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STUDIES OF THE THORIUM-URANIUM DICARBIDE PSEUDOBINARY SYSTEM IN THE PRESENCE OF EXCESS GRAPHITE

Jackie Lee Cook

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Submitted as a thesis to the Graduate Council of the University of Tennessee
in partial fulfillment of the requirements for the degree of Master of Science.

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METALS AND CERAMICS DIVISION

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SEPTEMBER 1965

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CHAPTER I

SUMMARY

Mixed carbides of thorium and uranium have potential use as highly refractory fuel and breeder material for graphite-based gas-cooled reactors. This study combined dilatometry, x-ray diffraction, metallography, thermal analysis and chemical analysis to provide a better understanding of the phase relationships for the $\text{UC}_2\text{-ThC}_2$ pseudobinary system. Thermal expansion as a function of temperature up to 1000°C was determined for UC_2 , ThC_2 , and the $(\text{U,Th})\text{C}_2$ alloys using drop-cast and machined specimens.

The microstructural features of all as-cast alloys were similar and all contained excess graphite. Only the anneals above 1500°C for extended periods of time caused significant changes in the microstructure of the carbide or the morphology of the graphite. Microhardness tended to increase slowly with annealing time at 1900°C but did not change significantly for lower temperature anneals.

Powder x-ray diffraction patterns were generally diffuse and did not improve appreciably with annealing for material that contained much excess graphite. Specimens of ThC_2 that contained only a trace of free graphite gave very sharp diffraction patterns after annealing at 1400°C and below. X-ray diffraction revealed that two distinctly different body-centered tetragonal structures coexisted for alloys in the composition range from 35 to 42 mole per cent ThC_2 .

Dilatometry and thermal analysis clearly showed two phase transformations in ThC_2 [$(1500 \pm 25)^\circ\text{C}$ and $(1430 \pm 10)^\circ\text{C}$], and the results indicated that the higher temperature transformation was diffusionless. Two similar transformations were observed for ThC_2 -rich alloys containing up to 25 mole per cent UC_2 . One phase transformation was observed for UC_2 at $(1768 \pm 25)^\circ\text{C}$. One solid-state transformation occurred for the $(\text{U,Th})\text{C}_2$ alloys in the composition range from 25 to 75 mole per cent ThC_2 . The transformation temperatures in this composition range passed through a minimum (approximately 1100°C) at approximately 46 mole per cent ThC_2 .

Two phase diagrams, either of which is consistent with experimental observations, were proposed for the UC_2 - ThC_2 pseudobinary system.

The mean coefficients of expansion for the $(\text{U,Th})\text{C}_2$ alloys between room temperature and 1000°C varied from $8.6 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ for ThC_2 to $13.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ for UC_2 .

CHAPTER II

INTRODUCTION

Interest in mixed carbides of uranium-thorium and plutonium-thorium continues to grow because of their potential as highly refractory fuel and breeder material for graphite-moderated, gas-cooled, high-temperature converter reactors. If advanced fuel elements are to be designed for higher operating temperatures, dimensional stability, lower fission-gas release, and improved irradiation behavior, it is essential that additional information be available on phase relationships, mechanism and kinetics of transformations, and chemical behavior of the mixed carbides. The objectives of this investigation are to more thoroughly determine the phase relationships in the thorium-uranium dicarbide pseudobinary system in the presence of excess graphite and to determine the thermal expansion as a function of temperature and composition. Emphasis is placed on alloys ranging in composition from 29 to 45 mole per cent thorium dicarbide.

Phase relationships in the uranium-carbon and the thorium-carbon binary systems, except for the regions near UC_2 and ThC_2 ,* are generally known. In spite of extensive investigations of both systems, the more

*The formulas UC_2 and ThC_2 are used to denote the phases without implying stoichiometry.

complicated phase relations in the UC_2 and ThC_2 regions have not been completely defined.

The Uranium-Carbon System

Since the complete uranium-carbon system has recently been the subject of several reviews (1-3),* only those features associated with the dicarbide will be considered here. The general areas of agreement for this system are presented in Figure 1 in the most recent phase diagram [reported by Storms (3)]. The low-temperature crystal structure of UC_2 was determined by neutron diffraction (4,5) to be CaC_2 -type body-centered tetragonal (abbreviated bct henceforth). The neutron diffraction data confirmed the structure determined by x-ray diffraction by Rundle (6) and Litz (7) and gave a more positive identification of the carbon positions. Wilson (8), using high-temperature x-ray diffraction, reported the high-temperature form of UC_2 to have a CaF_2 -type face-centered cubic (abbreviated fcc henceforth) structure and the transformation temperature to be approximately $1820^\circ C$. In recent neutron diffraction work at Los Alamos, Storms (3) reported the high-temperature crystal structure of UC_2 as a pyrite structure. The transformation temperature was determined by Wallace (9) to be $(1785 \pm 20)^\circ C$. One additional feature of the uranium-carbon binary is the UC_2 decomposition to $U_2C_3 + C$ at temperatures below $(1500 \pm 100)^\circ C$ (refs. 10-12). The rate of the decomposition is determined by the temperature. The

*Denotes reference. See pages 124 through 129.

Figure 1. Uranium-Uranium Dicarbide System Tentative Phase Diagram Reported by Storms^a.

^aE. K. Storms, A Critical Review of Refractories, Los Alamos Scientific Laboratory, LA-2942, 1964.

crystal structure of the sesquicarbide, U_2C_3 , was determined by x-ray and neutron diffraction to be body-centered cubic (4,13). The melting point of UC_2 in contact with graphite has been reported to be $(2450 \pm 50)^\circ C$ (ref. 3).

The Thorium-Carbon System

Prior work on the thorium-carbon phase diagram has been adequately reviewed and reported in the literature (3,14,15). Wilhelm and Chiotti (16) established that only two phases, ThC and ThC_2 , were stable in this system and that thorium sesquicarbide, Th_2C_3 , did not exist. The absence of Th_2C_3 has been supported recently by Krikorian (3) and Langer et al. (15).

Langer et al. (15), using their own data plus the original diagram of Wilhelm and Chiotti (16) and revisions suggested by Hansen and Anderko (17), constructed the tentative diagram presented in Figure 2. Hunt and Rundle (18), using x-ray and neutron diffraction, established that the room-temperature structure of ThC_2 was monoclinic. Krikorian (14) reviewed the crystal structure studies and presented transformation equations for indexing ThC_2 x-ray diffraction patterns as either monoclinic or pseudo-orthorhombic. Gantzel (19) refined the data of Krikorian and more accurately defined the parameters of the monoclinic structure. Langer et al. (15), using high-temperature x-ray diffraction and thermal analysis, observed that the monoclinic form transformed to tetragonal ThC_2 at $(1427 \pm 21)^\circ C$ and that, on further heating, the tetragonal form transformed to fcc ThC_2 at $(1481 \pm 28)^\circ C$.

Figure 2. Thorium-Carbon System Tentative Phase Diagram
Constructed by Langer et al.^a

^aS. Langer, N. Baldwin, P. Gantzel, F. Kester, and C. Hancock, Studies in the Thorium-Carbon Binary System, General Atomic, GA-5204, 1964.

Hill and Cavin (20), using high-temperature x-ray diffraction and differential thermal analysis, originally reported a single transformation from monoclinic ThC_2 to fcc ThC_2 at $(1415 \pm 10)^\circ\text{C}$ but later reported two transformations (21). The lower transformation from monoclinic to bct ThC_2 occurred over the temperature range from 800 to 1250°C . The upper transformation from tetragonal to fcc was reported to occur at $(1420 \pm 20)^\circ\text{C}$.

Although the results of Hill and Cavin do not completely agree with those of Langer, it is entirely possible that errors in the measurement of temperature may be responsible for the apparent discrepancy. Electrical resistivity measurements obtained by Chiotti et al. (22), using a carburized thorium wire, indicated the presence of two distinct transformations. The lower transition was spread from $(1300 \text{ to } 1340 \pm 35)^\circ\text{C}$, but the upper transition was relatively sharp and occurred at $(1425 \pm 35)^\circ\text{C}$. It would appear that two transformations are present. Further, Langer (15), Hill and Cavin (21), and Chiotti (22) have noted a distinct difference in the two transformations.

Chiotti observed through electrical resistivity measurements that the slope of the resistance versus temperature curve for the lower transformation was characteristic of an ordering-type transformation. Hill and Cavin observed a gradual merging of diffraction lines from 800 to 1250°C , which suggested that the lower transition is something other than a first-order phase change. Langer observed that the lower

transition absorbed or liberated a much smaller amount of heat than the upper transformation. All of the investigators implied that the upper transformation is a diffusionless, martensitic type, as suggested by Chang (24).

The UC₂-ThC₂ System

The first work reported on the UC₂-ThC₂ system was by Brett et al. (25). Using x-ray diffraction and metallography, they suggested that the ThC₂-UC₂ system was one of complete solid solubility. However, it is not completely clear whether they had equilibrated their specimens since they reported very diffuse x-ray diffraction patterns for all alloys. Benesovsky and Rudy (12) reported a miscibility gap which was approximately 30 mole per cent wide at 1700°C and persisted to 1900°C.

Work by Hill and Cavin (21) failed to reveal any indication of a miscibility gap at high temperature based on x-ray diffraction up to 2000°C and differential thermal analysis up to 1500°C. The tentative diagram that resulted from this study is shown in Figure 3 and indicates a completely miscible solid solution with an fcc structure at elevated temperatures. At low temperature, a single-phase solid solution, α , was found having the UC₂ bct structure from 0 to 30 weight per cent ThC₂. Between 30 and 38 weight per cent ThC₂, a two-phase region was identified by x-ray diffraction, one phase based on bct UC₂ with $a = 3.65$ angstroms and $c = 6.06$ angstroms for a 33 weight per cent ThC₂ alloy and the other, γ , based on bct ThC₂ with $a = 4.03$ angstroms and

Figure 3. The $\text{UC}_2\text{-ThC}_2$ System (in the Presence of Excess Carbon) Tentative Phase Diagram Resultant from Work by Hill and Cavin^a.

^aN. A. Hill and O. B. Cavin, Phase Equilibrium Studies in the System UC₂-ThC₂, Oak Ridge National Laboratory, ORNL-3668, 1964.

$c = 5.16$ angstroms for a 50 weight per cent ThC_2 alloy. The tetragonal phase based on ThC_2 was stable from approximately 40 to 75 weight per cent ThC_2 .

Langer et al. (23) studied the UC_2 - ThC_2 system using thermal analysis and x-ray diffraction in an attempt to resolve the question of mutual miscibility at high temperature. Preliminary conclusions from their work indicated that a narrow two-phase region between 33 and 40 weight per cent ThC_2 exists at low temperatures. They questioned the existence of complete miscibility up to 1600°C but agreed that above 1600°C the components were completely soluble.

