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URANIUM ALLOY DEVELOPMENT

PART III

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Metallurgy of Uranium

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ABSTRACT

The heat treatment of extruded uranium rod containing about 0.1 percent carbon as major impurity was continued further, and the maximum hardness obtained to date results from prolonged heating at 900° C and quenching in water. A possible mechanism for this behavior is given.

A summary of a suitable method for soft soldering and silver soldering uranium is given.

Some compression stress-strain curves are given for uranium biscuit metal. The earlier difficulty with securing satisfactory stress-strain results has been overcome, and it is believed that the new curves shown are correct.

A suitable melting and casting procedure for avoiding gravity segregation in uranium-molybdenum alloys has been developed. Presumably this method would give equally satisfactory results in other uranium-alloy systems.

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URANIUM ALLOY DEVELOPMENT - PART III

URANIUM-MOLYBDENUM ALLOYS

It will be recalled that severe segregation in most uranium alloys had been experienced when they were melted in a BeO crucible surrounded by a graphite heater crucible. This was true when the melt was allowed to freeze in the crucible. However, a few tests appeared to show that in the case of uranium-molybdenum alloys at least, the segregation was practically eliminated by bottom pouring. In an attempt further to improve conditions, particularly with respect to obtaining more predictable recoveries of added molybdenum, a 50-50 uranium-molybdenum alloy was made, crushed to pass a 20 mesh screen, and analyzed. The master alloy was found to contain 53.4 per cent molybdenum.

Two bottom-poured ingots, 1/2 in. diameter by about 2 in. long, were made in the regular vacuum melting furnace. These were both of 3 per cent Mo intended composition. One was sectioned at the bottom, center, and top in a lathe in such a way that chips were taken from the entire cross section of the cast rod. The other rod was sectioned similarly except that a distinction was made between the outer and the inner portions of the rod. The results are shown in Table I.

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Table I.
Chemical Analysis of the Two Uranium-Molybdenum Rods Sectioned for Segregation Tests

Sample No.	Intended composition	Composition by Analysis Per cent Mo		
		Bottom	Center	Top
2246	3% Mo	3.24	3.33	3.29
2247	3% Mo	3.42 (center)	3.43 (center)	3.42 (center)
		3.41 (outside)	3.36 (outside)	3.46 (outside)

While the molybdenum content was somewhat above the desired 3 per cent, the degree of segregation was practically nil. Hence it seemed feasible to proceed with making new, unsegregated uranium-molybdenum alloys. The work with the old segregated alloys was dropped. A series of new alloys containing from 0.5 per cent Mo to 10 per cent Mo was started using the bottom-pouring technique and the master alloy. The new castings were 9/16 in. in diameter by 2 in. long, and required a charge of about 200 grams, depending on the composition. So far there has been no opportunity to examine these alloys, but work on them will be commenced in the near future. As before, because of the limited power or the 3 KW induction furnace, the BeO crucible was used inside a graphite heater crucible. The melts were heated to about 1350° C., held for a few minutes, cooled to 1300° C., and poured into a graphite mold by means of a pointed 1/4 in. diameter graphite rod which plugged a hole in the bottom of the BeO crucible.

Two round-nose slugs of 3 per cent Mo alloy, 7/16 in. diameter by

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1-3/8 in. long (numbers 2244, 2245) were made for the Ordnance division. After quenching from 900° C. and reheating for 2 hours at 400° C., the Rockwell A hardness was 75 for one and 76 for the other.

COMPRESSION TESTS ON URANIUM AND OTHER METALS

At the time of the report of December 14, 1943, the status of the compression tests on uranium and its alloys was not satisfactory, as different elastic moduli were obtained for uranium and its alloys, and the moduli were lower than reported elsewhere. To check the compression jig with materials of well established moduli, specimens of mild steel and duraluminum were tested. The elastic moduli obtained were about one-half the established value in both the steel and the duraluminum. This meant that the method of test had to be revised, and that the only safe procedure would be to mount extensometers directly on the specimen instead of using dial gauges attached to the jig.

When this was done, using Tuckerman extensometers of one-inch gauge length on a specimen 3/8 in. diameter by 1½ in. long, correct values for steel were obtained. The same jig was used to secure nearly axial loading, but the openings in the side of the jig were enlarged to accommodate two Tuckerman gauges, one on either side of the specimen. The loading was found to be not far from axial, as the stress-strain curve obtained with the two extensometers generally agreed rather closely. In one case, within the limit or error of plotting, the same curve was obtained.

Figures 1, 2, 3 and 4 show compression stress-strain results on three different samples of uranium biscuit metal which assayed approximately

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99.9+ per cent uranium. Table II gives the elastic moduli obtained which agree well with similar results obtained elsewhere, and also gives the 0.02 per cent and 0.2 per cent offset yield strengths. Ultimate compression stress results could not be obtained as the column represented by the specimen was apparently too unstable, and it bent after reaching loads of several thousand pounds above the yield strength.

In plotting the results shown in Figures 1-4, average stress and average strain readings from the two Tuckerman extensometers were used, except in one case where the data fell on the same curve, and the points are plotted seperately, Figure 2. In no instance did the strain readings at constant stress differ by more than about 0.2 per cent strain, and in every case but one they were much closer together. While the moduli of the uranium and uranium-molybdenum samples shown in the report of December 14 were in error by a factor of 2 or more, it is believed that the yield strengths are of the correct order ot magnitude. However, these values will be checked in the near future.

