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RECRYSTALLIZATION OF THORIUM

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CLASSIFICATION CHANGED TO: DECLASSIFIED
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Contract No. W-7405-eng-26

METALLURGY DIVISION

RECRYSTALLIZATION OF THORIUM

F. H. Eckert and E. J. Boyle

Work carried out by: F. H. Eckert, D. S. Eaton
Metallography by: R. J. Gray
Fabrication by: J. H. Erwin

DATE ISSUED

FEB 18 1953

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RECRYSTALLIZATION OF THORIUM

F. H. Eckert E. J. Boyle

ABSTRACT

The recrystallization characteristics of Ames thorium and iodide thorium were determined. The effects of annealing temperature, annealing time, and cold work were followed by metallographic and hardness determinations. The results show that Ames thorium and iodide thorium recrystallize in the temperature range from 500 to 700°C. Iodide thorium recrystallizes at a slightly lower temperature than the Ames material.

Increasing the carbon content to 0.13% has no significant effect on the recrystallization temperature of iodide thorium. The higher carbon-content material shows some increase in hardness after completion of recrystallization. This behavior is believed to be a result of precipitation of carbon at the higher annealing temperatures.

INTRODUCTION

This investigation was undertaken to study the recrystallization characteristics of pure thorium that had been plastically deformed specified amounts and then isothermally annealed, with time, temperature, per cent reduction in area, and carbon content as variables. The effect of the carbon content was studied because carbon is the principal contaminant in thorium.

A previous study of the recrystallization of commercially pure Ames thorium was reported by Jackson and Beaver[1] at Battelle Memorial Institute. The present study include both high-purity iodide thorium and high-purity Ames thorium.

CONCLUSIONS

On the basis of the results of the present experimental work, the following conclusions appear to be justified:

1. Pure thorium, prepared from decomposition of the iodide, recrystallizes in the temperature range from 500 to 700°C after 20 to 80% reduction by cold rolling.
2. Ames thorium that has been cold rolled 20 to 80% recrystallizes in the temperature range from 520 to 700°C.
3. Carbon, which is the principal contaminant in Ames thorium, does not appear to have a significant effect on the recrystallization temperature range of the thorium. However, the hardness levels, following recrystallization, of alloys containing carbon in excess of the normal range for Ames thorium are considerably higher than those of the standard material.

EXPERIMENTAL PROCEDURE

Material. The Ames thorium used in this investigation was produced by calcium reduction of thorium fluoride at the Iowa State College at Ames, Iowa and was received as a 3-in.-dia cast billet (A-349B). The higher purity iodide thorium was produced at Battelle Memorial Institute by decomposition of thorium iodide on a hot filament. The analyses of this material and a thorium-carbon alloy series are shown in Table 1. The high oxygen and nitrogen contents in the thorium-carbon alloys with increasing carbon content are believed to be due to contamination by the master alloy used in preparing these alloys. Iodide thorium was used in their preparation, and all fabrication procedures were identical except for greater additions of the master alloy to the higher carbon alloys.

Fabrication. The thorium specimens were prepared in the following manner. The 3-in.-dia Ames thorium billet was extruded to 0.625-in.-dia rod, and four equal portions of this rod were cold rolled with various specified reductions. The rolled thorium plate was heated to 750°C for 30 min in a vacuum and furnace cooled to precipitate, initially, any impurities that might confuse the recrystallization studies by spurious hardening effects from precipitation. The annealed thorium plates were cold rolled to 20, 40, 60, and 80% reduction in area and sectioned into 0.071- by 0.75- by 0.50-in. specimens.

[1] L. R. Jackson and W. W. Beaver, *Journal of Metallurgy and Ceramics*, p. 74-88, TID-68 (Sept. 1949).

The iodide crystal-bar thorium was melted in an inert-atmosphere arc furnace by using a non-consumable tungsten electrode. The thorium melts were fabricated into 0.061- by 0.5- by 0.4-in. specimens with 20, 40, 60, and 80% cold work by the same procedure as outlined above for the Ames metal.

Thorium-carbon alloys were prepared by master alloy additions to the crystal-bar thorium in the inert-atmosphere arc furnace and were then cold reduced approximately 35%, homogenized 12 hr at 1275°C, furnace cooled to room temperature, and cold rolled to 70% reduction in area. Specimens, 0.07 by 0.6 by 0.5 in., were cut from the finished material.

Heat Treatment. A molten lead bath was found to be most suitable for heating the specimens since this provided a rapid way to attain bath temperatures and prevented oxidation of the thorium during the annealing experiments without the use of an inert atmosphere or a jacketing material. The lead was held in a resistance pot furnace with the temperature controlled to ±2°C by means of a chromel-alumel thermocouple.

Individual specimens of iodide and Ames thorium were isothermally annealed for varying times at temperatures ranging from 450 to 740°C for study of the effect of time on recrystallization. The specimens that had been cold worked 20, 40, and 60% and the thorium-carbon alloys were isothermally annealed for 1 hr at temperatures varying between 450 and 740°C for study of the effects of carbon and cold work on the recrystallization of thorium.

