Atomic Energy of Canada Limited

METALLURGICAL STRUCTURES IN A HIGH URANIUM-SILICON ALLOY

by

B.S. WYATT, L.C. BERTHIAUME and J.L. CONVERSI

Chalk River, Ontario

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ABSTRACT

The effects of fabrication and heat treatment variables on the structure of a uranium - 3.96 wt% silicon alloy have been studied using optical microscopy, quantitative metallography and hardness determinations.

It has been shown that an optimum temperature exists below the peritectoid temperature where the maximum amount of transformation to U_3Si occurs in a given period of time. The time required to fully transform an as-cast alloy at this optimum temperature is affected by the size of the primary U_3Si_2 dendrites. With a U_3Si_2 particle size of <12 μm complete transformation can be achieved in four hours.

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1. INTRODUCTION

Although the phase diagram of the uranium-silicon system and the crystal structure of the various phases have been fairly well established[1,2], there is very little information available on the metallurgical structure of uranium-silicon alloys. The emergence of the δ phase (U_3Si) as a possible nuclear fuel[3] has led to an increased interest in the structure of alloys with compositions close to that of U_3Si. Of particular interest are the effects of fabrication and heat treatment variables on the structure. This report describes the results of some experiments designed to study the effect of some of these variables on the alloy structure, and discusses the use of hardness determinations as a means of following the peritectoid transformation to U_3Si.

2. REVIEW

2.1 Phase Diagram

The original phase diagram for the uranium-silicon system was determined, in 1944, by Kaufmann et al.[1]. The crystal structures and the unit cell parameter of the phases observed by Kaufmann et al.were determined, in 1949, by Zachariasen[2]. The phase diagram determined by Kaufmann et al. was amended slightly, in 1965, by Blum et al.[4]. The uranium rich portion of the revised version is given in Figure 1.

2.2 Crystal Structure U_3Si

The crystal structure of the δ phase U_3Si was determined by Zachariasen[2]. Using the powdered crystal method Zachariasen

obtained diffraction data for the δ phase, which Kaufmann et al. had designated $U_{10}Si_3$. Zachariasen found that the data corresponded to a body-centered tetragonal translation lattice with unit cell dimensions of:

$$a = 6.029 \pm 0.002 \text{ Å}, \quad c = 8.697 \pm 0.003 \text{ Å}$$

and decided that the formula $U_{10}Si_3$ was not compatible with the diffraction data obtained. Zachariasen's unit cell had a volume of 316.03 $(Å)^3$ and because the extinctions he observed required the number of atoms per unit cell to be a multiple of four, he concluded that the formula should be U_3Si with four stoichiometric molecular units per unit cell. The structure of U_3Si as determined by Zachariasen and viewed along the fourfold axis of symmetry is given in Figure 2. The atom positions in a unit cell are given in Figure 3, and it can be seen that the body centered tetragonal structure of U_3Si is pseudocubic (Cu_3Au type). The calculated density for the U_3Si structure is $\rho = 15.58$ g/cm^3, and the stoichiometric composition is uranium-3.78 wt% Si.

Blum et al.[4], in 1965, observed that an allotropic transformation from tetragonal to cubic occurred in U_3Si, on heating, at 765°C. They found the cubic phase to be of the type Cu_3AuI with a lattice parameter a = 4.346 Å at 780°C. This phase, which they designated δ' (U_3Si) is stable to about 930°C, the peritectoid decomposition temperature of the U_3Si. Blum et al. found that not even the severest quench allowed the δ' to be retained at room temperature.

2.3 Micro-Structure of Alloys

U_3Si is formed by a peritectoid reaction at 930°C between γ uranium and the ∈ phase U_3Si_2[1]. This reaction is extremely sluggish and the structure of an as-cast alloy usually contains only a little U_3Si, even when slowly cooled. During cooling from the molten state to room temperature, a thin rim of U_3Si forms around the primary U_3Si_2 particles. The small U_3Si_2 particles in the eutectic matrix nearly all transform completely to U_3Si. A typical as-cast structure is shown in Figure 4.

The peritectoid reaction is normally activated by prolonged heating at a temperature 100-150°C below the peritectoid temperature. As in the case of many eutectoid transformations, the most rapid rate of transformation occurs at some degree of undercooling below the equilibrium temperature. Compositional variations such as the carbon concentration may also

affect the degree of undercooling required[5]. After an anneal of several days at a suitable temperature the structure of an alloy close to the U_3Si composition consists of a U_3Si matrix with small particles of U_3Si_2 or free uranium, depending on which side of the true U_3Si composition the alloy composition lies. A typical heat treated structure is shown in Figure 5.

Ambler[6], by careful polishing and etching of a sample of U_3Si, has shown that each U_3Si grain is made up of parallel bands which in turn contain fine sub-bands (Figure 6). He points out that identical structures are found in the systems indium-thallium, indium-cadmium, copper-manganese and chromium-manganese, which undergo martensitic transformations from face centered cubic to face centered tetragonal. By analogy with these systems, Ambler suggests that the bands seen in U_3Si could be the result of a martensitic transformation.

However, tests at CRNL on specimens transformed at temperatures below the f.c.c. → b.c.t. transition temperature reveal that the bands are still present. This suggests that they are not related to the f.c.c. → b.c.t. transition and may not be the result of a martensitic transformation. A separate hypothesis is that they are annealing twins which form as the U_3Si grains are growing.

2.4 The Composition of U_3Si

Considerable doubt still exists as to the true composition of U_3Si. The stoichiometric composition would be 3.78 wt% silicon but two workers have reported different values.

Kaufmann et al.[1] used the physical properties, hardness and density, to determine the composition. They measured the change in properties on fully transforming cast alloys with different silicon concentrations, reasoning that the maximum change in both hardness and density should occur at the U_3Si composition. From their results, Kaufmann et al. concluded that the composition of U_3Si was ~3.4 wt% silicon.

Isserow[7] on the other hand, reasoned that the alloy with the highest corrosion resistance should correspond to the U_3Si composition. He measured the corrosion resistance

of a number of alloys and found that the highest corrosion resistance was obtained with an alloy containing 3.97 wt% silicon.

3. AIMS OF THE PRESENT STUDY

As outlined in the preceding review, there is some information on the uranium-silicon phase diagram, the crystal structure of the phases and the micro-structure of a typical as-cast and transformed δ phase alloy. There is, however, no published information on the effect of casting and heat treatment variables on these structures.

Fabrication and heat treatment practices for producing transformed δ phase alloys have tended to be chosen empirically, with the result that in many cases it was very difficult to obtain complete transformation to U_3Si. The aim of this present study, therefore, was to investigate the effects of some of the variables encountered during casting and heat treatment, in order to determine the optimum conditions for obtaining fully transformed δ phase alloys.

Because of the wide differences between the experimentally determined values for the silicon concentration in U_3Si, a separate study has been conducted in an attempt to define the composition of U_3Si more precisely[8].

4. EXPERIMENTAL

4.1 Preparation of Specimens

The material used in this work contained 3.96 wt% silicon and 345 ppm carbon, and was supplied by Eldorado Nuclear Limited. Full analysis of the material is given in Table 1.

The alloy was vacuum induction melted and cast into ceramic moulds[9] in the form of rods 16 mm diameter. Slices, approximately 3 mm thick, were cut from the rods, and these were then cut into quarters to provide specimens for heat treatment. The specimens were tested, both in the as-cast condition and after a homogenizing treatment of three days at 960°C, followed by quenching into cold water. The structure of as-cast and homogenized specimens are shown in

Table 1

Analysis of U_3Si Alloy Heat 192

Silicon	3.96 wt%
Carbon	345 ppm
Ag	---
Al	300 ppm
As	---
B	0.1 ppm
Ba	---
Be	---
Bi	---
Ca	30 ppm
Cd	<0.1 ppm
Cr	30 ppm
Cu	15 ppm
Fe	300 ppm
Hf	2* ppm
K	---
Mg	
Mn	25 ppm
Mo	5 ppm
Na	---
Nb	1 ppm
Ni	75 ppm
Pb	---
Pd	---
Sb	---
Sn	5 ppm
Ti	75 ppm
V	10 ppm
W	---
Y	---
Zn	60 ppm
Zr	60* ppm

Code: (-) Looked for but not detected. Limit of detection - 1 ppm, except for B, Cd.

*Minimum values, as considerable losses could occur during sample preparation.

Figures 4 and 7. The homogenizing treatment caused coalescence of the primary U_3Si_2 particles and eliminated all of the small particles of U_3Si_2 from the eutectic matrix.

4.2 Heat Treatments

Specimens were sealed in quartz capsules under a vacuum of 5×10^{-6} Torr and given the following treatments:

a) Specimens were heat treated for a constant period, 72 hours, at temperatures between 700 and 900°C, to study the effect of heat treatment temperature on the transformation to the δ phase U_3Si.

b) In order to study the effect of time at temperature on the extent of transformation, specimens were heat treated for one to 144 hours at temperatures of 800 and 820°C and quenched into water, the capsules being broken during the quench.

c) The decomposition temperature of U_3Si was studied by annealing heat treated specimens (130 hours at 800°C) for a further one hour at temperatures between 700 and 950°C.

The effect of annealing time on the decomposition of U_3Si was studied by annealing similar specimens, for periods from one to 120 hours at 840 and 850°C.

d) In order to study the effect of cooling rate during casting from the melt, two rods of U_3Si were remelted and allowed to cool under different conditions. One rod was remelted under vacuum, in a thoria crucible and allowed to cool at a rate of ~1°C/min. A second rod was remelted and cast, under vacuum, onto a water cooled copper hearth. Samples of both castings were heat treated for one to 144 hours at a temperature of 800°C.

e) The effect of cooling rate after transformation was examined by heat treating three as-cast specimens for 72 hours at 800°C and cooling them at different rates. One specimen was furnace cooled in its capsule, a second was cooled in air in its capsule and the third was quenched into water, the capsule being

broken during the quench. An approximate estimate of the cooling rates was 2°C/min, 50°C/min and 3×10^4°C/min respectively.

4.3 Examination

All specimens obtained from the above experiments were examined by hardness measurements and optical microscopy. Hardness determinations were made on a metallographically polished cross section of the specimens using a 30 kg load on a Vickers pyramid diamond indentor. Optical microscopy was carried out on specimens prepared according to the methods outlined by Ambler (see Appendix 1).