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TABLE II. COMPRESSION TESTS ON URANIUM BISCUIT METAL, AS CAST

Alloy no.	Density, g/cm^3	0.2% offset yield, psi	0.02% offset yield	Youngs Modulus E, psi
2228	19.1	41,250	25,000	22.3×10^6
2239	18.5	40,250	27,000	22.5×10^6
2248	18.8	45,500	26,500	23.5×10^6
2263	18.9	38,750	23,500	25×10^6
Report CT-422 Hard rolled	----	------	------	19.0×10^6
Report CT-697 Annealed	----	------	------	22.3×10^6
Mild steel Cold rolled	----	------	------	27.6×10^6

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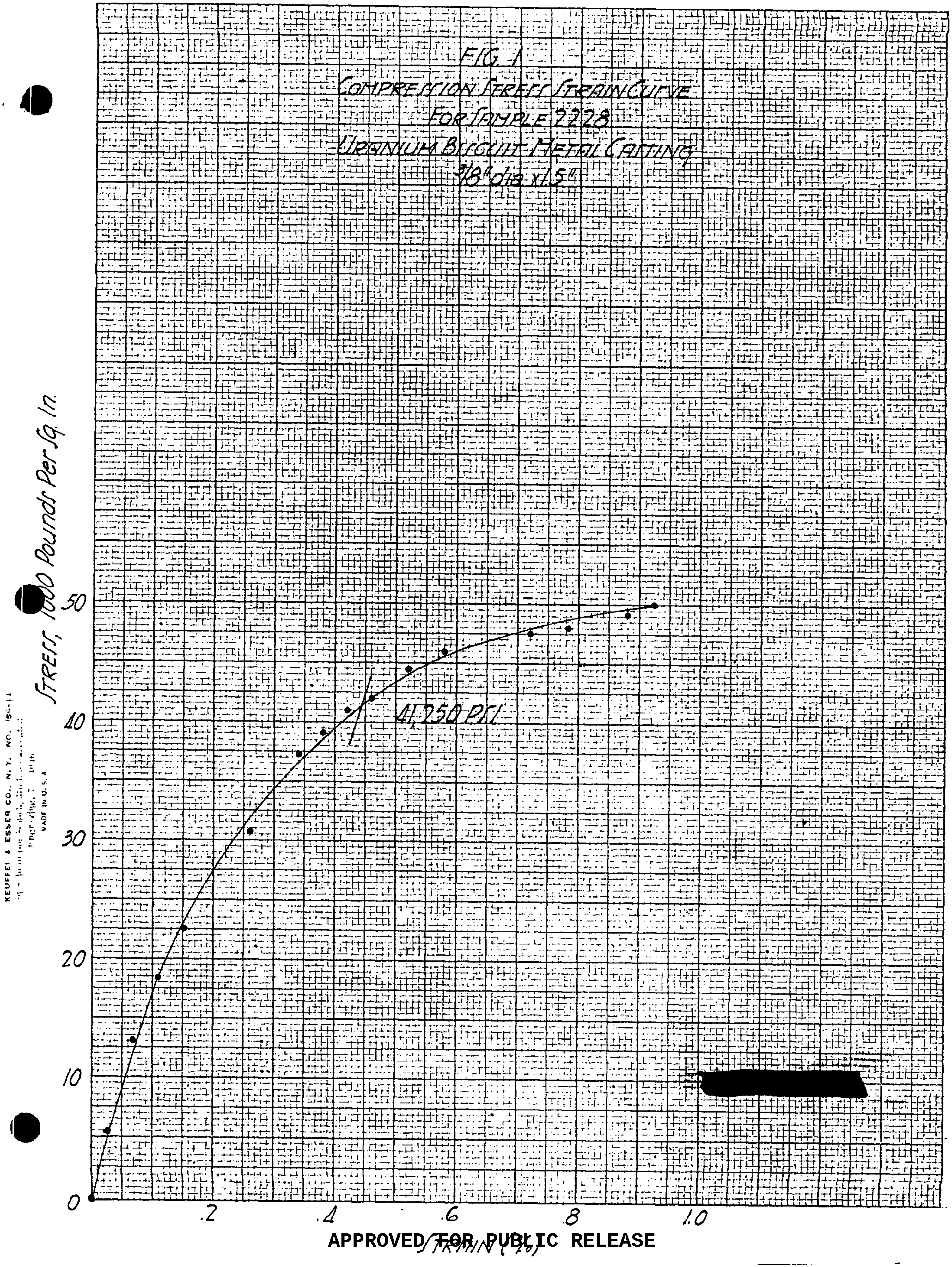