A portion of the results of previous investigators (21,23) of the ThC_2 - UC_2 system is summarized in Figure 4. These results indicate that carefully controlled experiments are needed to more precisely determine the phase relationships in the composition range associated with the two-phase region. Also, there remains the question of the existence of a miscibility gap above the transformation in the region from 30 to 40 mole per cent ThC_2 .

In the present investigation, dilatometry, thermal analysis, metallography, x-ray diffraction, and chemical analysis are used to provide a better definition of the phase relationships in the composition range 29 to 45 mole per cent ThC_2 .

Figure 4. A Portion of the ThC₂-UC₂ System (in the Presence of Excess Graphite) According to Hill and Cavin^a and Langer *et al.*^b

^aN. A. Hill and O. B. Cavin, Phase Equilibrium Studies in the System UC₂-ThC₂, Oak Ridge National Laboratory, ORNL-3668, 1964.

^bS. Langer, P. K. Gantzel, and N. L. Baldwin, "Solid Solutions of Uranium and Thorium Dicarbide" (paper presented at American Chemical Society Meeting, Chicago, Illinois, August 30-September 4, 1964).

CHAPTER III

EXPERIMENTAL DETAILS

Materials and Preparation

Uranium dicarbide, thorium dicarbide, and (U,Th)C₂ alloys were prepared from high-purity uranium and/or thorium metal and spectroscopic-grade carbon by arc melting and drop casting using a procedure similar to those described by Bradley et al. (26) and Stout (27). Chemical analyses of the starting materials are presented in Table I.

Small chips of thorium and/or uranium and carbon were placed in the water-cooled, copper "button mold" of the arc-melting furnace shown in Figure 5. The system was evacuated to a pressure of approximately 5×10^{-6} torr, and prior to melting, the pressure was increased to 500 torr by introducing a 50 per cent mixture of high-purity argon in helium. A zirconium getter button was melted initially to provide further purification of the environment. Using a graphite electrode and striker plate, individual buttons of ThC₂ and UC₂ were melted and turned several times to ensure homogeneity. The ThC₂ and UC₂ buttons were broken into small pieces and combined in amounts required for the (U,Th)C₂ alloys. After several meltings, the (U,Th)C₂ alloys were drop cast into a 1/2- or 3/8-inch-diameter by 2 1/2-inch-long copper mold. To prevent piping and cracking of these alloys, the casting mold was not water cooled. After cooling, the castings and all excess materials were

TABLE I
CHEMICAL ANALYSES OF STARTING MATERIALS

Starting Material	Impurity Element (ppm)																			
	Al	B	Be	C	Ca	Cu	Fe	H ^c	K	Mg	Mn	N ^c	Na	Ni	O ^c	Si	Th	U	W ^e	Zn
Thorium	11	2.8	1.6	160	2.7	3.5	36	6	7.7	2.2	0.9	15	6.8	a	140	a	99.68 x 10 ⁴ ^b	d	< 2	1.4
Uranium	8.0	0.2	0.2	140	1.1	4	48	2	0.8	1.1	4.0	22	2.7	1.6	32	a	d	99.94 x 10 ⁴ ^b	< 2	2.5
Carbon	a	a	a	99.94 x 10 ⁴ ^b	a	a	5	32	a	5	a	40	a	a	200	5	d	d	< 2	a

^aNot detected by spectrographic analysis.

^bDetermined gravimetrically.

^cDetermined by vacuum-fusion analysis.

^dNot determined.

^eDetermined by neutron activation.

Figure 5. Arc-Melting Furnace Used to Prepare $(\text{U,Th})\text{C}_2$ Alloys.

removed from the furnace and immediately placed under silicone oil in a dynamic-vacuum desiccator. A typical casting is shown in Figure 6.

The UC_2 and ThC_2 mixtures were prepared for melting with 9.3 to 9.36 weight per cent carbon (stoichiometric UC_2 contains 9.17 weight per cent carbon, and stoichiometric ThC_2 contains 9.38 weight per cent carbon). This amount of carbon, plus carbon picked up from the graphite electrode, ensured an excess of carbon in the final alloys.

The chemical compositions for all alloys are given in Table II. These analyses show that free carbon ranges from 0.9 to 2.6 weight per cent. Table III presents typical results for the major impurities found after arc melting. It is interesting to note that, by using a graphite electrode and striker plate, the tungsten content was less than ten parts per million in all alloys. The oxygen content ranged from 380 to 4900 parts per million, as shown in Table II, but no apparent correlation with thorium or uranium content was observed.

Sampling and Chemical Analysis

On the day of preparation, each alloy was sampled for chemical and x-ray diffraction analysis, metallography, and dilatometry. For the metallographic, x-ray, and chemical analyses, wafers approximately one-eighth inch thick were cut, with a high-speed, diamond, thin-sectioning saw and with the specimen submerged in silicone oil, from the center of the drop castings. The wafers remained in silicone oil until they were analyzed. X-ray analysis and metallography were initiated as soon as possible after the specimens were cut. However,

Figure 6. A Typical Drop Casting of Arc-Melted $(U,Th)C_2$. The casting is $3/8$ inch in diameter by $2\frac{1}{2}$ inches long.

TABLE II
CHEMICAL COMPOSITIONS OF ARC-CAST (U,Th)C₂ ALLOYS

Sample Designation	ThC ₂		UC ₂		Uranium (wt %)	Thorium (wt %)	Total Carbon (wt %)	Free Carbon (wt %)	Oxygen (ppm)
	(mole %)	(wt %)	(mole %)	(wt %)					
T-22	100.0	100.0	0	0	0	89.5	10.0	0.9	510
M-2	100.0	100.0	0	0	0	89.0	10.9	2.1	3500
M-3	87.7	87.5	12.3	12.5	11.1	77.1	9.9	-	4900
M-4	75.1	74.8	24.9	25.2	22.4	65.7	9.6	0.9	4800
T-21	62.3	61.9	37.7	38.1	34.2	55.0	10.2	1.4	1600
T-24	54.4	54.0	45.6	46.0	38.6	44.9	10.8	2.6	1200
T-20	51.1	50.6	48.9	49.4	44.0	44.8	10.4	1.8	1900
T-12	45.5	45.1	54.5	54.9	49.4	40.0	10.3	1.9	850
T-11	45.2	44.7	54.8	55.3	48.5	38.9	10.8	2.4	460
T-18	43.2	42.7	56.8	57.3	51.3	38.0	10.4	1.8	1000
T-7	41.7	41.3	58.3	58.7	51.8	36.1	10.8	2.0	1670
T-6	41.4	41.0	58.6	59.0	52.8	36.3	10.6	2.2	380
T-8	40.7	40.3	59.3	59.7	53.4	35.8	10.1	1.7	700
T-16	40.1	39.6	59.9	60.4	54.2	35.2	10.3	1.5	1400
T-14	37.6	37.2	62.4	62.8	56.4	33.1	10.1	1.4	1200
T-15	36.6	36.2	63.4	63.8	57.1	32.2	10.9	2.0	2000
T-3	35.3	34.9	64.7	65.1	57.7	30.6	10.9	2.3	1200
T-2	35.0	34.7	65.0	65.3	58.1	30.8	10.4	1.5	3900
T-5	35.0	34.7	65.0	65.3	58.2	30.7	10.5	2.3	600
T-17	34.0	33.7	66.0	66.3	59.6	30.0	10.2	1.4	970
T-19	32.6	32.2	67.4	67.8	60.9	28.6	10.3	1.6	740
T-10	29.7	29.4	70.3	70.6	63.0	26.0	11.0	2.4	850
T-9	29.3	29.0	70.7	71.0	63.1	25.6	10.6	2.0	1800
M-6	25.4	25.1	74.6	74.9	66.6	22.1	9.6	1.4	3700
T-23	0	0	100.0	100.0	88.9	0	9.9	1.4	470

TABLE III

MAJOR IMPURITIES IN ARC-MELTED (U,Th)C₂ ALLOYS

Element	Concentration ^a (ppm)
Ag	2
Al	10
Ca	12
Cd	< 10
Cr	5
Cu	4
Fe	30
H	22-86 ^b
Mg	2
Mn	< 2
Mo	< 4
N	30-85 ^b
Ni	6
O	380-4900 ^b
Sn	< 10
V	< 2
W	< 10 ^c
Zn	< 2

^aDetermined by spectrographic analysis unless otherwise indicated. The concentrations reported are typical values observed for all the compositions examined.

^bDetermined by vacuum fusion.

^cDetermined by neutron activation.

samples for chemical analysis were put into solution on the same day that they were prepared. For dilatometry, two specimens approximately one inch long were cut from the cast section. The remainder of the casting was stored in silicone oil in a dynamic-vacuum desiccator until needed for annealing studies and thermal analysis. Samples for chemical analysis weighed from two to five grams, and each sample was analyzed for uranium, thorium, total and free carbon, tungsten, oxygen, hydrogen, and nitrogen. The complete analytical procedures for the determination of uranium, thorium, and carbon in carbides are given in the literature (28-31). Tungsten was determined by neutron activation after dissolution with nitric acid and a trace of hydrofluoric acid. Vacuum fusion, using a platinum bath at 1900°C, was used to obtain the oxygen, hydrogen, and nitrogen contents. Precision of the analyses is estimated as follows:

Element Analyzed	Precision (%)
Uranium	±0.5
Thorium	±1.0
Total and free carbon	±5.0
Oxygen	±10.0
Hydrogen	±10.0
Nitrogen	±10.0
Tungsten	±5.0

The mass balance for the analyses given in Table II ranged from 98.2 to 101.1 weight per cent.

From analytically determined weight percentages of uranium and thorium, the mole percentages of UC_2 and ThC_2 were calculated according to Equations (1) through (3).

$$\frac{\text{weight fraction uranium}}{\text{atomic weight uranium}} = \text{moles uranium} = \text{moles UC}_2 . \quad (1)$$

$$\frac{\text{weight fraction thorium}}{\text{atomic weight thorium}} = \text{moles thorium} = \text{moles ThC}_2 . \quad (2)$$

$$\frac{\text{moles ThC}_2}{\text{moles (UC}_2 + \text{ThC}_2)} \times 100 = \text{mole per cent ThC}_2 . \quad (3)$$

The mole per cent to weight per cent conversion was made assuming a carbon-to-uranium atom ratio of 1.86 (refs. 32-34) and a carbon-to-thorium atom ratio of 2.0.

To check the sampling procedure and to determine whether the alloys were segregated, seven samples were cut from a casting containing 45.5 mole per cent ThC₂ (specimen T-12), as shown in Figure 7. Each sample was analyzed for uranium, thorium, and total and free carbon. The results given in Table IV showed that the uranium, thorium, and total-carbon contents were uniform within the limits of the chemical analyses but that the free-carbon content varied with sample position. The results indicated that the samples from other castings for chemical and x-ray analyses and metallography were indeed representative.