After heat treatment in the lead bath, all samples were quenched in water, and the progress of recrystallization was traced by Vickers hardness measurements and metallographic examination.

RESULTS AND DISCUSSION

The effect of annealing time on the recrystallization of Ames thorium and iodide thorium is shown in hardness vs. annealing time plots in Figs. 1 and 2. No apparent changes in microstructure were seen for the initial drop in hardness to 100 VHN in the Ames thorium, whereas the iodide specimens showed new strain-free grains forming at the first drop in hardness.

The effect of cold work on the recrystallization of Ames thorium and iodide thorium is shown in Figs. 3 and 4. Ames thorium exhibits a possible "recovery" zone, as shown by the break in the recrystallization curves, above which the microstructures revealed no evidence of recrystallization. This effect was not evident in the iodide thorium. The "recovery" zone in the Ames thorium may be due to the precipitation of another phase associated with the higher level of impurities in the Ames thorium. This hypothesis is supported by the fact that iodide thorium does not show this change under identical conditions. However, investigations have not been made to find out whether the iodide thorium shows this effect at lower temperatures.

Table 2 shows the annealing temperature for the start and completion of recrystallization with varying amounts of cold work. These temperatures were determined by metallographic examination. Examination of Table 2 and Figs. 3 and 4 shows that increasing cold work decreases the annealing temperature necessary for recrystallization to take place at any given time. Increased annealing time is necessary for recrystallization at lower temperatures.

It was generally observed that hardness after complete recrystallization was unaffected by previous cold work but was slightly higher, the lower the annealing temperature used. Iodide thorium recrystallizes at a slightly lower temperature than Ames thorium, all conditions being equal.

Figure 5 shows the effect of carbon content on the recrystallization of iodide thorium. Table 3 lists the temperatures at which recrystallization starts and finishes for different carbon alloys as determined by metallographic examination. It can be seen that the addition of carbon up to 0.13% apparently has no significant effect on the recrystallization temperature range of the iodide thorium. The increases in hardness in the temperature range from 675 to 740°C are believed to result from precipitation of carbides at these temperatures.

Photomicrographs showing the progress of recrystallization under various conditions are shown in Figs. 6 through 12. Figures 6 through 10 show the progress of recrystallization and grain growth in iodide thorium that was cold rolled 80% and annealed for various times at 600°C. Figures 11 and 12 show the structures obtained in Ames thorium treated according to some of the same conditions. It will be noted, in the case of the iodide thorium, that recrystallization started at the shortest annealing time. Ames thorium also shows the start of recrystallization at the shortest annealing time. However, the grain size of the Ames thorium after 1 hr at temperature is very much smaller than that of the iodide material, which indicates the grain growth restraining effect of the impurities in the former material.

TABLE 1. CARBON, VACUUM-FUSION, AND SPECTROGRAPHIC ANALYSES OF THORIUM

MATERIAL	COMPOSITION (ppm)								
	C	O_2	N_2	H_2	Al	Si	Fe	Be	Other Elements
Ames thorium billet* (A-349B)	370	120	150	<100	90	90	140	30	Faint traces
Iodide thorium	200	110	10	<100		Trace	Trace		Faint traces
Thorium-carbon alloy	1340	500	600	<100		Trace	Trace		Faint traces
	1010	400	70	<100		Trace	Trace		Faint traces
	850	200	40	<100		Trace	Trace		Faint traces
	490	<100	<10	<100		Trace	Trace		Faint traces

*Ames analysis, except carbon and oxygen.

TABLE 2. EFFECT OF COLD WORK ON RECRYSTALLIZATION OF THORIUM

REDUCTION IN AREA (%)	START OF RECRYSTALLIZATION (°C)		RECRYSTALLIZATION COMPLETE (°C)	
	Ames Thorium	Iodide Thorium	Ames Thorium	Iodide Thorium
80	520	500	625	575
60	520	510 to 520	625	600
40	540 to 550	520	650	600
20	550 to 575	550 to 575	650	700

TABLE 3. EFFECT OF CARBON ON RECRYSTALLIZATION OF IODIDE THORIUM*

CARBON CONTENT	START OF RECRYSTALLIZATION (°C)	FINISH OF RECRYSTALLIZATION (°C)
0.134	510 to 520	650
0.101	510 to 520	650
0.085	500 to 510	650
0.049	500 to 510	650

*70% reduction in area for all alloys.

