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Argonne, Illinois 60439

A STUDY OF THE URANIUM-ALUMINUM-SILICON SYSTEM

by

A. E. Dwight*

Materials Science Division

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*Resident Associate, Northern Illinois University, DeKalb

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A STUDY OF THE URANIUM-ALUMINUM-SILICON SYSTEM

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ABSTRACT

The U-Al-Si system was investigated in the composition range from U to U-80 at.% Al to U-75 at.% Si. More than 260 binary and ternary alloys were arc melted and studied by metallographic and x-ray powder diffraction techniques, in the as-cast condition and after thermal treatment at temperatures ranging from 700 to 1040°C. One or possibly two ternary compounds of undetermined crystal structure exist at or near U_2AlSi_2 .

The binary silicides U_3Si , U_3Si_2 , and USi can dissolve only small amounts of Al in solid solution, but the higher silicides U_3Si_5 , USi_{2-x} , and USi_3 dissolve increasingly larger amounts of Al.²⁻³ Of the three binary aluminides, UAl_2 can dissolve more than 12 at.% Si in solid solution. UAl_3 forms a continuous solid solution with USi_3 , and UAl_4 is not present in alloys containing more than 3 at.% Si.

U_3Si exhibits considerable ductility, which can be enhanced by partial substitution of Ge or Ga for Si. All other intermetallic compounds in the ternary system are brittle.

I. INTRODUCTION

One of the objectives of the Reduced Enrichment Research and Test Reactor (RERTR) Program is to develop a fuel alloy that contains low-enriched uranium (<20% ^{235}U). The space limitations imposed by reactors already in operation necessitate a high percentage of such uranium in the fuel alloy. Pure uranium is unsatisfactory because of its poor corrosion resistance and swelling characteristics. These requirements point to the need for a uranium-rich intermediate phase which is corrosion resistant and dimensionally stable under reactor conditions.

One of the compounds that has potential for this application is U_3Si , stoichiometrically ~3.9 wt.% Si. U_3Si has been recognized as a potential (solid) fuel alloy for some time, and a considerable amount of development work has been conducted on it. Of particular importance is the work of Canadian researchers, who found that the addition of small amounts of Al to U_3Si greatly improved aqueous corrosion resistance.

Furthermore, aluminum alloys are typically used for fuel element claddings in research reactors and, therefore, reactions between U_3Si and Al are of great technical interest.

The U-Al-Si ternary constitution diagram is a necessary tool for the development of fuel alloys based on U_3Si ; for that reason, the study described in this report was undertaken.

II. MATERIALS AND EXPERIMENTAL PROCEDURES

The alloys used in this investigation were made from depleted U (99.6 wt.% ^{238}U , 0.4 wt.% ^{235}U , and 100 ppm C), polycrystalline Si (Dow Corning, ≤ 3 ppba boron, ≤ 1.5 ppba donor) and Al rod (99.99 wt.%). Alloy samples, weighing 1-3 g, were arc melted on a water-cooled copper hearth with a water-cooled W electrode under an atmosphere of high-purity Ar (99.999+). No chemical analyses were conducted, but the nominal analyses are assumed to be correct inasmuch as the weight loss of the arc-cast ingot was always less than 1 wt.% of the charge.

Each arc-melted button was split into pieces with a cold chisel. One piece was retained in the as-cast condition; another piece was heat treated in an evacuated and sealed Vycor capsule suspended in a resistance-heated furnace. The specimens were cooled by quenching the capsule in water, without breaking, as there appeared to be no need for a more drastic quench. Temperature control during heat treatment was $\pm 5^\circ C$.

Metallographic specimens were mounted in Bakelite, ground through 6/0 paper, polished with diamond paste and alumina powder, and etched with a solution containing 85 cm³ H₂O, 36 cm³ HNO₃, 0.5 cm³ HF, and 1.7 g citric acid. Debye-Scherrer powder cameras were used to obtain X-ray diffraction patterns from material that had been crushed in a closed carbide die and passed through a 325-mesh sieve. Some problems with pyrophoric powder were encountered, but only with the most uranium-rich alloys. X-ray patterns were taken using several wavelengths, but Cu K α radiation proved to be the most useful. The hexagonal and tetragonal structures were indexed by a graphical method. To verify the crystal structure, intensities were obtained from a computer program for comparison with observed intensities. The density of a few alloys was measured by immersion in bromobenzene.

The isothermal sections of the U-Al-Si system were constructed from optical metallographic studies and x-ray powder diffraction data.

III. LIMITING BINARY DIAGRAMS

A. U-Al

The diagram shown in Fig. 1 is taken from Ref. 2. We have verified the crystal structures reported for the three binary compounds. UAl_2 has the $MgCu_2$ -type structure with $a_0 = 7.765 \text{ \AA}$, UAl_3 has the

Fig. 1. U-Al Phase Diagram. From Constitution of Binary Alloys by M. Hansen and K. Anderko. Copyright (c) 1958. Used with the permission of McGraw-Hill Book Company.

Cu_3Au -type structure with $a_0 = 4.265 \text{ \AA}$, and for UAl_4 , the diffraction pattern agrees with that calculated from the b.c. orthorhombic unit cell constants given in Ref. 2. UAl_2 , with a measured density of 8.27 g/cm^3 , is very brittle and tends to shatter when subjected to thermal shock. This characteristic is typical of many Laves phases.

B. U-Si

The diagram shown in Fig. 2 is the best estimate of the correct diagram for U-Si; the limited experimental data obtained in the present investigation are consistent with this diagram. The uranium-rich portion of the diagram up to U_3Si_2 is well established, but in the region from 50 to 80 at.% Si, the data in the early literature are conflicting, with different formulas given for the various phases.

The nomenclature of the phases has caused confusion. U_3Si was first called epsilon, then changed to delta. In the present report, Greek-letter designations are only used for α -, β -, and γ -U, and all intermediate phases are referred to by formula.

Fig. 2. U-Si Phase Diagram.

