UNCLASSIFIED

[redacted]

L A REPORT 68

[redacted] copies

February 15, 1944

This document contains 30 pages

URANIUM ALLOY DEVELOPMENT

PART IV

WORK DONE BY:

A. U. Seybolt

L. B. Stark

W. F. Arnold

F. J. Schnettler

REPORT WRITTEN BY:

A. U. Seybolt

Metallurgy, All - Uranium

[redacted]

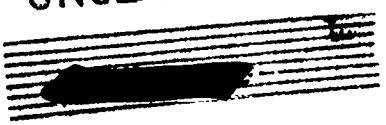
[redacted]

[redacted]

ABSTRACT

Analyses are given of the bottom-poured uranium-molybdenum alloys mentioned in LA-55, and show practically no segregation. Compression stress-strain tests have been made on unalloyed uranium castings and on extruded bar. The results show no significant difference between these two types of material, the 0.2 percent off-set yield strength being about 31,000 lbs/sq in, and the moduli of elasticity ranging from 19,000,000 lbs/ sq in to 25,000,000, with most of the values falling close to 22,000,000. Alloys of uranium with Ti, Zr, Cr, Cb, Ni, and Pt at 2 atomic percent concentrations in each case were made in 200 gram lots. Columbium had little effect on the hardness. Cr. Ni, and Pt increased the hardness considerably, and in the cases of Cr and Pt, X-ray diffraction films showed structures different from the low temperature modification of uranium. Microphotographs of the structures are given. One gram alloys of uranium with V, Au, Pd, Os, Ru, Ir, Re, Rh, and Ga were made to test the techniques for making and studying such small samples and to make alloys with new elements. The alloys of all of these elements at 2 atomic percent showed appreciably greater hardness than uranium. The alloys with Pd, Os, Ru, Rh, and Ga appeared to contain two phases (besides a little carbide). X-ray diffraction patterns of the Ir and Re alloys showed the new structure previously found in the Cr and Pt alloys. Present interpretation is that these alloys contain a higher temperature modification of uranium, the hard brittle nature of the alloys further suggesting that this is the beta phase which in pure uranium is stable between 660 and 770° C. Microphotographs of these alloys are given.

[redacted]



URANIUM ALLOY DEVELOPMENT - PART IV

URANIUM-MOLYBDENUM ALLOYS

The series of uranium-molybdenum alloys mentioned in the report of January 14, 1944, have been analyzed and some of them have been examined under the microscope in the as-cast condition. Table I shows the intended composition and chemical analysis of the six samples. Most of the ingots were analyzed at the top and bottom, but in one case, through some analytical error, the top sample had to be discarded. In another case, severe segregation made the determination useless. The segregation in this case was probably due to an insufficiently high alloying temperature.

Table I. Compositions and As Cast Rockwell A Hardness of Bottom Poured 200 g Uranium-Molybdenum Alloys

Alloy No.	Rockwell A Hardness	Intended Composition Percent	Composition by Analysis		Percent Carbon Content
			Top	Bottom	
2264	65	0.5 Mo	0.70 Mo	0.71 Mo	.037
2267	64	1.0 "	---	1.09 "	.041
2266	69	2.0 "	2.28 "	2.23 "	.046
2268	71	3.0 "	3.13 "	3.10 "	.045
2269	--	4.0 "	4.23 "	4.08 "	.059
2273	--	5.0 "	Severe segregation		---

It will be observed that in all cases where top and bottom of the ingots were analyzed, there was no significant difference in the molybdenum content.

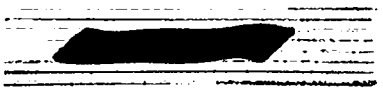
The Rockwell A hardness of those specimens tested show a more or less regular increase in hardness with molybdenum content.

Microstructure

The same microstructures shown before in earlier reports are seen again in Figs. 1-4, representing the range of as cast structures observed from 0.5 to 3 percent molybdenum.

COMPRESSION STRESS-STRAIN TESTS ON URANIUM

Several stress-strain curves for cast uranium biscuit metal were shown in the report of January 14, 1944. More recently, stress-strain curves for as-received 1¼ in. diam. extruded rod and as-cast 2 in. diam. bars have been obtained. In each case, the 5/8 in. by 1½ in. compression specimens were machined from a volume approximately half way between the outside and the center of the bar. The results are shown in Figs. 5-11. The yield strengths of the extruded bar and the cast rod (melted and cast in graphite) are surprisingly lower than in the case of the presumably purer biscuit metal: about 30,000 psi as against 40,000 psi obtained with biscuit metal castings. On the other hand, the moduli of elasticity agree quite well with those determined for the biscuit metal castings, falling in the range of 20-26 million pounds per sq. in.



As before, the stress-strain curves plotted are the average of two Tuckerman strain gauges mounted on opposite sides of the specimens. Table II shows the yield strengths and moduli of elasticity observed for the seven uranium compression samples, as well as the carbon content of the samples tested.

URAMIUM ALLOYS OF TWO ATOMIC PERCENT TITANIUM, ZIRCONIUM, CHROMIUM, COLUMBIUM, NICKEL, AND PLATINUM

It will be recalled that the above alloys of uranium were prepared in intended concentrations of 5 weight percent and the results described in the report of November 9, 1943. While the molybdenum alloy containing 2-3 weight percent molybdenum appears to be the most promising of uranium alloys examined to date, it was thought that six of the alloying elements previously investigated in the 5 percent alloys would bear further investigation in lower concentrations. These alloying elements are the following: titanium, zirconium, chromium, columbium, nickel, and platinum. These elements were chosen on the basis of their effect on the hardness of uranium and on the type of microstructure observed. Accordingly 2 atomic percent alloys of these metals with uranium were prepared in approximately 200 gram amounts, using the same bottom pouring technique described previously. The 9/16 in.diameter, 2 in. long cast rods were cut up into 3/16 in. thick discs for heat treatment, microscopic examination, X-ray diffraction and hardness tests. Sections of the top and bottom of each ingot were sent in for chemical analysis of the added metal. Table III shows the intended composition of these alloys, the hardness values, and X-ray diffraction results after quenching from 900° C.