The amount of U_3Si_2 was determined in certain specimens using a Quantitative Television Microscope. This instrument is capable of distinguishing selected features in an image and performing a range of measurements on these features, e.g. the number, area, average size and size distribution.

5. RESULTS AND DISCUSSION

5.1 Effect of Heat Treatment Temperature

Hardness determinations on as-cast and homogenized samples heat treated for 72 hours at temperatures between 700 and 900°C are plotted against temperatures in Figure 8. Minima occur in both curves; at a temperature of 800°C for the as-cast specimens and at 820°C for the homogenized specimens. Metallographic examination showed that the minima in the curves occurred at the temperature at which the maximum amount of transformation to U_3Si had taken place; no free uranium remained in the structure of either as-cast or homogenized specimens heat treated at their optimum temperature, (Figure 5), whereas free uranium was present in specimens heat treated at temperatures both above and below the optimum, (Figures 9 and 10).

The amount of U_3Si_2 remaining in the structures of samples after heat treatment was determined using the Quantitative Television Microscope. Normally the amount of U_3Si being

formed would be the best parameter for following the peritectoid transformation, but in alloys containing excess U_3Si_2 (i.e. hyperstoichiometric alloys) the decrease in U_3Si_2 can be utilized. Measurements of U_3Si_2 content are also more convenient, since, when plotted against time and temperature, they follow the transformation in a similar manner to those of hardness. The amounts of U_3Si_2 remaining in both as-cast and homogenized samples after the 72 hour treatment are plotted against temperature in Figure 11.

It can be seen from Figures 8 and 11 that the curves of hardness and volume % U_3Si_2 versus temperature are very similar. The minimum amount of U_3Si_2 occurs at the same temperatures as the minimum hardness, 800°C in the case of the as-cast material and 820°C in the case of homogenized. This is further evidence that the maximum amount of transformation has occurred at these particular temperatures.

There appears to be a slight difference in the homogenized samples; the optimum transformation temperature for the homogenized material being about 20°C above that for the as-cast material. This however is probably due to the slightly larger U_3Si_2 particle size in the homogenized specimens.

5.2 Effect of Time at Temperature

Hardness determinations on specimens heat treated for one to 144 hours, at 800°C in the case of the as-cast material, and 820°C in the case of homogenized material, are plotted against time in Figure 12. For both materials the curves level off after ~48 hours, suggesting that transformation is essentially complete. Metallographic examination of the specimens showed that after 48 hours at 800 and 820°C respectively there were no areas of free uranium visible in the structures of either the as-cast or homogenized specimens.

The amount of U_3Si_2 remaining in both as-cast and homogenized samples after heat treatment are plotted against time in Figure 13. The curve for as-cast material follows a similar pattern to that of hardness. The amount of U_3Si_2 levels off after approximately 50 hours, although there is a continuous slight decline up to 144 hours. Assuming that U_3Si is stoichiometric, the amount of U_3Si_2 present after 144 hours (8-8.5 vol%) is still above the equilibrium

amount (6.5 vol%) for an alloy of this composition (3.96 wt% Si), suggesting that the material is approaching equilibrium only very slowly.

In the case of the homogenized material, leveling off of the amount of U_3Si_2 does not occur until approximately 80 hours although by this time the amount of residual U_3Si_2 is close to the equilibrium amount.

The amount of U_3Si_2 present in the respective samples explains why the homogenized material has a lower hardness than the as-cast material after approximately 48 hours at their respective optimum temperatures.

The rate of transformation up to 48 hours is essentially the same in both sets of samples if allowance is made for the amount of U_3Si already present in as-cast samples prior to heat treatment.

5.3 Decomposition of U_3Si

Hardness determinations on heat treated specimens subsequently annealed at temperatures between 700 and 930°C, are plotted against temperature in Figure 14. It can be seen that the hardness begins to increase in samples annealed above 850°C. Metallographic examination revealed that this increase in hardness coincided with the appearance of free uranium at the U_3Si/U_3Si_2 interface (Figure 15). This free uranium was not present in samples annealed for one hour at 840°C.

Increasing the time at the decomposition temperature did not markedly affect the hardness of the alloy. The hardness did not increase significantly after six hours at 840, but did increase from 255 VPN after one hour at 850°C to 268 VPN after six hours. There was no significant change in hardness between six and 120 hours at 850°C. Examination of the structures showed that decomposition had not occurred at 840°C even after six hours, but, at 850°C, slightly more free uranium was present at the U_3Si/U_3Si_2 interface after six hours than after one hour. There did not appear to be any increase in the amount of uranium formed between six and 120 hours at 850°C. It was concluded from these results that the onset of decomposition occurred very close to 850°C and that a 3-phase region exists below the peritectoid temperature with a lower limit, for this particular alloy, of 850°C.

Both partially transformed and partially decomposed structures, will have hardnesses above the minimum obtained on a fully transformed structure. There is, however, a distinct difference in the structures obtained. In partially transformed alloys (i.e. Figure 10) a layer of U_3Si normally separates U_3Si_2 from free uranium, whereas in partially decomposed alloys (Figure 16) free uranium separates U_3Si from U_3Si_2. It is therefore possible to determine whether alloys are partially transformed or partially decomposed by metallographic examination, but not using hardness determinations.

5.4 Effect of Cooling Rate

5.4.1 During Casting

The structure of the alloy cast into a thoria crucible and cooled at a rate of ~1°C/min is shown in Figure 17. Massive U_3Si_2 dendrites were formed with an average width of approximately 900 μm, although dendrites as large as 1500 μm were observed in some areas. Each of the massive primary dendrites of U_3Si_2 was surrounded by a rim of U_3Si and the small U_3Si_2 particles in the eutectic had transformed to U_3Si during cooling.

In the alloy cast onto a water cooled copper hearth, the U_3Si_2 particles were extremely small, <15 μm (Figure 18), and it was difficult to differentiate between the primary U_3Si_2 dendrites and the U_3Si_2 particles in the eutectic.

Subsequent heat treatment showed that the slowly cooled alloy was only partially transformed even after 144 hours at the optimum transformation temperature of 800°C (Figure 19). The rapidly cooled alloy, however, was fully transformed after only four hours at 800°C, (Figure 20).

It was not possible to study the rate of transformation of the slowly cooled alloy using hardness determinations because of the extreme brittleness of the large U_3Si_2 particles. These particles fractured when a load was applied by means of the macro hardness indentor. The rate of transformation of the rapidly cooled alloy is shown in Figure 21. Even though metallographic examination of the alloy indicated complete transformation after four hours at 800°C, the hardness continued to drop as the time at temperature was increased. This suggests that transformation

is not necessarily complete when free uranium is no longer visible in the micro-structure, but may continue extremely slowly for a considerable period of time.

It is evident from these results that the cooling rate during casting, and the resultant U_3Si_2 particle size, has a significant effect on the time required to fully transform an as-cast alloy of U_3Si.

A more comprehensive study of the effect of U_3Si_2 particle size on transformation is the subject of a separate report[10]

5.4.2 After Transformation

The rate of cooling after transformation did not affect the structure or hardness of the three samples cooled at different rates. The hardness figures were almost identical and the banded structure observed by Ambler[6] was present irrespective of the rate of cooling.

5.5 Effect of Homogenization

Examination of Figure 8 shows that the optimum temperature for transformation is increased from 800 to 820°C if the as-cast material is homogenized at 960°C prior to carrying out the transformation. The only visible effect of the homogenizing treatment is to cause coalescence of the primary U_3Si_2 particles and the small U_3Si_2 particles in the eutectic matrix. This effectively increases the size of the primary U_3Si_2 particles and would, therefore, be expected to decrease the amount of transformation in a given period of time, compared to as-cast material. The only known factor which affects the optimum transformation temperature is carbon[5], and since the carbon contents of the as-cast and homogenized samples were the same (~350 ppm), there is no obvious explanation for the behaviour noted here.

5.6 The Use of Hardness Determinations

From the results of this work it appears that there is a good correlation between macro hardness and the amounts of the various phases present in alloys close to the U_3Si composition. Thus, only heat treatments which can vary the amounts of the different phases have a significant effect on the hardness of the alloys.

The micro hardnesses of the various phases which are present in alloys containing less than 40 at% Si (the composition of U_3Si_2) are:

U_3Si_2	600-700 VPN
U_3Si	180-200 VPN
α-uranium	280-400 VPN

Because of their differences in hardness it is possible to follow the peritectoid transformation to U_3Si using hardness as the criterion. As-cast alloys, containing the maximum amounts of U_3Si_2 and uranium, are the hardest alloys, whereas fully transformed alloys, containing the maximum amount of U_3Si, are the softest.

The criterion of hardness can, however, only be used consistently to follow the transformation when the amounts of the various phases are undergoing a significant change. Hardness may or may not detect the very small changes which occur as the transformation nears completion.

Hardness figures alone will not necessarily establish the condition of a particular alloy or the heat treatment it has received. Comparison of Figures 8 and 14 show that alloys which have been partially transformed may have the same hardness as fully transformed alloys which have begun to decompose, although their structures are quite different (see section 5.3).

6. CONCLUSIONS

1) An optimum transformation temperature exists below the peritectoid temperature, at which the maximum amount of transformation to U_3Si can be achieved in a given period of time.

   For the particular alloy studied the optimum temperature is 800°C for as-cast material, and 820°C for material homogenized for 72 hours at 960°C and rapidly quenched.

2) Complete transformation, estimated by reference to Kaufmann's equilibrium diagram for material containing 100 ppm carbon, was achieved in the homogenized material by heat treatment for 80 hours at 820°C. Heat treatment of the as-cast material at 800°C, however, did not bring about complete transformation in 144 hours.

3) Decomposition of the U_3Si phase began in this alloy, on heating, at 850°C. Only a small amount of decomposition occurs in periods up to 120 hours at this temperature, suggesting that a 3 phase field exists at temperatures over about 850°C.

4) Cooling rate during casting controls the size of the primary U_3Si_2 dendrites. Increasing the cooling rate reduces the dendrite size, and hence decreases the time needed to bring about complete transformation to U_3Si.

5) Cooling rate after transformation has no effect on the structure of the alloy.