The potential use of U_3Si as a reactor fuel was recognized early in the development of nuclear reactors, and the crystal structure and properties of this compound have been the subject of many subsequent studies. The crystal structure reported by Zachariasen⁴ in 1949 was accepted and reproduced in many reference works. More recent work by Kimmel⁵ has established that the space group reported earlier was correct, but assignment of U and Si to the lattice sites was incorrect. The revised crystal structure is shown in Fig. 3. The base layer contains all U atoms, the next layer is half U and half Si, and the sequence repeats. The layer at $\frac{1}{2}$ the cell height is rotated slightly relative to the base layer; it is this rotation which enables us to classify the structure as a distorted variant of Cu_3Au -type. No close Si-Si bonding occurs; only U-U and U-Si bonds are present. This is the feature which probably accounts for the unusual "ductility" of the alloy. As will be discussed in more detail later, Zr is the only element that can substitute for part of the U, but Ga, Ge, and Sn can be substituted for part of the Si without destabilizing the crystal structure.

Fig. 3. The U_3Si Structure (tI16).

Kimmel et al.⁶ have reported that the tetragonal structure undergoes an orthorhombic distortion at 120 K. Blum et al.⁷ established that the tetragonal U_3Si transforms to a cubic Cu_3Au -type at 765°C. Thus, the succession of phase changes on cooling is cubic to tetragonal to orthorhombic.

U_3Si_2 has a congruent melting point at about 1665°C and undergoes no transformations in the solid state. The compound has a primitive tetragonal structure (Fig. 4) with $a_0 = 7.3299 \pm 4 \text{ \AA}$, $c = 3.9004 \pm 5 \text{ \AA}$, $c/a = 0.532$, and 10 atoms per unit cell. All published data by recent workers are in agreement with the early work of Zachariasen.⁴ The structure may be considered as a deformed Cu_3Au -type with pairs of Si atoms replacing a single atom. There are close Si-Si bonds and the compound is brittle.

The equiatomic compound USi is the most controversial of the binary silicides. The compound was reported by Zachariasen⁴ to be orthorhombic, of the FeB structure type. His results were based on diffractometer data taken on powder. In later work, Laugier et al.⁸ report that pure USi has a tetragonal structure with $a_0 = 10.61$ and $c_0 = 24.42 \text{ \AA}$, as determined from a Weissenberg pattern on a small single crystal. They further state that the orthorhombic structure is stabilized by 0.5-1.0 wt.% oxygen. The large unit cell constants and the lack of crystallographic details make it difficult to evaluate the validity of the tetragonal form of USi.

Fig. 4. The U_3Si_2 Structure (tP10).

In the present work, binary alloys were made with 48, 50, and 52 at.% Si. After heat treatment at 900°C, Debye-Scherrer patterns were taken. These patterns were complex and poorly resolved, but did not agree with either the published orthorhombic or tetragonal unit cells. However, metallographic examination confirms that a USi compound does exist and supports the assumption that it is formed peritectically. The microstructure of a 50 at.% Si alloy is shown in Fig. 5; the crystal structure of USi could not be definitely established.

The compound U_3Si_5 is hexagonal, hP3, AlB_2 -type, with U atoms in the 1a sites and Si atoms partially filling the 2d sites; other 2d sites are vacant. Figure 6 shows the crystal structure. Data for an alloy with 62.5 at.% Si gave $a_0 = 3.896$, $c_0 = 4.017$, and $V/m = 52.8 \text{ Å}^3$. Other binary alloys which varied only slightly in composition gave unit cell constants slightly different from these, which suggests a narrow range of solid solubility for the phase. This is the compound which the JCPDS file 13-108 refers to as $USi_{1.67}$. The metallographic appearance is distinctive with a striated structure as shown in Fig. 7.

The next compound, USi_{2-x} , is tetragonal, tI12, $ThSi_2$ -type, with U atoms in the 4a site and Si atoms in the 8e site. Some of the Si sites are vacant, and like other $ThSi_2$ -type compounds, USi_{2-x} has a defect structure. The crystal structure is illustrated in Fig. 8. The data for an alloy with 65 at.% Si show that $a_0 = 3.99$, $c_0 = 13.15$, and $V/m = 52.29 \text{ Å}^3$. These values are in fair agreement with the JCPDS file 13-547, which lists the composition as $U_4Si_{9.5}$. Pearson¹⁰ refers to this phase as $USi_{1.88}$. The microstructure is banded, as shown in Fig. 9.

Fig. 5. Microstructure of USi (U + 10.55 wt.% Si) Heat Treated for 5 days at 700°C.

Fig. 6. The U_3Si_5 Structure (hP3, $a_0 = 3.896$ and $c_0 = 4.017 \text{ \AA}$).

Fig. 7. Microstructure of U_3Si_5 in an Alloy of $U_{37.5}Al_5Si_{57.5}$ ($U + 1.26 \text{ wt.}\% \text{ Al} + 15.13 \text{ wt.}\% \text{ Si}$) Heat Treated for 3 days at 900°C .

Fig. 8. The USi_{2-x} Structure (tI12, $a_0 = 3.99$ and $c_0 = 13.15 \text{ \AA}$).

Fig. 9. Microstructure of USi_{2-x} (Striated Phase) in an Alloy of $U_{35}Al_{12.5}Si_{52.5}$ (U + 3.32 wt.% Al + 14.54 wt.% Si), Heat Treated for 5 days at 900°C.

The compound USi_2 , with exact 1:2 stoichiometry, is stable below 450°C. It has the AlB_2 -type structure, as in U_3Si_5 . Our data for an alloy with 66.6 at.% Si gave unit cell constants of $a_0 = 3.839$, $c_0 = 4.72$, and $V/m = 51.97 \text{ Å}^3$. These constants are very similar to those for U_3Si_5 . The downward trend in V/m for the three phases U_3Si_5 , USi_{2-x} , and USi_2 is consistent with the increasing content of the smaller Si atom.

The silicon-rich compound USi_3 has the cubic Cu_3Au -type structure, cP4. Our data show $a_0 = 4.036$ and $V/m = 65.74 \text{ Å}^3$. This compound usually has a white microstructure after etching, with no distinctive substructure.

C. Al-Si

The Al-Si diagram² is reproduced in Fig. 10. There are no intermediate phases in the system, and the binary diagram is of interest only because the eutectic at 577°C gives rise to a eutectic valley in the ternary U-Al-Si system.