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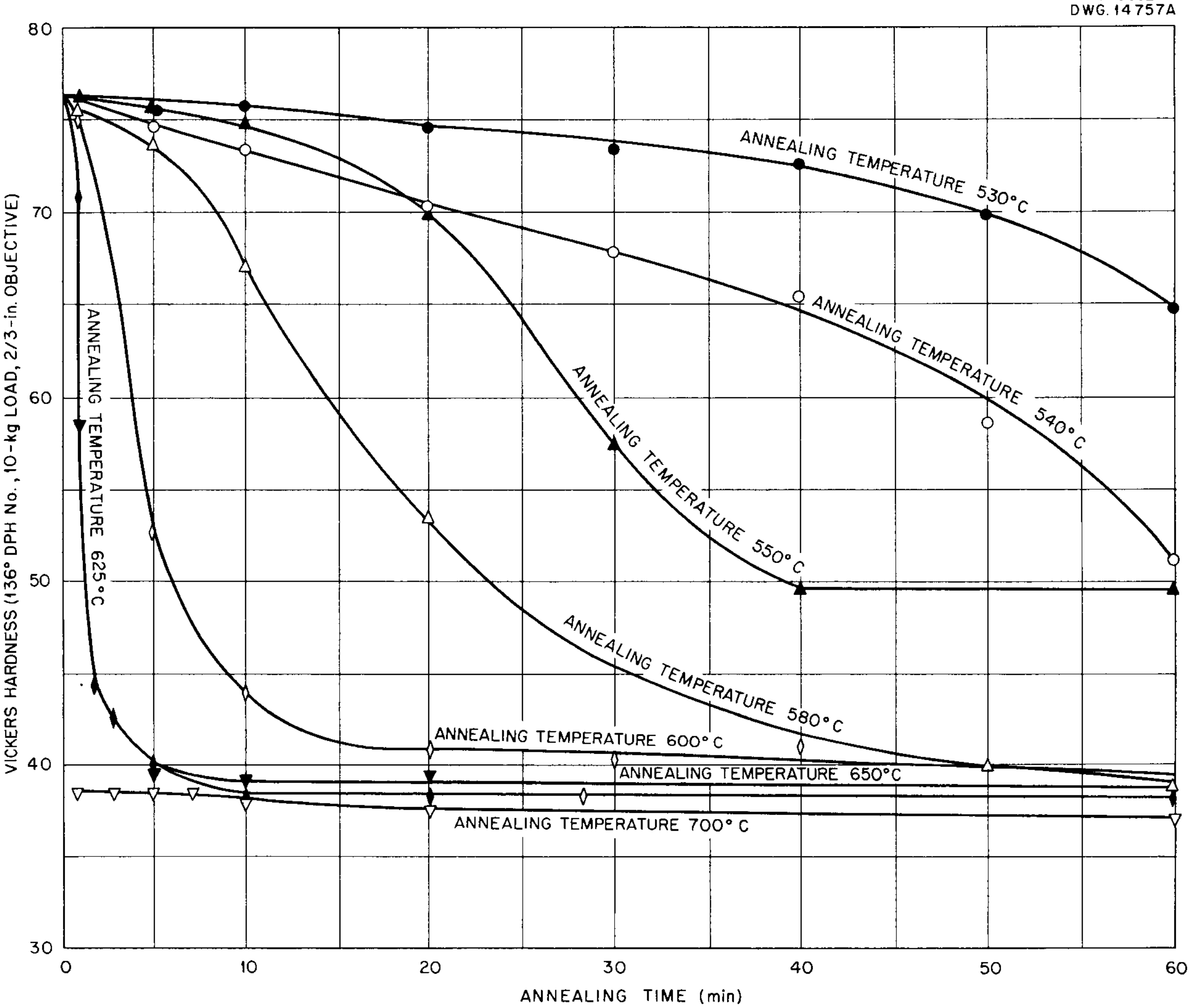

Fig. 1. Isothermal Recrystallization Curves for Iodide Thorium.

