	0.8661	0.8656	92
2385	0.9258	0.7333	0.7331	79

Stationary Bomb Reduction Vacuum Remelts

1234	0.9505	------	0.9325	98
2362	0.9524	------	0.9306	99
2379	0.9617	------	0.9461	98
2381	0.9481	------	0.6435	68
2382	0.9631	------	0.9534	99
2386	0.9474	------	0.9415	99

[redacted]

Future Work

More uranium alloys will be prepared in 200 g amounts for studying the effects of the alloying metals formerly studied only in one gram samples.

Fig. 7. Vacuum Remelted Centrifuged Bomb Reduction. 0.08% Fe.
Etched electrolytically in 2% Oxalic Acid

2360-1-1 x 250

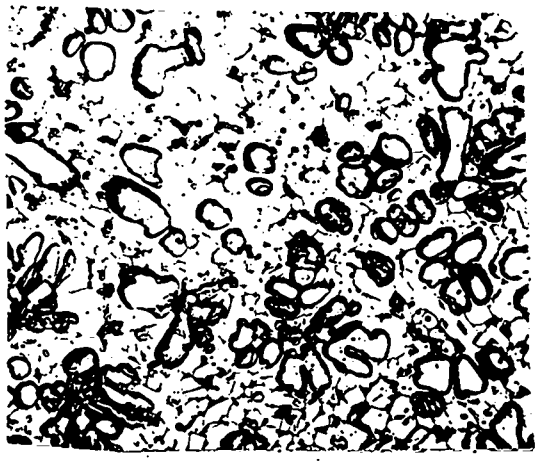

Fig. 8. Vacuum Remelted Centrifuged Bomb Reduction. 0.1% Fe.
Etched in 1:1 nitric-acetic acids

2363-1-0 x 250

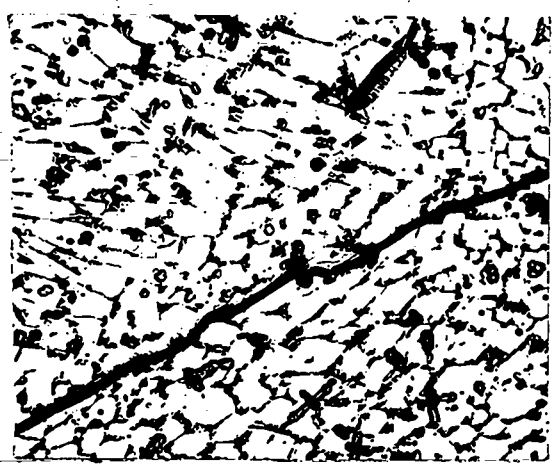

Fig. 9. Vacuum Remelted Centrifuged Bomb Reduction.
Unetched

2374-1-0 x 250

Fig. 10. Vacuum Remelted Centrifuged Bomb Reduction. >0.1% Fe.
Unetched

2375-1-0 x 250

[redacted]

Fig. 11. Vacuum Remelted Centrifuged Bomb Reduction.
Etched Electrolytically in 2% Oxalic Acid

2377-1-0 x 250

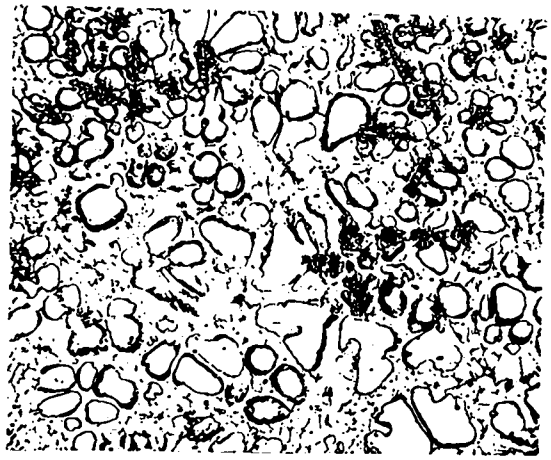

Fig. 12. Vacuum Remelted Centrifuged Bomb Reduction.
Unetched

2378-1-0 x 250

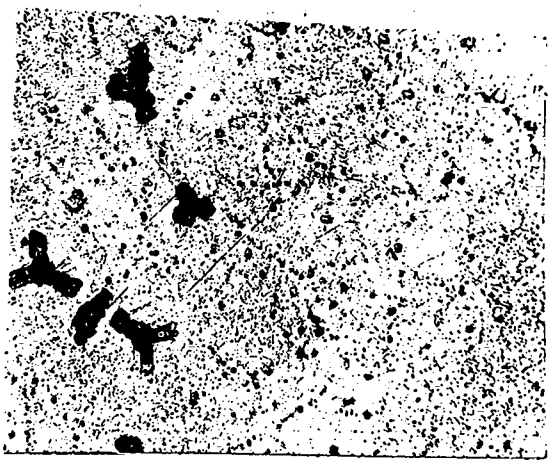

Fig. 13. Vacuum Remelted Stationary Bomb Reduction. 0.002% Fe, 0.0035% Ca.
Etched Electrolytically in 2% Oxalic Acid