Fig. 10. The Al-Si System. From Constitution of Binary Alloys by M. Hansen and K. Anderko. Copyright (c) 1958. Used with the permission of McGraw-Hill Book Company.

IV. U-Al-Si TERNARY SYSTEM

A. Previous Work

The U-rich region extending to the quasi-binary U_3Si_2 - UAl_2 was investigated by Petzow and Kvernes.^{11,12} They presented data² on the melting surface, three isothermal sections at 950°, 850°, and 650°C, and four vertical sections. The "melting surface" reported by Petzow and Kvernes¹¹ is reproduced in Fig. 11. The 950°C and 850°C isotherms are shown in Figs. 12 and 13. The present work confirms the phase relations illustrated in these figures and disagrees only in that we find a solid solubility of at least 10 at.% Si in UAl_2 .

Phase relations in the U-rich corner up to 4 at.% Al and 4 at.% Si were reported by Svistonova and Ivanov.¹³ They constructed isothermal sections at 900°, 800°, 770°, and 740°C. The 900°C isotherm is reproduced in Fig. 14. No attempt was made to verify their work.

A paper by Zygmunt¹⁴ reports a ternary $UAlSi$ compound which was not confirmed in the present study.

B. Present Study

In this investigation, more than 260 binary and ternary alloys were made and prepared for metallographic examination. The compositions are listed in the Appendix and shown as circles in Fig. 15. More than 230 Debye-Scherrer x-ray diffraction patterns were taken. The alloys were

Fig. 11. Melting Surface in the $\text{U}-\text{U}_3\text{Si}_2-\text{UAl}_2$ System. Reproduced from Ref. 11 with the permission of the copyright holder.

Fig. 12. 950°C Isothermal Section in the $\text{U}-\text{U}_3\text{Si}_2-\text{UAl}_2$ System. Reproduced from Ref. 11 with the permission of the copyright holder.

Fig. 13. 850°C Isothermal Section in the U-U₃Si-UAl₂ System. Reproduced from Ref. 11 with the permission of the copyright holder.

Fig. 14. 900°C Isothermal Section in the U-rich Corner. Reproduced from Ref. 13.

Fig. 15. Alloy Compositions Examined in the Present Investigation.

studied both in the as-cast condition and after the heat treatments listed in the Appendix. The 900°C isothermal section is reproduced in Fig. 16. The formation of two binary compounds (USi_2 at 450°C and UAl_4 at 730°C) necessitates a 400°C isothermal, which is shown in Fig. 17.

The aluminides are of interest to the RERTR project because they would be the terminal products of a reaction between a silicide fuel and the Al matrix and clad. In the aluminide richest in U, UAl_2 , Si can replace Al to at least 12 at.% Si at 900°C and there are indications that in the as-cast condition even higher Si substitutions are possible under non-equilibrium conditions. These data conflict with those of Petzow and Kvernes, who state that UAl_2 shows no extension into the ternary region, and with those of Ludwig, who reported that UAl_2 will dissolve only a very small amount of Si. Figure 18 shows our data on the lattice parameter for $\text{U}(\text{Al},\text{Si})_2$ as a function of at.% Si.

The second aluminide, UAl_3 , has the cubic Cu_3Au -type structure. UAl_3 is isostructural with USi_3 and our data indicate complete miscibility between UAl_3 and USi_3 at 900°C, although there are indications that a

Fig. 16. 900°C Isothermal Section of U-Al-Si System. The microstructures of the alloys indicated by circles are shown in the figures designated by the adjacent numbers. The dotted line (Ref. 11) shows the path of the eutectic.

Fig. 17. 400°C Isothermal Section of U-Al-Si System.

Fig. 18. The Unit Cell Parameters of $U(Al,Si)_2$ (cF24, $MgCu_2$ -type).

miscibility gap might exist at some lower temperature. Figure 19 shows a smooth transition in unit cell parameter from UAl_3 to USi_3 and emphasizes the fact that Si has a smaller atomic volume than Al when alloyed with U.

The third aluminide, UAl_4 , is formed at $730^\circ C$ by a peritectic reaction, $liquid + UAl_3 \rightarrow UAl_4$. The mechanism of the transformation has been reported by Lee and Leighly,¹⁶ and the crystal structure of UAl_4 by Borie.¹⁷ In the present work an arc-melted alloy of UAl_4 , heat treated for three days at $600^\circ C$, gave an x-ray powder pattern that was consistent with the crystal structure reported by Borie. We did not attempt to determine the solubility of Si in UAl_4 , nor the effect of Si on the peritectic temperature, but the pattern of UAl_4 was not observed for any ternary alloy.

The uranium-rich silicide, U_3Si , has been studied by many investigators; the crystal structure is described in Sec. III.B above. Bonding in U_3Si differs from that in other silicides, as Zachariasen points out in Ref. 4: "In U_3Si there is, in contrast to the other silicides, no bond between silicon atoms. In all structures except U_3Si covalent bonds between silicon atoms occur." It may be coincidental,³ but U_3Si is also the only silicide that exhibits ductility and resistance to fracture.

Fig. 19. The Unit Cell Parameters of $U(Al,Si)_3$ (cP4, Cu_3Au -type).

U_3Si has only limited solubility for Al. The 650°C isotherm reported by Petzow and Kvernes¹¹ shows about 1 at.% Al dissolved in U_3Si . We did not attempt to determine the limit of solid solution, but a comparison of x-ray powder patterns for U_3Si and $U + 1.5$ wt.% Al + 3.5 wt.% Si shows a small shift in lattice spacings, indicating that enough Al is in solution to slightly expand the unit cell. The figure of 0.5 wt.% Al reported by Ross¹⁸ seems rather high. Our x-ray and metallographic data verify that two three-phase areas exist, i.e., ($U_3Si + \gamma-U + UAl_2$) and ($U_3Si + U_3Si_2 + UAl_2$).

The second silicide, U_3Si_2 , may be considered as a deformed Cu_3Au -type with pairs of Si atoms replacing a single Si atom. Si-Si bonds are present, and the alloy is brittle. X-ray diffraction patterns show no measurable solution of Al in U_3Si_2 , as indicated by the absence of a shift in the d spacings of high-angle reflections. The compound always occurs as a dendritic structure in as-cast alloys.