6) Hardness determinations are a valid means of following the transformation to U_3Si provided the relative amounts of the phases present are undergoing a significant change.

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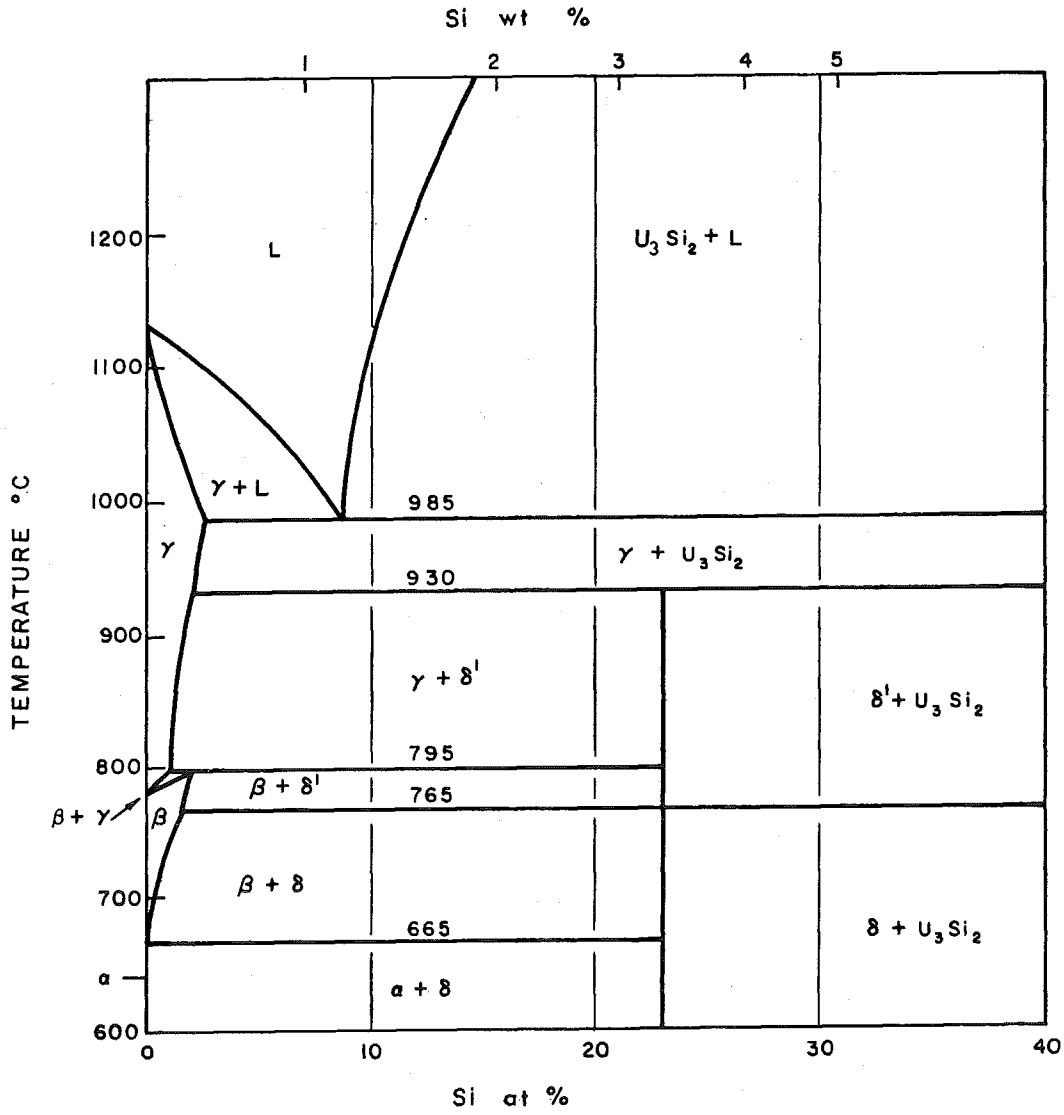


Fig. 1. Uranium-silicon phase diagram [4]).

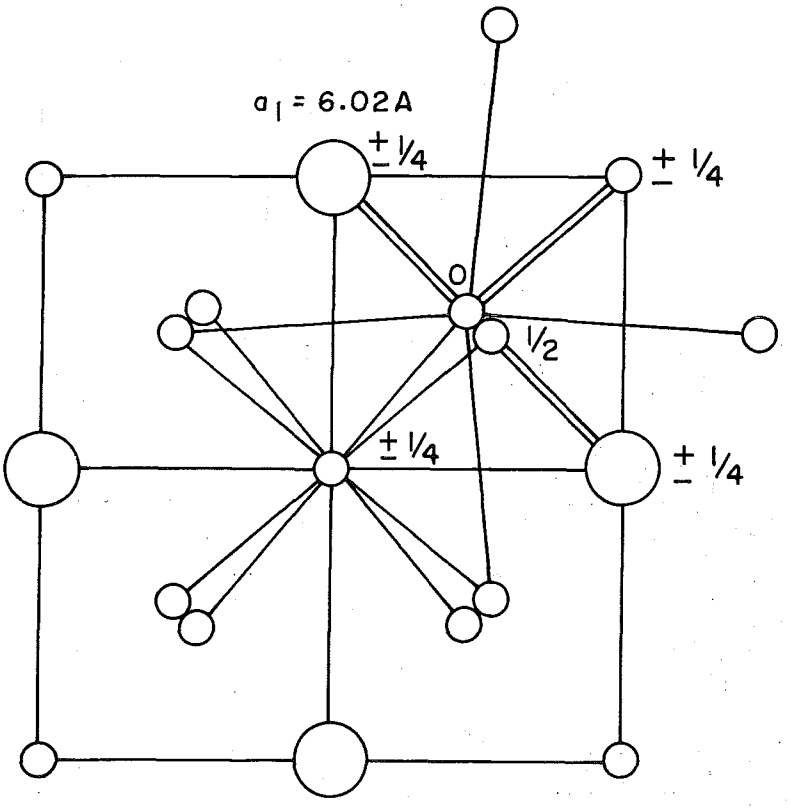


FIGURE 2

THE U_3Si STRUCTURE VIEWED ALONG THE FOUR-FOLD AXIS.
LARGE CIRCLES – Si ATOMS
SMALL CIRCLES – URANIUM ATOMS
ACCORDING TO ZACHARIASEN (1949).

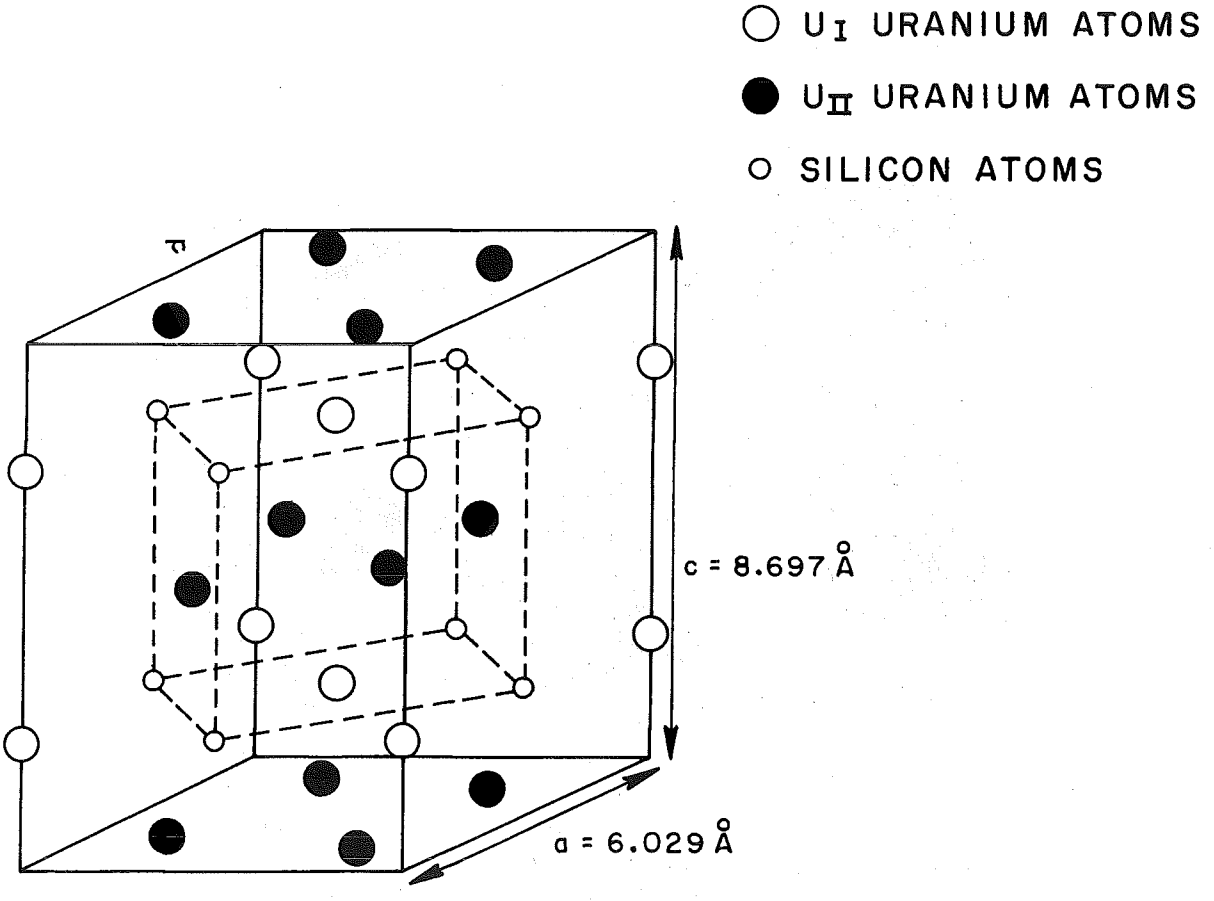


FIGURE 3

THE BODY CENTERED TETRAGONAL STRUCTURE OF δU_3Si. THE BROKEN LINE LATTICE REPRESENTS THE FACE CENTERED CUBIC Cu_3Au TYPE STRUCTURE OF δ' U_3Si, PRODUCED WHEN $c/a = \sqrt{2}$ AND THE U_{II} ATOMS ARE AT 1/4 AND 3/4 THE WAY ALONG THE DIAGONALS OF THE (001) AND (002) PLANES OF THE BODY CENTERED TETRAGONAL LATTICE.