Using an x-ray fluorescence spectrograph with a one-millimeter-diameter x-ray beam, the homogeneity of the samples was examined on a finer scale. The thorium and uranium intensities were measured at one-eighth- to one-fourth-inch intervals along the length of the casting. Using known compositions of (U,Th)C₂, it was possible to convert the intensity ratios to thorium-to-uranium weight ratios. The weight ratios for five alloys were observed to vary less than the counting statistics along the entire length of the castings.

Figure 7. Sample Position for the Determination of Compositional Variations in a $(U,Th)C_2$ Drop Casting.

TABLE IV
COMPOSITIONAL VARIATIONS OF AN ARC-CAST, (U,Th)C₂ SPECIMEN (T-12)
AS MEASURED BY CHEMICAL ANALYSIS

Sample Designation	Uranium (wt. %)	Thorium (wt. %)	Total Carbon (wt. %)	Free Carbon (wt. %)	ThC ₂		UC ₂	
					(mole %)	(wt. %)	(mole %)	(wt. %)
T-12A	49.78	40.41	10.29	2.75	45.43	45.01	54.57	54.99
T-12B	49.22	40.32	10.28	1.78	45.66	45.24	54.34	54.77
T-12C	49.48	40.29	10.34	1.71	45.51	45.09	54.49	54.91
T-12D	49.23	40.18	10.34	1.74	45.57	45.15	54.43	54.85
T-12E	49.60	40.12	10.34	1.78	45.34	44.92	54.66	55.08
T-12F	49.36	40.18	10.34	1.62	45.50	45.08	54.50	54.92
T-12G	49.39	40.09	10.40	1.99	45.43	45.01	54.57	54.99
Average value	49.44	40.23	10.33	1.91	45.49	45.07	54.51	54.93
Maximum value	49.78	40.41	10.40	2.75	45.66	45.24	54.66	55.08
Minimum value	49.22	40.09	10.28	1.62	45.34	44.92	54.34	54.77
Standard deviation	0.20	0.12	0.04	0.39	0.10	0.10	0.10	0.10

Using an electron microprobe with a two-micron-diameter beam, an intensity trace for thorium, uranium, and carbon in a 35.0 mole per cent ThC₂ alloy (specimen T-5) was made. The trace across the three-eighth-inch-diameter specimen is given in Figure 8. X-ray fluorescence and electron microprobe analyses support the chemical findings that the thorium and uranium were uniformly distributed while the excess carbon exhibited a large degree of segregation.

Isothermal Annealing

Isothermal anneals were carried out at 1000, 1100, 1200, 1300, 1500, and 1900°C for times ranging from 150 to 1750 hours. Irregular pieces of (U,Th)C₂, each weighing from one to three grams, were chipped under silicone oil from the castings. Samples were taken from the cast section and from the top of the casting (see Figure 6, page 17). Following the simultaneous removal of all samples from the silicone oil, most of the oil was wiped off with a paper towel. The samples were then rinsed lightly with carbon tetrachloride, loaded into a graphite crucible, and completely covered with 177- to 250-micron-diameter, graphitized coke particles. The crucible was fitted with a lid which had a 0.060-inch-diameter hole in the center for gas release and temperature measurement.

For anneals at 1100°C and below, the loaded graphite crucibles were put in a tantalum tray and placed in a Marshall Products wire-wound furnace capable of continuous operation at 1200°C. The furnaces shown in Figure 9 were fitted with two-inch-diameter by

Figure 8. An Electron-Microprobe Relative-Intensity Scan for Uranium, Thorium, and Carbon in a 35 Mole Per Cent (U,Th)C₂ Alloy.

Figure 9. Furnaces for Vacuum Annealing at 1000°C and Below

twenty-four-inch-long McDanel's-30-grade mullite tubes and were sealed by Pyrex taper joints to a vacuum manifold. The pressure in this system was maintained at 5×10^{-6} torr. Temperature was sensed with a Pt-10 per cent Rh versus platinum thermocouple placed between the furnace liner and the mullite tube and controlled by a Wheelco Model 402 capacitrol time-proportioning controller. Temperature of the specimens was determined by inserting a Pt-10 per cent Rh versus platinum thermocouple into an alumina thermocouple well positioned directly over the specimens. The measuring thermocouples were calibrated by periodic comparison with a standard thermocouple of the same composition which had been calibrated at the melting points of gold, silver, aluminum, and zinc. The precision of temperature measurements in these furnaces was $\pm 5^\circ\text{C}$ or less. The hot zone for these furnaces was uniform within $\pm 5^\circ\text{C}$ for eight inches at 1000°C , and the maximum cooling rate, without forced cooling, between 1000 and 700°C was 3.6°C per minute.

Anneals above 1100°C were performed in graphite resistance furnaces having concentric-tube heating elements. One such furnace is shown schematically in Figure 10. Tantalum radiation shields, shown in Figure 10, were used in furnace No. 1 [for annealing at temperatures between 1200 and 2000°C (Figure 11)] and in furnace No. 2 [for annealing at temperatures between 1200 and 1800°C (Figure 12)]. Carbon felt, National Carbon VDF-grade, was used to insulate furnace No. 3 [for annealing at temperatures between 1200 and 2500°C (Figure 13)].

Furnaces Nos. 1 and 2 were identical except for the outer shells. Furnace No. 1 had an aluminum, double-wall, water-cooled shell, whereas

Figure 10. Schematic Representation of the Concentric-Tube, Resistance-Heated, Graphite, Vacuum Furnaces Used for Annealing at Temperatures Between 1200 and 2500°C.

Figure 11. Concentric-Tube, Resistance-Heated, Graphite, Vacuum Furnace No. 1 Used for Annealing at Temperatures Between 1200 and 2000°C.

Figure 12. Concentric-Tube, Resistance-Heated, Graphite, Vacuum Furnace No. 2 Used for Annealing at Temperatures Between 1200 and 1800°C.

Figure 13. Concentric-Tube, Resistance-Heated, Graphite Furnace No. 3 Used for Annealing at Temperatures Between 1200 and 2500°C.

furnace No. 2 had a quartz shell. The graphite heating element for these two furnaces was 1 1/4 inches in diameter by 9 inches long and had a $\pm 5^{\circ}\text{C}$ uniform 3 1/2-inch-long hot zone that was displaced approximately 3/4 inch from center. The hot zone was determined by placing a Pt-10 per cent Rh versus platinum thermocouple inside an alumina thermocouple well that was passed through the furnace from one end as shown in Figure 12. Temperature was manually controlled using a 220-volt constant-voltage transformer, a step-down transformer, and an autotransformer. The constant-voltage transformer eliminated most of the line voltage fluctuation, and the temperature at a given level was observed to vary less than $\pm 5^{\circ}\text{C}$ during a 500-hour test. The power requirement was 2.6 kilowatts at 1600°C . Temperatures were measured with a Pyrometer Instrument Company micro-optical pyrometer by sighting directly on the samples through a hole in the crucible lid. All temperatures were corrected for absorption due to the pyrometer, sight glasses, and necessary prisms according to the data given in Appendix A, page 130. These data were obtained by the Standards Laboratory of the Oak Ridge National Laboratory. Each furnace was maintained at a pressure equal to or less than 1×10^{-5} torr. The maximum cooling rate for furnaces Nos. 1 and 2 was 150°C per minute between 1500 and 800°C .

Furnace No. 3 was composed of a three-inch-diameter by fifteen-inch-long heater and a $\pm 20^{\circ}\text{C}$ uniform eight-inch hot zone. Temperature was manually controlled with a saturable reactor and step-down transformer. Variations for a 200-hour test were $\pm 25^{\circ}\text{C}$ at 1900°C .

Power requirement at 1900°C was 3.5 kilowatts. This system was maintained at a pressure of 5×10^{-5} torr at temperature and had a maximum cooling rate of 45°C per minute between 1900 and 1200°C.

The operating procedure for annealing was to place the samples into an appropriate furnace, evacuate to a pressure of 5×10^{-5} torr or less, and heat as rapidly as possible to the annealing temperature while maintaining the desired vacuum. Upon completion of an anneal, the furnace was cooled at the maximum rate by shutting off the power or at a slow cool by slowly reducing the power level. Rates ranging from the maximum to 4°C per minute were obtained. After the sample reached room temperature, the crucible was taken from the furnace and three pieces were removed from each composition for x-ray diffraction, metallography, and thermal or chemical analysis. The crucibles with the remaining pieces were reloaded and the annealing was continued.

Dilatometry

Specimens for thermal expansion were approximately one inch long and from one-fourth to one-half inch in diameter for most of the compositions given in Table II, page 18. Specimens were prepared either by cutting a cylindrical section from the drop castings as described previously or by electrical-arc-discharge machining an arc-melted button under transformer oil. Specimens were successfully machined by this method from arc-melted ThC_2 and alloys containing 25.4, 29.7, 40.7, 75.1, and 87.7 mole per cent ThC_2 . For detection of internal

defects prior to measurement, all specimens were radiographed with a one million-volt x-ray source.

Measurements at pressures from 2×10^{-6} to 1×10^{-5} torr were made with a fused-silica dilatometer of the type shown in Figure 14 and by a system described by Fulkerson (35). Approximately linear heating rates of from 2.0 to 2.5°C per minute were obtained with a Pt-40 per cent Rh, wire-wound resistance furnace and a variable-speed motor-driven autotransformer. Temperature was measured with a calibrated Pt-10 per cent Rh versus platinum thermocouple, which was positioned approximately one thirty-second inch from the cylindrical surface of the specimen. The furnace was observed to have a $\pm 5^\circ\text{C}$ uniform four-inch-long hot zone, and specimens were positioned in the center of the hot zone, as shown in Figure 15.

Dilation measurements from 40 to 1000°C were made during heating by reading a dial gage (smallest division - 0.0001 inch) at temperature intervals of approximately 60°C. Each specimen was measured a minimum of three times, and average values were calculated. The precision for a determination was estimated to be ± 2 per cent.

A computer program,* given in Appendix B, page 133, was written for calculating, averaging, and plotting the cumulative expansion and the mean and instantaneous temperature coefficients of expansion. Typical data and plots for the program are given in

*The initial computer program was written by W. A. Laing, Jr., and the final program was written by S. E. Atta.

Figure 14. Schematic Representation of the Dilatometers Used to Measure the Thermal Expansion of Arc-Cast $(U,Th)C_2$ Alloys.

Figure 15. Cross Section of Thermal Expansion Furnace.

Appendix C, page 150. Cumulative expansions and the mean temperature coefficients of expansion were calculated according to Equations (4) and (5).

$$\text{cumulative expansion (in./in.)} = \frac{1}{L_0} (\Delta L) . \quad (4)$$

$$\text{mean temperature coefficient (}^{\circ}\text{C}^{-1}\text{)} = \frac{1}{L_0} \left(\frac{\Delta L}{\Delta T} \right) . \quad (5)$$

L_0 refers to the initial specimen length at room temperature, and ΔL refers to the change in length for a corresponding change in temperature, ΔT . Using the method of least squares, the mean temperature coefficients and the cumulative expansion data as a function of temperature were fitted to a first-, second-, or third-order polynomial. The equation expressing cumulative expansion as a function of temperature above the reference level was determined and subsequently differentiated to obtain the instantaneous coefficients of expansion.