The third silicide, USi , also has a very low solubility for Al. X-ray data are not conclusive for identification of USi in a ternary alloy. Figure 5 illustrates the metallographic appearance of a binary alloy that is nearly single-phase USi .

U_3Si_5 , also referred to as $USi_{1.67}$, marks the composition at which the silicides begin to take Al into solid solution. This compound has a distinctive metallographic structure (see Fig. 7), which can be

recognized in two- and three-phase fields of the ternary system. The x-ray diffraction pattern is sharp and easily recognized. The crystal structure is a defect type and a ternary element (e.g., Al) could either enter an empty lattice site or displace Si atom. No attempt was made to locate the Al atoms in the unit cell.

The next silicide, USi_{2-x} , also has a defect structure, but of the tetragonal ThSi_2 -type. At least 16 at.% Al can be dissolved in the compound, which has a distinctive banded structure (Fig. 9). This structure is visible in the three-phase region ($\text{USi}_{2-x} + \text{U}(\text{Si},\text{Al})_3 + \text{T}$), as shown in Fig. 20.

Stoichiometric USi_2 is a controversial compound. We have accepted the work of Brown and Norreys,¹⁹ who report that USi_2 is stable up to 450°C and has the hexagonal AlB_2 -type structure. Because we do not find this compound in ternary alloys, we assume that it "disappears" by way of a three-phase region, as shown in Fig. 17. USi_3 exhibits complete solid solubility with UAl_3 at 900°C, and as a true pseudobinary section effectively separates the ternary system into two parts. The few alloys that were made with >75 at.% Si gave x-ray patterns in which $\text{U}(\text{Si},\text{Al})_3$ was the dominant phase.

A ternary intermetallic compound was found with composition corresponding to the formula U_2AlSi_2 or, possibly, $\text{U}_2\text{Al}_2\text{Si}_3$. In the as-cast condition, samples in this composition region yielded a sharp x-ray diffraction pattern, but the crystal structure has not yet been solved. Table I lists the observed d spaces and intensities of reflections. After heat treatment at 900°C and lower, a completely different diffraction pattern was found (Table II). This pattern is also unsolved, although an effort was made to index both patterns by use of the Visser Powder Indexing Program.²⁰ For a successful computer solution, 40 reflections are needed, and we could not read 40 with sufficient resolution.

The transformation from the as-cast structure is shown in Figs. 21 and 22. Figure 21 shows the ternary phase and a eutectic of ternary phase + UAl_2 in an alloy of $\text{U}_{37.5}\text{Al}_{27.5}\text{Si}_{35}$. After three days at 900°C, the same alloy (Fig. 22) has some residual untransformed compound in the center, surrounded by a transformation layer. At longer times the reaction is complete, but more study is needed to determine the nature of the reaction. It was found that Ge could be substituted for Si to form U_2AlGe_2 , which is isostructural with heat-treated U_2AlSi_2 .

C. U + 3.5 wt.% Si + 1.5 wt.% Al Alloy

Extensive research by Canadian investigators has shown that addition of 1.5 wt.% Al to U_3Si greatly increases the corrosion¹⁸ resistance. Their findings are summarized in a Canadian patent¹⁸ as follows: "The U alloy suitable for nuclear fuels contains 3.2-3.7 wt.% Al, except that when the Al content is <1.5% the Si content is <3.5%. The structure consists of essentially delta U_3Si (containing ~0.5 wt.% dissolved Al), minor amount of UAl_2 , and isolated particles of U_3Si_2 . The alloy is cast at 1500-1600°, rapidly cooled to give a fine microstructure,

Fig. 20. Microstructure of $U_{35}Al_{20}Si_{45}$ (U + 5.32 wt.% Al + 12.47 wt.% Si), Heat Treated for 5 days at 900°C.

Table I. Observed d Spaces and Intensities (Cu K α Radiation)
for As-Cast U₂AlSi₂

2 θ	d	I	I ₁₀₀	2 θ	d	I	I ₁₀₀
16.38	5.41	5	10	53.71	1.71	17	35
19.92	4.46	2	4	55.86	1.65	6	12
21.94	4.05	2	4	56.44	1.63	5	10
23.27	3.82	24	49	57.90	1.59	5	10
27.43	3.25	49	100	58.59	1.58	2	4
27.96	3.19	7	14	59.43	1.56	2	4
28.62	3.12	26	53	61.61	1.51	2	4
33.06	2.71	34	69	64.61	1.44	7	14
33.45	2.68	10	20	66.46	1.41	7	14
34.65	2.60	2	4	69.70	1.35	2	4
35.83	2.51	14	29	71.71	1.32	7	14
36.21	2.48	30	61	74.55	1.27	6	12
37.29	2.41	33	67	79.37	1.21	2	4
38.75	2.32	4	8	81.56	1.18	5	10
39.98	2.26	4	8	84.15	1.15	3	6
44.95	2.02	1	2	85.67	1.13	2	4
47.60	1.91	4	8	90.55	1.09	1	2
49.94	1.83	11	22	93.38	1.06	2	4
50.72	1.80	27	55	95.40	1.04	3	6
52.93	1.73	3	6	100.44	1.00	3	6

^aThe diffraction pattern shown in this table corresponds to the single-phase portion of Fig. 21.

Table II. Observed d Spaces and Intensities (Cu K α Radiation)
for Heat-treated^a U₂AlSi₂^b

2 θ	d	I	I ₁₀₀	2 θ	d	I	I ₁₀₀
18.61	4.77	21	41	39.76	2.27	11	22
21.55	4.12	6	12	40.26	2.24	8	16
23.52	3.78	47	92	41.15	2.19	10	20
24.82	3.59	7	14	43.79	2.07	4	8
27.08	3.29	36	71	45.45	2.00	3	6
27.53	3.24	11	22	50.03	1.82	13	26
28.88	3.09	10	20	51.26	1.78	9	18
31.23	2.86	11	22	51.62	1.77	6	12
31.67	2.83	8	16	52.42	1.75	7	14
32.60	2.75	34	67	53.70	1.71	7	14
32.77	2.73	27	53	54.91	1.67	10	20
34.16	2.62	12	24	55.69	1.65	10	20
34.59	2.59	51	100	56.56	1.63	12	24
35.93	2.50	10	20	57.25	1.61	9	18
36.22	2.48	11	22	57.81	1.59	16	31
36.74	2.45	11	22	60.33	1.53	4	8
37.55	2.40	8	16	62.21	1.49	7	14
38.12	2.36	29	57	63.11	1.47	4	8
38.30	2.35	19	37	65.78	1.42	5	10
39.43	2.29	24	47	68.98	1.36	11	22

^a6 days at 900°C.