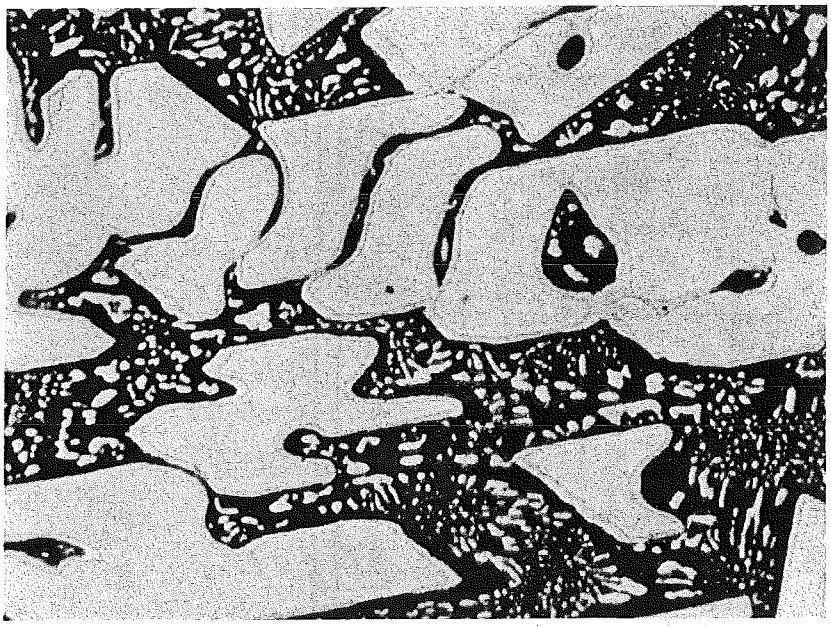


Figure 4 1000X
Met J1-N2 Typical As-Cast U_3Si Structure. Large white particles - U_3Si_2. Black - uranium. Grey rim around large white particles and small grey particles in uranium matrix - U_3Si.

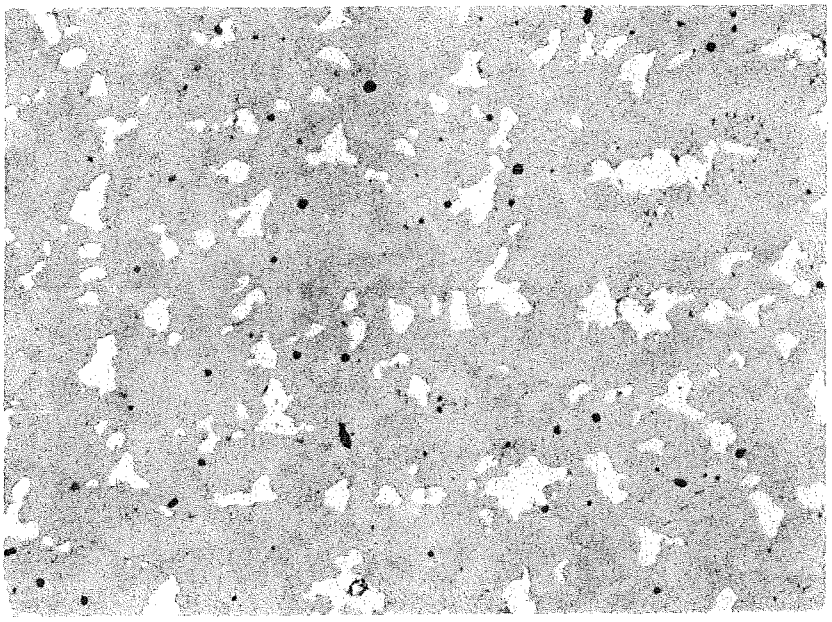


Figure 5 250X
Met J86-L1 Typical Heat Treated Structure After 72 Hours at 800°C. White - U_3Si_2. Grey - U_3Si. Dark spots - UO_2.

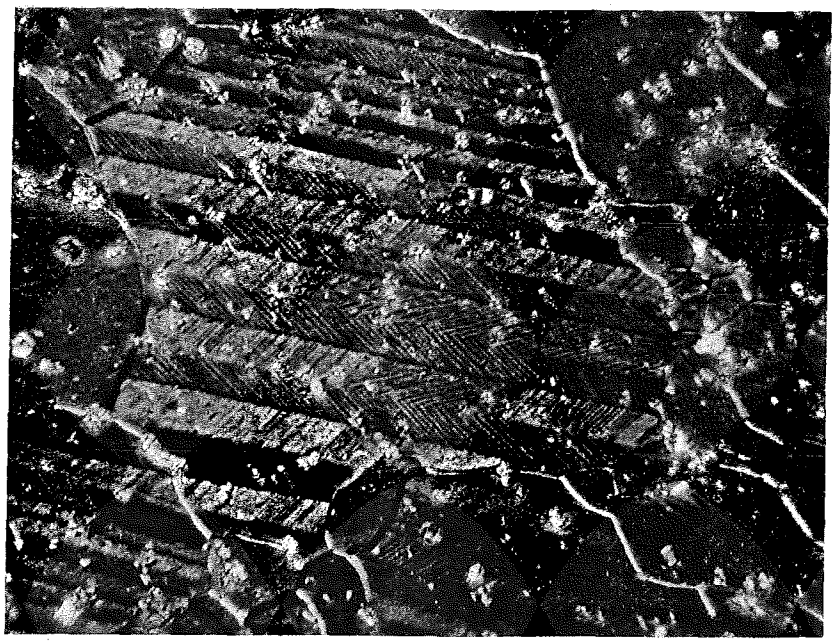


Figure 6 1000X
Met I141-A5 Bands and Subbands in Grains of U_3Si Attack Polished and Chemically Etched; Polarized Light[6].

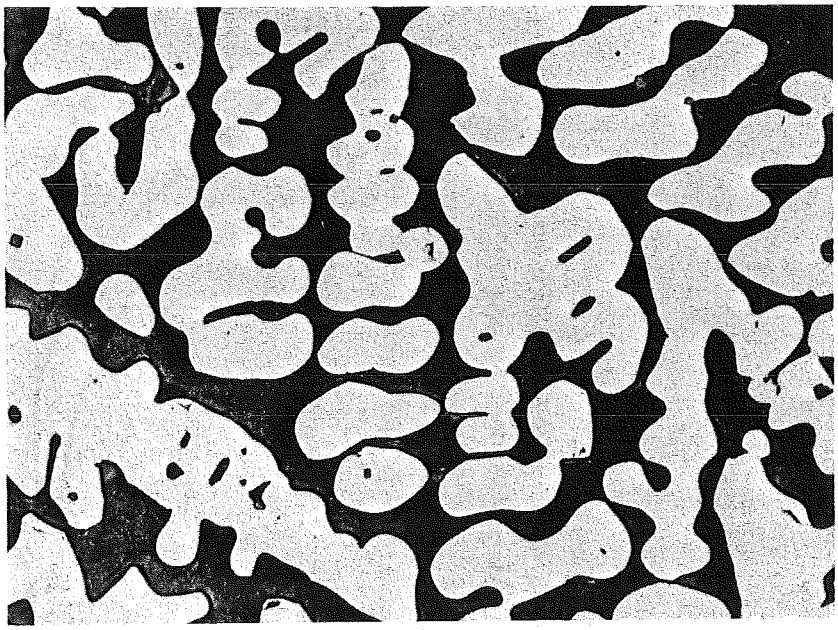


Figure 7 250X
Met JI-FF1 Heat 192, Homogenized 72 Hours at 960°C, Water Quenched.