A correction for the silica dilatometer was determined using high-purity tungsten and silica as standards. The corrections, shown in Appendix C, page 150, were a linear function of temperature, and the computer was instructed to interpolate and apply the necessary corrections to all data. The calibration was checked three times during this investigation and was not observed to change.

Thermal expansion for the above samples was also recorded as a function of time and temperature using tantalum and graphite dilatometers shown in Figures 16 and 17, respectively. Length changes, determined with a linear variable differential transformer, and temperatures, determined with a Pt-10 per cent Rh versus platinum

Figure 16. The Tantalum Dilatometer Capable of Manual Operation Using the Dial Gage or Automatic Recording with a Linear Variable Differential Transformer.

Figure 17. The Graphite Dilatometer Capable of Automatic or Manual Operation.

thermocouple, were recorded simultaneously as a function of time using equipment described by Fulkerson (35). Both traces were marked simultaneously at fifteen-minute intervals to provide a correlation between dilation and temperature. Inflections in the dilation versus time curves were used to determine phase transformations, and temperatures of these transformations were obtained from the temperature versus time trace. Temperature precision was no more than $\pm 5^\circ\text{C}$. Using a furnace and control system as described above, each sample was heated in vacuum (less than 5×10^{-6} torr) to temperatures from 1400 to 1600°C at rates of 0.32 to 6.5°C per minute. Each sample was examined a minimum of two times for phase changes, but no attempt was made to determine expansion coefficients from the data obtained in the graphite or tantalum dilatometer.

The ability of the above systems to accurately determine transformation temperatures was verified using a sample of high-purity iron.* The alpha-gamma transformation in iron was observed to occur at $(916 \pm 5)^\circ\text{C}$ on heating and $(905 \pm 5)^\circ\text{C}$ on cooling. These transformation values compare favorably with the literature transformation values of "about" 910°C on heating and 900°C on cooling (36).

Thermal Analysis

In addition to dilatometry, solid-state transformations were determined by observing inflection points on the temperature versus

*High-purity iron supplied by J. P. Moore, Physical Properties Group, Metals and Ceramics Division, Oak Ridge National Laboratory.

time curves obtained on heating and cooling various compositions. With equipment (Figure 18) similar to that described by Rupert (37), samples contained in a graphite crucible (Figure 19) were heated inductively in vacuum (1×10^{-6} torr) using a 20-kilowatt, 360-kilocycle-per-second power supply and current concentrator. The radiation, or light, emitted through a one-sixteenth-inch-diameter sight hole in the lid of the crucible passed through two quartz sight glasses into an optical system that focuses approximately 70 per cent of the incident light on a light sensing element (photodiode). The remaining 30 per cent is reflected at right angles to the incident beam to permit simultaneous determination of the sample temperature using a micro-optical brightness pyrometer. The optical pyrometer was periodically calibrated with a reference National Bureau of Standards strip lamp, and absorption due to sight glasses and the beam splitter was determined. The correction data are given in Appendix D, page 161. Temperatures determined with the micro-optical pyrometer were reported with a $\pm 25^{\circ}\text{C}$ uncertainty.

The photodiode output provided the input to the thermal analyzer system shown schematically in Figure 20. Utilizing an oscilloscope and/or an X-Y plotter, the light intensity, which is correlative with temperature, was plotted versus time. Also, the derivative of the temperature curve as a function of time (dT/dt) was provided through the use of an electronic differentiator circuit. Small changes in the slope of temperature versus time curve due to a small heat of transformation were amplified significantly on the derivative curves, thus

Figure 18. A Photograph of the Thermal Analysis System.

Figure 19. Schematic Drawing of the Thermal Analysis Furnace.

Figure 20. Schematic Representation of the Thermal Analysis Circuit.

allowing more accurate determination of the transformation temperatures. The derivative, dT/dt , was plotted, simultaneously with temperature, versus time or versus temperature when linear heating or cooling rates were used.

Although differential thermal analysis was not used in this work, the system was designed for such. Through the use of two photodiodes, the temperature versus time curve of two separate samples can be displayed on a dual-beam oscilloscope or a two-pen X-Y plotter, and either the differential or difference signal between the two photodiodes or the derivative of either of the curves can be simultaneously displayed on the same trace.

The power-control system was designed to provide the following types of temperature control:

1. Manual steady-state control at a fixed temperature.
2. Automatic steady-state control at a fixed temperature based on crucible brightness or output power.
3. Incremental changes in temperature between predetermined steady-state levels.
4. Linear changes of temperature at rates adjustable between 3 and 500°C per minute in the temperature range of 1000 to 2800°C.

The control system consisted of a photodiode detector for observation of brightness of the crucible wall, or a power feed-back loop, the power-control system, a magnetic amplifier, the high-frequency generator, and the load coil and current concentrator, which heat the crucible.

As a calibration, the melting point of nickel was determined to be $(1460 \pm 25)^{\circ}\text{C}$ on heating at a rate of 10°C per minute and the melting point of copper was observed to be $(1080 \pm 25)^{\circ}\text{C}$ on heating at a rate of 25°C per minute. These values compare favorably with the literature values of 1453°C and 1083°C for nickel and copper, respectively.

Metallography, Microhardness, and X-Ray Diffraction

Samples of the as-cast and annealed materials were examined by metallography and x-ray diffraction using standard techniques.

Metallographic specimens were prepared according to the procedure described by Kegley (38). Examination was made at 100 to 1000x using bright field illumination and polarized light.

Using a Kentron microhardness tester with a Knoop indenter under a 100-gram load, microhardness was measured on all metallographic samples. For each specimen, five to ten impressions were measured, and the average values and the spread of the data were reported.

X-ray diffraction patterns were obtained with a Norelco, 114.6-millimeter Debye-Scherrer camera and a Norelco high-angle diffractometer fitted with a scintillation detector and pulse-height analyzer circuit. Nickel-filtered CuK_{α} radiation ($\lambda = 1.5418$ angstroms) was used. Specimens for Debye-Scherrer or powder patterns were crushed and loaded into 0.3-millimeter-diameter soft glass capillaries in an argon-filled glove box. The capillaries were sealed with Apiezon and transferred from the box and fused with a match. Specimens examined

on the diffractometer were ground under silicone oil on 400-grit silicon carbide paper. A film of oil was left on the sample, and the sample was mounted in an aluminum window frame holder and held in place with molding clay. Diffractometer traces were made scanning at one degree 2θ per minute between twenty-four and sixty degrees 2θ .

The powder patterns and the diffractometer traces were compared with UC_2 and ThC_2 standards for phases present. Very few measurements were made since most of the powder patterns were too diffuse to resolve at angles higher than $2\theta = 90$ degrees.

CHAPTER IV

RESULTS AND DISCUSSION

Two types of results were obtained for the arc-cast materials having the chemical composition given in Table II, page 18.

1. By microstructure, microhardness, density, x-ray diffraction, and thermal expansion, alloys were characterized as a function of composition.
2. By dilatometry and thermal analysis, solid-state transformations were detected as a function of temperature and composition.

The characterization of the material was necessary for a better understanding of the solid-state transformations, and these results will be presented first.

Microstructure

The microstructures of arc-cast ThC_2 , UC_2 , and $(\text{U,Th})\text{C}_2$ alloys were very similar, as shown in Figures 21 through 27. To prevent complications by the monocarbide phase, all alloys prepared for this investigation contained between 0.9 and 2.6 weight per cent free carbon. The chemical analyses, given in Table IV, page 23, and metallography showed that the free-carbon content was not homogeneous. Typical variations in the morphology of the free carbon, or more precisely, the free graphite, are presented in Figure 21. The morphology of the

Figure 21. Arc-Melted ThC_2 , UC_2 , and $(\text{U,Th})\text{C}_2$ Alloys Containing Excess Graphite. Bright field. Etchant: 50 parts HNO_3 , 50 parts H_2O . 100X. (a) ThC_2 containing 2.1 weight per cent free carbon. (b) $(\text{U,Th})\text{C}_2$ containing 35.3 mole per cent ThC_2 and 2.3 weight per cent carbon. (c) $(\text{U,Th})\text{C}_2$ containing 51.1 mole per cent ThC_2 and 1.4 weight per cent carbon. (d) UC_2 containing 1.4 weight per cent free carbon.

Figure 22. Arc-Melted $(U,Th)C_2$ Alloy Containing 34.0 Mole Per Cent ThC_2 . Determined by x-ray diffraction to contain graphite and a bct UC_2 solid solution. Etchant: 50 parts HNO_3 , 50 parts H_2O . 500X. (a) Bright field. (b) Polarized light.

Figure 23. Arc-Melted (U,Th)C₂ Alloy Containing 36.6 Mole Per Cent ThC₂. Determined by x-ray diffraction to contain graphite and approximately equal amounts of a bct UC₂ phase and a bct ThC₂ phase. Etchant: 50 parts HNO₃, 50 parts H₂O. 500X. (a) Bright field. (b) Polarized light.

Figure 24. Arc-Melted (U,Th)C₂ Alloy Containing 45.2 Mole Per Cent ThC₂. Determined by x-ray diffraction to contain graphite and a bct ThC₂ solid solution. Etchant: 50 parts HNO₃, 50 parts H₂O. 500X. (a) Bright field. (b) Polarized light.

Figure 25. Arc-Melted (U,Th)C₂ Alloy Containing 75.1 Mole Per Cent ThC₂. Determined by x-ray diffraction to contain graphite and a bct ThC₂ solid solution. Etchant: 50 parts HNO₃, 50 parts H₂O. 500x. (a) Bright field. (b) Polarized light.

Figure 26. Arc-Melted UC_2 Alloy Containing Excess Graphite.
Bright field. Etchant: equal parts HNO_3 , H_2O , and CH_3COOH . 250X.

Figure 27. Arc-Melted ThC_2 Alloy Containing Excess Graphite. Etchant: 50 parts HNO_3 , 50 parts H_2O . 500X. (a) Bright field. (b) Polarized light.

free graphite varied in an apparently random manner with respect to uranium or thorium content. Frequently, the alloys contained a combination of the typical-type distributions of graphite shown in Figure 21. For example, some alloys contained large, curved graphite flakes separated by small graphite platelets in a particular region and a dispersion of predominately medium-sized flakes or fine flakes in adjacent regions, as shown in Figure 21. All alloys contained both the relatively large graphite flakes or stringers and finely divided platelets, as shown in Figure 22. Figures 22 through 25 show typical microstructures for the arc-cast $(U,Th)C_2$ alloys. Microstructures for UC_2 and ThC_2 in the presence of excess graphite are shown in Figures 26 and 27, respectively. It was not possible to distinguish by metallographic means the microstructures of $(U,Th)C_2$ alloys that x-ray diffraction showed to contain a bct UC_2 or a bct ThC_2 solid solution. This can be seen by comparing Figures 22 and 24. Similarly, alloys that x-ray diffraction showed to contain both the UC_2 and the ThC_2 tetragonal phases appeared as a single-phase alloy plus graphite (Figure 23).