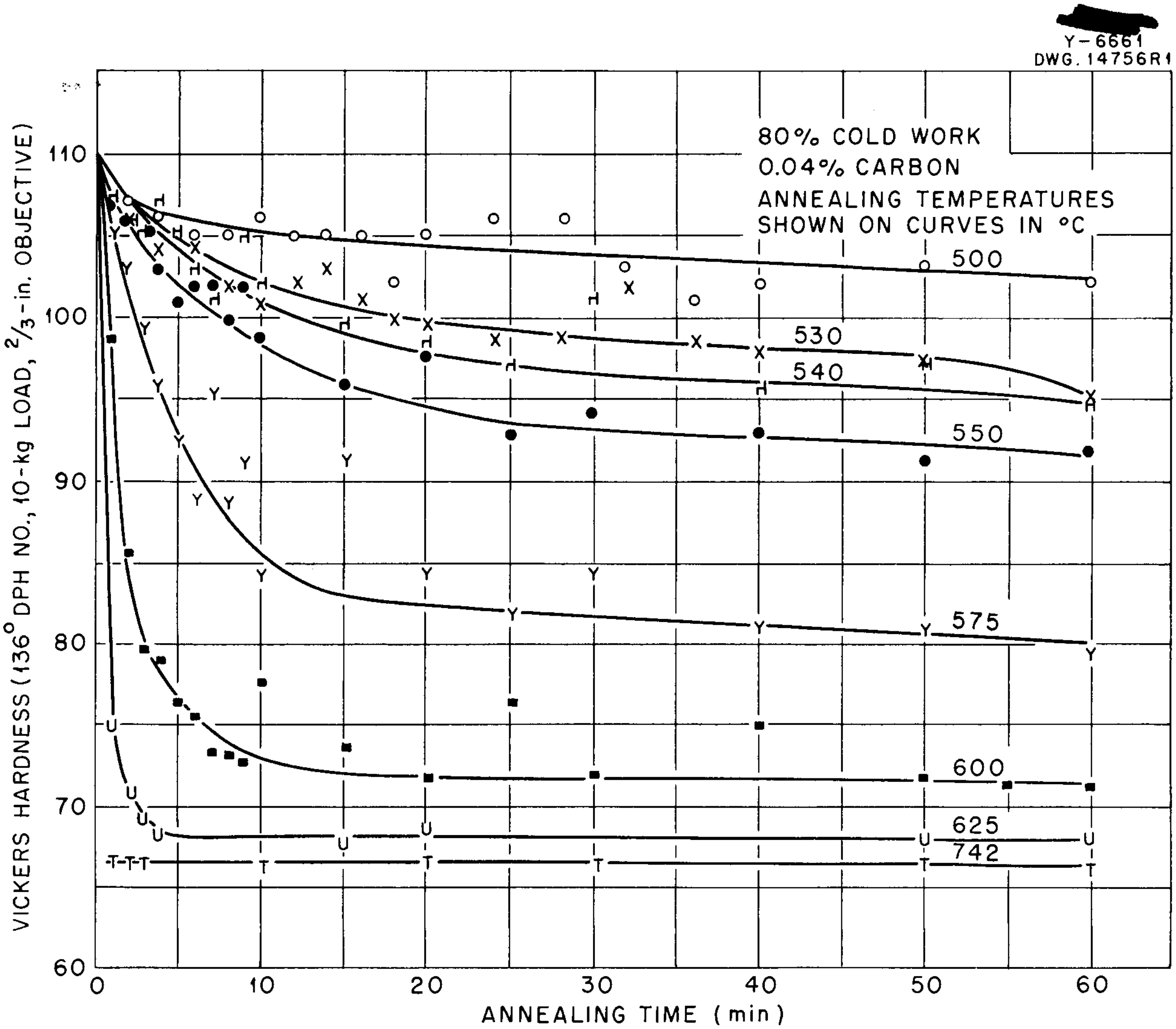

Fig. 2. Recrystallization Curves for Ames Thorium.