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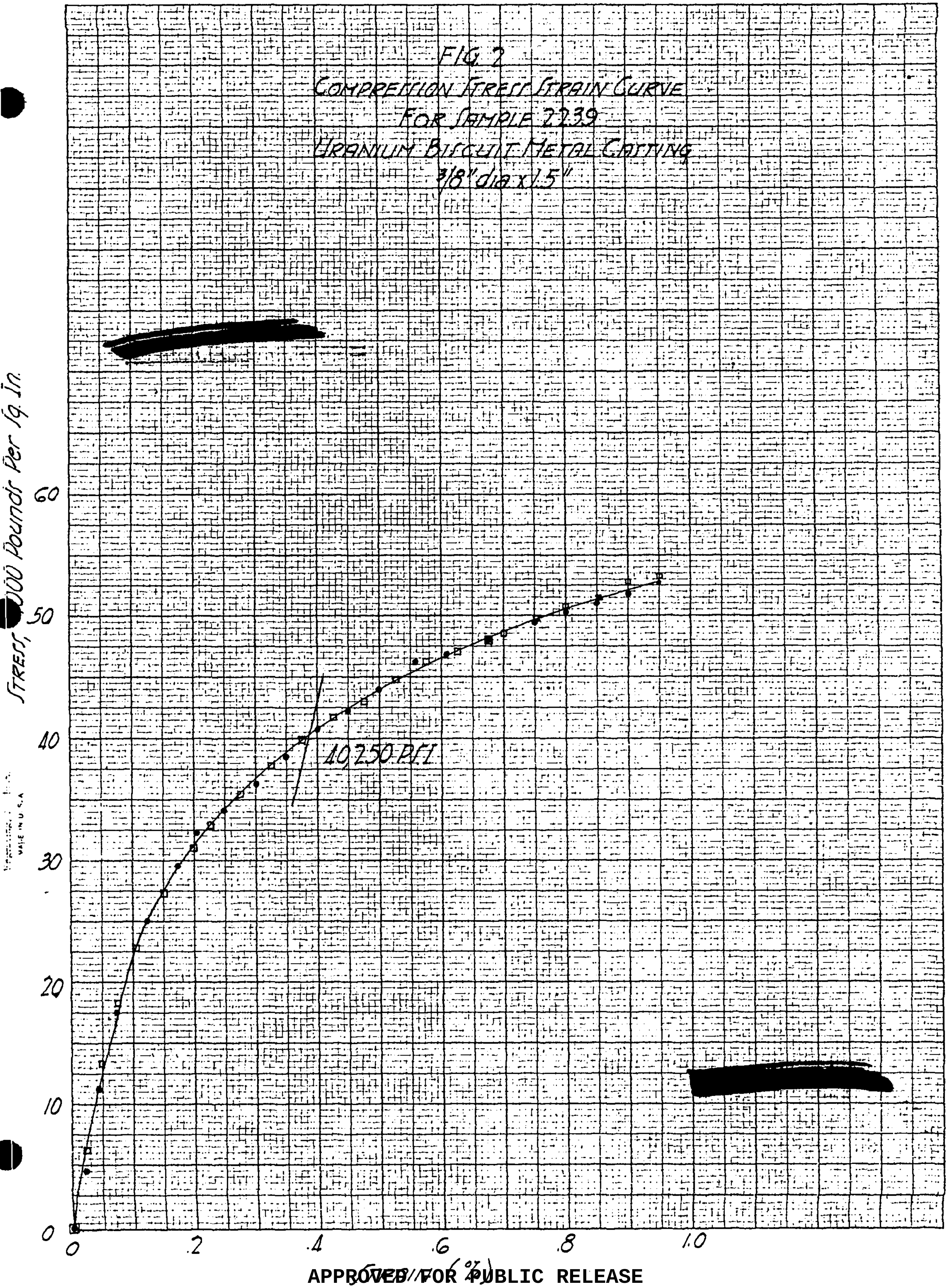
FIG. 2
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 2239
URANIUM BISCUIT METAL CASTING
3/8" dia x 1.5"