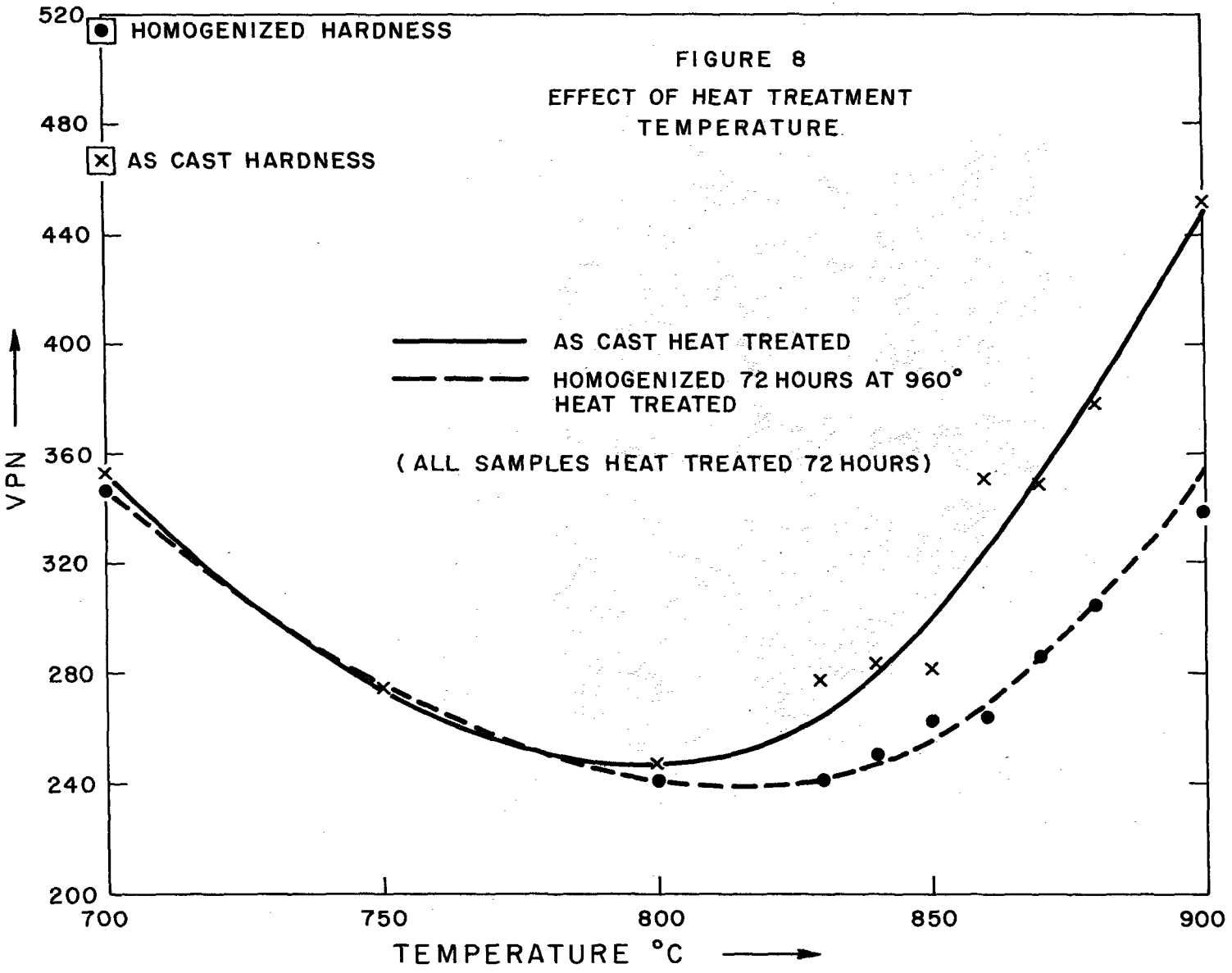


FIGURE 8

EFFECT OF HEAT TREATMENT TEMPERATURE

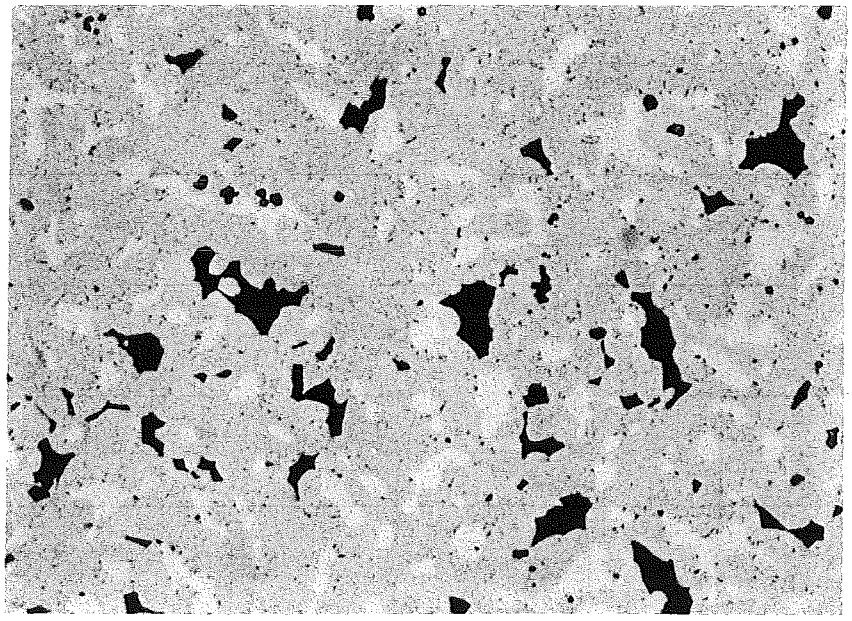


Figure 9 250X
Heat Treated 72 Hours at 830°C. White - U_3Si_2. Light grey - U_3Si. Black - uranium.

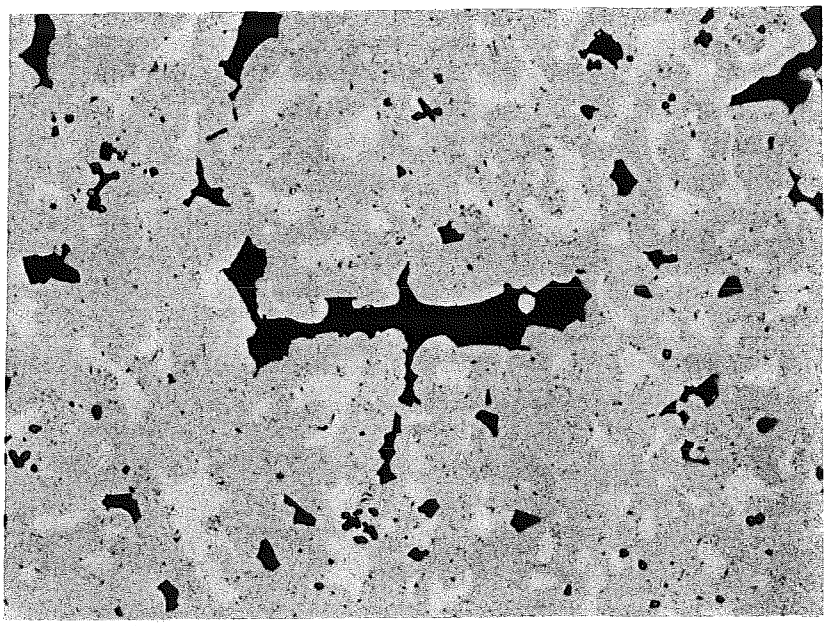


Figure 10 250X
Heat Treated 72 Hours at 750°C. White - U_3Si_2. Light grey - U_3Si. Black - uranium.

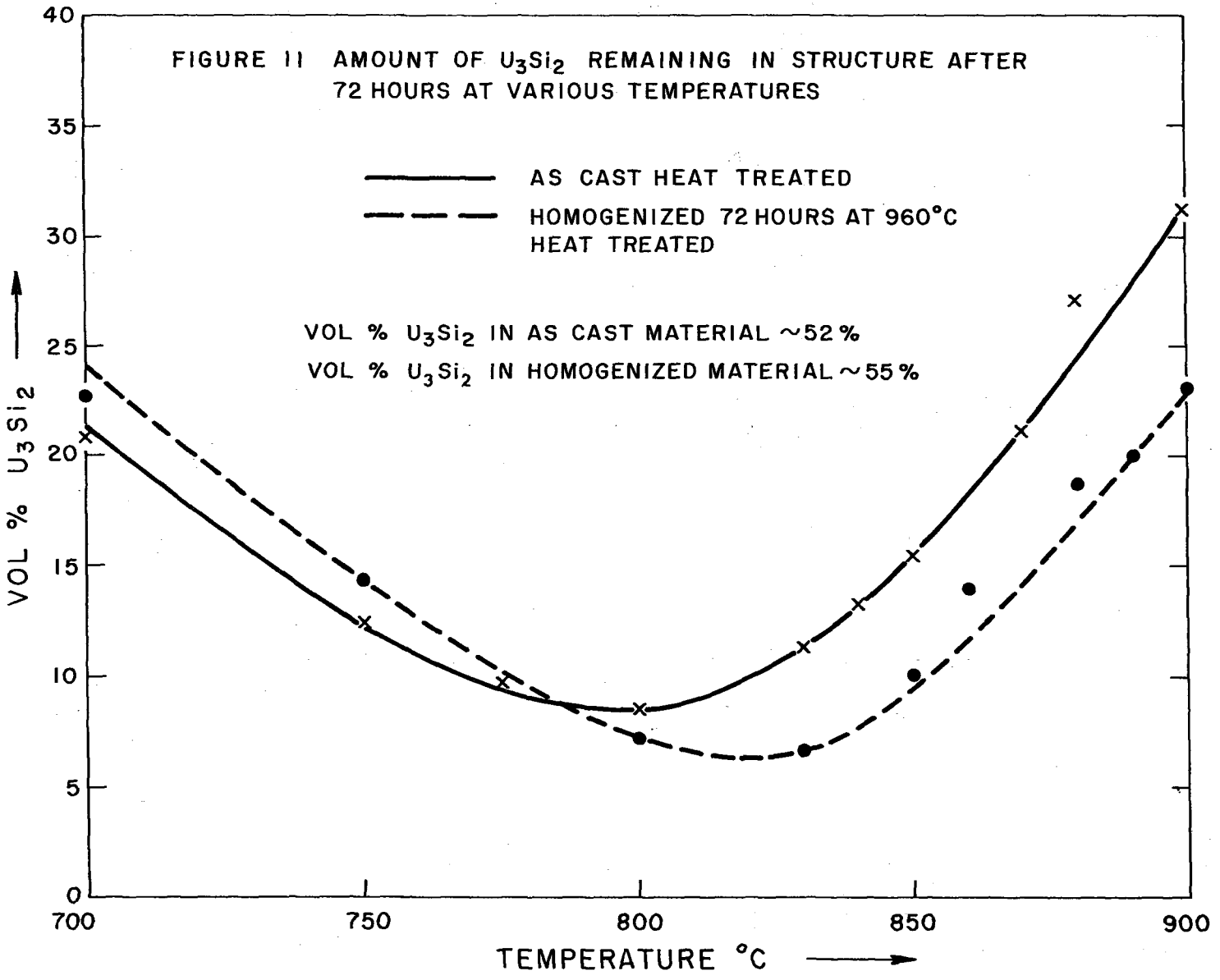


FIGURE 11 AMOUNT OF U_3Si_2 REMAINING IN STRUCTURE AFTER 72 HOURS AT VARIOUS TEMPERATURES
AS CAST HEAT TREATED
HOMOGENIZED 72 HOURS AT 960°C HEAT TREATED
VOL % U_3Si_2 IN AS CAST MATERIAL ~52 %
VOL % U_3Si_2 IN HOMOGENIZED MATERIAL ~55 %
VOL % U_3Si_2
TEMPERATURE °C
0
5
10
15
20
25
30
35
40
700
750
800
850
900