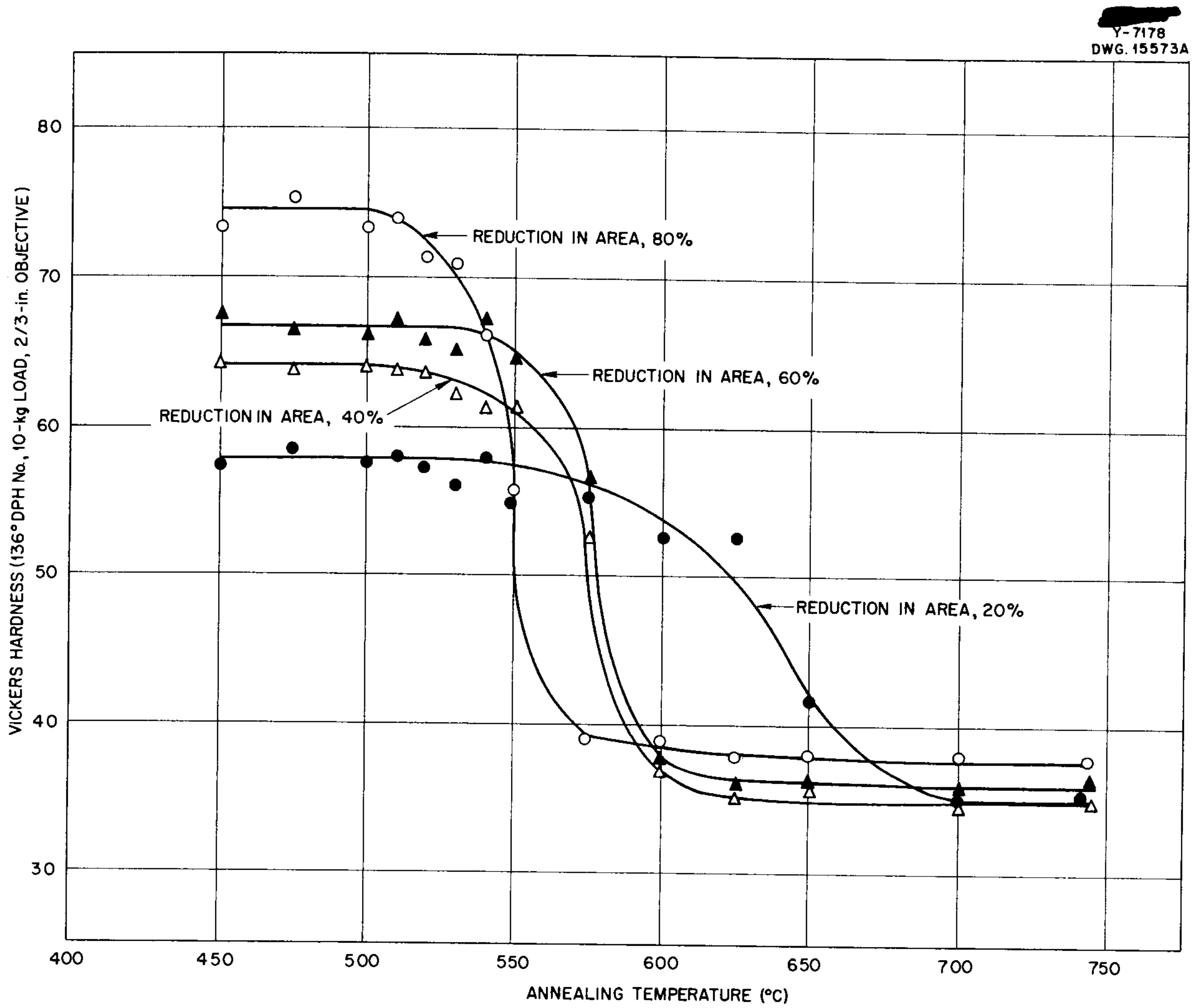

Fig. 3. Effect of Cold Work on Recrystallization of Iodide Thorium.

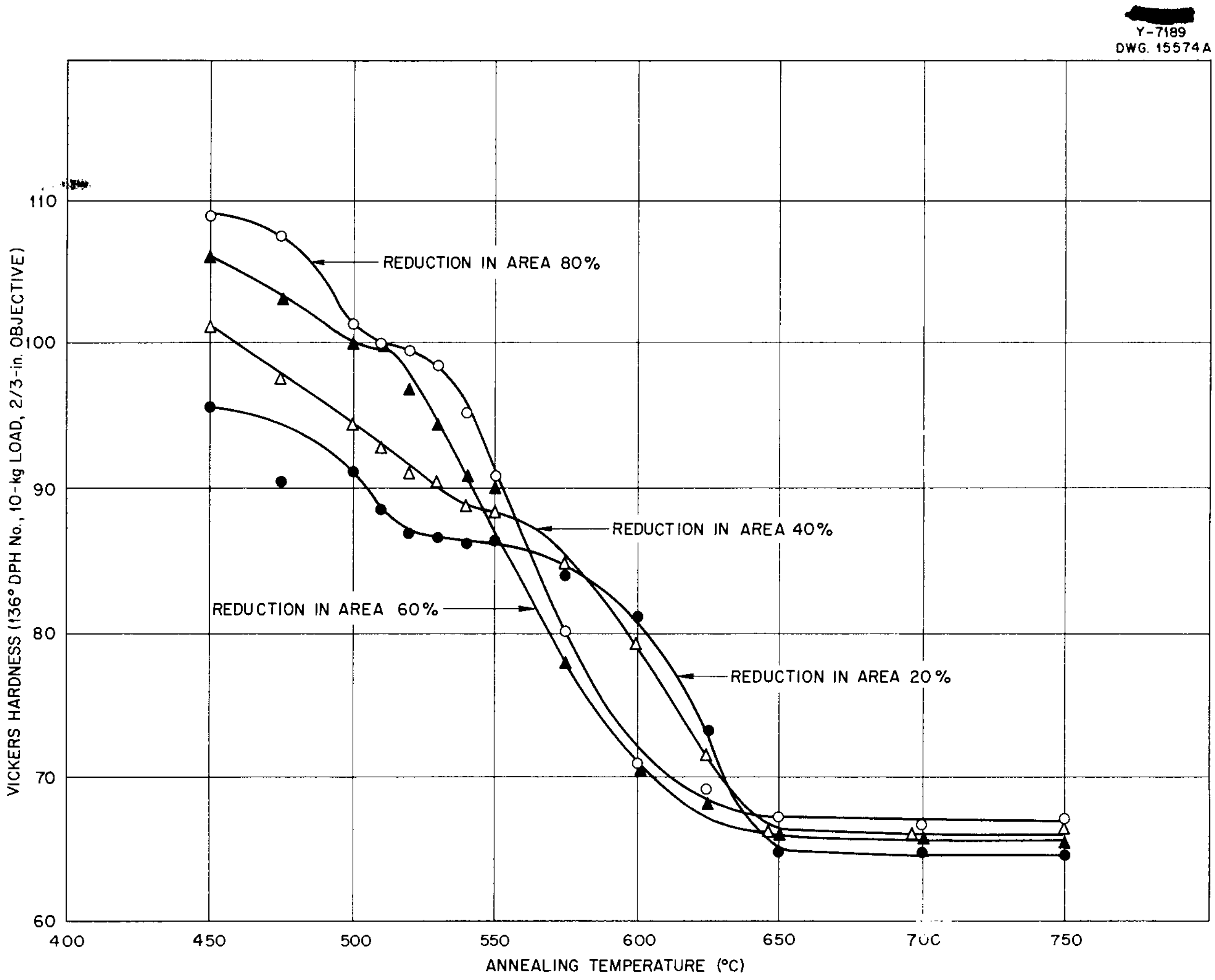

Fig. 4. Effect of Cold Work on Recrystallization of Ames Thorium.