2381-1-1 x 250

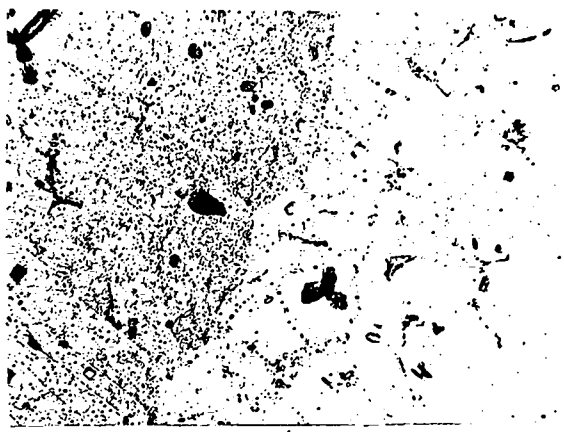

Fig. 14. Vacuum Remelted Stationary Bomb Reduction.
Etched Electrolytically in 2% Oxalic Acid

2382-1-1 x 250

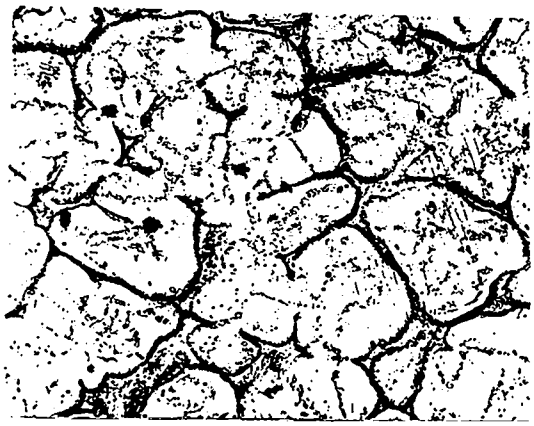

Fig. 15. Vacuum Remelted Centrifuged Bomb Reduction. ThO_2 Crucible. 0.1% Fe, 0.004% Ca.
Etched electrolytically in 2% Oxalic Acid

2385-1-1 x 250

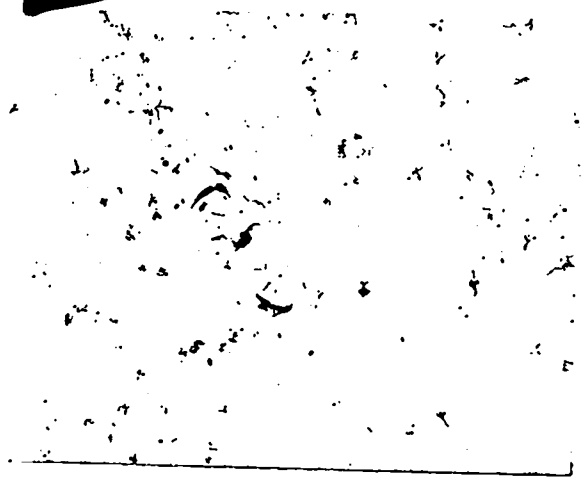

Fig. 16. Vacuum Centrifuged Centrifuged Bomb Reduction. Li Reduction. 0.04% Fe, 0.01% Li
Etched electrolytically in 2% Oxalic Acid

2388-1-1 x 250

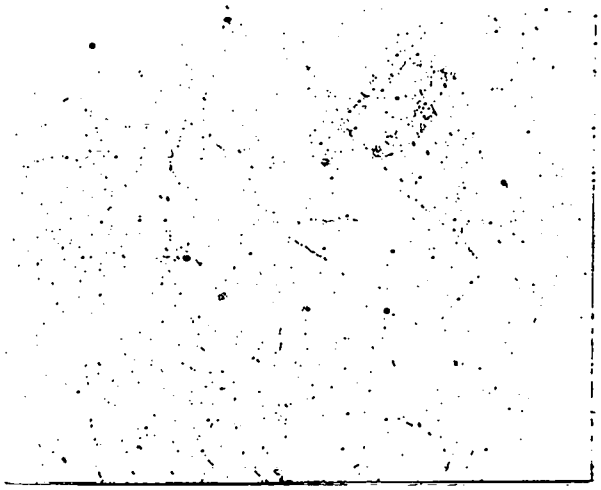

Fig. 17. Centrifuged Bomb Reduction Not Vacuum Melted. Li Reduction. 0.04% Fe, 0.001% Li.
Etched electrolytically in 2% Oxalic Acid