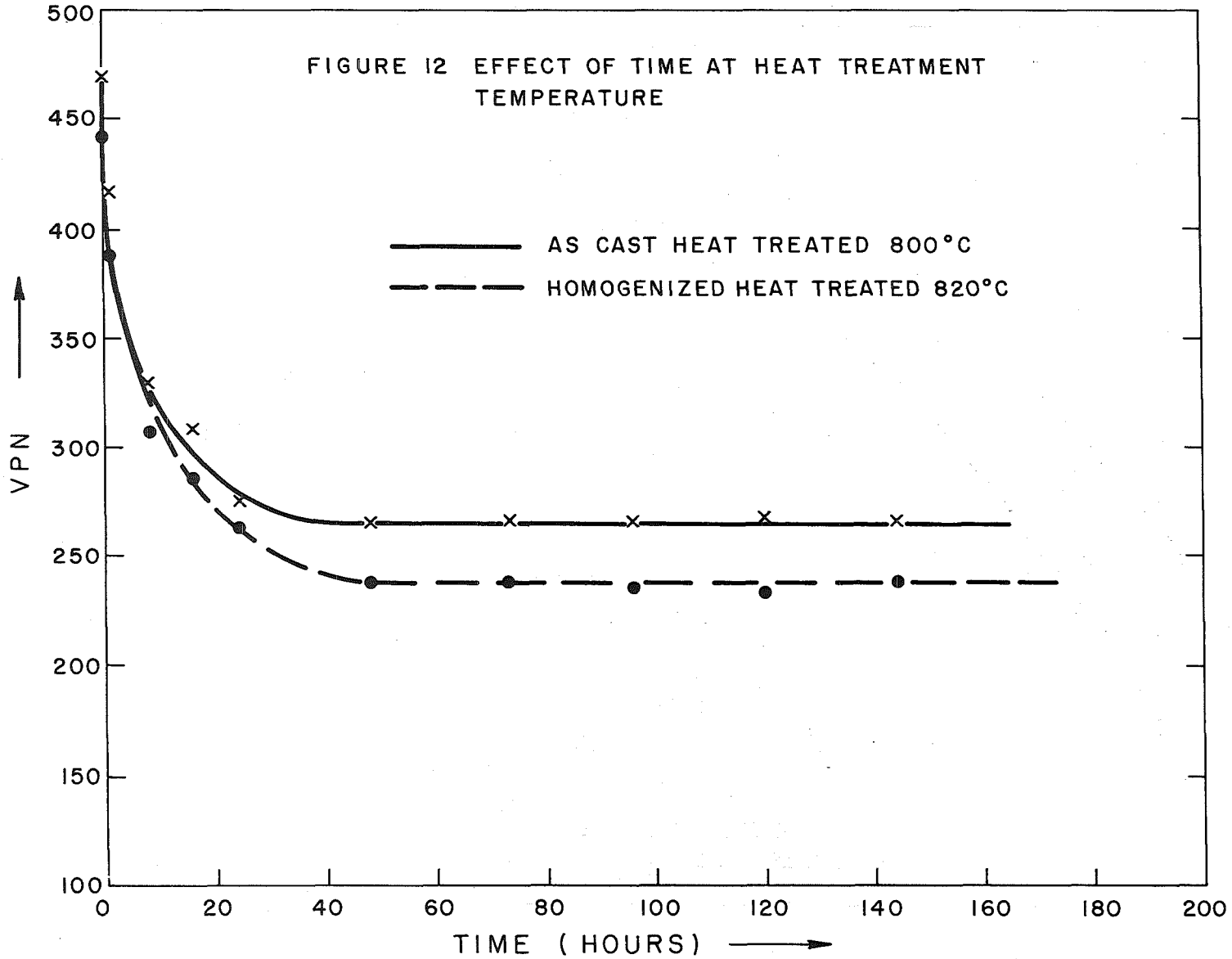


FIGURE 12 EFFECT OF TIME AT HEAT TREATMENT TEMPERATURE
AS CAST HEAT TREATED 800°C
HOMOGENIZED HEAT TREATED 820°C
VPN
500
450
400
350
300
250
200
150
100
0
20
40
60
80
100
120
140
160
180
200
TIME (HOURS)

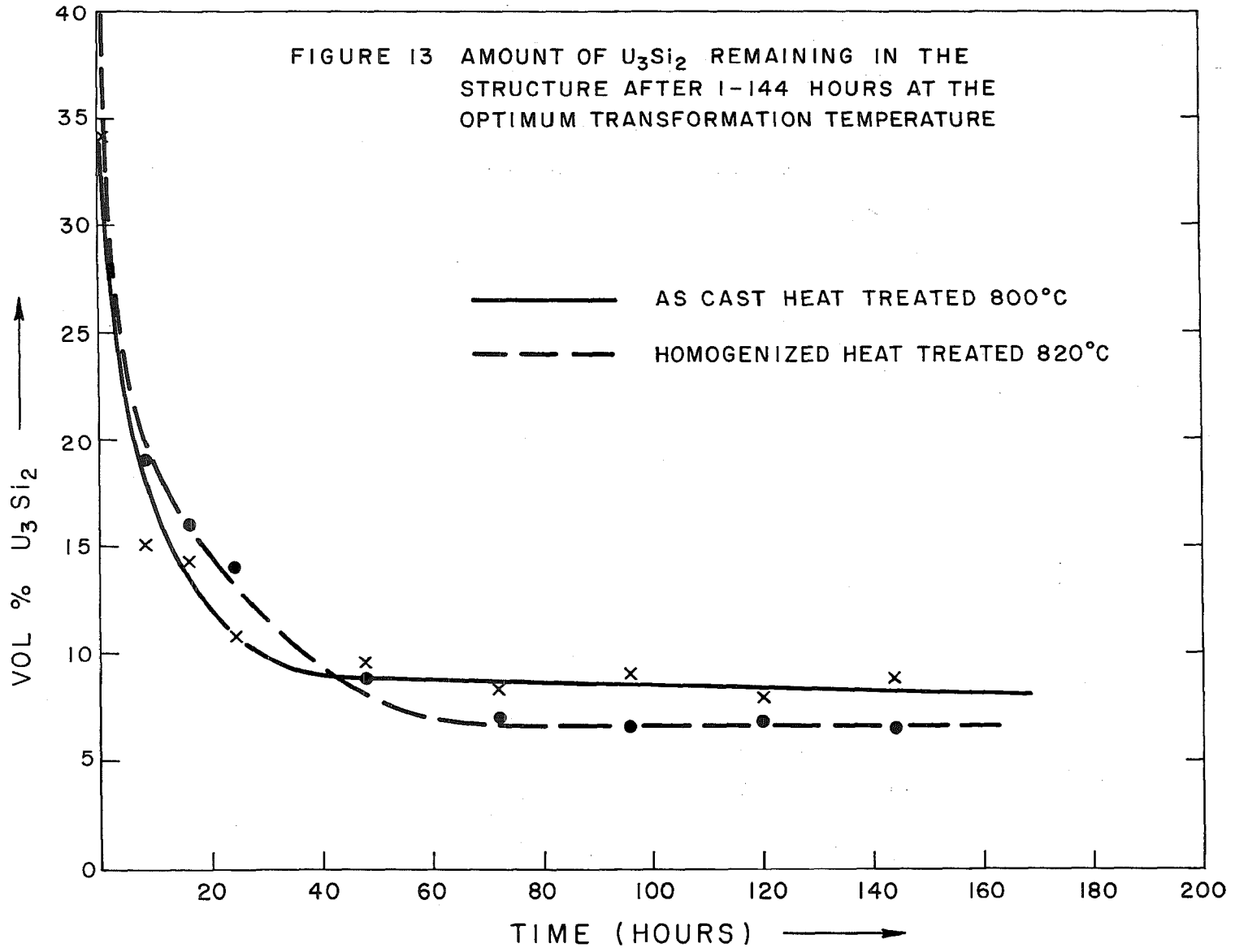


FIGURE 13 AMOUNT OF U_3Si_2 REMAINING IN THE STRUCTURE AFTER 1-144 HOURS AT THE OPTIMUM TRANSFORMATION TEMPERATURE
AS CAST HEAT TREATED 800°C
HOMOGENIZED HEAT TREATED 820°C
VOL % U_3Si_2
TIME (HOURS)
0
5
10
15
20
25
30
35
40
20
40
60
80
100
120
140
160
180
200

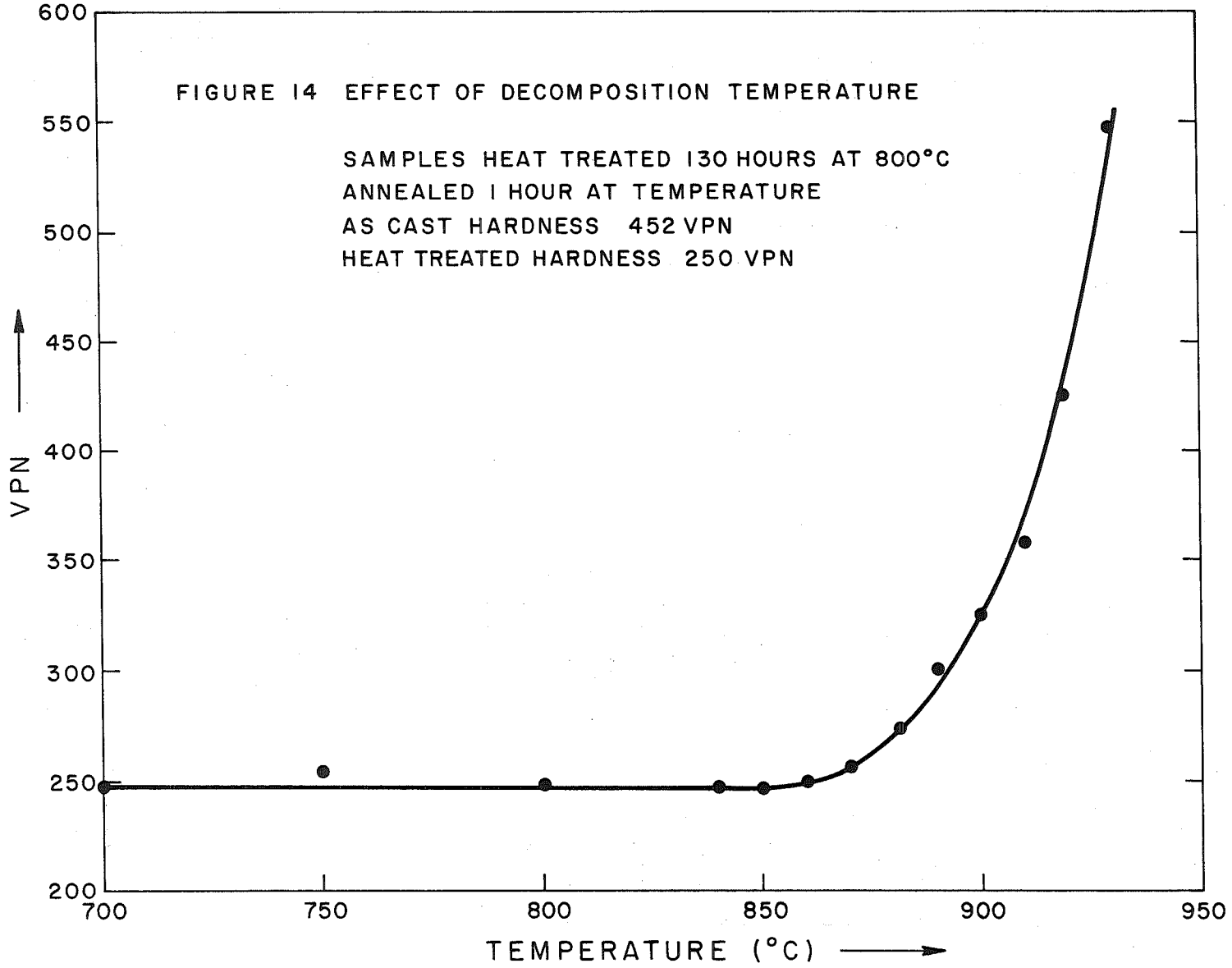


FIGURE 14 EFFECT OF DECOMPOSITION TEMPERATURE
SAMPLES HEAT TREATED 130 HOURS AT 800°C
ANNEALED 1 HOUR AT TEMPERATURE
AS CAST HARDNESS 452 VPN
HEAT TREATED HARDNESS 250 VPN
VPN
600
550
500
450
400
350
300
250
200
700
750
800
850
900
950
TEMPERATURE (°C)

