ELDORADO MINING AND REFINING LIMITED

RESEARCH AND DEVELOPMENT DIVISION

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HEAT TREATMENT OF DILUTE ALLOY URANIUM RODS.

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ABSTRACT

Samples of cast uranium rods containing small additions of iron aluminum and carbon were beta heated, quenched and alpha annealed to determine the effect on grain size and structure. Minimum average grain size obtained was 60 micron. High alpha annealing failed to produce a recrystallized grain structure from the beta quenched samples.

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1. INTRODUCTION

Uranium metal fuel rods for nuclear reactors should be as dimensionally stable as possible and have a minimum of surface irregularities if they are to have a reasonable life under reactor irradiation conditions. To obtain these properties it is desirable to have a finished rod with a small equiaxed randomly-oriented grain structure, and limited grain growth during irradiation. This type of structure can be obtained by adding small quantities of impurities to the uranium metal and subjecting it to various heat treatments.

This investigation deals specifically with the addition of iron and aluminum to vacuum cast uranium metal, and the results of subsequent heat treatment on the grain structure of the dilute alloy rods.

2. EXPERIMENTAL:

Vacuum casting of the dilute alloys was carried out at National Lead, Albany, N.Y. Five heats of approximately 100 lbs. each were made and fifteen castings, $1\frac{1}{2}$" diameter by about 20" long, were produced -3 per heat. Each casting was sampled bottom and top and analyzed for iron, aluminum and carbon. Average analyses are shown in Table 1. Details of the casting and complete analyses are reported in E.M. & R., Research and Development, "Report on Dilute Alloy Production" by E.B.Spice, March 1961. There appeared to be no significant segregation from top to bottom of individual castings, and no significant difference between castings in a heat.

Fifteen samples of 2" minimum length, (one from each casting) were received for heat treatment. Photomicrographs of the as-cast structures are shown in Figure 1.

Heat treatment was carried out in a Houghton 980 salt bath, resistance heated and temperature controlled to $\pm$ 10°C. The thermocouple was located as close as possible to the sample. Beta heat temperature was 735°C. Delay times in air before water quenching were varied from about two seconds (immediate quench) to three minutes. Some samples were air cooled from the beta. Annealing of the beta treated samples was carried out at 640°C for up to 26 hours.

All samples from beta treatment were cut in half transversely. The cut face was polished and examined microscopically at 100x under polarized light. Photomicrographs showing the effect of delay quenching and double quenching on the beta structure are shown in Figures 2 and 3.

The samples from beta treatment were annealed and the original cut face again examined. Photomicrographs of the annealed specimens are shown in Figures 4 and 5.

To determine the effect of variations in iron, aluminum and carbon in the dilute alloy at least one sample from each heat was beta treated and alpha annealed. Comparison of the five heats is shown in Figures 6 and 7. Only one photomicrograph was used to represent heat #1702 and 1705 as their analyses were quite similar.

At least two samples from each heat were examined microscopically at 15x for porosity. Out of fourteen samples examined nine showed considerable porosity, while five showed very little porosity. The porosity was generally fairly equally distributed across a transverse section. Samples were not identified as to which end of the casting they were cut from. It was therefore impossible to determine if there was any difference in porosity between heats. Figure 8 shows a macro-photograph of a sample from heat #1705 which contains considerable porosity.

3. DISCUSSION:

(a). Effect of Beta Quenching

Comparing Figures, 1, 2, 3 and 6 there is no doubt that beta quenching produces considerable grain refinement, with the finest grain being produced at the shorter delay times. Double beta quenching does not appear to cause further refinement.

To determine the effect of iron and aluminum on the degree of grain refinement some comparative figures were obtained for beta quenched NRX size rod and for beta quenched 1.5" diameter cast billets from the type III rod program.[1] Grain sizes are shown in Table 2. Grain size of the immediately quenched dilute alloy samples (Figures 3 & 6) ranged from 60 to 130 microns with an average of 91 microns. Carbon levels in the dilute alloy were either lower than or in the same range as carbon levels in NRX rod. Thus it appears that the presence of iron and aluminum produces a greater degree of grain refinement than is normally obtained in beta quenched NRX size uranium rod. Data were insufficient to determine the effect of variations in iron, aluminum and carbon content on the degree of grain refinement.

(b). Effect of Alpha Annealing

From Figure 4 it is apparent that little or no recrystallization takes place upon annealing at 640°C for up to twenty-six hours. In one case areas of recrystallized grains were obtained at the surface of a rod

TABLE 1. ANALYSIS OF DILUTE ALLOY

Heat No.	PPM Fe	PPM Al	PPM C
1702	228	646	231
1705	223	686	229
1709	174	673	347
1712	281	655	297
1716	267	655	454

Analyses are averages of six samples per heat analyzed by three different laboratories.