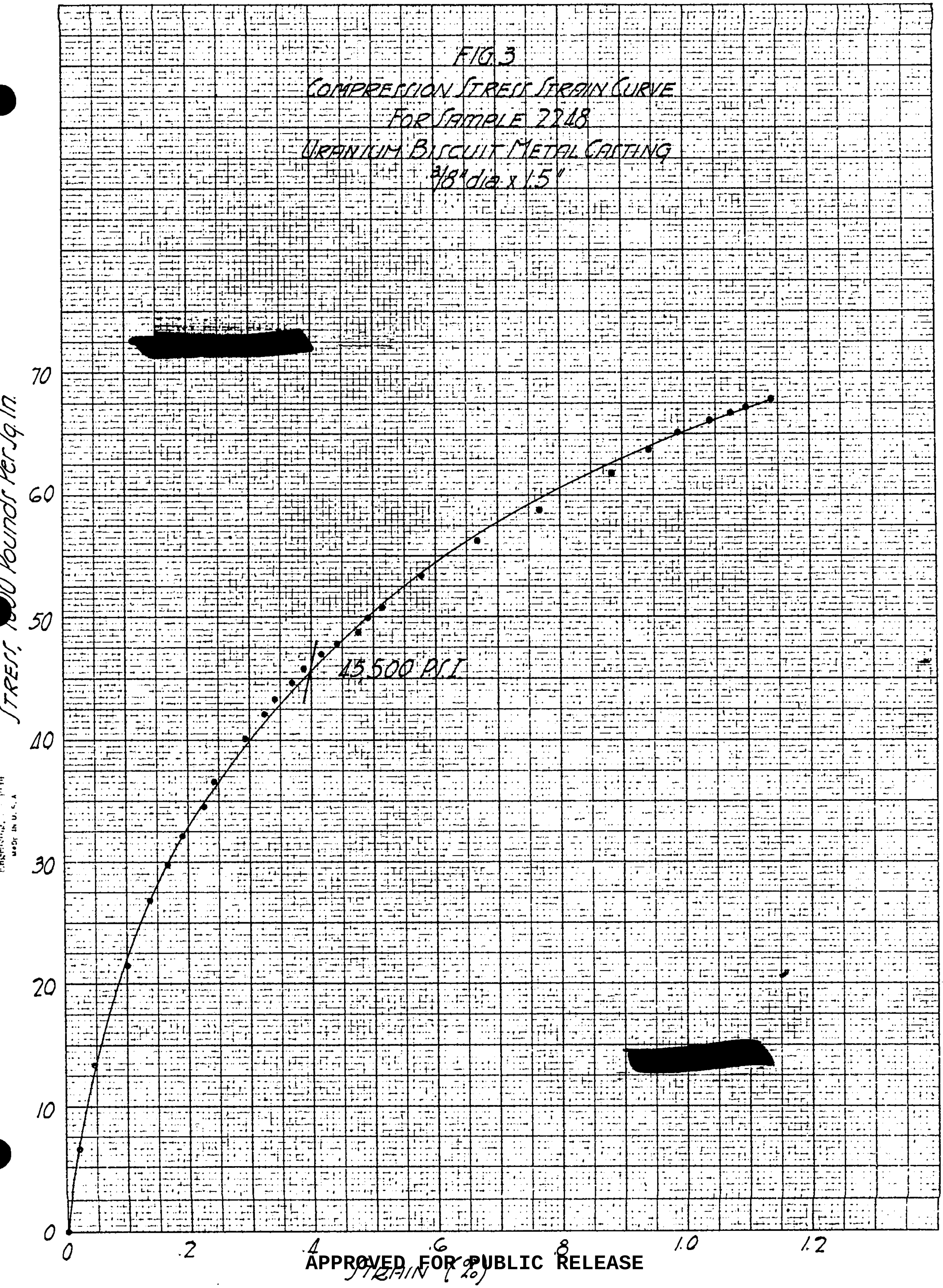


FIG. 3
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 7248
URANIUM BISCUIT METAL CASTING
3/8" dia. x 1.5"
[redacted]
45,500 P.S.I.
[redacted]
STRESS, 1000 Pounds Per Sq. In.
0
10
20
30
40
50
60
70
0
.2
.4
.6
.8
1.0
1.2
STRAIN (%)

FIG. 4
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 2263
URANIUM BISCUIT METAL CASTING
3/8" dia x 1.5"

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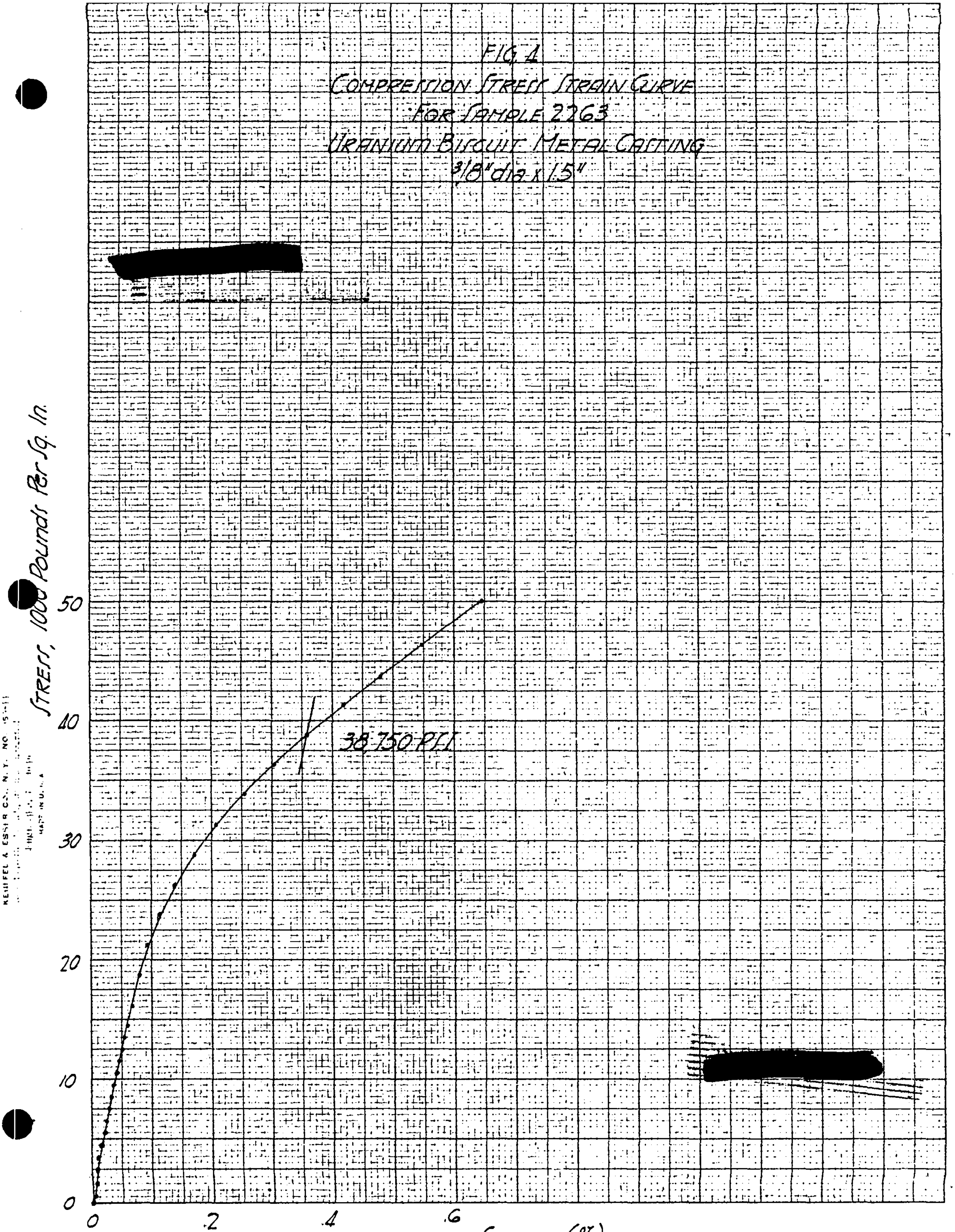

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HEAT TREATMENT OF EXTRUDED URANIUM ROD

The heat treatments on samples of the extruded rod described in the report of December 14 were continued with the view of obtaining more information concerning the mechanism of hardening. It is still postulated that the hardening is due to the carbon impurity, but definite proof of this is lacking at present.