[redacted]

TABLE II. COMPRESSION TESTS ON UNALLOYED URANIUM SAMPLES

Alloy No.	Carbon Content %	Specimen Machined from	Rockwell A Hardness	Density, g/cm^3	0.2% offset yield psi	.02% offset yield psi	Youngs Modulus E psi
2304	.062	2" dia. cast rod, S. F. 4	--	18.7	31,000	16,000	22×10^6
2306	.062	2" dia. cast rod, S. F. 4	--	18.7	30,000	15,000	19.9×10^6
2308	.066	2" dia. cast rod, S. F. 4	--	18.9	31,500	16,000	21.7×10^6
2315	.051	$1\frac{1}{8}$" dia. extruded rod, RU 677	57	18.9	32,750	17,000	25.7×10^6
2332	.053	$1\frac{1}{8}$" dia. extruded rod, RU 677	55	18.7	30,500	16,500	22.3×10^6
2336	.058	$1\frac{1}{8}$" dia. extruded rod, RU 677	53	18.9	31,250	15,000	21.9×10^6
2337	.058	$1\frac{1}{8}$" dia. extruded rod, RU 677	56	18.9	32,750	16,000	21.6×10^6

[redacted]

APPROVED FOR PUBLIC RELEASE

UNCLASSIFIED

TABLE III. COMPOSITIONS, HARDNESS VALUES, AND QUALITATIVE X-RAY DIFFRACTION RESULTS OF THE TWO ATOMIC PER CENT URANIUM ALLOYS QUENCHED FROM 900° C.

Alloy No.	Atomic % Intended	Weight % Intended	Analysis Weight %		Structure	Rockwell A Hardness*	Eberbach Microhardness D.P.N.	Rockwell A Hardness from Eberbach
			Top	Bottom				
2277 - 1	2 Ti	0.42 Ti	0.40	0.23	Alpha uranium	56	240	58
2281 - 1	2 Zr	0.776 Zr	2.7	<0.1	" "	55	219	55
+ 2282 - 1	2 Cr	0.45 Cr	-- 0.45++ --		Structure X	69	425	73
2283 - 1	2 Cb	0.80 Cb	1.39	0.25	Alpha uranium	59	240	58
+ 2292 - 1	2 Ni	0.50 Ni	0.42	0.48	" "	67	345	66
+ 2293 - 1	2 Pt	1.67 Pt	--	--	Structure X	66	372	69

+ Showed Quenching cracks

* Hardness unalloyed uranium varies from 50 - 70 Ra

++ Owing to analytical difficulties, top and bottom analyses were not obtained. The figure given is an average of 4 pieces of the rod chosen at random. The figures ranged from .41 to .55% Cr.

APPROVED FOR PUBLIC RELEASE

Microstructure

None of the sections cut from the above alloys were examined in the as-cast condition, but the first samples to be studied were held at 900° C for two hours and quenched in water. The microstructure of the alloys in this condition are shown in Figs. 12-17.

Figs. 12 and 13 showing the structure of the titanium and zirconium alloys respectively appear to exhibit the regular alpha uranium phase with spots of the carbide phase. It is probable that the majority of the titanium and zirconium is tied up in combination with carbon and little or none is left to be in solution in the uranium. Because of the segregation in the zirconium alloy and in the titanium alloy, it is uncertain how much of the alloying metals are actually present in the metallographic samples. In any case, it is likely that in order to secure interesting alloys of uranium with zirconium or titanium, more than 2 atomic percent must be used.

Fig. 14, the uranium-chromium alloy, shows an entirely different matrix from alpha uranium. Etching shows nothing not visible in the unetched structure, and the alloy is quite brittle and subject to quenching cracks, which are clearly visible. There are also dendrites of a second phase, which may be particles of uranium carbide, UC, but this is uncertain. It is possible that chromium has the property of retaining the beta phase of pure uranium on quenching from a high temperature.

Both the nickel and platinum alloys (Figs. 16 and 17 respectively) show matrices which seem to be some other phase than alpha uranium. However X-ray diffraction results showed that the nickel alloy was alpha uranium.

Again, as in the case of chromium, these alloys tend to be quite brittle as quenched from 900° C, and show quenching cracks.

The columbium alloy, Fig. 15, shows the usual alpha uranium structure with a copious sprinkling of a second phase. Apparently if columbium dissolves in solid uranium at 900° C in the amount of 2 atomic percent (0.80 weight percent), it does not stay in solution. However, some of the particles observed are probably the carbide phase.

To sum up, it appears that while none of the alloying metals investigated in the 200 g heats shows much promise on the basis of the 900° C heat treatment, it is possible that further investigation may show something interesting, particularly in the case of chromium, nickel, and platinum.

X-ray Diffraction Results

The same six samples of uranium alloys, micrographs of which have been shown in Figs. 12-17, were also used for back-reflection X-ray diffraction to find out if (1) there was any evidence of solid solubility of the alloying metal, or (2) if an entirely new phase were formed. Small amounts of a second phase could not be detected, but if the structure of the matrix was altered by alloying, this would be readily shown.

The results in a qualitative way are shown in Table III; the photograms have not been measured as yet. As noted in the table, only two structures were seen, that of alpha uranium and another structure which may be that of a high temperature modification of uranium.

It is unlikely that the new structure could be due to a second phase in each system where it occurs, because the alloys are so dilute. Furthermore, the fact that the structure is the same in five cases (the two above and three others in the next section) strongly suggests that it is due to a change in uranium; i.e. one of the high temperature forms of uranium. Since gamma uranium is known to be ductile when hot and beta uranium to be brittle, the new structure is probably due to beta uranium.

Hardness

The Rockwell A hardness values of the samples used both for metallographic and X-ray examination have already been given in Table III. Besides Rockwell A hardness, Eberbach micro hardness values were determined. This hardness scale is supposed to be equivalent to Vickers hardness, as a Vickers type diamond pyramic penetrator is used. Because of the small loads used in forcing the penetrator into the sample, the mark made is small enough to measure the hardness of micro constituents. In this case, the hardness of the matrix or major constituent was tested.

The last column in Table III gives the Rockwell A hardness obtained from the Eberbach Vickers hardness by conversion using an average curve established by testing a series of about fifteen uranium alloy samples of a range of hardness by both Rockwell A and by the Eberbach micro method. In general, the agreement is satisfactory.

[redacted]

ONE GRAM BEATS OF URANIUM ALLOYS

A series of one gram melts were made using 0.040 in. uranium wire and various alloying elements previously not investigated. The purpose of the one gram melts was twofold:

1. to get used to working with small amount of material, and
2. to investigate other alloying metals.

All of the most likely alloying elements for uranium have now been used, either in the 200 g melts or in the one gram melts. Table IV gives the intended compositions of the one gram melts.

Table IV. Compositions and Hardness Values of the One Gram Melts

Alloy No.	Intended Composition Atomic Percent	Weight Percent	Eberbach Micro Hardness[1] D.P.H.
3294	2 V	0.45 V	410
3296	2 Au	1.66 Au	460
3297	2 Pd	0.913 Pd	404
3298	2 Os	1.61 Os	±70
4301	2 Ru	0.868 Ru	445
3302	2 Ir	1.63 Ir	440
3303	2 Re	1.57 Re	555
3305	2 Rh	0.877 Rh	440
3307	2 Ga	0.59 Ga	324
3316	2 Re (3 g melt)	1.57 Re	542 center & bott 543 top

[1] Hardness values for alloys quenched from 900° C.