^bThe diffraction pattern shown in this table corresponds to the transformed portion of the single-phase platelet shown in the lower part of Fig. 22.

Fig. 21. Microstructure of As-cast $U_{37.5}Al_{27.5}Si_{35}$ (U + 6.97 wt.% Al + 9.23 wt.% Si).

Fig. 22. Microstructure of Same Alloy as in Fig. 21, after 3 days at 900°C. Transformation is incomplete. The irregular layer in the center of the platelet is untransformed residual phase.

and heat treated at 800-850°. The corrosion rate is 0.02-0.03 kg/m²-hr in flowing steam at 823°C, 1 atm, and ≥ 2 hr exposure."

A topical report by Feraday et al.²¹ on the irradiation behavior of U + 3.5 wt.% Si + 15. wt.% Al contains data which indicate that the Al addition improved irradiation stability. The same report includes a photomicrograph of the alloy after 72 h at 800°C. The phases are identified as U₃Si, U₃Si₂, and UAl₂. This microstructure is consistent with the ternary phase diagram reported by Petzow and Kvernes¹¹ and with Fig. 16 of the present work. The microstructure of our alloy, heat treated for 72 h at 800°C, is shown in Fig. 23; it consists of U₃Si with small amounts of uniformly dispersed U₃Si₂ and UAl₂.

D. Isopleths from U₃Si to Al and from U₃Si₂ to Al

The series of alloys from U₃Si to Al are of interest to the RERTR project in that they display the reaction products which could result from a complete reaction between the fuel (U₃Si) and matrix (Al powder). The alloy U₆₀Al₂₀Si₂₀ would result from reaction to equilibrium of a mixture containing 96.49 wt.% U₃Si and 3.51 wt.% Al (82.96 vol.% U₃Si, 17.04 vol.% Al). Equilibrium could only be attained in the absence of oxygen or other contaminants and after a relatively long time at an elevated temperature, requisites not likely to be found in either an as-fabricated or an overheated reactor fuel element.

The microstructure of U₆₀Al₂₀Si₂₀ (Fig. 24) shows isolated particles of UAl₂ and a very small amount of U₃Si₂ in a matrix of U₃Si. Another alloy on the section with higher Al content is U₄₅Al₄₀Si₁₅. The microstructure of this alloy (Fig. 25) shows UAl₂ and lesser amounts of U₃Si and U₃Si₂. In U_{37.5}Al₅₀Si_{12.5} (Fig. 26), with higher Al content, grains of UAl₂ are separated by a eutectic of U₃Si₂ and UAl₂. In U₃₀Al₆₀Si₁₀ (Fig. 27), with an even higher Al content, the microstructure shows two phases, UAl₂ and UAl₃, both containing Si in solid solution. The second silicide, U₃Si₂, is also a potential fuel alloy as a powder dispersed in an Al matrix. A series of alloys in the section or isopleth from U₃Si₂ to Al are shown in Figs. 28 through 33, with microstructures described in the captions. Phase identification was made from x-ray powder patterns, supplemented by etching characteristics. The angular, straight-sided nature of the U₃Si₂ dendrites is noticeable in several photomicrographs.

V. DISCUSSION

A. Properties of Alloys

The densities of selected binary and ternary alloys, measured by immersion in bromobenzene, are listed below:

(a)

20 μm

(b)

Fig. 23. Microstructures of U_3SiAl (U + 3.5 wt.% Si + 1.5 wt.% Al).
(a) As-cast; (b) heat treated (72 h at 800°C).

Fig. 24. Microstructure of U₆₀Al₂₀Si₂₀ (U + 3.51 wt.% Al + 3.65 wt.% Si) Heat Treated for 5 Days at 900°C.

Fig. 25. Microstructure of U₄₅Al₄₀Si₁₅ (U + 8.8 wt.% Al + 3.45 wt.% Si) Heat Treated for 5 Days at 900°C.

Fig. 26. Microstructure of $U_{37.5}Al_{50}Si_{12.5}$ (U + 12.70 wt.% Al + 3.30 wt.% Si) Heat Treated for 5 Days at 900°C.

Fig. 27. Microstructure of $U_{30}Al_{60}Si_{10}$ (U + 17.9 wt.% Al + 3.10 wt.% Si) Heat Treated for 5 Days at 900°C.

Fig. 28. Microstructure of $U_{54}Al_{10}Si_{36}$ (U + 1.91 wt.% Al + 7.15 wt.% Si) Heat Treated for 5 days at 900°C.

Fig. 29. Microstructure of $U_{48}Al_{20}Si_{32}$ (U + 4.19 wt.% Al + 6.99 wt.% Si) Heat Treated for 5 Days at 900°C.

Fig. 30. Microstructure of $U_{42}Al_{30}Si_{28}$ (U + 6.98 wt.% Al + 6.78 wt.% Si) Heat Treated for 5 Days at 900°C.

Fig. 31. Microstructure of $U_{36}Al_{40}Si_{24}$ (U + 10.45 wt.% Al + 6.53 wt.% Si) Heat Treated for 5 days at 900°C.

Fig. 32. Microstructure of $U_{30}Al_{50}Si_{20}$ (U + 14.9 wt.% Al + 6.21 wt.% Si).

Fig. 33. Microstructure of $U_{24}Al_{60}Si_{16}$ (U + 20.8 wt.% Al + 5.78 wt.% Si).

at.%			g/cm ³	at.%			g/cm ³
U	Al	Si		U	Al	Si	
60	20	20	12.33	37.5	35	27.5	8.95
50		50	10.86	35	35	30	8.64
48	20	22	10.49	33.3	59.2	17.5	8.51
40	35	25	9.46	33.3	51.7	15	8.45
40	20	40	9.38	33.3	66.6		8.27

These data show no systematic trend with respect to Al or Si because the density of the alloy is overwhelmingly controlled by the U content.