The results on various samples of extruded rod RU-5205 are shown in Table III. All samples except 2150 were 3/16 in. thick slabs cut from $1\frac{1}{4}$ in. diameter extruded bar RU-5205. Specimen no. 2150 was 3/8 in. diameter by 1 in. long, and was machined from a 3/4 in. diameter vacuum cast rod. The carbon content of RU-5205 was formerly found to be 0.1 per cent, but this is being checked again. The carbon content of 2150 has not been reported as yet.

TABLE III. HARDNESS OF UNALLOYED URANIUM SAMPLES AFTER VARIOUS HEAT TREATMENTS

Specimen No.	Temperature	Time	Quench	Rockwell A Hardness and Remarks
2134-10	800° C furnace cool	——	none	59
-11	740° C furnace cool	——	none	60
-12	700° C furnace cool	——	none	61
-27	900° C	2 hr.	cold H_2O	67 successive heat treatment
-27	300° C	2 hr.	cold H_2O	
2150	900° C	2 hr.	cold H_2O	56 3/8" dia x 1" specimen cut from 3/4" dia casting
2150	300° C	2 hr.	cold H_2O	56
2150	900° C	5 hr.	cold H_2O	57
2134-28	900° C	2 hr.	cold H_2O	66 successive heat treatment
" "	300° C	15 min	" "	68
" "	300° C	30 min	" "	68

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TABLE III (continued)

Specimen No.	Temperature	Time	Quench	Rockwell A Hardness and Remarks
2134-28	300° C	2 hr.	cold H_2O	69 successive heat treatment
" "	300° C	3 hr.	" "	68
" "	900° C	5 hr.	" "	70
2134-10	900° C	5 hr.	iced brine	70 heat treatment continued from previous condition
-15	900° C	5 hr.	" "	71
-25	900° C	5 hr.	" "	69
-10	300° C	2 hr.	cold H_2O	69
-15	300° C	2 hr.	" "	69
-25	300° C	2 hr.	" "	69
-10	900° C	5 hr.	boiling brine 98° C	62
-15	900° C	5 hr.	" " "	63
-25	900° C	5 hr.	" " "	63
-10	300° C	2 hr.	cold H_2O	63
-15	300° C	2 hr.	" "	63
-25	300° C	2 hr.	" "	63
-10	300° C	16 hr.	air cool	64
-15	300° C	16 hr.	" "	64
-25	300° C	16 hr.	" "	64
-10	900° C	1 hr.	iced brine	67
-15	900° C	1 hr.	" "	68
-25	900° C	1 hr.	" "	68
-10	900° C	2 hr. total	" "	65 specimens showed very fine cracks
-15	900° C	2 hr.	" "	69
-25	900° C	2 hr.	" "	68
2150	900° C	16 hr.	" "	59 3/8" dia x ½" specimen 61 3/8" dia x 3/16" specimen
2134-2	900° C	2 hr.	" "	65
2134-24	900° C	2 hr.	cold H_2O	66
2150	900° C	2 hr.	" "	59 3/8" dia x ½" specimen 60 3/8" dia x 3/16" specimen
2134-2	900° C	4 hr. total	" "	66
2134-24	900° C	4 hr. "	" "	67
2134-2	900° C	8 hr. "	" "	66 some readings as high as 70
-24	900° C	8 hr. "	" "	69 some readings as high as 72
-2	900° C	16 hr. "	" "	68 one reading as high as 71
-24	900° C	16 hr. "	" "	71 one reading as high as 72

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Several conclusions can apparently be drawn from these results:

1. Samples of unalloyed uranium respond differently to identical heat treatments, presumably because of differences in carbon content.
2. Longer soaking periods ("solution treatment"), up to 16 hours, result in higher hardness as-quenched from 900° C, but there is some indication that over about 5 hours causes no large increase in ultimate hardness.
3. Aging time at 300° C following a 2 hour 900° C solution treatment is of little importance if it exceeds 15 minutes.
4. Quenching in iced brine (-3° C) results in higher as-quenched hardness than quenching in boiling brine (98° C).
5. Aging at 300° C, following a 5 hour soak at 900° C and quench, results in no increase in hardness (unlike aging after a 2 hour soak at 900° C).
6. Smaller specimens, about 3/16 in. thick, show higher as-quenched hardness values than larger, 3/8 in. diameter x ½ in., specimens.
7. Quenching from 900° C after 5 hours or more at temperature results in, thus far, maximum hardness: 70-71 Rockwell A.

Microstructure of Heat-Treated Extruded Uranium Rod Samples. Photomicrographs of several of the heat-treated samples described in Table III are shown in Figs. 5-22. The first seven pictures, Figs. 5-11, whow the two types of structure observed in specimens of extruded rod which were soaked for extended periods (over 5 hours) at 900° C and water quenched. The outside of the specimen shows a two-phase structure apparently caused by retaining either the gamma or beta phase at room temperature. It is mildly suggestive of a martensitic structure, but is not so pronounced and clear-cut as the martensite developed in steels. The interior portion of the specimens seem to show only a single (probably alpha) uranium phase with particles of

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the carbide constituent.