[redacted]

Procedure

The uranium wire and the alloying metal (mostly in the form of powder) were charged into a BeO crucible about 1/2 in. high and about 0.16 in. diameter I.D. The wall thickness of the crucible was about 0.13 in.

A vacuum tube oscillator type of high frequency induction furnace was used for melting, and has a frequency of about 540 kilocycles. No conducting heater crucible was used, but the heating was induced directly into the charge. Melting occurred in less than 20 seconds, when pieces of uranium wire about 3.8 in. long were used. However, when a larger number of short pieces were used, difficulty in melting was experienced because of poor coupling. Later this difficulty was overcome by redesigning the fused quartz furnace tube to allow closer approach of the coil to the crucible.

During the making of the one gram alloys, a coil about 1¼ in. I.D. was used, and the maximum temperature obtainable was limited to about 1350° C. With the smaller coil in use now, nearly 1600° C may be reached. The temperature limitation caused no difficulty in preparing the alloys except in the case of rhenium and iridium, both of which appeared to raise the melting point of uranium to the neighborhood of the maximum temperature obtainable. However, subsequent microscopic examination seemed to indicate that the alloys were all reasonably homogeneous. No difficulty was experienced in securing good consolidation of the small melts. Apparently the speed of melting was fast enough to prevent appreciable oxidation. The pressure during melting usually did not exceed 2 microns.

Microscopic Examination

The entire one gram samples were mounted in bakelite and polished longitudinally after heat treating for 2 hours at 900° C and water quenching. Figs. 18-30 show the structures observed at 250 diameters.

The vanadium structure resembles somewhat the one observed for U-Ti and U-Zr. Apparently a vanadium carbide is formed, and the carbide may be seen projecting up from the polished surface in Fig. 18. However, some of the vanadium appeared to go into solid solution, since a few quenching cracks, Fig. 19, were formed suggesting some hardening due to the vanadium.

Gold apparently goes into solid solution in uranium, although there is some question whether the structure is a single phase one, owing to the ambiguity of Fig. 20. The stains observed in Fig. 20 appear to be rather reproducible and Fig. 21 of the unetched structure show suggestive light spots corresponding in size and shape to the dark etching areas.

Fig. 22 shows the structure of the palladium alloy which is definitely a two phase structure, characterized by more or less dark circular patches.

Osmium seems to produce a rather indistinct fine structure hard to describe (Fig. 23). It seems to consist of two phases.

The ruthenium alloy seems to show a second phase. There are small needle-like particles, and light colored, irregularly shaped areas (Fig. 24).

The iridium alloy (Figs. 25 and 26) probably consists of a single phase, in which a sort of sub grain boundary structure is observed inside the large grains developed on electropolishing. It is possible, however, that a small amount of some precipitate outlines the sub grain boundaries.

The structure of the uranium-rhodium alloy (Figs. 27-28) is somewhat similar to that of the ruthenium alloy. It is apparently a two-phase alloy showing a rather indistinct fine structure.

The gallium alloy (Fig. 29) seems to consist of nearly equal amounts of two phases which resemble the result of a eutectoid inversion. At least the structure is reminiscent of that observed in the case of aluminum bronze, for example.

The rhenium alloy structure (Figs. 30-31) is difficult to interpret. If it were not for two types of areas observed in both the one gram sample and the three gram sample, one of which etches dark and the other light, it would clearly be a single phase structure. The grain boundaries are well developed. The dark etching area, the majority of the field in Fig. 29, and the minority in Fig. 30, appear again and again, both during polishing and etching. The disposition of the dark etching area appears to bear no relation to the grain boundaries. It is possible that the dark etching areas are places where some fine precipitate has formed. This sample could not be satisfactorily polished electrolytically and hence mechanical polishing was used.

X-ray Diffraction Results

The structures observed in the X-ray patterns are noted in Table V. Because of lack of time, only the results for seven of the alloys are given.

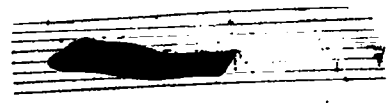


Table V. Qualitative X-ray Diffraction Results

Sample No	Intended Composition		Structure
2302-1	2 atomic percent	Ir	Structure X
2305-1	"	Rh	Alpha uranium
2307-1	"	Ga	Alpha uranium
2298-1	"	Os	Alpha uranium
2316-1	"	Re	Structure X
2301-1	"	Ru	Structure X
2297-1	"	Pd	Alpha uranium

The same two structures observed in the larger 2 atomic percent alloys were seen here. Structure X is again the same new structure referred to previously, possibly the beta phase structure.

Hardness

The micro hardness values in Vickers units D.P.N. (diamond pyramid number) is given in Table IV. No comparable Rockwell A values are given because a series of suitable samples in this hardness range have not as yet been tested by both methods. Direct Rockwell A values were not attempted because of the thinness of some of the samples and because several of the ingots showed cracks.

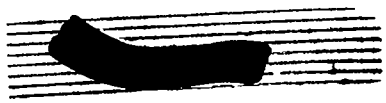
Comparing the micro hardness values given in Table III for the 200 g atomic percent alloys, it will be noted that the hardness of the one gram alloys are nearly all higher than the alloys listed in Table III.

Many of these alloys show high enough hardness to warrant further investigation.

UNCLASSIFIED

FUTURE WORK

More attention will be paid to the technique of producing and testing one gram melts and, if time allows, some of the more interesting alloys will be made up in larger amounts for further study.



UNCLASSIFIED

Fig. 1. As Cast Structure of 0.5 Molybdenum Alloy

2264-1 Etched* x 250

Fig. 2. As Cast Structure of 1 % Molybdenum Alloy

2267-0 Etched* x 250

Fig. 3. As Cast Structure of 2 % Molybdenum Alloy

2266-1 Etched* x 250

Fig. 4. As Cast Structure of 3 % Molybdenum Alloy

2268-1 Etched* x 250

* All etched specimens were etched electrolytically in 10 per cent oxalic acid unless otherwise indicated