The only ductile compound in the ternary system is U_3Si . All others are brittle enough that powder can be prepared by crushing in a closed carbide die. Sharp x-ray diffraction patterns were obtained without stress relieving the powder. Alloys containing 80 wt.% or more Al and/or Si are too soft to permit preparation of powder by this method, but no effort was made to investigate this region of the diagram.

All alloys were stable in air at room temperature except those containing uranium solid solution as one of the phases. Even when U_{SS} existed, however, the oxidation rate was very low. Precautions were necessary to avoid ignition of fine powder by impact in U-rich alloys.

B. Transformation Kinetics

U_3Si is produced by a sluggish peritectoid reaction:
 $U_3Si + \gamma U \rightarrow U_3Si$ at 925°. An as-cast alloy always contains metastable U_3Si_2 and αU ; a prolonged heat treatment is necessary to produce U_3Si . We confirmed early in the investigation that 3 days at 800°C is more than adequate to drive the peritectoid reaction to completion.

A detailed study of the kinetics of U_3Si formation in cast U_3Si has been published by Kimmel et al.²² They found that "the growth of U_3Si at temperatures below 760°C is controlled by interdiffusion of silicon and uranium and probably by a vacancy mechanism. The high (50 kcal/mole) activation energy of the diffusivity causes a drastic slowdown of the growth-rate when the temperature decreases." The kinetics of U_3Si formation between 750 and 850°C were studied by Honma et al. (Ref. 23). These authors found that the most suitable transformation temperature was about 800°C, and that the period of 90% transformation was 85 hours.

C. Substitution for Si in U_3Si

In a 1970 study,²⁴ three series of ternary U_3Si -base alloys were investigated. One was the $U_{75}Ga_{25-x}Ge_x$ series; in the other two, Ga or Ge was partially substituted for Si. The study showed that Ga or Ge could

partially replace Si, and that a ternary $U_{75}Ga_{10}Ge_{15}$ compound was isostructural with U_3Si . Compositions are shown in the area-of-stability plot of Fig. 34. The compounds that contain Ga or Ge have an expanded unit cell, as expected from the larger atomic volume of Ga and Ge.

The outstanding feature of the substituted or ternary compounds is their ductility. An arc-melted button of $U_{75}Ga_{10}Ge_{15}$, annealed at $800^{\circ}C$, was cold hammered into a thin disk without intermediate anneals. Another annealed button was cold rolled into 1/16" strip with an intermediate anneal. The investigation was terminated before quantitative data could be obtained, but these alloys clearly exhibit an extremely high room-temperature malleability.

D. Alloy Chemistry

The controlling factor that determines the nature of a binary U-X diagram is the position of the alloying element (X) in the Periodic Table. The alloy chemistry for binary phase diagrams of thorium, uranium, and plutonium was reported earlier.²⁵ The larger number and greater complexity of ternary uranium phase diagrams would require an analysis beyond the scope of the present report. Certain trends can nevertheless be observed. It was noted from the study of binary diagrams that uranium-rich compounds crystallize in uncommon crystal structures, but uranium-poor compounds have structures that are commonly found in transition element compounds. It was also noted that uranium forms compounds with definite stoichiometric ratios and never forms sigma phases or other electron compounds that exist over a range of composition.

Both of the above observations hold true for ternary as well as binary uranium compounds. A trend that is unique to ternary systems is the occurrence of ternary intermetallic compounds that contain three distinct crystallographic lattice sites, e.g., $ThCu_2Si_2$. This structure requires large, medium, and small atoms in a 1:2:2 ratio, and also requires a narrowly restricted range of electron concentrations. (A UAl_2Si_2 compound was reported,¹⁴ but our research indicates that it was actually $U(Al,Si)_3$, Cu₃Au-type.) Ternary uranium diagrams, like binaries, can be grouped into several types, and it is of interest to determine which group the U-Al-Si system resembles. Three groups are considered:

1. X-Al-Si

Ternary diagrams of varying degrees of completeness have been published for X-Al-Si systems in which X is Ca, Sr, Ba, Ce, Y, Ti, Zr, Nb, Ta, Cr, Mo, W, Mn, Re, and Fe. The occurrence of ternary compounds (and extensive solubility in binary compounds) is most prevalent at the AB_2 level. All XAl_2 binary compounds (X = Ca through the rare earths) are Laves phases, while no XSi_2 compounds are Laves phases. This precludes a continuous range of solution between XAl_2 and XSi_2 . Usually, the solubility of Al in XSi_2 is greater than the solubility of Si in XAl_2 . The Nb-Al-Si system,²⁶ shown in Fig. 35, bears only a slight resemblance to the U-Al-Si system. Nb_3Si has the Ti_3P -type structure (stable above $1600^{\circ}C$), and U_3Si is a distorted variant of Cu_3Au . Some resemblance of the two ternaries was expected, and Nb does form a continuous series of solid solutions with the bcc polymorph of U.

Fig. 34. Area-of-Stability Plot for U_3Si -based Ternary Compounds. Ga or Ge can be substituted for Si up to $U_{75}Si_{10}Ga_{15}$ (or $U_{75}Si_{10}Ge_{15}$). The ternary compound $U_{75}Ga_{13}Ge_{12}$ is isostructural with the tetragonal form of U_3Si .

Fig. 35. Nb-Al-Si Constitution Diagram. Reproduced from Ref. 26 with the permission of the copyright holder.

At the AB_3 level there is continuity between USi_3 and UAl_3 , both of which have the Cu_3Au -type structure. No other X-Al-Si system exhibits this mutual solid solubility, and, in fact, XAl_3 and XSi_3 binary $CuAu_3$ -type compounds are rather scarce.

In those systems which resemble U-Al-Si (i.e., Nb-Al-Si, Zr-Al-Si,²⁷ and Mo-Al-Si²⁸), the number of true ternary compounds is very limited. Similarly, only two true ternary compounds were found in the U-Al-Si system.