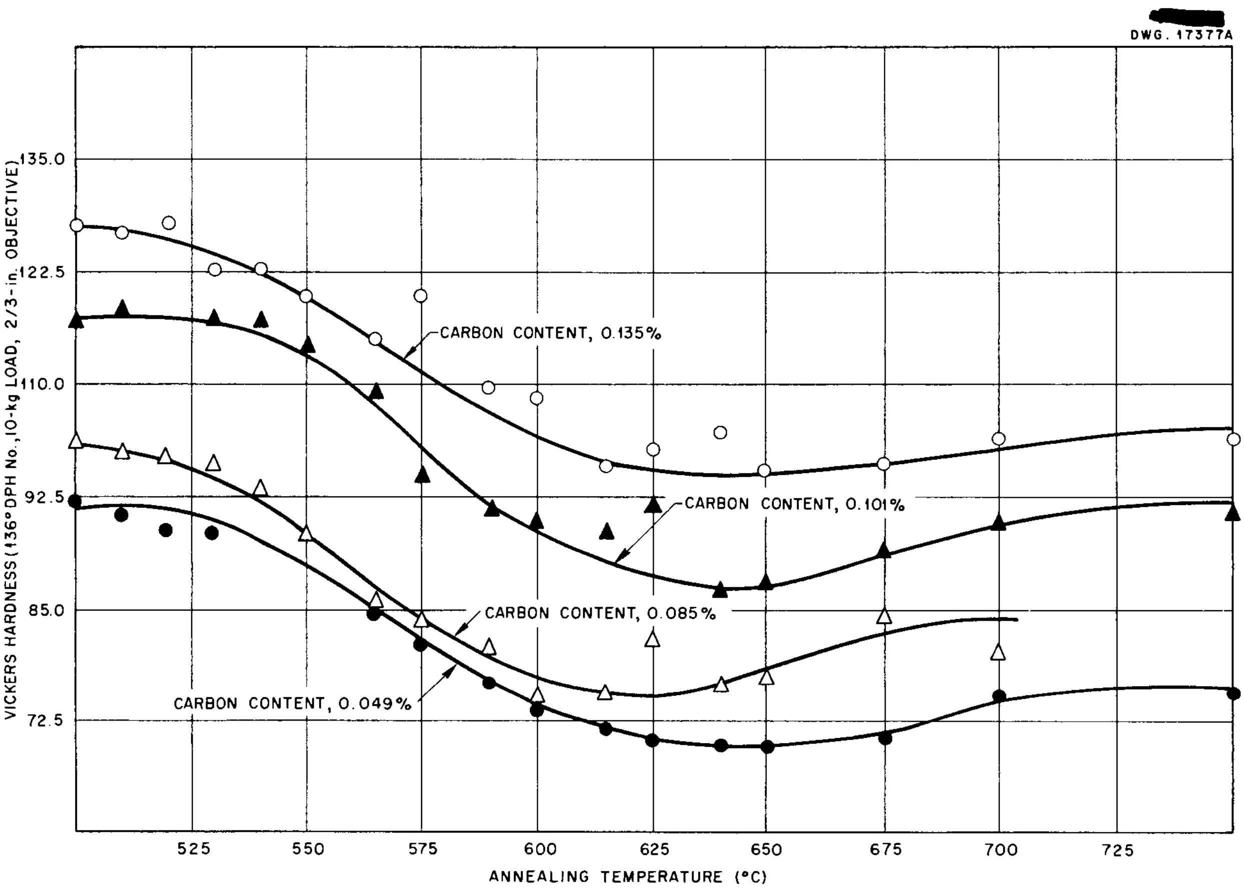

Fig. 5. Effect of Carbon Content on Recrystallization of Iodide Thorium.