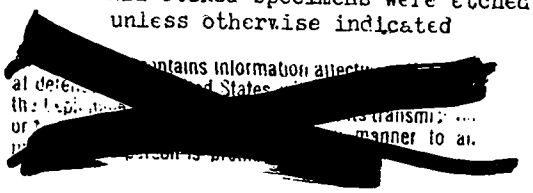


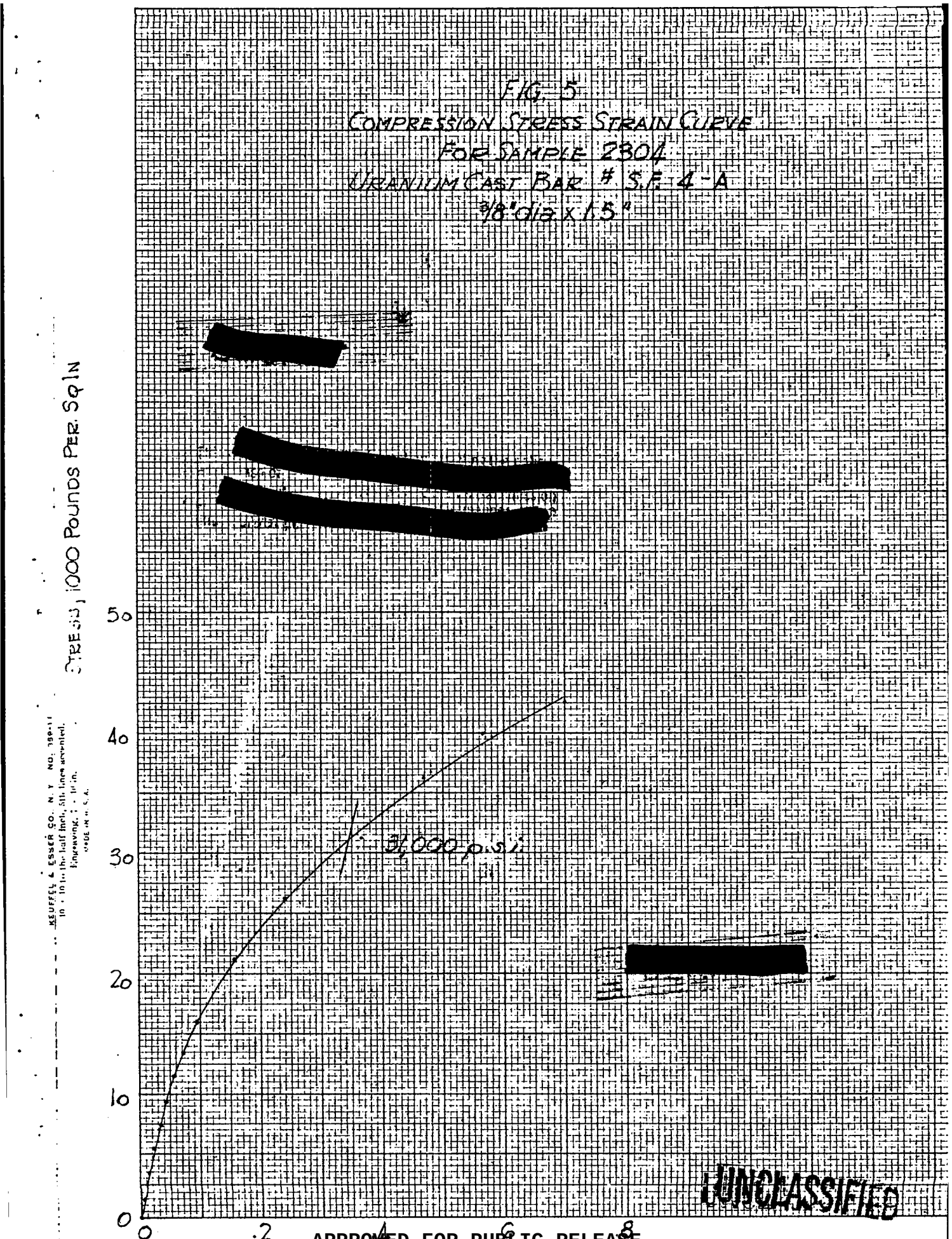
FIG. 5

COMPRESSION STRESS STRAIN CURVE

FOR SAMPLE 2304

URANIUM CAST BAR # S.F. 4-A

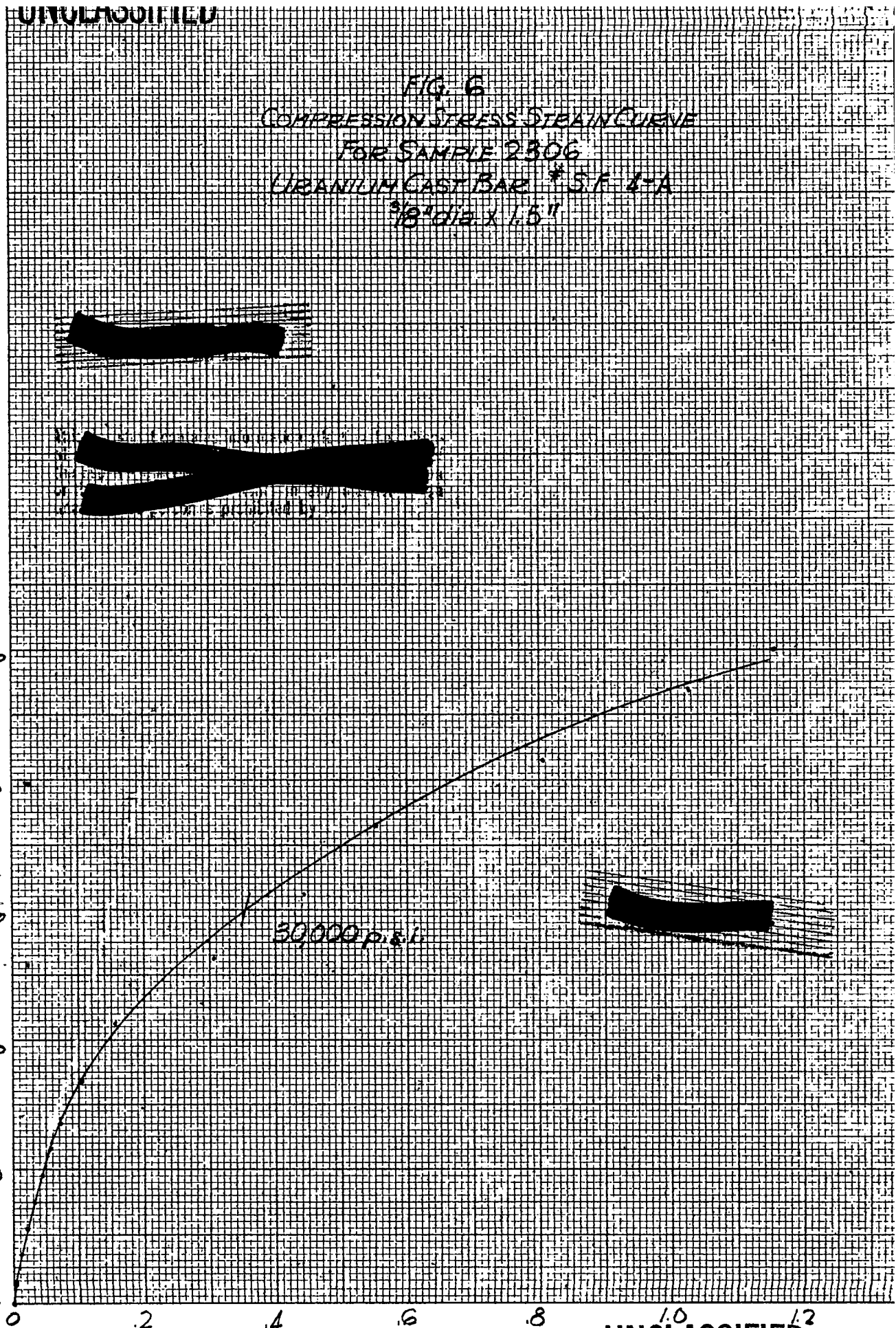
3/8" dia x 1.5"



UNCLASSIFIED

UNCLASSIFIED

FIG. 6
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 2306
URANIUM CAST BAR # S.F 4-A
3/8" dia. x 1.5"



KEUFFEL & ESSER CO., N.Y. NO. 359-11
MADE IN U.S.A.