2. U-X-Si

The U-Zr-Si ternary system has been reported by Farkas et al.²⁹ These authors report no indication of zirconium solubility in U_3Si , but a fairly extensive range of solid solution for Zr in U_3Si_2 (about 20 at.% Zr at 705°C).

There are numerous ternary compounds in the U-Fe-Si,³⁰ U-Co-Si,²⁸ and U-Ni-Si²⁸ systems with some solid solubility in compounds at the AB_2 level.

The U-Be-Si system,³¹ reproduced in Fig. 36, is of particular interest in that Be can replace Si to an even greater degree than can Al, in both U_3Si_5 and in USi_{2-x} . Data are lacking on related ternaries, especially U-In-Si and U-Ga-Si. The U-rich corner of the U-C-Si system has been investigated by Guinet and coworkers.^{32,33} They report that the maximum amount of C dissolved in U_3Si is 1500 ppm at 810°C. At 815°C there is an equilibrium among four solid phases:

The points of interest to the RERTR project are that C lowers the peritectoid temperature for formation of U_3Si from 925°C to 815°C, and that C goes into solid solution in U_3Si_2 but not appreciably into U_3Si .

3. U-Al-X

The U-Al-Sn ternary diagram (Fig. 37), reported by Marazza et al.,³⁴ resembles the U-Al-Si diagram in that UAl_3 and USn_3 form continuous solid solutions. There are no analogs of U_3Si_2 or U_3Si in the U-Sn binary system, and apparently no solid solubility of Sn in UAl_2 . Marazza et al. note the poor oxidation resistance of Sn-rich alloys. This observation is in agreement with the present author's experience, i.e., oxidation resistance is worse for ternary compounds with Sn or Pb than for those with Si or Ge.

Petzow and co-workers³⁵ have studied the Al-rich part of the U-Zr-Al ternary system and have found extensive replacement by Zr or U in UAl_2 and UAl_3 . They also report that the solubility of Zr in UAl_4 is very small. The solubility of Zr in UAl_2 and UAl_3 is to be expected from the rules of alloy chemistry, in that Zr has a larger atomic volume than U and increases the R_A/R_B ratio toward a value closer to the ideal ratio.³⁶ Partial constitution diagrams have been reported for the U-Mn-Al,

Fig. 36. U-Be-Si Constitution Diagram. Reproduced from Ref. 31 with the permission of the copyright holder.

Fig. 37. U-Al-Sn Constitution Diagram. Reproduced from Ref. 34 with the permission of the copyright holder.

U-Fe-Al,³⁷ U-Co-Al,³⁸ and U-Ni-Al³⁹ systems. A dominant feature of all four systems is a low-melting eutectic in the U-(Mn, Fe, Co or Ni) binary system. The number of ternary intermetallic compounds for Mn, Fe, and Co is 1, 1, and 3, respectively (the number for Ni is uncertain). This increase in the number of ternary compounds as one proceeds from Group VII through Group VIII has been noted in a study of rare-earth ternary systems. The number of ternary compounds decreases for Cu and Zn.

The ternary compound U_2Co_2Al is of interest in that it is isostructural with U_3Si_2 . It is probable that Co occupies the 4h sites (Si in U_3Si_2) and Al occupies the 2a sites (U in U_3Si_2). This provides a guide to the selection of ternary elements which might enter into solution in U_3Si_2 . (We have already discussed the solubility of Zr in U_3Si_2 .) Only an atom of small atomic volume such as C could be expected to replace Si in U_3Si_2 .

In summary, the U-Al-Si constitution diagram shows little resemblance to any other X-Al-Si system, but does resemble the U-Be-Si and U-Al-Sn systems. The empirical rules of alloy chemistry correctly predict relatively few ternary compounds in the U-Al-Si system.

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APPENDIX

Alloy Compositions and Heat Treatments

Spec. No.	Atomic Percent			Temp. (°C)	Spec. No.	Atomic Percent			Temp. (°C)
	U	Al	Si			U	Al	Si	
1	33.3	33.3	33.3	943	44	30	35	35	800
2	50	20	30	943	45	30	30	40	800
3	62	20	18	800	46	25	50	25	800
4	50	40	10	800	47	25	25	50	800
5	47.5	22.5	30	810	48	40	25	35	800
6	45	25	30	810	49	37.5	25	37.5	800
7	40	30	30	810	50	30	32.5	37.5	800
8	35	35	30	810	51	30	27.5	42.5	800
9	30	40	50	810	52	35	20	45	1040
10	52	28	28	900	53	35	25	40	1040
11	50	30	20	900	54	30	25	45	1040
12	45	35	20	900	55	40	15	45	1040
13	40	40	20	900	56	35	15	50	1040
14	35	45	20	900	57	30	20	50	1040
15	25	65	10	860	58	35	27.5	37.5	800
16	25	60	15	860	59	25	32.5	42.5	800
17	25	55	20	860	60	50	10	40	800
18	25	50	25	860	61	37.5	17.5	45	800
19	25	45	30	860	62	37.5	15	47.5	800
20	52	20	28	900	63	40	12.5	47.5	800
21	44	40	16	900	64	42.5	12.5	45	800
22	40	50	10	900	65	42.5	15	42.5	800
23	30	70		900	66	40	10	50	800
24	25	70	5	900	67	45	5	50	800
25	48.3	5	46.7	900	68	50	5	45	800
26	46.6	10	43.3	900	69	55	5	40	800
27	45	15	40	900	70	47.5	15	37.5	800
28	43.3	20	36.7	900	71	45	17.5	37.5	800
29	41.6	25	33.3	900	72	50	2.5	47.5	800
30	33.3	66.6		800	73	52.5	2.5	45	800
31	33.3	61.6	5	800	74	55	2.5	42.5	800
32	33.3	56.6	10	800	75	45	2.5	52.5	800
33	33.3	51.6	15	800	76	47.5	2.5	50	800
34	33.3	46.6	20	800	77	57.5	2.5	40	800
35	33.3	41.6	25	800	78	37.5	20	42.5	800
36	25	40	35	800	79	40	17.5	42.5	800
37	25	35	40	800	80	35	17.5	47.5	800
38	25	30	45	800	81	32.5	20	47.5	800
39	45	30	25	800	82	32.5	22.5	45	800
40	40	35	25	800	83	35	22.5	42.5	800
41	35	40	25	800	84	37.5	12.5	50	800
42	30	45	25	800	85	32.5	17.5	50	800
43	35	30	35	800	86	27.5	22.5	50	800