TABLE 2. GRAIN SIZE OF BETA QUENCHED NRX SIZE URANIUM ROD

Description	Number Examined	Grain Size	
		Range	Ave.
As rolled 18M series NRX rod beta quenched at AMF	3	180 - 200	187
$1\frac{1}{2}$" long samples of NRX rod beta treated at Ottawa - immediate quench	4	100 - 180	129
$1\frac{1}{2}$" diameter cast billets beta quenched at AMF	7	112 - 205	149
$1\frac{1}{2}$" diameter cast billets double beta quenched at AMF	1	123 - 287*	233

* Billet was cut into six sections for examination giving the above grain size variation.

annealed for only twelve hours. This anomaly could not be explained since longer anneals failed to show any signs of recrystallization.

Grain size measurements on the annealed samples indicate definite grain growth upon annealing the delay quench and double quench samples. However four of the six samples immediately quenched from the beta showed no grain growth upon annealing. Data were insufficient to determine the effect of variations in the iron, aluminum and carbon content on annealing.

4. CONCLUSIONS

Iron and aluminum, in concentrations of approximately 250 and 650 ppm respectively can be successfully added to uranium metal to produce cast rods which when water quenched from the beta will display a fine grained beta structure. The degree of grain refinement is affected by the delay time in air before quenching. Annealing of the beta quenched samples at 640°C for up to 26 hours does not produce a recrystallized structure.

5. ACKNOWLEDGMENT

The author wishes to express his thanks to Mr. G. Sims for conducting the heat treatment program and assisting in the subsequent metallographic evaluation.

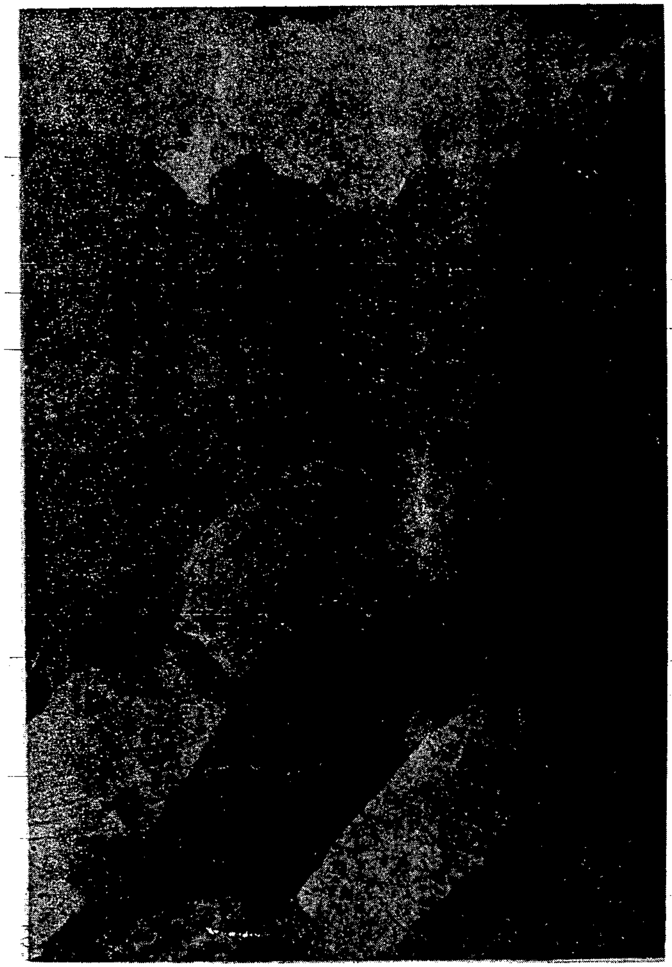
6. REFERENCES:

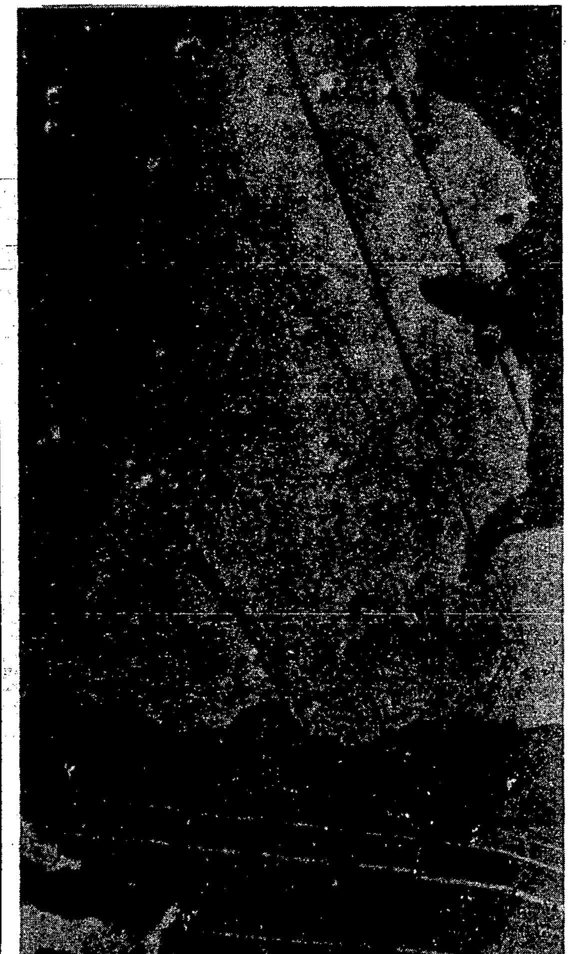
(1). Eldorado Mining and Refining, Research and Development Report T 60-25, "The Metallographic Examination of Specimens From the First Rolling of Type III Fuel Elements" - J.C. Anderson.