UNCLASSIFIED

FIG. 7
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 2308
URANIUM CAST BAR # S.F. 4 A
3/16" dia. x 1.5"

STRESS, 1000 POUNDS PER SQ IN.

50
40
30
20
10
0

31,500 p.s.i.

0 .2 .4 .6 .8

STRAIN (IN/IN)

KEUFFEL & ESSER CO., N.Y. NO. 359-11
10 × 10 to the ½ inch, fifth inch accented
Engraving, 7 × 10 In
MADE IN U.S.A.

UNCLASSIFIED

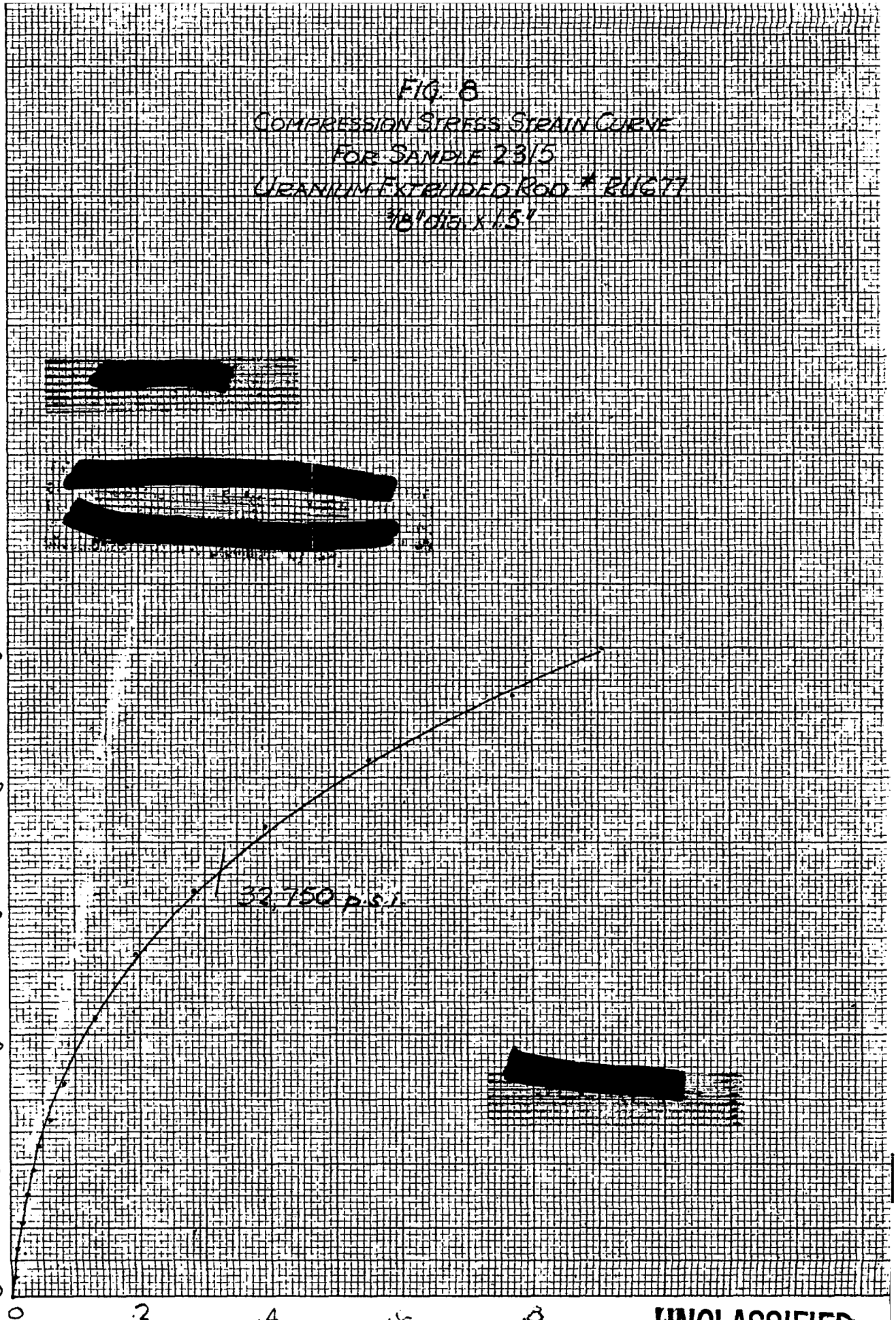
FIG. 8

COMPRESSION STRESS STRAIN CURVE

FOR SAMPLE 2315

URANIUM EXTRUDED ROD # BUG 77

3/8" dia. x 1.5"



KEUFFEL & ESSER CO., N. Y. NO. 389-11
10 X 10 to the ½ inch, Six lines accented
Engraving, 7 - 1111h.
MADE IN U. S. A.

UNCLASSIFIED

FIG. 9
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 2332
URANIUM EXTRUDED ROD * EUG17
3/8" DIA. X 1.5"