Figs 12-15 show the structure of two furnace-cooled samples, one cooled from 900° C and the other cooled from 740° C. Fig 15, 740° C furnace-cooled structure, shows the commonly observed single-phase matrix with dispersed carbide phase. The 900° C furnace-cooled structure, Figs. 12-14, however, seems to show two phases other than the carbide phase. In Fig. 14 some of the carbide phase appears to precipitate partly in needles as well as small rounded particles.

The rest of the pictures show structures observed as a result of quenching from 740° C, 700° C, 600° C, and 300°C. The 700° C and 600° C quenched samples show essentially the same structure: carbides in a single phase field, although there is a possibility that some of the finely divided constituent observed in Figs. 16 and 21 may be something else. The only other structure which is different from those already observed in the coarse banded structure in Fig. 20 for the 740° C quenched sample observed near the corner of the specimen. This may indicate some beta retention, but it is not certain.

<u>Discussion of Hardening of Extruded Uranium Rod</u>. The microstructure of the extruded rod containing about 0.1 per cent carbon suggests that a martensite-type hardening mechanism is responsible for the maximum hardness observed on quenching the rod after a sufficiently long soak at 900° C to get most of the carbon in solid solution. Shorter solution treatment at 900° C apparently does not dissolve enough carbon to obtain the martensite reaction on quenching, but enough stays in solution after 2 hours at 900° C to get subsequent increases in hardness on aging at 250-400° C.

While this explanation is only provisional, it appears to explain the observed facts and will serve as a point of approach for any future work on the subject. Because of the necessity for quite drastic quenching to secure maximum hardness on

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the small pieces used in this work, it appears unlikely that large pieces of uranium containing about 0.1 carbon can be satisfactorily hardened by heat treatment.

SOLDERING URANIUM

A few experiments were carried out with the purpose of finding a suitable procedure for both soft soldering and silver soldering uranium to other metals. After experimenting with various hot-dip coatings on uranium to supply a suitable base for soft soldering, it was found that an alloy of 80 per cent Cd and 20 per cent Zn provided an excellent coating. The piece of uranium was cleaned in 50 per cent HNO_3 and dipped into the Cd-Zn alloy bath through the flux layer. After dipping up and down through the flux layer a few times, the uranium was completely coated and showed no bare spots.

A satisfactory method of silver soldering consisted of copper plating the uranium, and then applying silver solder in the usual manner. The copper plate showed no tendency to peel off during heating, and the method appeared to be capable of making a satisfactory joint between uranium and other metals. A more complete summary of this work has been prepared as a seperate report.

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Plate I

Fig. 5. Extruded Rod Sample No. 2134-2. Quenched after various periods of time at 900° C. At periphery of specimen.

2134H2-3 Etched* x 250

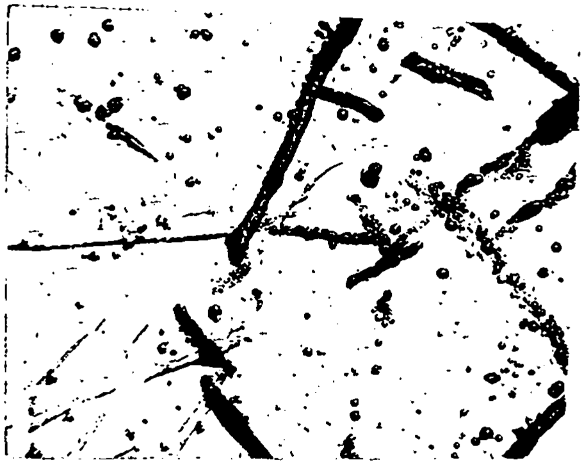

Fig. 6. Same as Fig. 5. Different field: Near corners, shows cracks

2134H2-2 Etched* x 250

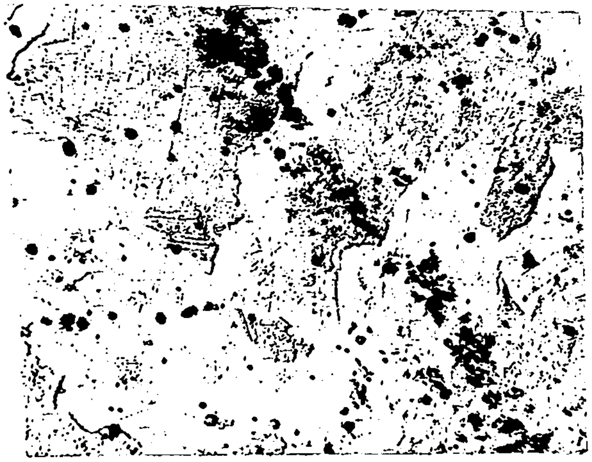

Fig. 7. Same as Fig. 5
Different field: Center Area

2134H2-1 Etched x 250

[redacted]

* All etched specimens were etched electrolytically in 10 per cent oxalic acid.

Plate II [redacted]

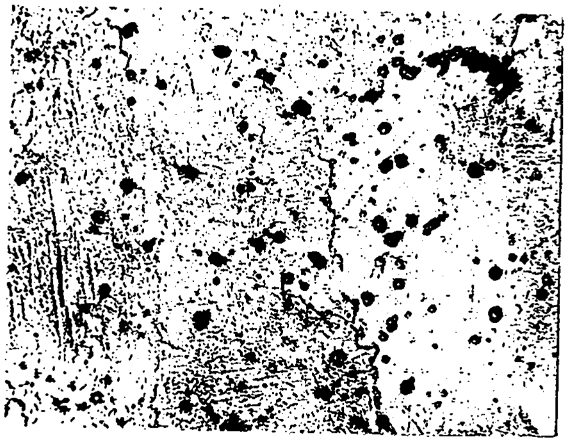

Fig. 8. Extruded Rod Sample No. 2134-28 900° C. 5 hrs. Water quench. Center area.