FIGURE I 100X

AS CAST DILUTE ALLOY

Heat No. 1702	Heat No. 1705	Heat No. 1709



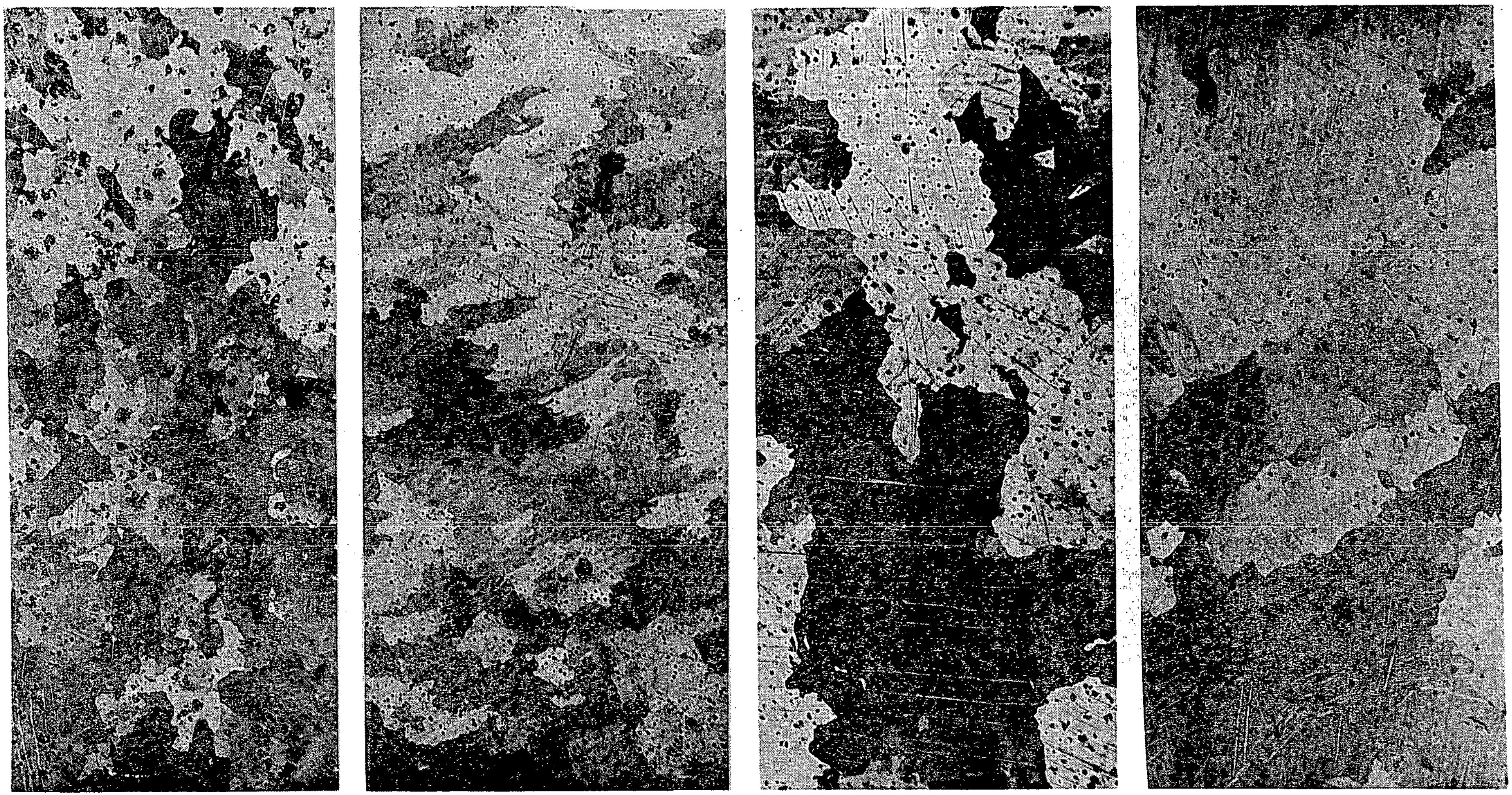


	A-60-435	A-61-17	A-61-10
Ave. Grain Size (μ)	300	300	500

FIGURE 2 100X

EFFECT OF DELAY QUENCHING ON STRUCTURE OF BETA HEATED DILUTE ALLOY.

Heat Nos 1702 and 1705 Beta Treatment - 15' at 735°C



	A-61-167A	A-61-168A	A-61-172A	A-61-170A
	Immediate Quench	1 min. Delay	3 min. Delay	Air Cooled
Ave. Grain Size(μ)	65	80	175	240

FIGURE 3 100X

EFFECT OF DOUBLE BETA QUENCHING ON STRUCTURE OF DILUTE ALLOY.