Spec. No.	Atomic Percent			Temp. (°C)	Spec. No.	Atomic Percent			Temp. (°C)
	U	Al	Si			U	Al	Si	
87	30	22.5	47.5	800	131	52.5	15	32.5	800
88	42.5	5	52.5	800	132	47.5	17.5	35	800
89	40	7.5	52.5	800	133	50	17.5	32.5	800
90	37.5	10	52.5	800	134	50	12.5	37.5	800
91	35	12.5	52.5	800	135	52.5	12.5	35	800
92	32.5	15	52.5	800	136	33.3	26.6	40	800
93	30	17.5	52.5	800	137	27.5	32.5	40	800
94	27.5	20	52.5	800	138	40	20	20	800
95	25	22.5	52.5	830	139	31.25	21.25	42.5	800
96	35	16	49	830	140	50		50	600
97	33.3	17.5	49.2	830	141	45		55	600
98	32.5	25	42.5	830	142	36.9	51.6	11.5	800
99	37.5	22.5	40	830	143	33.3	41.6	25	800
100	32.5	27.5	40	830	144	25	35	40	800
101	33.3	16.6	50	800	145	25	30	45	800
102	36.6	13.3	50	800	146	45	30	25	800
103	42.5	10	47.5	800	147	25	27.5	47.5	800
104	45	10	45	800	148	25	22.5	52.5	800
105	52.5	7.5	40	800	149	25	70	5	800
106	50	7.5	42.5	800	150	25	65	10	800
107	47.5	7.5	45	800	151	35	12.5	52.5	900
108	45	7.5	47.5	800	152	33.3	17.5	49.16	900
109	35	12.5	32.5	800	153	50	20	30	900
110	33.75	21.25	45	800	154	37.5	7.5	55	900
111	42.5	7.5	50	800	155	35	10	55	900
112	45	12.5	42.5	800	156	40	5	55	900
113	47.5	12.5	40	800	157	32.5	10	57.5	900
114	42.5	17.5	40	800	158	42.5	2.5	55	900
115	37.5	45	17.5	800	159	40	2.5	57.5	900
116	37	3	60	800	160	37.5	5	57.5	900
117	53.3	6.6	60	800	161	35	7.5	57.5	900
118	33.3	10	56.6	800	162	45	27.5	27.5	900
119	40	20	40	1040	163	42.5	27.5	30	900
120	45	20	35	1040	164	40	27.5	32.5	900
121	50	15	35	1040	165	37.5	27.5	35	900
122	56.5	5	42.5	1040	166	35	27.5	37.5	900
123	30	15	55	800	167	32.5	27.5	40	900
124	55		45	800	168	30	27.5	42.5	900
125	52.5	2.5	45	800	169	51	15	34	900
126	50	5	45	800	170	42	30	28	900
127	47.5	7.5	45	800	171	33.3	44.4	22.2	900
128	47.5	20	32.5	800	172	25	65	10	900
129	47.5	25	27.5	800	173	25	58.3	16.6	900
130	57.5		42.5	800	174	64	15	21	900

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Spec. No.	Atomic Percent			Temp. (°C)	Spec. No.	Atomic Percent			Temp. (°C)
	U	Al	Si			U	Al	Si	
175	52.5	30	17.5	900	220	41.25	23.25	35	900
176	41.5	45	13.75	900	221	41.25	25	37.75	900
177	30	60	10	900	222	40	20	40	900
178	33.3	56.6	10	900	223	37.5	25	37.5	900
179	33.3	51.6	15	900	224	40	41.25	38.75	900
180	42.5	25	32.5	900	225	40	18.75	41.25	900
181	40	25	35	900	226	37.5	20	42.5	900
182	37.5	25	37.5	900	227	35	20	45	900
183	33.3	16.6	50	900	228	33.3	49.16	17.5	900
184	45	20	35	900	229	30	52.5	17.5	900
185	42.5	20	37.5	900	230	25	57.5	17.5	900
186	42.5	22.5	35	900	231	54	10	36	900
187	40	22.5	37.5	900	232	48	20	32	900
188	35	30	35	900	233	42	30	28	900
189	37.5	30	32.5	900	234	36	40	24	900
190	40	30	30	900	235	30	50	20	900
191	45	22.5	32.5	900	236	24	60	16	900
192	47.5	20	32.5	900	237	60	20	20	900
193	50	17.5	32.5	900	238	45	40	15	900
194	25	10	65	900	239	37.5	50	12.5	900
195	25	20	55	900	240	22.5	70	7.5	900
196	47.5	5	47.5	900	241	40	35	25	900
197	45	10	45	900	242	35	35	30	900
198	42.5	21.25	36.25	800	243	30	35	35	900
199	40	23.75	36.25	800	244	40	20	40	900
200	40	21.25	38.75	800	245	40	20	20	900
201	37.5	23.75	38.57	800	246	38	30	32	900
202	38.75	21.25	40	800	247	35	30	35	900
203	36.25	23.75	40	800	248	30	40	30	900
204	37.5	21.75	41.25	800	249	37	50	13	900
205	35	23.75	41.25	800	250	36	50	14	900
206	45	18.75	36.25	900	251	35	50	15	900
207	43.75	18.75	37.5	900	252	40	35	25	900
208	42.5	18.75	38.75	900	253	37.5	35	27.5	900
209	41.25	18.75	40	900	254	35	35	30	900
210	60		40	900	255	33.3	51.6	15	900
211	41.25	20	38.75	900	256	60	20	20	900
212	42.5	20	37.5	900	257	48	20	32	900
213	41.25	21.25	37.5	900	258	33.3	66.6		900
214	40	22.5	37.5	900	259	33.3	59.16	7.5	900
215	38.75	22.5	38.75	900	260	48		52	700
216	42.5	15	42.5	900	261	50		50	700
217	41.25	16.25	42.5	900	262	52		48	700
218	42.5	16.25	41.25	900	263	37.5	25	37.5	900
219	41.25	17.5	41.25	900					