2389-1-1 x 250

Fig. 18. Vacuum Remelted Centrifuged Bomb Reduction. Li Reduction.
Etched Electrolytically in 10% Chromic Acid

2390-1-2 x 250

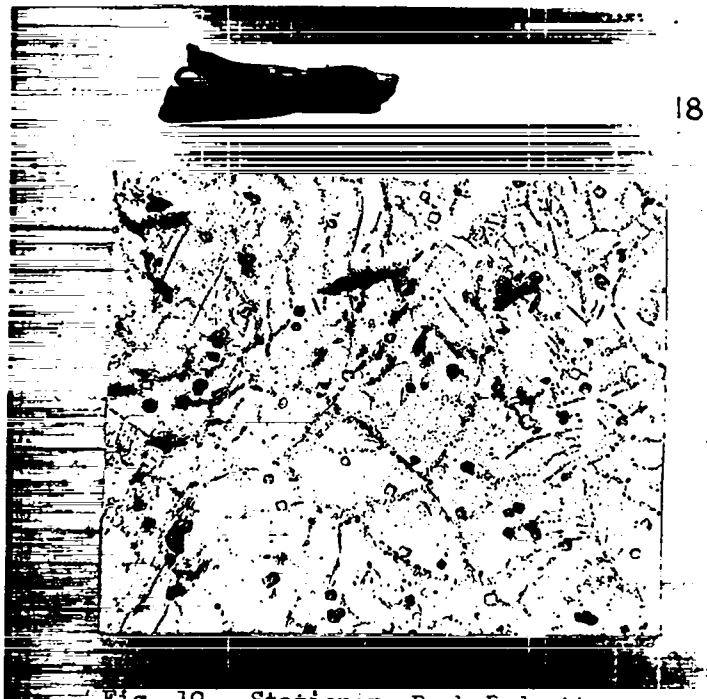

Fig. 19. Stationary Bomb Reduction
Not Remelted.
Etched Electrolytically in 2% Oxalic Acid

2393-1-1 x 250

Fig. 20. Stationary Bomb Reduction
Not Remelted.
Etched Electrolytically in 2% Oxalic Acid.

2394-1-1 x 250

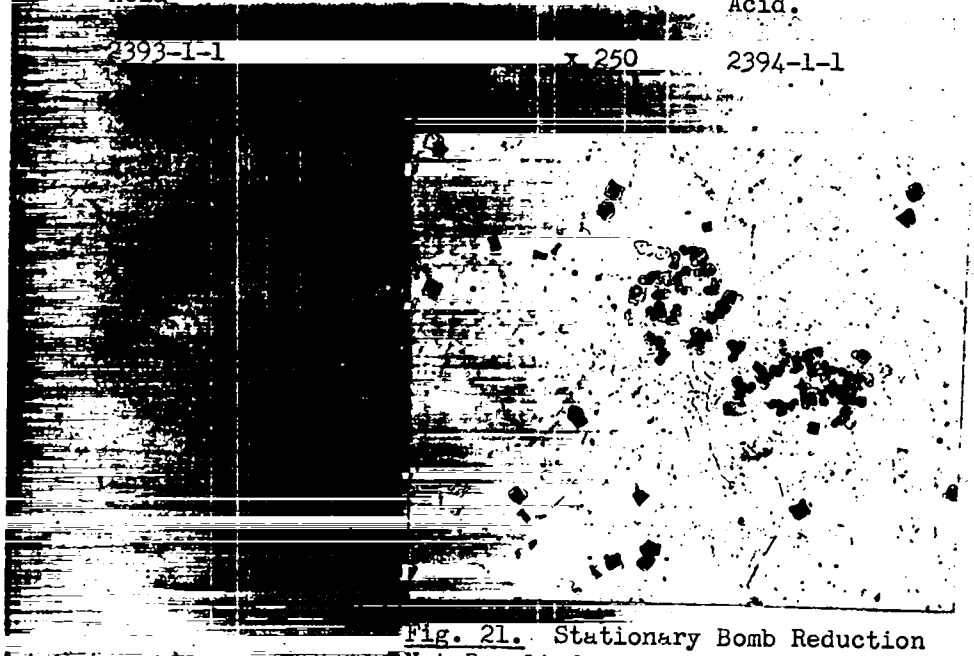

Fig. 21. Stationary Bomb Reduction
Not Remelted.
Etched Electrolytically in 2% Oxalic Acid

2395-1-1 x 250

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