Heat Nos. 1702 & 1705 BETA TREAT- 15' at 735°C

A-61-167A
Single Heat Immediate Quench

A-61-174A
Double Heat Immediate Quench each time

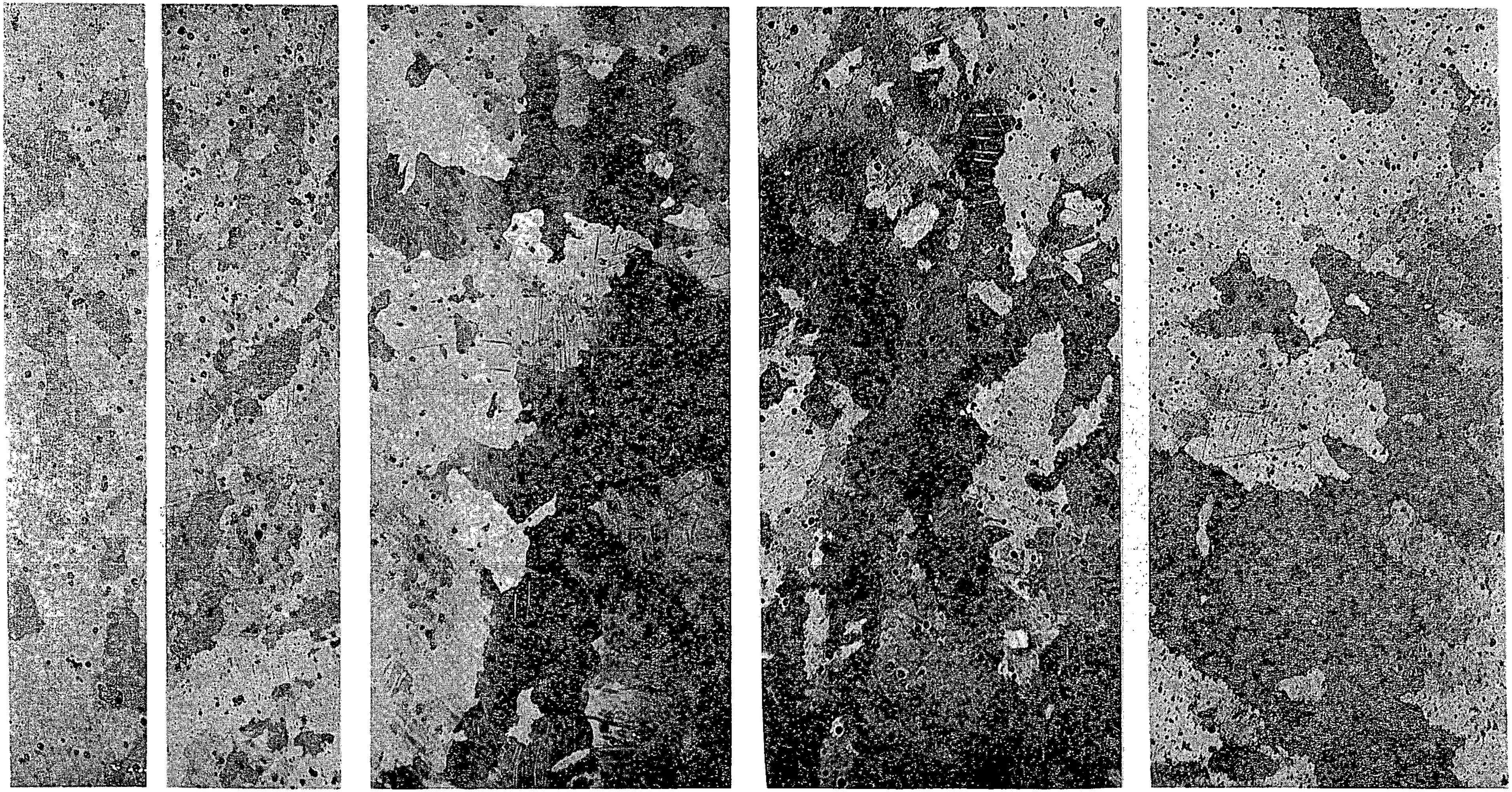
Ave. Grain Size (μ)	65	100

FIGURE 4 100X

EFFECT OF ANNEALING TIME ON BETA TREATED DILUTE ALLOY.

Heat Nos 1702 & 1705

Beta Treat - 15' at 735°C Immediate Quench
Anneal Temperature - 640°C



	A-61-166A	A-61-166C	A-61-176A	A-61-173A	A-61-175A
	edge	centre			Double Beta Quench
	12 hour anneal		24 hour anneal	26 hour anneal	24 hour anneal
Ave. Grain Size (μ)	65		115	65	140