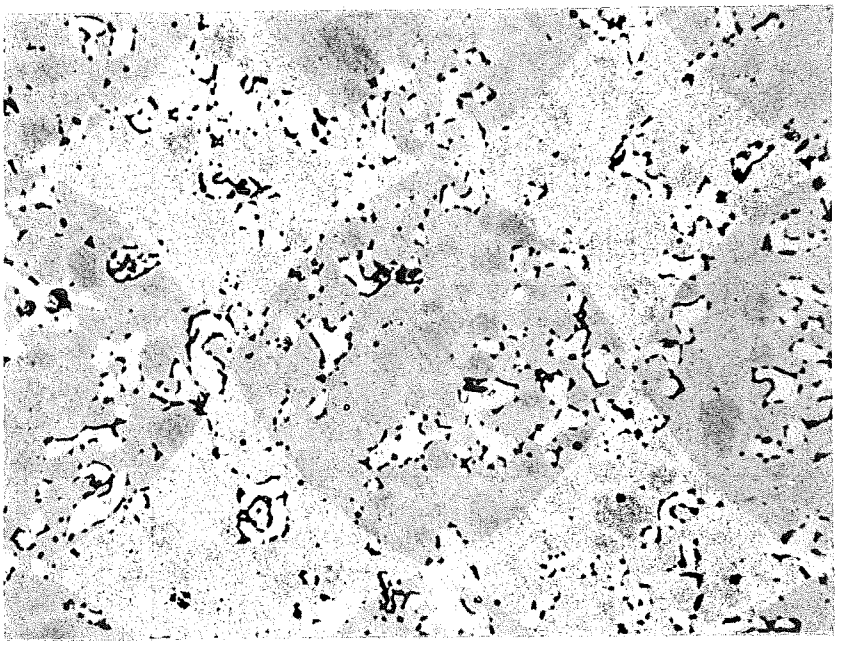


Figure 15 250X

Met I229 J1 Heat Treated 130 Hours at 800 °C, Then One Hour at 850 °C.

White - U_3Si_2

Grey - U_3Si

Black - Uranium

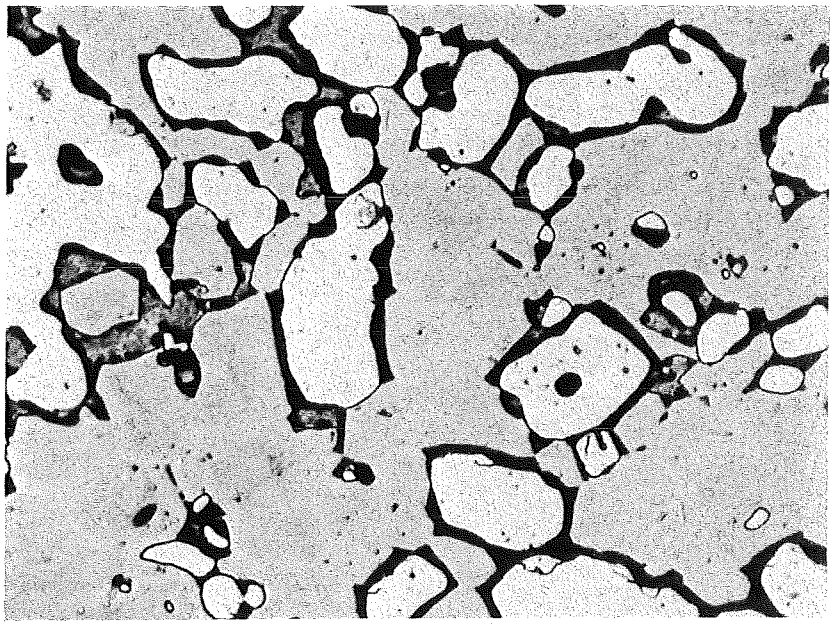


Figure 16 1000X
Heat Treated 130 Hours at 800°C. Partially decomposed one hour at 900°C. White - U_3Si_2. Black - uranium. Grey - U_3Si.

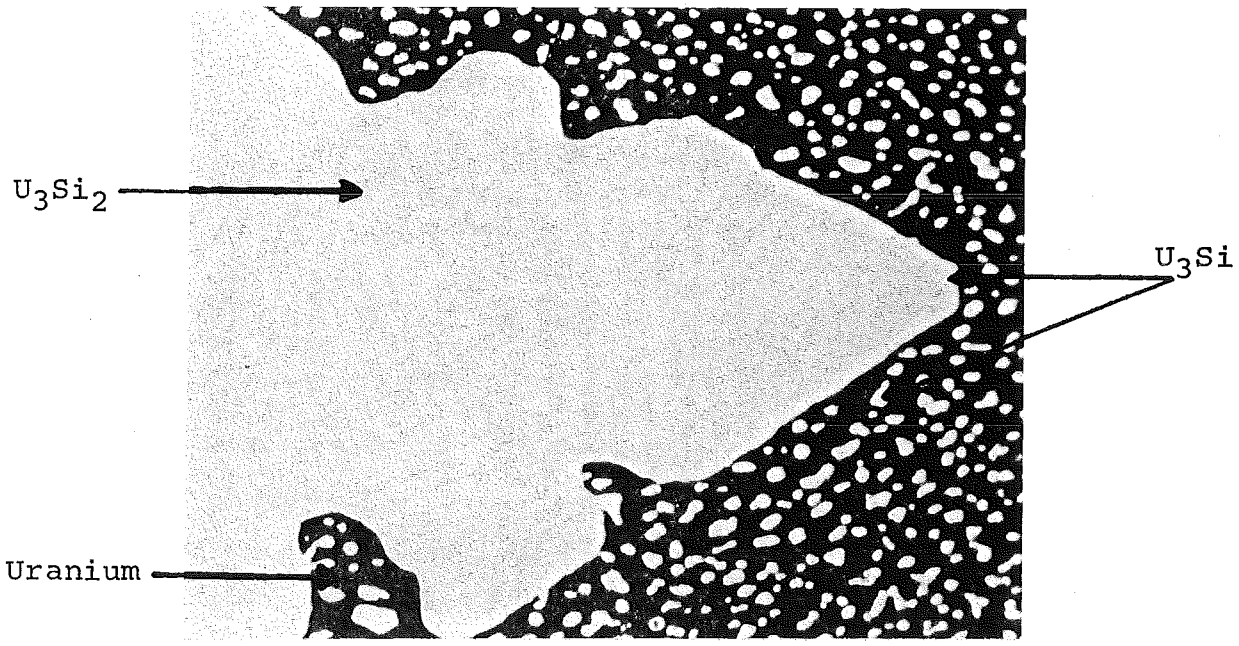


Figure 17 250X
Met J120-W1 Slowly Cooled Alloy - As-Cast
Phases as Marked.