The characteristic feature of all the microstructures for the arc-melted UC_2 , ThC_2 , and the $(U,Th)C_2$ alloys was the banded substructure. This substructure was composed of main bands and sub-bands, and both assumed shapes ranging from lath to lenticular. The bands were not readily apparent in bright-field illumination but responded to polarized light in a manner characteristic of an anisotropic material. The lenticular shape of many of these bands in Figures 22 and 26 suggest that they were nucleated inside a grain and propagated in two

opposite directions; this would account for the larger width dimension in the center of lenticular plates. The bands generally terminated at grain boundaries and graphite flakes, but occasionally bands were stopped by intersection with another band. In samples that contained a large number of free graphite platelets (Figure 27), the banded substructure was very fine except in sample areas that were devoid of excess graphite. This is consistent with the previous observation that graphite flakes serve as terminal points for growing bands. The other extreme in substructure growth is shown in Figure 28 for a sample of ThC_2 that contained only a small quantity of free graphite.* Details of some sub-bands are shown in Figure 28(b).

Interpretation of these banded microstructures in terms of the phase diagram is complex. As shown in Figure 3, page 10, the alloys of UC_2 and ThC_2 form a continuous series of fcc solid solutions at elevated temperatures, and at low temperatures, except for the thorium-rich alloys, the structure is bct. Thorium dicarbide and alloys containing up to 25 mole per cent UC_2 have been observed to transform from fcc to bct and subsequently to monoclinic on cooling (21,23). All the alloys undergo at least the cubic to tetragonal transformation during cooling from the melt, and this transformation is accompanied by a volume change of less than 1 per cent for all compositions according to Hill and Cavin (21). This small volume change, plus the appearance of the main

*This sample and the photomicrograph shown in Figure 28(b) were made available by M. J. Bradley and T. M. Kegley, Jr.

Figure 28. Arc-Melted ThC_2 Alloys. Polarized light. Etchant: 50 parts HNO_3 , 50 parts H_2O . (a) Contains 0.3 weight per cent carbon. 1000X. (b) Contains 0.06 weight per cent carbon. 250X.

bands, suggests that the transformation is probably diffusionless, as was first suggested by Chang (39). Further support for this type of transformation is the marked similarity in banded microstructures of the (U,Th)C₂ alloys and the diffusionless transformation from cubic to tetragonal in the indium-thallium alloys (40), the chromium-manganese alloys (41), and the copper-manganese alloys (42). Also, the microstructures are not greatly different from the diffusionless transformations from cubic to hexagonal close packed for the cobalt-molybdenum alloys (43).

A particularly interesting comparison between the microstructures of ThC₂ (Figure 28) and the indium-thallium alloys was observed. The main bands in the indium-thallium alloys contained fine sub-bands, which were similar to those seen in Figure 28(b). The two sets of bands for the indium-thallium alloys were explained in terms of a double-shear mechanism (40). A similar explanation can be applied to the ThC₂ microstructure shown in Figure 28(b). The main bands that are almost vertical in this photomicrograph were probably formed by the initial shear, and the uniform sub-bands were probably formed by a second shear as the material transforms from fcc to bct. According to Figure 2, page 7, a second polymorphic transformation from bct to monoclinic occurs on further cooling. However, this transformation is accompanied by both less atomic rearrangement and a smaller volume change than the first transformation and does not appear to change the microstructure significantly. An additional feature of the microstructure, Figure 28(b), was the randomly spaced lenticular plates that cross the sub-bands but are

retained within the main bands. These plates are probably deformation twins that result from the anisotropic expansion and/or contraction of the monoclinic material during heating or cooling. In an attempt to separate the portions of the microstructure that were a result of the cubic to tetragonal transformation from the tetragonal to monoclinic transformation, anneals were carried out at temperatures below and above the lower transformation. Samples of ThC_2 having the microstructure seen in Figure 28 were annealed at 1350°C for 200 hours, 1400°C for 368 hours, and 1455°C for 370 hours. The resultant microstructures are shown in Figures 29 through 31. The sample that was heated in the ThC_2 monoclinic solid solution region, Figure 29, had a more equiaxed grain structure with some lenticular plates, probably deformation twins. Heating to 1400°C (Figure 30), still in the ThC_2 monoclinic solid solution region according to Langer et al. (23), gave an equiaxed grain structure that contained more lenticular bands than the previous anneal. For an explanation consistent with the phase diagram shown in Figure 2, page 7, all of these bands observed in Figure 30 must also be deformation twins. Even after this anneal, there is still some of the original as-cast structure that has not "recrystallized." Annealing at 1455°C , which according to the phase diagram of Figure 2, page 7, is in the ThC_2 bct solid solution region, yielded the microstructure shown in Figure 31. Nothing in the room-temperature microstructure for this anneal can be definitely associated with the tetragonal to monoclinic transformation. Some of the larger bands may be transformation bands, but their similarity to the twins makes a distinction impossible. The

Figure 29. Sample of ThC_2 after Annealing 200 Hours at 1350°C . Polarized light. Etchant: 50 parts HNO_3 , 50 parts H_2O . (a) 1000x. (b) 200x.

Figure 30. Sample of ThC₂ after Annealing 368 Hours at 1400°C. Polarized light. Etchant: 50 parts HNO₃, 50 parts H₂O. (a) 100x. (b) 500x.

Figure 31. Sample of ThC₂ after Annealing 370 Hours at 1455°C. Polarized light. Etchant: 50 parts HNO₃, 50 parts H₂O. (a) 100X. (b) 500X.

results from these three anneals support the explanation given for the arc-cast microstructure (Figure 28, page 58). It is interesting to note that the sub-bands were present only in ThC_2 and $(\text{U,Th})\text{C}_2$ alloys containing less than 25 mole per cent UC_2 , which corresponds to the composition range where the ThC_2 bct solid solution undergoes a transformation to monoclinic on cooling. Further, it is interesting that the anneals yielded microstructures that are similar to recrystallization in a plastically deformed material.

The large amount of grain growth shown in Figures 29 and 31 was not attained for comparable anneals on ThC_2 or $(\text{U,Th})\text{C}_2$ alloys that contained large amounts of free graphite. This is not too surprising in view of the large number of relatively immobile graphite plates that are present and act as barriers to grain boundary migration. The typical effect of annealing time and temperature on the microstructure of the arc-melted $(\text{U,Th})\text{C}_2$ alloys containing large amounts of free graphite is shown in Figure 32. Below 1500°C , essentially no change in the microstructure was observed for the times shown. At 1500°C , some rearrangement of the fine graphite platelets was observed after 665 hours, and after 1165 hours, they appear to be almost completely aligned along certain crystallographic directions similar to a Widmanstätten structure. At 1900°C , the fine platelets have almost disappeared after 500 hours, and the large graphite flakes are considerably larger than for the as-cast alloys. Although it appears that the carbon solubility for these alloys was slightly increased at 1900°C , it is impossible to confirm metallographically without the use of such quantitative

Figure 32. The Effect of Annealing on the Microstructure of a $(U,Th)C_2$ Alloy Containing 35.0 Mole Per Cent ThC_2 and 2.3 Weight Per Cent Free Graphite. Bright field. Etchant: 50 parts HNO_3 , 50 parts H_2O . 250X. Reduced 71%.

techniques as linear analysis for the amount of free graphite. Another observation that suggested an increase in carbon solubility at 1900°C is illustrated in Figure 33. Graphitized coke particles, which were used as packing material for annealing, stuck to the specimens and were apparently absorbed by the specimens. Prior to the anneal the coke particles were spheroids.

Microhardness and Density

Densities for the as-cast alloys were determined as a function of composition using silicone oil and an immersion technique. The results are presented and compared with a calculated density in Table V. The calculated densities are not to be interpreted as theoretical densities since they were obtained by using the analytical chemistry results for the UC_2 , ThC_2 , and free carbon of the respective alloys. The precision of the calculated value is therefore approximately equal to the sum of the precisions of the three analyses, or ± 6.5 per cent. The precision of the density determination is ± 0.5 per cent. All specimens were radiographically inspected for defects prior to measurement, and only one sample, T-12A, was found to be defective. Since these densities were determined on solid, arc-melted and drop-cast samples that should have a density between 97 and 99 per cent of theoretical, errors in the calculated densities probably cause the slightly low values for the per cent of calculated.

The microhardness values, Knoop Hardness Numbers (abbreviated KHN henceforth), for the arc-melted materials are given in Table VI.

Figure 33. A (U,Th)C₂ Alloy Containing 41.7 Mole Per Cent ThC₂ That Was Annealed at 1900°C for 150 Hours. Prior to annealing, the material contained 2.0 weight per cent free graphite, but the graphitized coke particles are apparently being absorbed by the carbide. Etchant: 50 parts HNO₃, 50 parts H₂O. 250X. (a) Bright field. (b) Polarized light.

TABLE V
OIL-IMMERSION DENSITIES FOR (U,Th)C₂ ALLOYS

Sample Designation	Composition		Density		Per Cent of Calculated
	ThC ₂ (wt %)	Free Carbon (wt %)	Calculated ^a (g/cm ³)	Experimentally Determined ^b (g/cm ³)	
T-22A	100	0.90	9.34	8.90	95.2
M-2	100	2.1	9.00	8.69	96.5
M-3	87.5	-	-	9.13	-
M-4	74.8	0.9	9.76	9.42	96.5
T-21B	61.9	1.4	9.82	9.37	95.4
T-20B	50.6	1.8	9.88	9.56	96.7 ^c
T-12A	45.1	1.9	10.36	9.56	92.2 ^c
T-11A	44.7	2.4	9.79	9.64	98.4
T-11B	44.7	2.4	9.79	9.72	99.2
T-18B	42.7	1.8	10.02	9.79	97.7
T-7A	41.3	2.0	9.99	9.75	97.5
T-7B	41.3	2.0	9.99	9.73	97.3
T-6A	41.0	2.2	9.92	9.79	98.6
T-6B	41.0	2.2	9.92	9.79	98.6
T-8A	40.3	1.7	10.11	9.80	96.9
T-8B	40.3	1.7	10.11	9.86	97.5
T-16A	39.6	1.5	10.19	9.83	97.4
T-16B	39.6	1.5	10.19	9.67	94.8
T-14B	37.2	1.4	10.27	9.84	95.8
T-15A	36.2	2.0	10.08	9.60	95.2
T-15B	36.2	2.0	10.08	9.76	96.8
T-5	34.7	2.3	9.99	9.83	98.3
T-2B	34.7	1.5	10.29	9.97	96.8
T-17A	33.7	1.4	10.34	9.99	96.6
T-19B	32.2	1.6	10.29	10.07	97.8
T-10A	29.4	2.4	9.98	9.93	99.4
T-10B	29.4	2.4	9.98	9.89	99.0
T-9	29.0	2.0	10.21	10.12	99.1
M-6	25.1	1.4	10.50	10.47	99.7

^aCalculated using a UC₂ density of 11.68 grams per cubic centimeter, a ThC₂ density of 9.60 grams per cubic centimeter, and a carbon density of 2.26 grams per cubic centimeter.