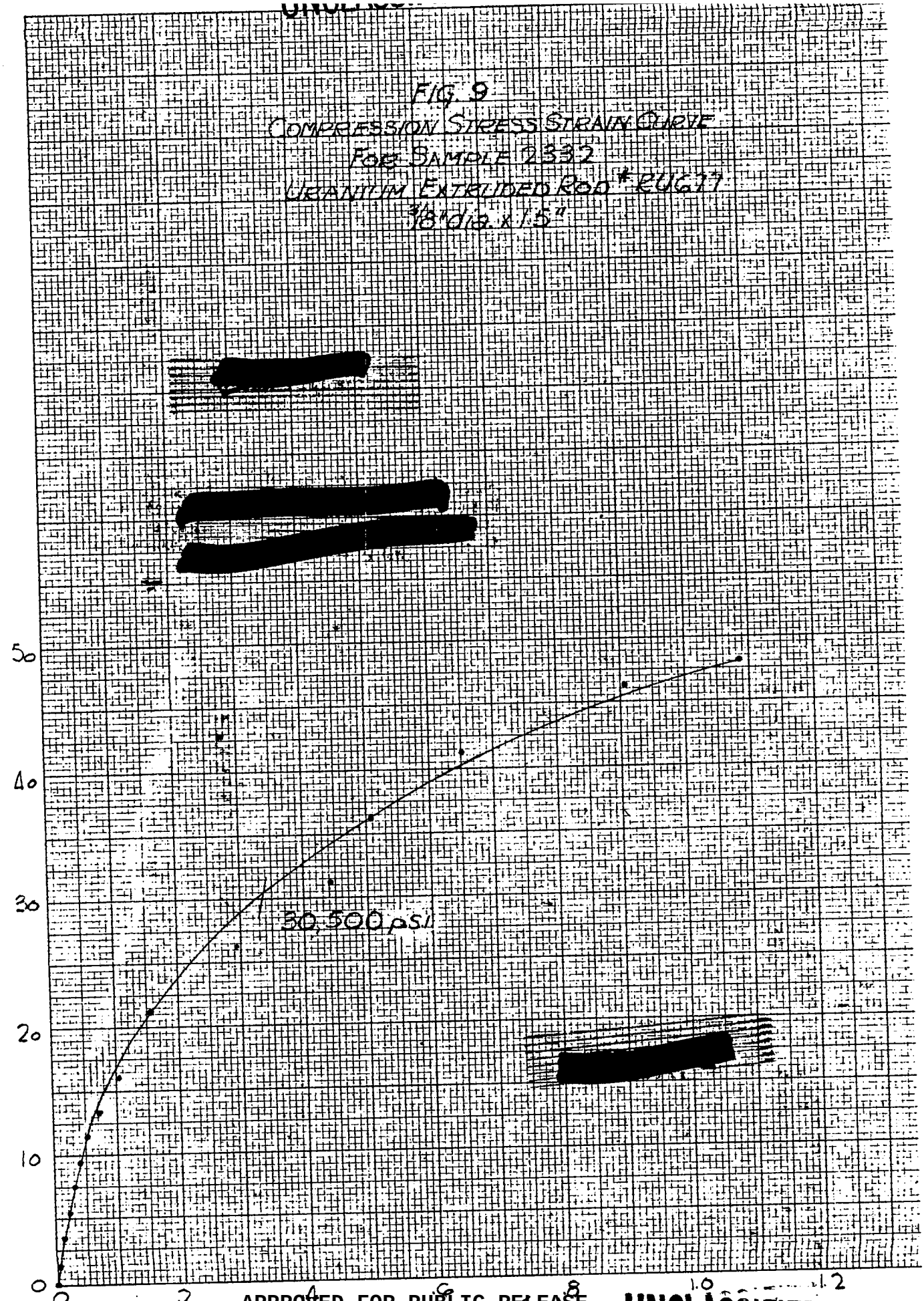


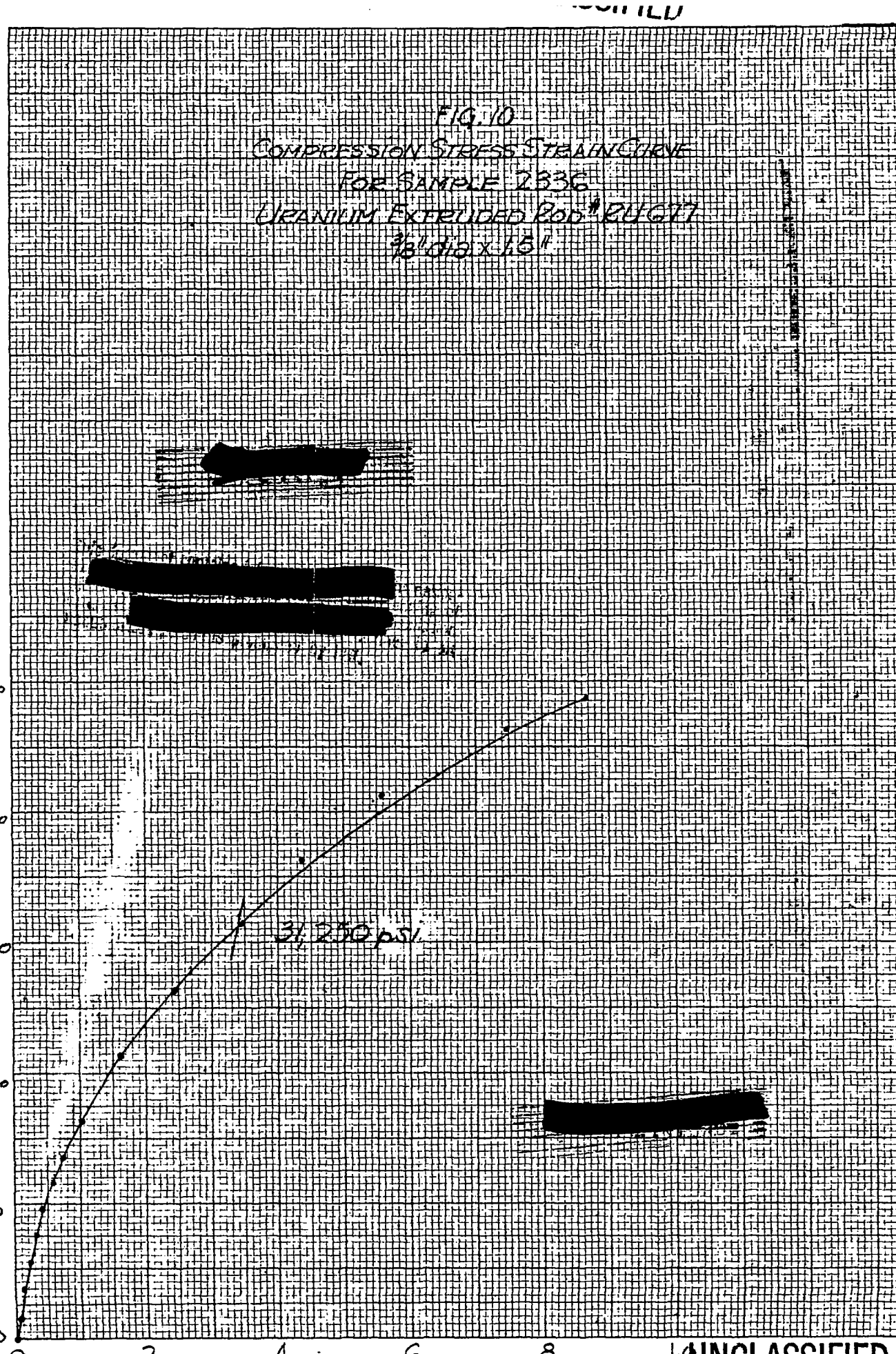
FIG. 10

COMPRESSION STRESS STRAIN CURVE

FOR SAMPLE 2336

URANIUM EXTRUDED ROD #RUG17

3/8" dia x 1.5"



KEUFFEL & ESSER CO., N. Y. NO. 359-11
10 to the half inch, 5th lines accented.
Engineering, 7 - 10-54.
MADE IN U.S.A.