Fig. 6. Iodide Thorium Cold Rolled 80%. 250X

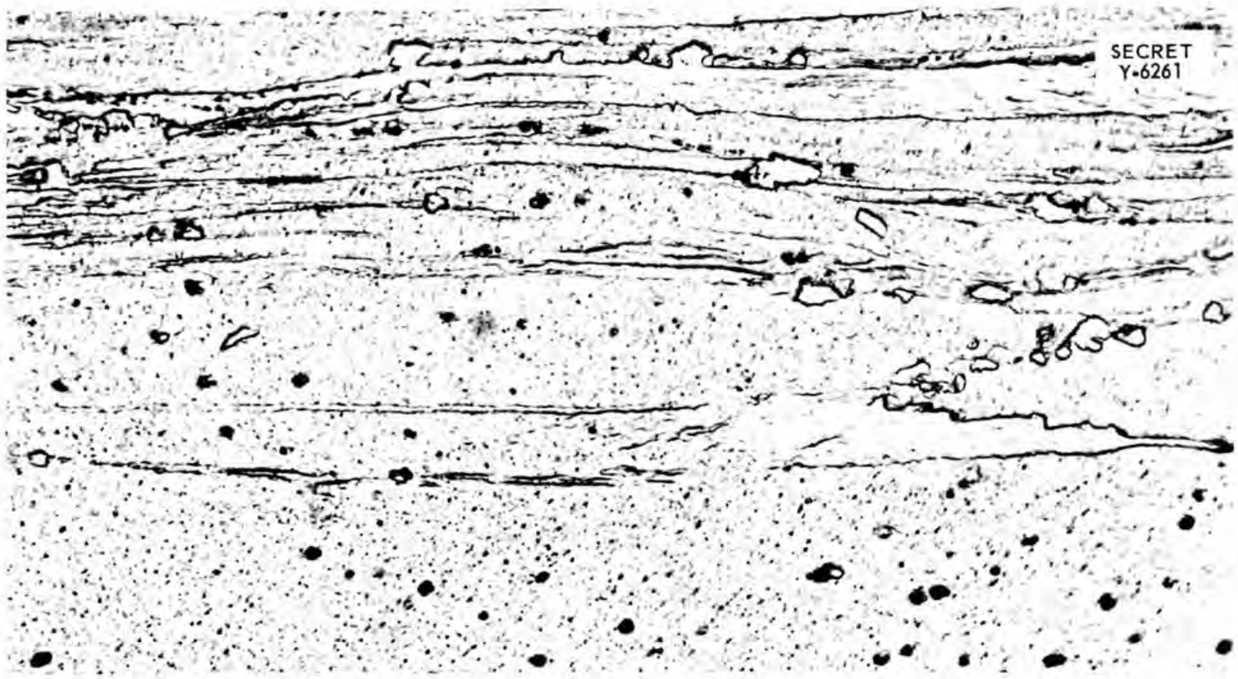

Fig. 7. Iodide Thorium Cold Rolled 80% and Annealed 1 min at 600°C. 250 X

Fig. 8. Iodide Thorium Cold Rolled 80% and Annealed 5 min at 600°C. 250X

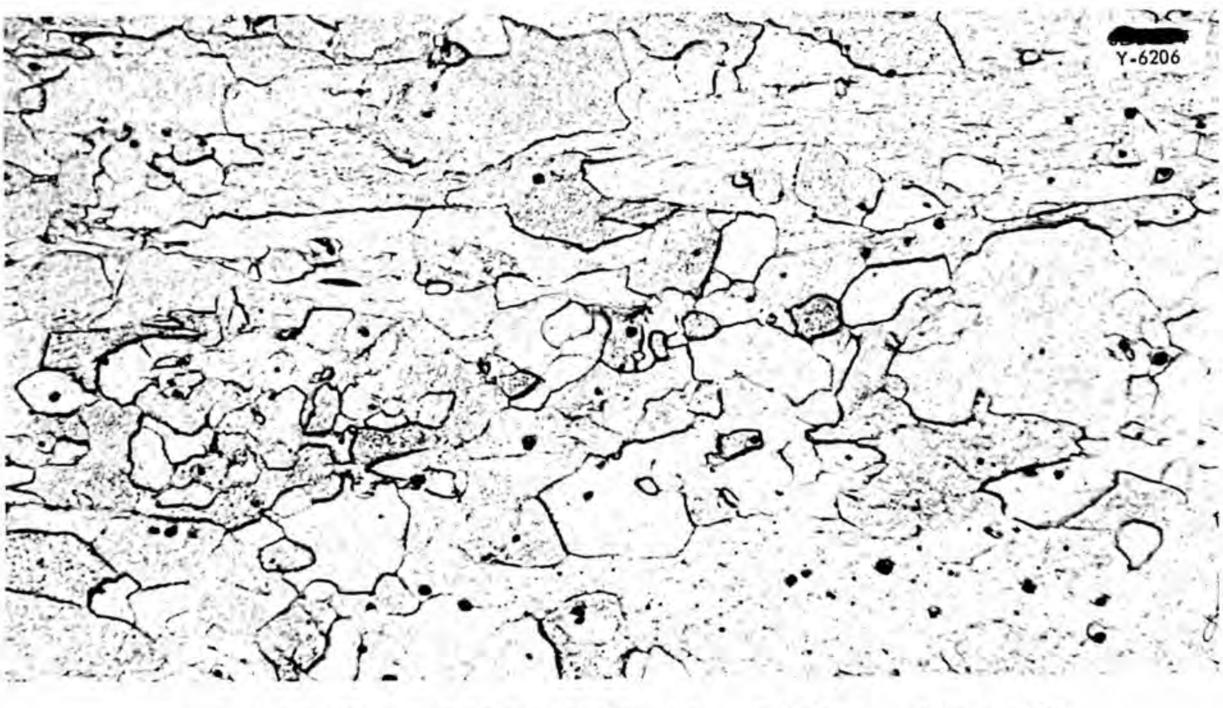