^bDetermined on one-inch-long, three-eighth- or one-half-inch-diameter specimens by oil immersion using silicone oil (density = 1.095 grams per cubic centimeter).

^cRadiography revealed a large cavity in the center of this specimen.

TABLE VI
MICROHARDNESS FOR VARIOUS COMPOSITIONS OF AS-CAST (U,Th) C_2 ALLOYS

Specimen Designation	Composition (mole % ThC ₂)	Microhardness	
		Range (KHN)	Average (KHN)
T-22	100	359-495	439
ThC ₂ -8C	100	583-631	609
M-3	87.7	484-907	749
T-21	62.3	298-438	368
T-24	54.4	278-510	367
T-20	51.1	435-495	470
T-12	45.5	317-386	347
T-11	45.2	295-612	508
T-18	43.2	250-394	291
T-7	41.7	435-549	497
T-6	41.4	492-547	523
T-8	40.7	268-384	317
T16	40.1	307-361	336
T-14	37.6	317-583	463
T-15	37.6	293-389	375
T-3	35.3	429-488	461
T-2	35.0	481-547	498
T-5	35.0	484-675	545
T-17	34.0	346-465	396
T-19	32.6	396-524	466
T-10	29.7	389-528	470
T-9	29.3	472-558	512
T-23	0.0	429-598	520

The range of hardness values for a given specimen was larger than the variations between average values as a function of composition. Variations such as these are common for brittle materials that have high hardness values. It was impossible to make hardness impressions in the (U,Th)C₂ alloys with loads greater than 100 grams without cracking the specimens. Yet, the 100-gram load gives extremely small impressions that cause errors in measurement of the impression to be magnified in the hardness number. Another possible source of error for these hardness values was the large number of fine graphite platelets present in the microstructure. It was impossible to make impressions in most of the alloys without hitting these platelets. This is probably a random error, but it is generally expected that the hardness will decrease with increasing free-carbon content.

Hardness was measured for many of the annealed specimens, and a complete tabulation of the results is given in Appendix E, page 164. For all the alloys examined, the general trends in hardness were:

1. Specimens annealed at 1500°C and below for times up to 1165 hours did not show a significant change in hardness.
2. Specimens annealed at 1900°C were much harder than the as-cast materials, and the hardness increased slowly with annealing time.

The apparent hardness increase for the 1900°C anneals may be due to the removal of the fine graphite platelets from a large portion of the microstructure, as shown in Figures 32, page 65, and 33, page 67. If

these fine platelets interfere with the measurement of "alloy" hardness, it is consistent with the microstructure that the observed hardness does not change for the specimens annealed at 1500°C and below.

Thermal Expansion

The technique described in Chapter III was used to determine the thermal expansion parameters between 40 and 1000°C for most of the compositions given in Table II, page 18. Using the computer program given in Appendix B, page 133, the appropriate corrections for the silica dilatometer were applied and plots of the type shown in Figure 34 were obtained. The instantaneous and the mean temperature coefficients were calculated from these data and are compared in Table VII. Typical plots of the mean temperature coefficient as a function of temperature are shown in Figure 35. The mean temperature coefficients as a function of composition are tabularly presented in Table VIII and graphically presented in Figure 36. The cumulative expansion varied with composition in the manner shown in Figure 37. A complete tabulation of the thermal expansion parameters for each specimen is given in Appendix F, page 170

A search of the literature showed that only two thermal expansion values were available for similar material (44,45). These mean temperature coefficients of expansion for UC_2 (ref. 44) and a (U,Th) C_2 alloy containing 10 mole per cent UC_2 (ref. 45) are compared with the present data in Figure 36. Both of the previously determined values were higher than those observed during this work. Metallographic examination of the

Figure 34. Average Cumulative Expansion Versus Temperature for UC_2 , ThC_2 , and Two (U,Th) C_2 Alloys.

TABLE VII
COMPARISON BETWEEN INSTANTANEOUS AND MEAN TEMPERATURE COEFFICIENTS OF
EXPANSION FOR ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Instantaneous and Mean Temperature Coefficients ^a ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) for the Given Temperature ^b					
	600°C		800°C		1000°C	
	I	M	I	M	I	M
100	8.46	7.83	9.60	8.03	11.16	8.57
87.7	8.46	8.31	9.64	8.49	11.72	8.92
75.1	9.94	8.94	11.02	9.34	12.13	10.00
62.3	11.71	9.59	13.40	10.49	15.11	11.26
51.1	12.40	10.00	14.28	10.87	16.15	11.94
45.2	12.75	10.21	14.69	11.32	16.51	12.37
43.2	13.18	10.48	15.12	11.49	17.07	12.44
41.7	13.21	10.86	14.80	11.72	16.56	12.57
41.4	12.94	10.60	14.57	11.45	16.43	12.27
40.7	12.88	10.61	14.52	11.42	15.87	12.15
40.1	13.20	10.69	15.29	11.65	17.08	12.60
37.6	13.24	10.44	15.46	11.49	17.67	12.56
36.6	12.84	10.40	14.56	11.02	16.36	12.24
35.0	13.38	10.70	15.32	11.69	17.26	12.88
34.0	13.37	11.95	15.22	11.87	17.08	12.73
32.6	13.64	10.91	15.74	11.85	17.84	12.59

TABLE VII (continued)

Composition (mole % ThC ₂)	Instantaneous and Mean Temperature Coefficients ^a ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) for the Given Temperature ^b					
	600 $^{\circ}\text{C}$		800 $^{\circ}\text{C}$		1000 $^{\circ}\text{C}$	
	I	M	I	M	I	M
29.7	13.56	11.63	14.32	12.65	15.48	12.89
29.3	14.03	11.45	15.72	12.38	17.41	13.25
25.4	13.36	12.27	13.96	12.56	14.50	13.23
c	13.49	12.88	13.49	12.63	13.49	13.51

^aThe instantaneous coefficient is designated by I and the mean coefficient is designated by M.

^bThe mean coefficient was determined using the initial temperature of 30 to 50 $^{\circ}\text{C}$ and the upper temperature indicated.

^c100 per cent UC₂.

Figure 35. Typical Plots for the Mean Temperature Coefficient Versus Temperature for ThC_2 and $(\text{U,Th})\text{C}_2$ Alloys.

TABLE VIII

MEAN TEMPERATURE COEFFICIENT OF EXPANSION FOR ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Mean Temperature Coefficient of Expansion ^a ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) for the Given Temperature Range ^b						
	40 to 400°C	40 to 500°C	40 to 600°C	40 to 700°C	40 to 800°C	40 to 900°C	40 to 1000°C
100	7.35	7.51	7.83	7.94	8.03	8.24	8.57
87.7	8.49	8.35	8.31	8.31	8.49	8.66	8.92
75.1	8.59	8.73	8.94	9.17	9.34	9.57	10.00
62.3	9.06	9.19	9.59	10.02	10.49	10.92	11.26
51.1	9.06	9.47	10.00	10.47	10.87	11.30	11.94
45.2	9.16	9.61	10.21	10.76	11.32	11.84	12.37
43.2	9.53	9.99	10.48	10.96	11.49	12.06	12.44
41.7	9.94	10.38	10.86	10.31	11.72	12.12	12.57
41.4	9.60	10.04	10.60	11.03	11.45	11.86	12.27
40.7	9.69	10.11	10.61	11.02	11.42	11.84	12.15
40.1	9.76	10.14	10.69	11.22	11.65	12.21	12.60
37.6	9.41	9.80	10.44	10.99	11.49	12.04	12.56
36.6	9.61	9.93	10.40	11.02	11.35	11.72	12.24
35.0	9.82	10.13	10.70	11.22	11.69	12.21	12.88
34.0	10.16	10.52	10.95	11.38	11.87	12.37	12.73
32.6	9.45	10.39	10.91	11.40	11.85	12.42	12.59
29.7	10.78	11.28	11.63	12.21	12.65	12.75	12.89
29.3	10.41	10.86	11.45	11.90	12.38	12.80	13.25
25.4	11.79	11.89	12.27	12.41	12.56	12.90	13.23
c	12.66	12.98	12.88	12.32	12.63	13.04	13.51

^aEach value reported is the average of at least three determinations per specimen on one to three specimens of the same composition.

^bThe temperature of the specimen at the start of each run ranged from 30 to 50°C. Actual temperatures were used in the calculations.

^c100 per cent UC₂.

Figure 36. The Average Temperature Coefficient of Expansion Versus Composition for $(\text{U,Th})\text{C}_2$ Alloys. The average coefficients between room temperature and 800°C and room temperature and 1000°C are shown.

Figure 37. The Cumulative Expansion Versus Composition for $(\text{U,Th})\text{C}_2$ Alloys.

UC₂ used by Carlsen (44) showed that the free-carbon content was much lower than in the presently investigated UC₂ samples. Such an observation was not possible for the (U,Th)C₂ examined by Griesenauer (45). It is not unreasonable that the variations in carbon content change the expansion characteristics of these materials. The microstructural studies (Figures 21 through 27, pages 49 through 55) showed the distribution of free carbon to be inhomogeneous. This could also account for the scatter shown in Figures 36 and 37 for a given composition. Repeat measurements on the same specimen agreed within ± 2 per cent, which is insufficient to account for the observed differences.

Several compositions were examined between 32 and 42 mole per cent ThC₂, which should lie in a two-phase region according to the phase diagram shown in Figure 4, page 12. Inspection of both the cumulative expansion and the mean temperature coefficients for these alloys as a function of temperature failed to show any evidence for a phase change up to 1000°C. This suggests that either the expansion rates of the UC₂ and ThC₂ solid solutions are only slightly different or that the phase boundaries are almost vertical from room temperature to 1000°C. The data presented in Figures 36 and 37 clearly show that the expansion rates of ThC₂ and UC₂ are very different, and even the difference in rates for alloys between 25 and 50 mole per cent ThC₂ is significant. From the expansion results, it would therefore seem more probable that the boundaries of the two-phase region are almost vertical between room temperature and 1000°C.

X-Ray Diffraction

Debye-Scherrer-type x-ray diffraction patterns and diffractometer traces for the as-cast and the annealed specimens were examined for phases present by comparison with standard patterns available from previous work (21). The room-temperature x-ray diffraction results, given in Table IX, revealed three crystal structures for (U,Th)C₂ alloys:

1. A ThC₂ monoclinic structure existed for alloys containing more than approximately 75 mole per cent ThC₂.
2. A ThC₂ bct structure existed between approximately 42 and 55 mole per cent ThC₂.
3. A UC₂ bct structure, with a 20 to 30 per cent larger c_o/a_o ratio, existed for alloys containing less than 35 mole per cent ThC₂.