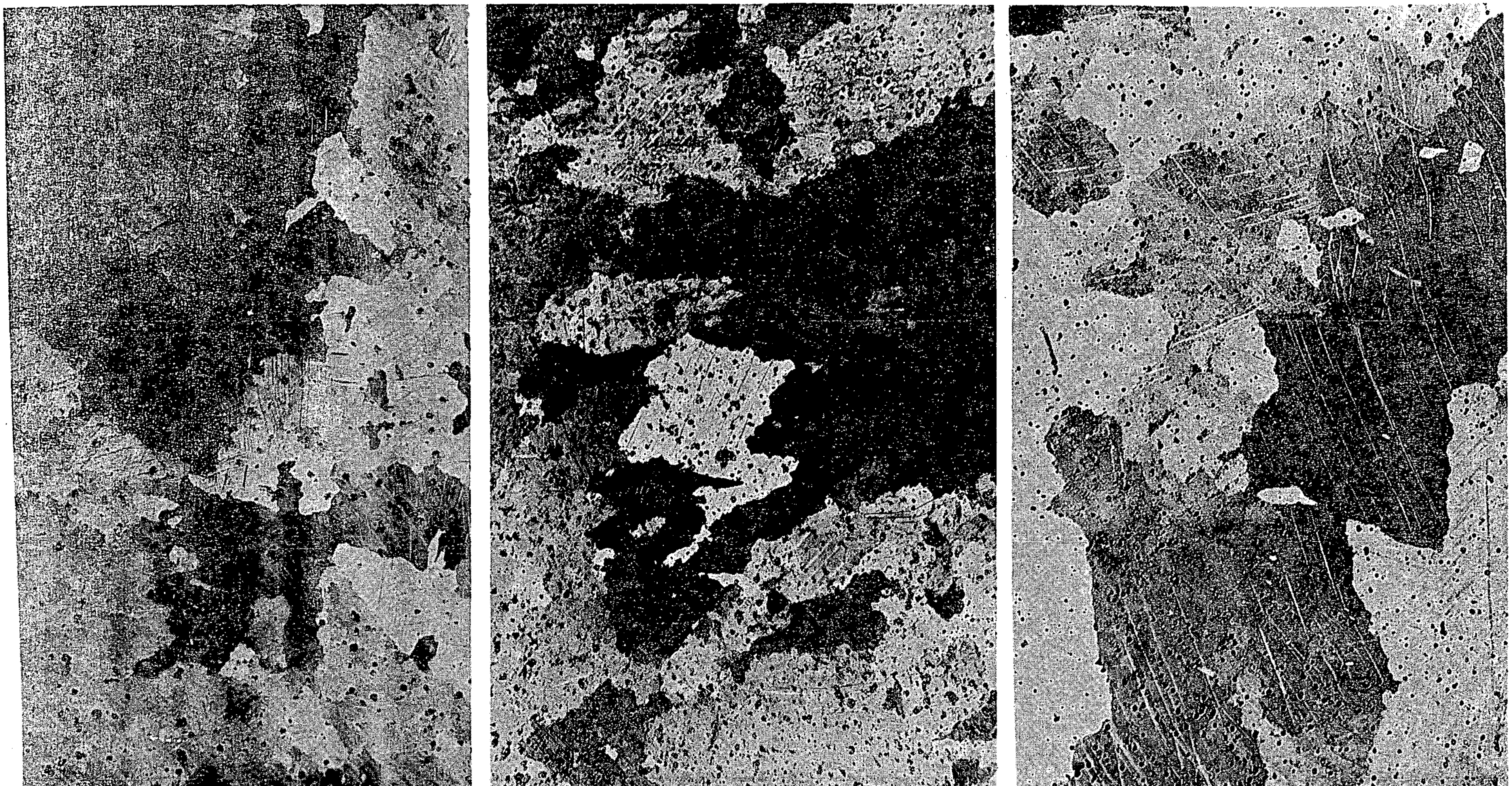
FIGURE 5 100X

EFFECT OF ANNEALING ON DELAY QUENCH BETA TREATED DILUTE ALLOY.

Beta Treat - 15' at 735° C

Anneal - 24 hours at 640°C

Heat Nos 1702 & 1705



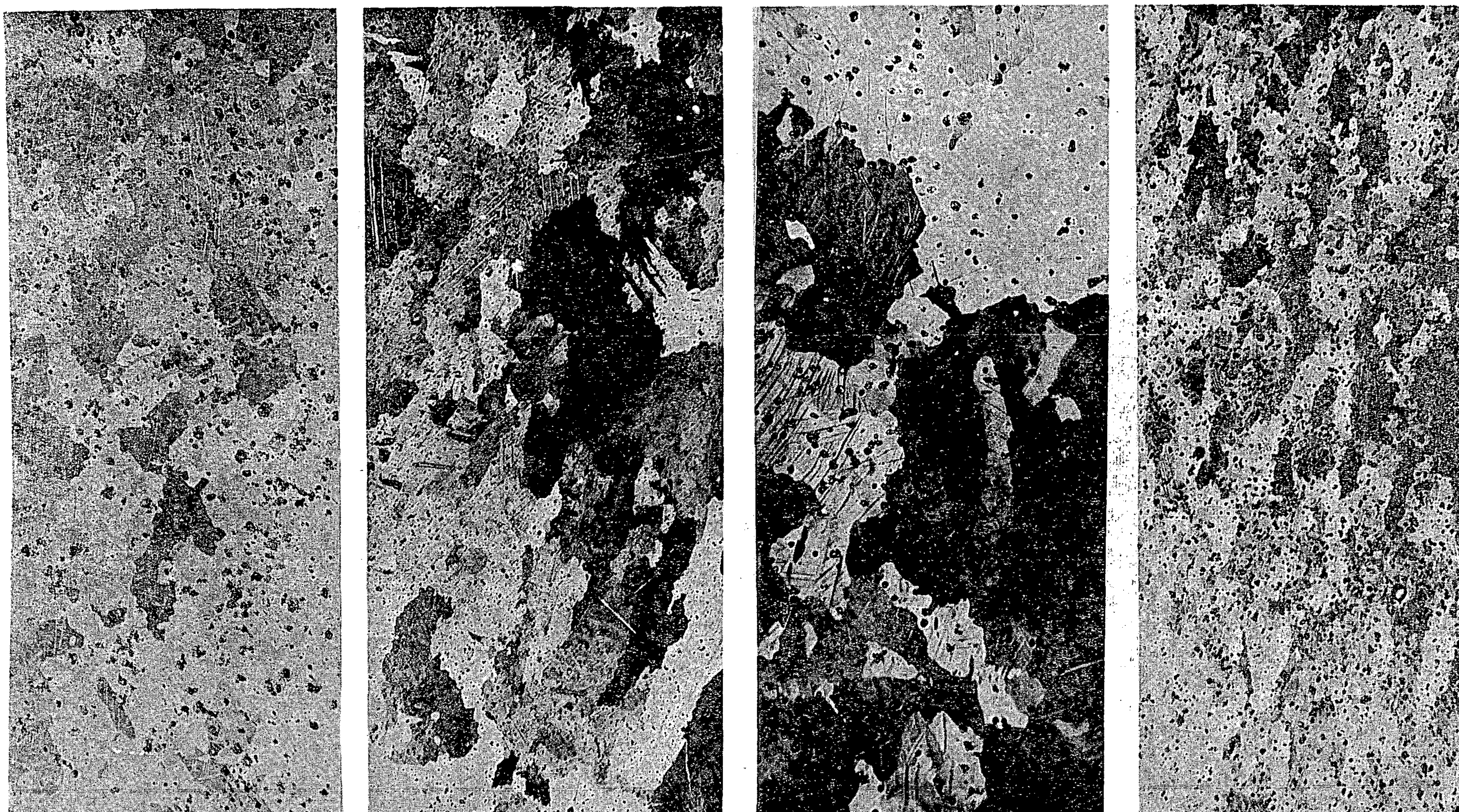
	A-61-176A	A-61-222A	A-61-224A
	Immediate Quench	1 min. Delay	3 min. Delay
Ave. Grain Size (μ)	115	120	220