FIG 11
COMPRESSION STRESS STRAIN CURVE
FOR SAMPLE 2337
URANIUM EXTRUDED ROD #EW 677
3/8" dia. x 1.5"

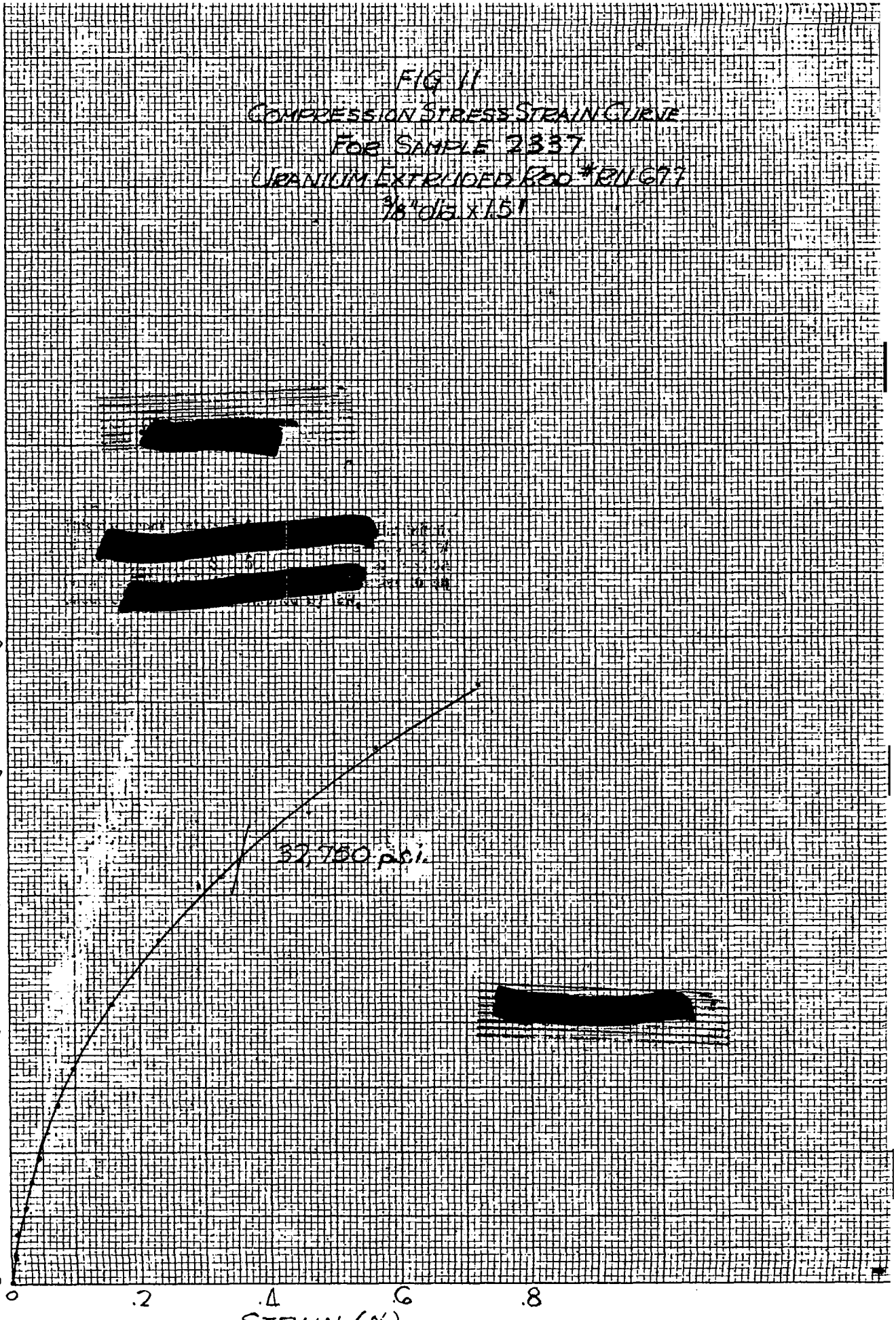


Fig. 12. 2 atomic per cent Ti-U Alloy as Quenched from 900° C.

2277H1-1 Etched* x 250

Fig. 13. 2 atomic per cent Zr-U Alloy as Quenched from 900° C.

2281H1-1 Etched* x 250

Fig. 14. 2 atomic per cent Cr-U Alloy as Quenched from 900° C.

2282H1-0 Not Etched x 250

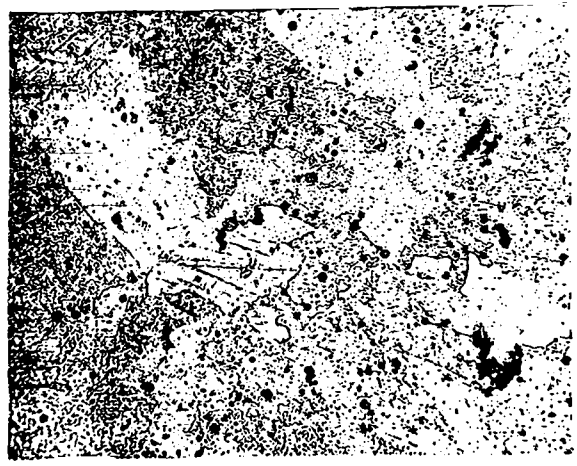


Fig. 15. 2 atomic per cent Cb-U Alloy as Quenched from 900° C.

2283H-1 Etched* x 250

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

Fig. 16. 2 atomic per cent Ni-U Alloy Quenched from 900° C.

~~2292~~-1-1 x 250

Fig. 17. 2 atomic per cent Pt-U Alloy Quenched from 900° C.

~~2293~~-1-1 Etched x 250

Fig. 18. 2 atomic per cent V-U Alloy as Quenched from 900° C.

2294-1-1 Etched x 250

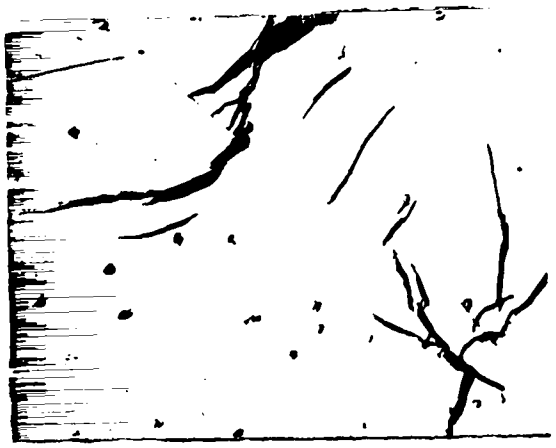


Fig. 19. Same as Fig. 18. Different Field.