The two tetragonal regions were separated by a two-phase field that was approximately 7 mole per cent wide (35 to 42 mole per cent ThC₂).

Because of the large difference in the c_o/a_o ratios for the two bct structures, specimens containing two phases gave two distinct sets of diffraction lines.

Definition of the two-phase region separating monoclinic and tetragonal ThC₂ was less certain. Only one as-cast alloy (75.1 mole per cent ThC₂) had diffraction lines common to both the bct and the monoclinic structures prior to annealing. Apparently, the fast cooling associated with drop casting retained the ThC₂ bct structure for the alloys with compositions between 55 and 75 mole per cent ThC₂, but

TABLE IX

PHASES DETECTED BY X-RAY DIFFRACTION AT ROOM TEMPERATURE ON ARC-MELTED AND ANNEALED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a		
100	T-22		As cast	Strong ^b		-	ThC ₂ monoclinic
100	T-22	1300	216	Strong ^b		120	ThC ₂ monoclinic
100	T-22	2100 ^c	130 ^c	Strong ^b		-	ThC ₂ monoclinic
100	ThC ₂ -6		As cast	Strong ^b		-	ThC ₂ monoclinic
100	ThC ₂ -6	1350	200	Strong ^b		100	ThC ₂ monoclinic; lines
100	ThC ₂ -6	1400	368	Strong ^b		110	very sharp ThC ₂ monoclinic; lines
100	ThC ₂ -6	1455	370	Strong ^b		110	very sharp ThC ₂ monoclinic; lines
87.3	M-3		As cast	Strong ^b		-	very sharp ThC ₂ monoclinic
75.1	M-4		As cast	Strong		-	ThC ₂ monoclinic and bct
	M-4	1000	1100	Strong ^b		-	ThC ₂ monoclinic and bct
62.3	T-21		As cast	Strong ^b		-	ThC ₂ bct
	T-21	1300	216	Strong ^b		22	ThC ₂ monoclinic and bct
62.3	T-21	2100 ^c	130 ^c	Strong ^b		-	ThC ₂ bct
54.4	T-24		As cast	Strong ^b		-	ThC ₂ bct
	T-24	1300	216	Strong ^b		-	ThC ₂ monoclinic and bct
51.1	T-20		As cast	Strong ^b		-	ThC ₂ bct
51.1	T-20	1300	216 ^b	Strong ^b		22	
51.1	T-20	2100 ^b	130 ^b	Strong ^b		-	
45.5	T-12		As cast	Strong ^b		-	
45.5	T-12	1200	446	Strong ^b		10	
45.5	T-12	1200	760	Strong ^b		4	Slightly spotty lines
45.5	T-12	1500	445	Strong ^b		20	
45.5	T-12	1500	445	Strong ^b		45	Under mechanical pressure
45.5	T-12	2100 ^c	130 ^c	Strong ^b		-	in a graphite crucible

TABLE IX (continued)

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a		
45.2	T-11	1000	As cast	Strong _b		-	
			190	Strong _b		20	
			453	Strong _b		20	
			743	Strong _b		5	
			950	Strong _b		5	
			1750	Strong _b		20	
			446	Strong _b		10	
			760	Strong _b		4	
45.0	T-11	1500	445	Strong _b		45	
			350	Strong _b		45	
			516	Strong _b		45	
			830	Strong _b		45	Slightly spotty lines
			1190	Strong _b		45	Slightly spotty lines
				Strong _b		45	Spotty line
43.2	T-18	2100 ^c	As cast	Strong _b		-	
41.7	T-7	1000	As cast	Strong _b		-	
			190	Strong	Weak	20	
			453	Strong	Weak	20	
			743	Strong	Weak	5	
			950	Strong	Weak	5	
			1750	Strong	Weak	20	
			450	Strong	Weak	140	
			744	Strong	Very weak	140	
			907	Strong	Weak	8	
			1243	Strong	Medium	8	
			150	Strong	Weak	120	
			425	Strong	Weak	120	
			665	Strong	Weak	12	

TABLE IX (continued)

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a		
41.7	T-7	1900	925	Strong	Weak	12	
			1165	Strong _b	Weak	12	
			150	Strong _b		45	
			500	Strong _b		45	
			667	Strong _b		45	
			990	Strong _b		45	
			1350	Strong _b		45	
41.4	T-6	1000	As cast	Strong	Weak	-	Spotty lines
			190	Strong	Weak	20	
			453	Strong	Weak	20	
			743	Strong	Weak	5	
			950	Strong	Weak	5	
		1200	1750	Strong	Weak	20	
			150	Strong	Weak	140	
			450	Strong	Weak	140	
			744	Strong	Strong	140	
			907	Strong	Very weak	8	
		1500	1243	Strong	Very weak	8	
			150	Strong	Weak	120	
			425	Strong	Weak	120	
			665	Strong	Weak	12	
			925	Strong	Weak	12	
41.4	T-6	1900	1165	Strong _b	Very weak	12	
			150	Strong _b		45	
			500	Strong _b		45	
			667	Strong _b		45	
			990	Strong		45	

TABLE IX (continued)

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a		
40.7	T-8	1000	As cast	1350	Strong ^b	45	Spotty lines Spotty lines
					Strong	Very weak	
				190	Strong	Very weak	
				453	Strong	Very weak	
				743	Strong	Very weak	
				950	Strong	Very weak	
		1200		1750	Strong	Very weak	
				446	Strong	Very weak	
				445	Strong	Weak	
				516	Strong	Very weak	
				830	Strong	Weak	
				1190	Strong	Very weak	
		2100 ^c	As cast		Strong	Weak	
			As cast		Strong	Medium	
40.1	T-16				Strong	Strong	
37.6	T-14				Strong	Strong	
36.6	T-15				Strong	Strong	
35.3	T-3				Strong	Strong	
		1000	As cast		Very weak	Strong ^b	
				190		Strong ^b	20
				453		Strong ^b	20
				743		Strong ^b	5
				950		Strong ^b	5
				1750		Strong ^b	20
		1200		150		Strong	140
				450	Very weak	Strong	140
				744	Strong	Strong	140
				904	Strong	Strong	8
				1243	Strong	Medium	8

TABLE IX (continued)

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a		
35.3	T-3	1500	150		Strong ^a	120	Held under mechanical pressure in a graphite crucible
			445	Strong	Weak	45	
		1900	925	Strong	Strong	12	
			1165	Strong	Weak	12	
			150	Strong	Very weak	45	
			500	Strong	Very weak	45	
			669	Strong	Very weak	45	
			990	Strong	Very weak	45	
			1350	Strong	Very weak	45	
					Strong ^b	-	
35.0	T-2	1100	150	Strong	Strong	100	Oxidized, UC ₂ lines and oxide lines UC ₂ and ThC ₂ and oxide lines
			465			100	
		1300	750			100	
			990		Strong ^b	100	
			1200	Strong	Strong	14	
			150	Strong	Strong	120	
			200	Strong	Medium	120	
			150	Strong	Weak	45	
			500	Strong	Weak	45	
35.0	T-2	1900	667	Strong	Weak	45	
		1900	990	Strong	Medium	45	
		2000	1350	Strong	Strong	45	
			50	Strong	Strong	45	
35.0	T-5	1000	190		Strong ^b	-	
					Strong ^b	20	

TABLE IX (continued)

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a		
34.0	T-17	1200	453	Very weak	Strong ^b	20	Spotty lines
			743		Strong ^b	5	
			950		Strong	5	
			1750		Strong	20	
			150	Very weak	Strong ^b	140	
			450		Strong ^b	140	
			907	Strong	Strong	8	
			1243	Strong	Medium	8	
		1500	150	Strong	Strong	120	
			425	Weak	Strong	120	
			665	Strong	Strong	12	
			925	Strong	Weak	12	
		1900	1165	Strong	Weak	12	
			150	Strong	Weak	45	
			500	Strong	Strong	45	
			667	Strong	Strong	45	
		2100 ^c	990	Strong	Strong	45	
			1350	Strong	Strong	45	
			As cast		Strong ^b	-	
			130 ^c		Strong ^b	-	
29.7	T-10	1000	As cast		Strong ^b	-	Sharp and spotty lines
			190		Strong ^b	20	
			453		Strong ^b	20	
			743		Strong ^b	5	
		1200	950		Strong ^b	5	
			1750		Strong ^b	20	
			446		Strong ^b	10	
			760		Strong ^b	4	

TABLE IX (continued)

Composition (mole % ThC ₂)	Sample Designation	Thermal History		Relative Amount of Phases		Approximate Cooling Rate (°C/min)	Remarks	
		Annealing Temperature (°C)	Annealing Time (hr)	ThC ₂ ^a	UC ₂ ^a			
29.3	T-9	As cast	1500	445	Very weak	Strong ^a	45	
			1900	516		Strong ^b	45	Sharp and spotty lines
			830	Strong ^b		45	Sharp and spotty lines	
			1000	190		Strong ^b	-	
			453	Strong ^b		20		
			743	Strong ^b		20		
			950	Strong ^b		5		
			950	Strong ^b		5		
			1750	Strong ^b		20		
			1200	446		Strong ^b	10	
			760	Strong ^b		4		
29.3	T-9	1500	445	Strong ^b	45			
		1900	350	Strong ^b	45			
		1900	830	Strong ^b	45	Sharp and spotty lines		
		1190	Strong ^b	45	Sharp and spotty lines			
0.0	T-23	As cast			Strong ^b	-		
			1300	216	Strong	120	UC ₂ and weak U ₂ C ₃	

^aThe UC₂ and ThC₂ crystal structures are bct unless otherwise specified.

^bSingle phase.

^cCooled at 20°C per minute from 2100 to 1100°C and held for an additional 130 hours and cooled to room temperature at approximately 25°C per minute.

after extended anneals at 1000 to 1400°C the powder patterns showed lines common to both the bct and the monoclinic structures. However, the near superposition of many of these lines and the absence of several of the low-intensity lines common only to the monoclinic structure made determination of the two-phase boundaries extremely difficult. Hill and Cavin (21), using a pseudo-orthorhombic cell for the thorium-rich alloys, observed a uniform variation in the cell parameters with compositions between 50 and 100 mole per cent ThC_2 . Consequently, they showed the region separating the two structures as a shaded area on the diagram in Figure 3, page 10. Langer et al. (23) observed a gradual change from the monoclinic to the tetragonal structure with increasing UC_2 content. The diffuseness of the diffraction patterns and the small separation of the low-angle diffraction lines prevented complete indexing of the bct or the monoclinic structures in this region. Thus, until sharper patterns with resolvable, high-angle diffraction lines are available, it is possible only to estimate the boundaries of the two-phase region between monoclinic and tetragonal. After considering the high-temperature dilatometry results, to be discussed later, the two-phase region was estimated to extend from 55 to 75 mole per cent ThC_2 .