FIGURE 6 100X

EFFECT OF SMALL VARIATIONS IN IRON, ALUMINIUM AND CARBON ON BETA TREATED DILUTE ALLOY.

BETA TREAT-15' at 735°C Immediate Quench

Heat No.	1702 or 1705	1709	1712	1716



	A-61-167A	A-61-219A	A-61-220A	A-61-221A
Ave. Grain Size (μ)	65	100	130	60

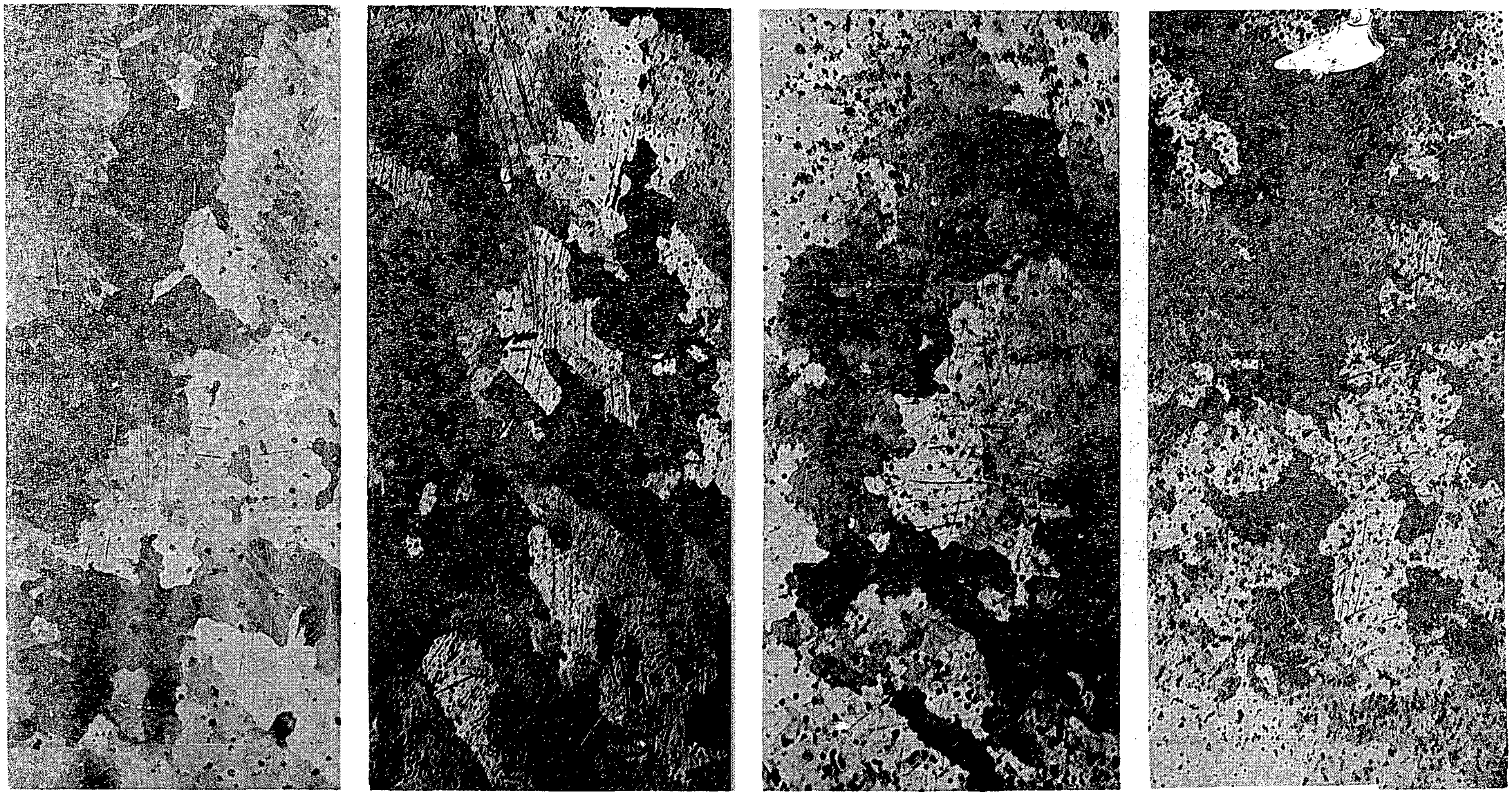
FIGURE 7 100X

EFFECT OF SMALL VARIATIONS IN IRON, ALUMINIUM & CARBON ON