2294-1-0 Unetched x 250

Fig. 20. 2 atomic per cent Au-U Alloy as Quenched from 900° C. Etched in 1:1 nitric acetic acid.

2296-1-1 x 250

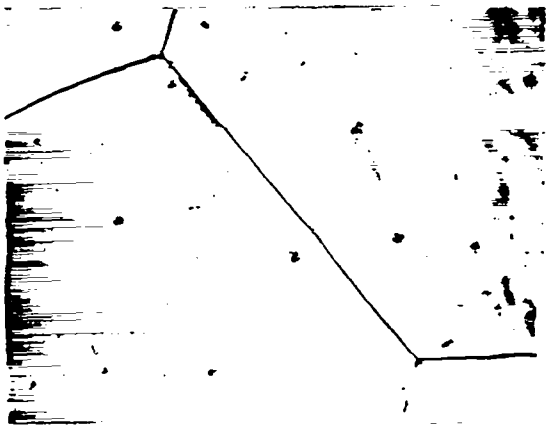


Fig. 21. Same as Fig. 20. Different Field

2296-1-0 Unetched x 250

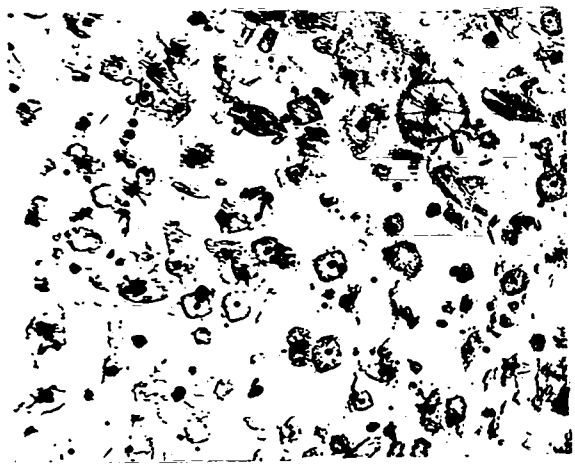


Fig. 22. 2 atomic per cent Pd-U Alloy as Quenched from 900° C.

2297-1-1 Etched x 250

Fig. 23. 2 atomic per cent Os-U Alloy as Quenched from 900° C. Etched 1:1 nitric acetic acid.

2298-1-1 x 250

Fig. 24. 2 atomic per cent Ru-U Alloy as Quenched from 900° C.

2301-1-2 Unetched x 250

Fig. 25. 2 atomic per cent Ir-U Alloy Quenched from 900° C. Etched in 1:1 nitric acetic acid.

2314-1-1 x 250

Fig. 26. Same as Fig. 25. Different Field.

2314-1-0 x 250

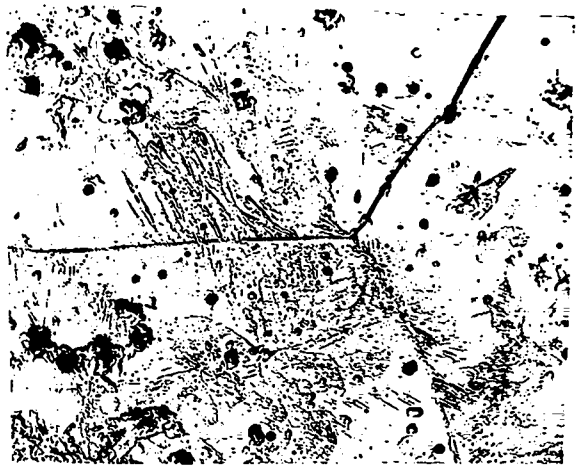


Fig. 27. 2 atomic per cent Rh-U Alloy Quenched from 900° C.

2305-1-1 Etched x 250

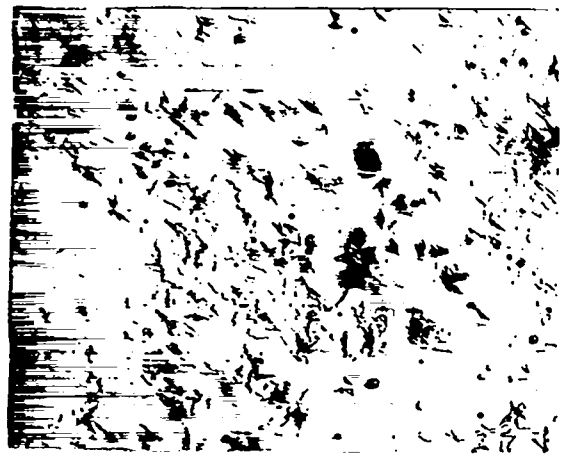


Fig. 28. Same as Fig. 27. Different Field.

2305-1-0 Unetched x 250

Fig. 29. 2 atomic per cent Ga-U Alloy Quenched from 900° C. Etched in 1:1 nitric acetic acid.

2307-1-1 x 250

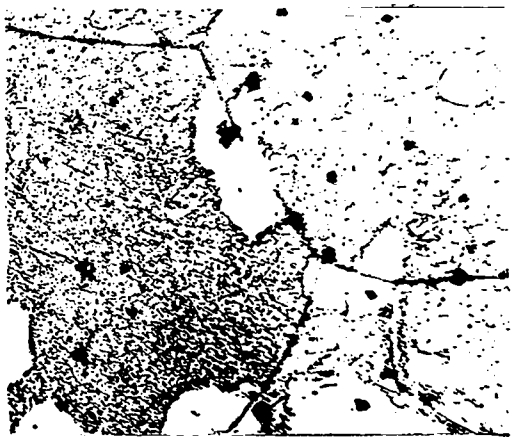


Fig. 30. 2 atomic per cent Re-U Alloy Quenched from 900° C. Thre gram sample. Etched in 1:1 nitric acetic acid.

2316-1-2 x 250

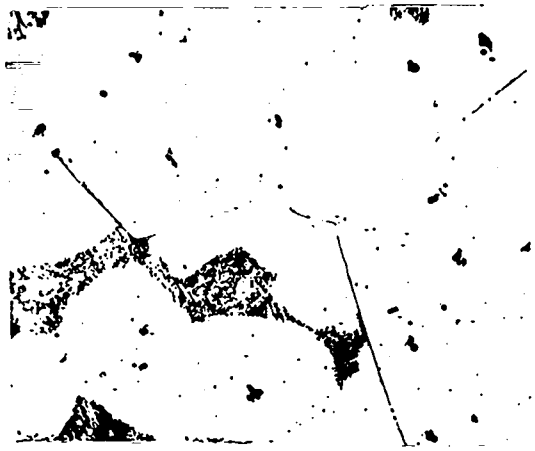


Fig. 31. 2 atomic per cent Re-U Alloy Quenched from 900° C. One gram sample. Etched in 1:1 nitric acetic acid.

2303-1-2 x 250

UNCLASSIFIED

UNCLASSIFIED

5575