2134H28-1 Etched x 250

Fig. 9. Same as Fig. 8. Different field: At periphery of specimen.

2134H28-2 Etched x 250

Fig. 10. Extruded Rod Sample No. 2134-24 Quenched after various periods of time at 900° C. Center area.

2134H24-2 Etched x 250

Fig. 11. Same as Fig. 10. Different field: At periphery of specimen.

2134H24-1 Etched x 250

[redacted]

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Plate III

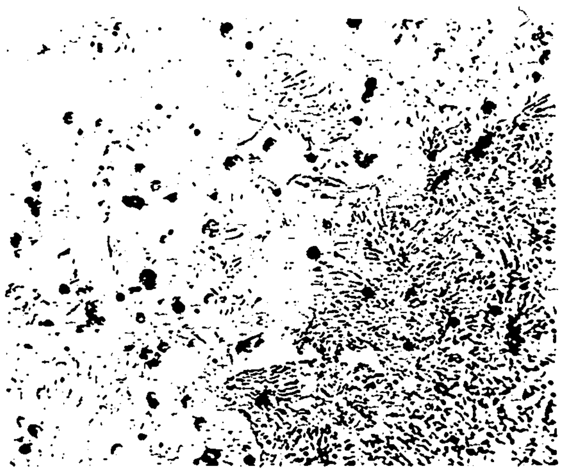

Fig. 12. Extruded Rod Sample No. 2134-9
Furnace cooled from 900° C. Center area.

2134H9-2 Etched x 250

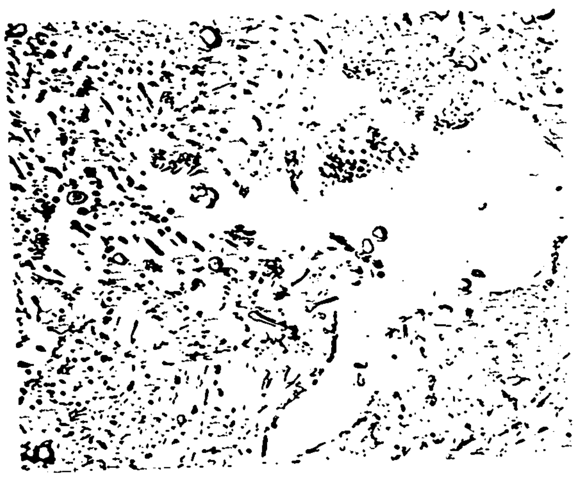

Fig. 13. Same as Fig. 12.
Different field: Center area.

2134H9-0 Unetched x 250

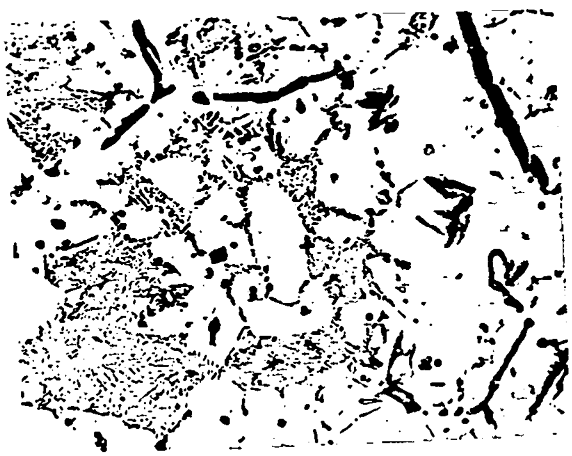

Fig. 14. Same as Fig. 12.
Different field: Near corners, edges

2134H9-1 Etched x 250

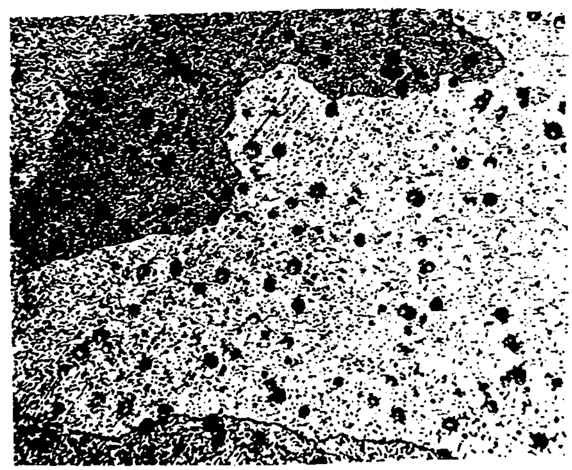

Fig. 15. Extruded Rod Sample No.
2134-11. Furnace cooled from 740° C.