BETA TREATED AND ALPHA ANNEALED DILUTE ALLOY.

Beta Treat - 15' at 735°C immediate quench
Anneal - 24 hrs at 640°C

Heat No.	1702 or 1705	1709	1712	1713

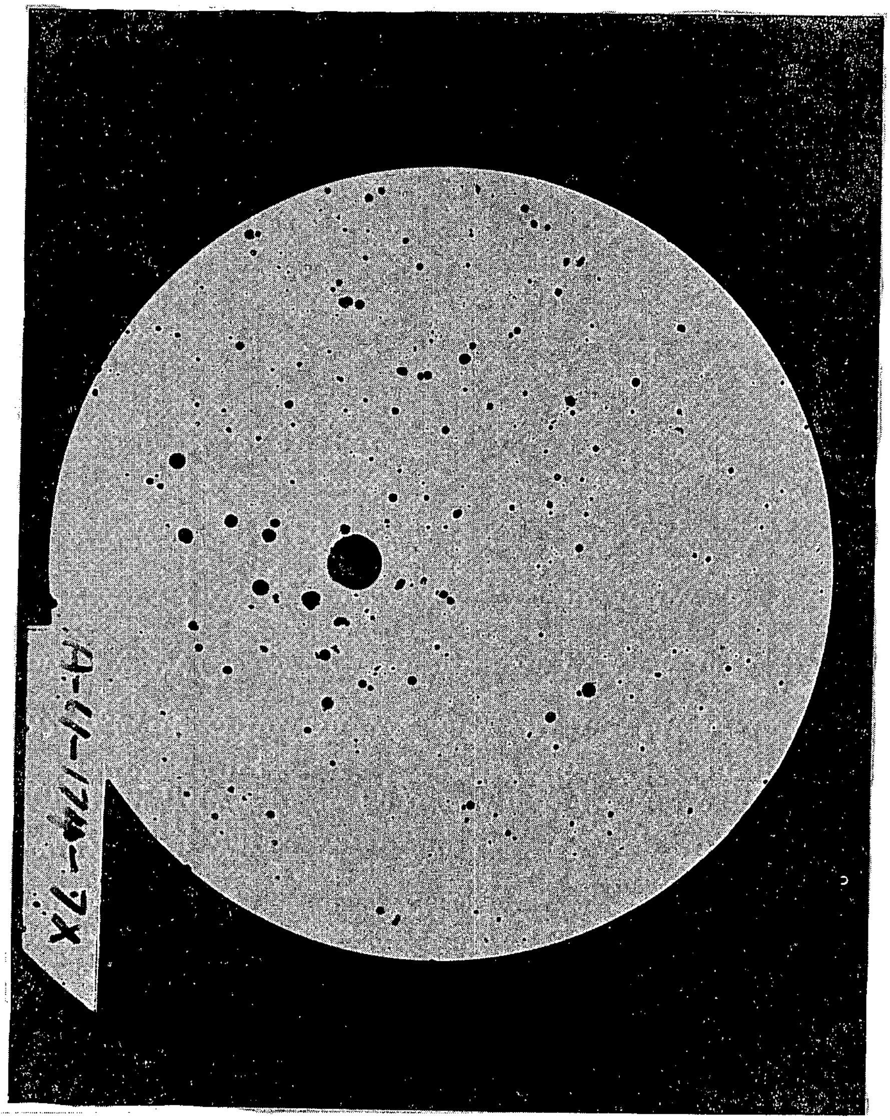


	A-61-176A	A-61-225A	A-61-228A	A-61-230A
Ave. Grain Size (μ)	115	100	130	85

FIGURE 8 7X

MACRO PHOTOGRAPH SHOWING POROSITY IN DILUTE ALLOY ROD

Heat No. 1705



A-61-174