Fig. 9. Iodide Thorium Cold Rolled 80% and Annealed 10 min at 600°C. 250X

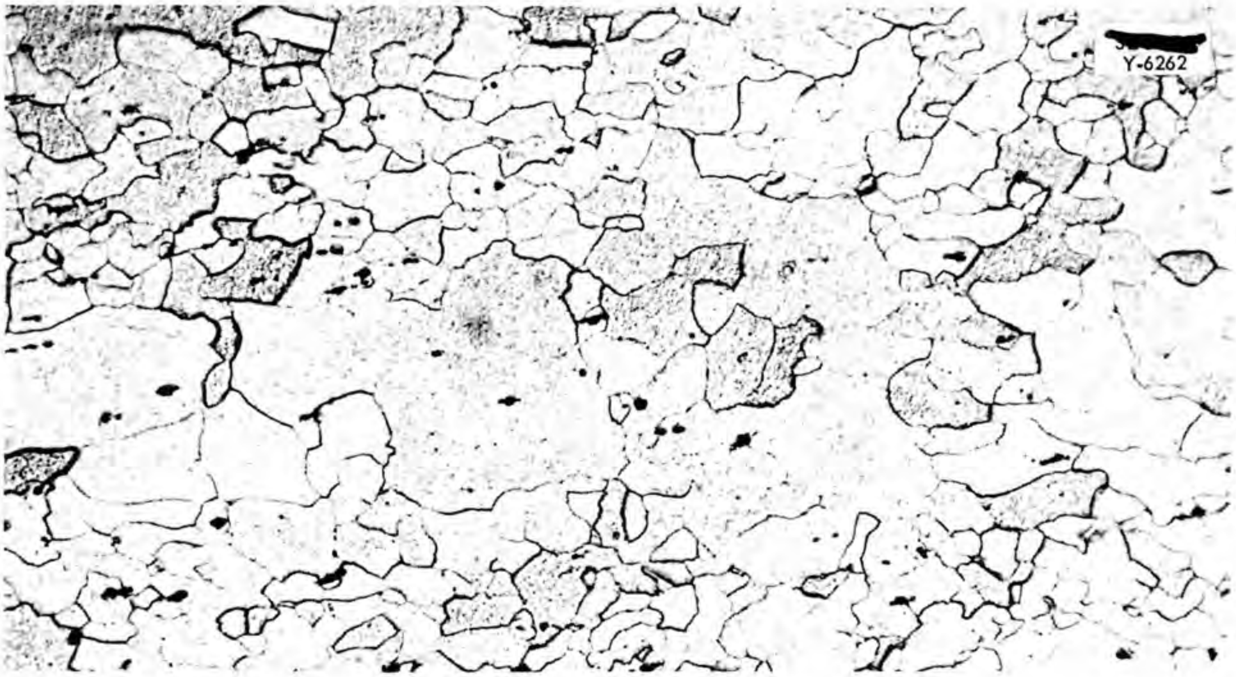

Fig. 10. Iodide Thorium Cold Rolled 80% and Annealed 60 min at 600°C. 250X

Fig. 11. Ames Thorium Cold Rolled 80%. 250X

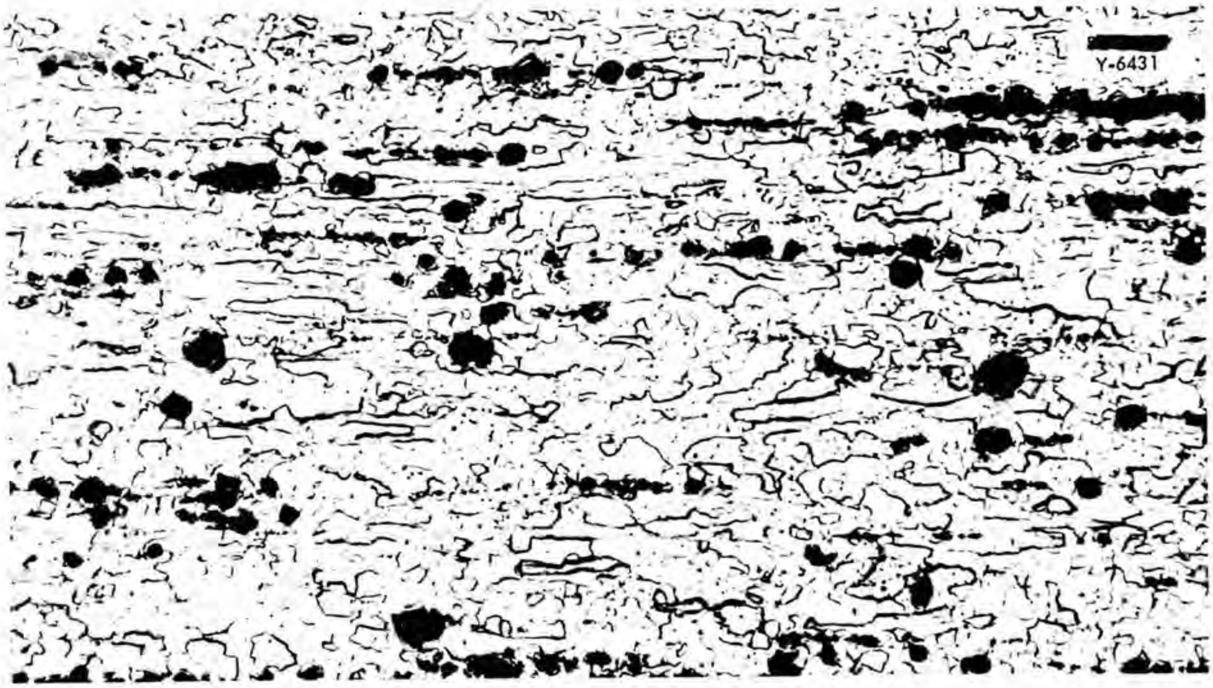

Fig. 12. Ames Thorium Cold Rolled 80% and Annealed 60 min at 600°C. 250X