2134H11-0 Etched x 250

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Plate IV

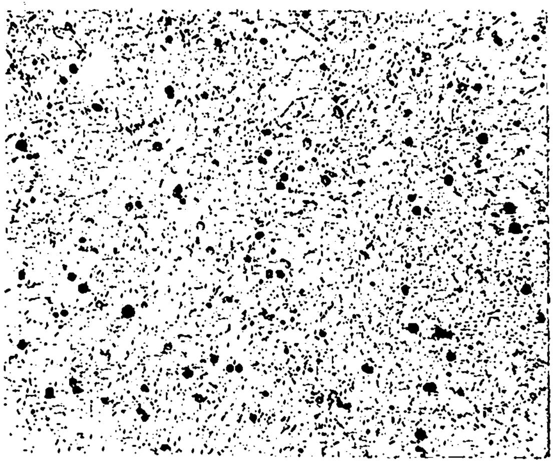

Fig. 16. Extruded Rod Specimen No. 2134-5
Quenched from 600° C. Center Area

2134H5-0 Unetched x 250

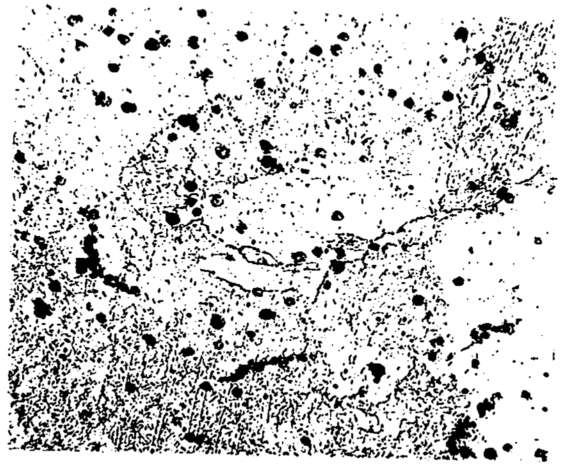

Fig. 17. Same as Fig. 16.
Different field: Center area

2134H5-1 Etched x 250

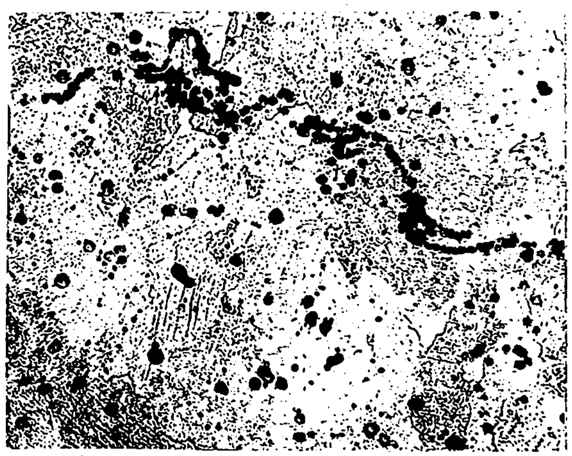

Fig.18. Extruded Rod Specimen No. 2134-8
Quenched from 300° C. Center area.

2134H8-1 Etched x 250

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Plate V

Fig. 19. Extruded Rod Sample No. 2134-3
Quenched from 740° C. Center Area.

2134H3-1 Etched x 250

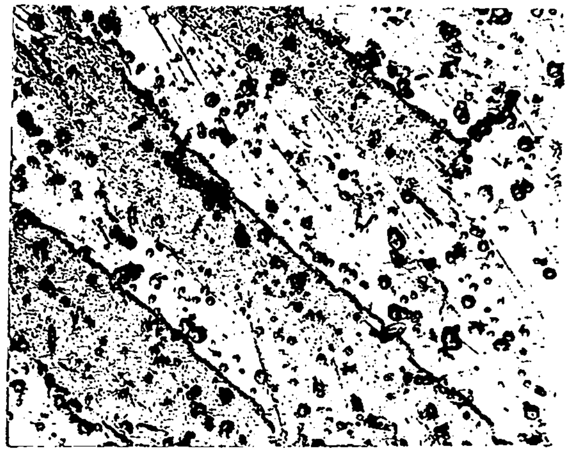

Fig. 20. Same as Fig. 19.
Different field: Near corners.

2134H3-2 Etched x 250

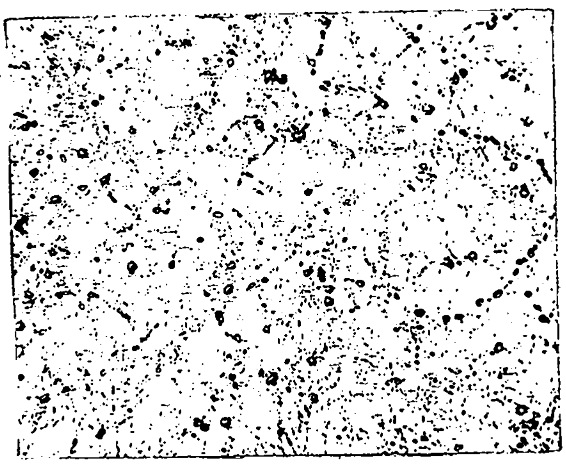

Fig. 21. Extruded Rod Specimen No. 2134-4
Quenched from 700° C. Center Area.

2134H4-0 Unetched x 250

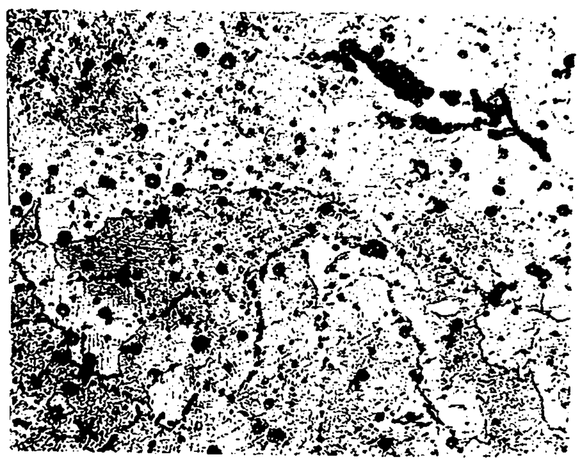

Fig. 22. Same as Fig. 21.
Different field: Center area

2134H4-1 Etched x 250

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