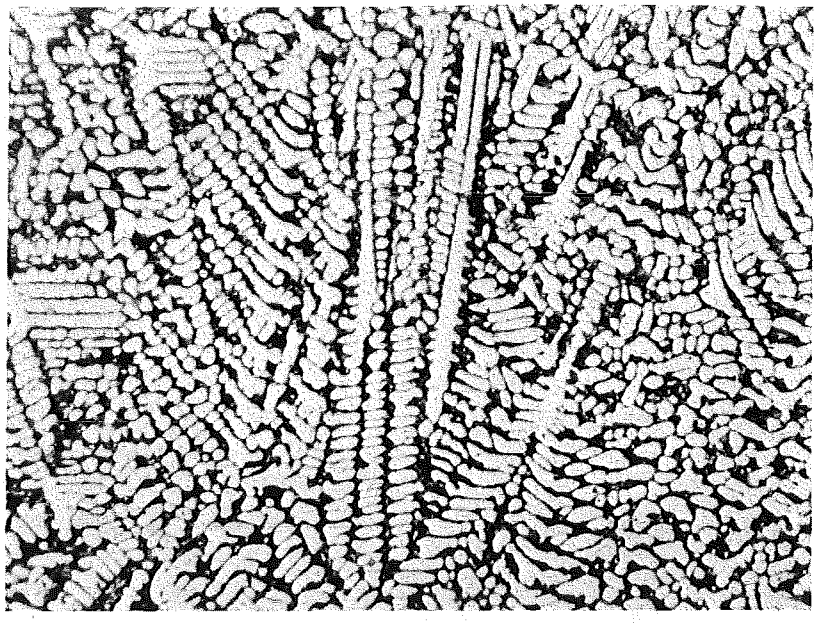
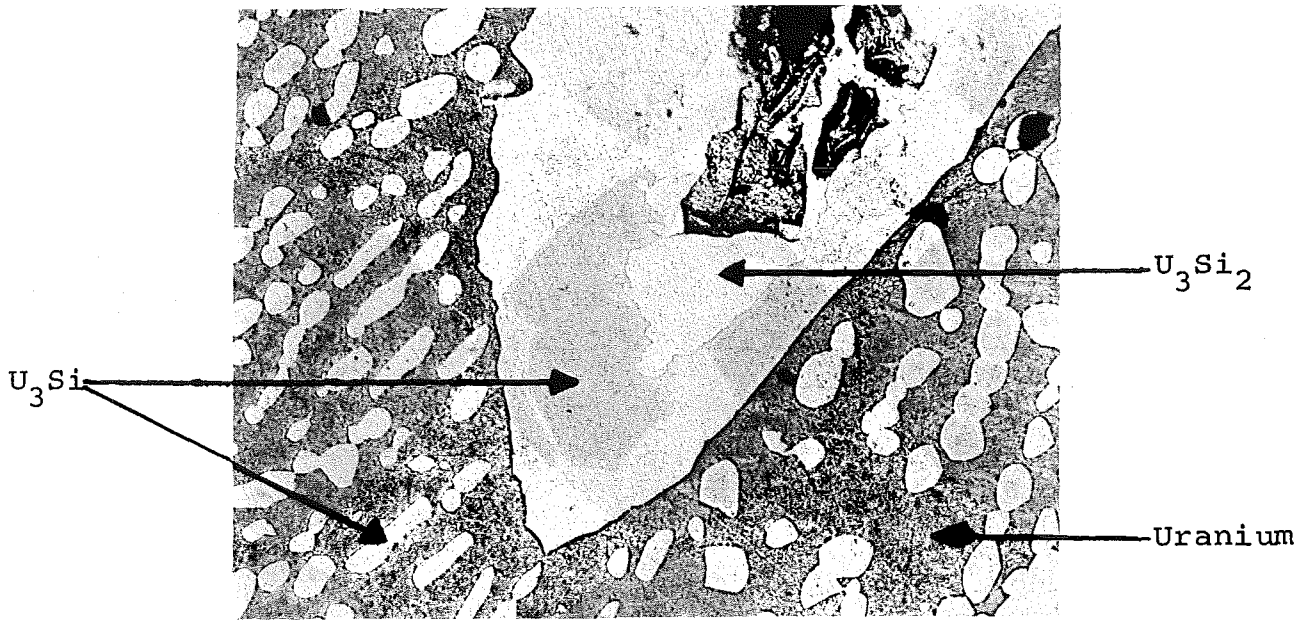


Figure 18 250X
Met J172-A5 Rapidly Cooled Alloy - As-Cast.
White - U_3Si_2. Black - uranium.



Treated 144 Hours at 800°C. The phases are as marked. The black area in the U_3Si_2 is caused by some of the very brittle U_3Si_2 phase being torn out during preparation of the samples.

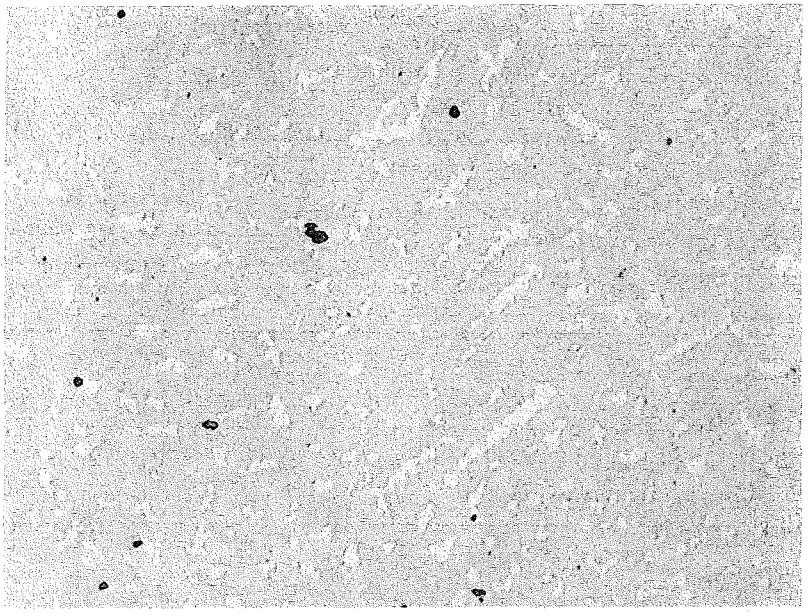


Figure 20 250X
Met J237-A3 Rapidly Cooled Alloy Heat Treated Four Hours at 800°C. White - U_3Si_2. Grey - U_3Si. Small black particles - UO_2.

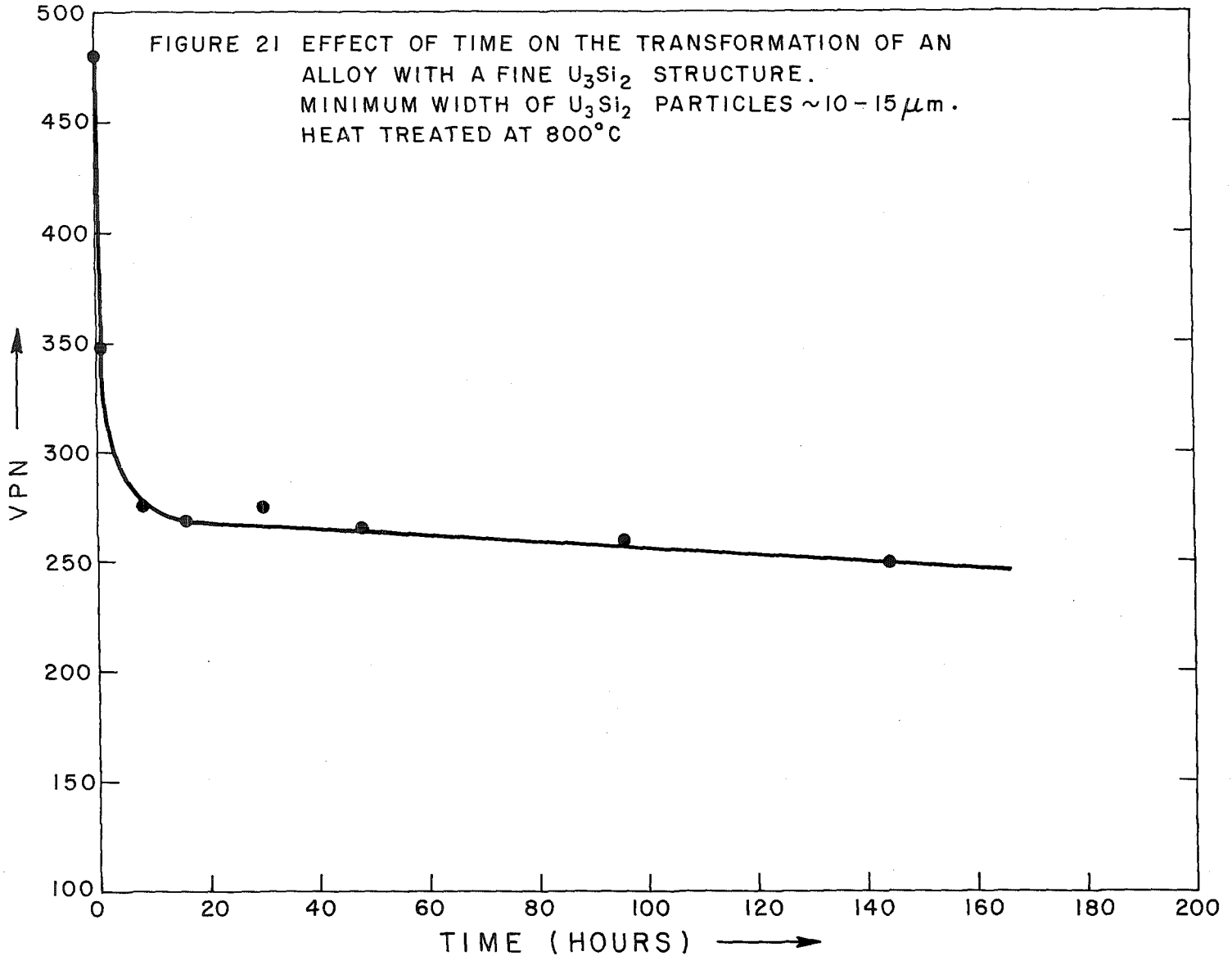


FIGURE 21 EFFECT OF TIME ON THE TRANSFORMATION OF AN ALLOY WITH A FINE U_3Si_2 STRUCTURE. MINIMUM WIDTH OF U_3Si_2 PARTICLES $\sim 10-15\mu m$. HEAT TREATED AT 800°C

APPENDIX

THE METALLOGRAPHIC POLISHING AND ETCHING OF U_3Si

J.F.R. Ambler

The following is a summary of the methods used at CRNL* for the metallographic polishing and etching of U_3Si:

1. Mounting

   Samples can be hot or cold mounted.

2. Grinding

   After Belt Grinder, 220, 320, 400 and 600 silicon carbide papers with Varsol lubrication.

3. Mechanical Polishing

   6 μm, 2 μm, 1 μm and 1/4 μm diamond cloths with Varsol lubrication. For the routine examination of as-cast specimens and for the determination of the efficiency of the "Deltizing" heat treatment, the sample can be etched with reagent 5(a) at this stage.

4. Attack Polishing

   30% Hydrogen Peroxide Solution and ignited Ammonium Dichromate for 10-20 minutes. This is necessary for the examination of "deltized" specimens using polarized light and prior to grain boundary etching in solution 5(b).

5. Etching

   a) Murakami's Reagent (10% solution of Potassium Ferricyanide and 10% Potassium Hydroxide Solution). Stains free uranium rapidly, U_3Si less rapidly and U_3Si_2 slowly. Used to reveal as-cast structures and relative amounts of U_3Si and U_3Si_2 after "deltizing". Etching time approximately 1 minute at room temperature.

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* Chalk River Nuclear Laboratories

b) 3.4 grams Citric Acid, 72 ccs Nitric Acid, 170 ccs Water and 1 cc 48% Hydrofluoric Acid. This is a grain boundary etch used to reveal the grain structure of U_3Si in "deltized" specimens. Also reveals fine structures in U_3Si grains. Stains U_3Si_2. Used on Attack-Polished specimens. Etching time up to 6 seconds at room temperature.