As-cast alloys with compositions between 42 and 55 mole per cent ThC_2 had the ThC_2 bct structure, which was not affected by the anneals described in Table IX. Annealing for 500 hours at 1900°C caused these alloys to give spotty x-ray diffraction lines; this is consistent with the increase in grain size shown metallographically in Figure 32,

page 65.

An alloy containing 41.7 mole per cent ThC_2 was single-phase bct ThC_2 after casting, but specimens annealed at 1000, 1200, and 1500°C gave a two-phase x-ray diffraction pattern (bct ThC_2 plus bct UC_2) at room temperature. However, the anneals at 1900°C resulted in a room-temperature diffraction pattern that was again bct ThC_2 . These results suggest that the alloy is very near a boundary between a two-phase and a one-phase region. This observation is supported by the results of an x-ray diffraction study on an alloy containing 41.4 mole per cent ThC_2 . The study showed this specimen to be a two-phase structure after casting and after annealing except at 1900°C. At a composition of 40.7 mole per cent ThC_2 , the two-phase structure was observed, by room-temperature x-ray diffraction, for all the anneals shown in Table IX. The x-ray diffraction results on the above three compositions placed the uranium-rich phase boundary of the bct ThC_2 structure very close to 41.7 mole per cent ThC_2 . The center of the two-phase field, as seen in Table IX, is between 36.6 and 37.6 mole per cent ThC_2 . As-cast alloys containing 35 mole per cent ThC_2 were bct UC_2 , but when annealed at 1000 to 1900°C, they reverted to a two-phase structure. However, alloys containing 34 mole per cent ThC_2 and less were bct UC_2 after casting and after all subsequent anneals. Therefore, the thorium-rich boundary of the bct UC_2 structure is probably between 34 and 35 mole per cent ThC_2 . The results of x-ray diffraction on the annealed specimens agree with Langer et al. (23) and Hill and Cavin (21) on the presence of an immisible region approximately 7 mole per cent wide between the bct UC_2 and the bct ThC_2 structures at low temperatures.

However, the two-phase region is placed, by this work, at a slightly higher ThC_2 content than previously reported.

The as-cast UC_2 specimen had a bct structure that is metastable according to Figure 1, page 5. After annealing at 1300°C for 216 hours, very weak lines associated with the body-centered cubic U_2C_3 were present along with the bct UC_2 lines. No such evidence for a sesquicarbide was seen for any of the annealed $(\text{U,Th})\text{C}_2$ alloys. A 25.4 mole percent ThC_2 alloy contained the highest uranium concentration examined.

Few parameter measurements were made for the $(\text{U,Th})\text{C}_2$ alloys because of the diffuseness of the powder patterns. This diffuseness and the associated line broadening were probably a result of strain in the material, which was probably caused by one or more of the following mechanisms:

1. Mechanical deformation during specimen preparation.
2. Anisotropic expansion and/or contraction during thermal treatments.
3. Changes in volume associated with phase transformation.
4. Presence of the large amount of graphite.
5. Oxidation of the specimen during examination.

An annealing study on ThC_2 showed Mechanism 4 to be the main contributor to the poor patterns. Specimens of ThC_2 containing various amounts of free graphite (from 0.06 to 2.1 weight per cent) were annealed at 1300°C for 216 hours. Prior to annealing, all of these alloys gave typical poor patterns. Diffraction patterns of the

heat-treated material improved significantly with decreasing carbon content. Strain produced by Mechanism 4 is indicated. The monoclinic cell parameters determined for the annealed sample containing 0.06 weight per cent free graphite are given below:

<u>Parameter</u>	<u>Value</u>
a	(8.296 $\pm$ 0.005) angstroms
b	(4.232 $\pm$ 0.002) angstroms
c	(10.583 $\pm$ 0.037) angstroms
β	(90.23 $\pm$ 0.037) degrees

A 1400°C, 370-hour anneal on the material having 0.06 weight per cent free carbon gave patterns only slightly poorer than those obtained after the 1300°C anneal. This observation showed that the strain contribution due to the anisotropy of the material was small, if present. The anneal for 370 hours at 1455°C, which is above the monoclinic to tetragonal transformation, indicated an only slightly increased strain over the 1300°C anneal. One concludes that the volume change associated with this transformation in ThC₂ does not cause an excessive amount of strain. These results are consistent with the microstructures of the above described samples (Figures 28 through 31, pages 57 and 61 through 63). Hence, the large quantity of excess graphite strained the alloys as they cooled from the melt and/or as they transformed from fcc to bct structures. However, the effects of the transformation from the bct to the monoclinic structure and of the anisotropy were small. The effects of the fcc to bct transformation alone were not evaluated. Anneals on the alloys described in Table II, page 18, at 1000 to 1900°C

for times up to 1750 hours failed to give a pattern comparable to the 1300°C anneal on the ThC₂ containing 0.06 weight per cent free carbon.

The effect of oxides formed during handling and examination does not contribute significantly to the diffuseness of the pattern. This was determined by placing an unprotected, prepared, metallographic specimen on the x-ray diffractometer and observing the traces obtained over a period of time. A large amount of oxide formed on the specimen after a forty-eight-hour exposure. The oxide was removed and a diffractometer trace was determined. No oxide lines were observed, and the carbide pattern was apparently unaffected by the low-temperature surface oxidation. A powder pattern on the oxide showed only one, very broad, weak diffraction band characteristic of an amorphous material. With the electron microscope, the diameter of the oxide particles was estimated to be less than twenty angstroms. One concludes that room-temperature oxidation does not affect the quality of diffraction patterns; the resultant oxide lines are too weak and too broad to be detected in the presence of the carbide.

Phase Transformations Detected by Dilatometry and Thermal Analysis

Hill and Cavin (21) and Langer et al. (15,23), using some form of thermal analysis to determine the transformation temperature and high-temperature x-ray diffraction to determine the crystal structures, observed a high-temperature transformation from bct to fcc structure for UC₂, ThC₂, and (U,Th)C₂ alloys. The independent results of these investigators agreed on the crystal structures, but as shown in

Figure 4, page 12, there was a difference between 50 and 100°C in the transformation temperatures. For the present investigation, dilatometry and thermal analysis were used to detect the solid-state phase transformations.

With the high-temperature graphite or tantalum dilatometer described in Chapter III, the transformations from a cubic to a tetragonal structure were observed for ThC_2 and several $(\text{U,Th})\text{C}_2$ alloys. A summary of the results is given in Tables X and XI, and a typical dilation versus time curve for the $(\text{U,Th})\text{C}_2$ alloys is shown in Figure 38. A similar curve for ThC_2 is given in Figure 39. A complete tabulation of the data is given in Appendix G, page 181.

Transformation temperatures, obtained by thermal analysis, for some of these samples are presented in Table XII [UC_2 and the $(\text{U,Th})\text{C}_2$ alloys] and Table XIII (ThC_2). Heating and cooling rate curves for UC_2 , ThC_2 , and $(\text{U,Th})\text{C}_2$ alloys are given in Figures 40 through 46.

A summary of the data from dilatometry, thermal analysis, and x-ray diffraction are presented in Figure 47. Both the thermal analysis and dilatometry data showed considerable hysteresis — transformation temperatures on heating were higher than those obtained on cooling. The largest hysteresis (196°C) occurred for the $(\text{U,Th})\text{C}_2$ alloys containing between 42 and 52 mole per cent ThC_2 , and the effect decreased uniformly as the composition approached the terminal compounds, UC_2 and ThC_2 . This maximum in the hysteresis corresponded to the minimum for the transformation temperatures. Further, the transformation temperatures

TABLE X
AVERAGE TEMPERATURES DETERMINED BY DILATOMETRY FOR SOLID-STATE TRANSFORMATIONS IN (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Sample Designation	Number of Determinations	Transformations Observed for Heating			Transformations Observed for Cooling			Temperature Difference between Heating and Cooling	
			Lower Temperature, T _{LH} (°C)	Upper Temperature, T _{UH} (°C)	T _{LH} - T _{UH} (°C)	Upper Temperature, T _{UC} (°C)	Lower Temperature, T _{LC} (°C)	T _{UC} - T _{LC} (°C)	T _{LH} - T _{LC} (°C)	T _{UH} - T _{UC} (°C)
62.3	T-21A	2	1244	1278	34	1202	1182	20	62	76
54.4	T-24B	2	1196	1223	27	1184	1158	26	38	39
51.1	T-20A	2	1178	1216	38	1047	1001	46	177	169
45.5	T-12A	5	1168	1195	27	999	979	20	189	196
45.5	T-12A-20-118 ^e	2	1175	1200	25	f	f	-	-	-
45.2	T-11B	2	1184	1210	26	f	f	-	-	-
41.7	T-7A	3	1187	1202	15	1055	1037	18	150	147
41.7	T-7A-17-114 ^g	2	1174	1193	19	1055	1030	25	144	148
41.4	T-6B	4	1191	1212	21	1061	1042	19	149	151
40.1	T-16B	4	1176	1216	40	1079	1054	25	122	137
37.6	T-14B	2	1237	1249	12	1134	1124	10	113	115
35.0	T-5	7	1242	1270	28	1175	1152	23	90	95
35.0	T-5-20-118 ^e	2	1252	1268	16	1176	1162	14	90	92
35.0	T-2A	2	1246	1266	20	1180	1168	12	78	86
35.0	T-2B	4	1248	1274	26	1186	1164	22	84	88
35.0	T-2B-17-114 ^g	3	1240	1273	33	1188	1134	54	106	85
34.0	T-17B	2	1270	1286	16	1200	1182	18	88	86
32.6	T-19B	2	1278	1298	20	1220	1200	20	78	78
29.7	T-10A	6	1326	1352	26	1280	1254	26	72	72
29.7	T-10A-17-114 ^g	4	1304	1340	36	1276	1248	28	56	64
29.3	T-9	4	1309	1340	31	1269	1248	21	61	71
29.3	T-9-20-118 ^e	2	1326	1344	18	1280	1263	17	63	64
25.4	M-6	2	1390	1414	24	1343	1321	22	69	71

^aThe lower transformation temperature observed on heating, schematically shown in Figure 38.

^bThe upper transformation temperature observed on heating, schematically shown in Figure 38.

^cThe upper transformation temperature observed on cooling, schematically shown in Figure 38.

^dThe lower transformation temperature observed on cooling, schematically shown in Figure 38.

^eAnnealed 118 hours at 2000°C prior to examination.

^fNot determined because of instrument trouble.

^gAnnealed 114 hours at 1700°C prior to examination.

TABLE XI

TEMPERATURES FOR SOLID-STATE TRANSFORMATIONS DETERMINED BY DILATOMETRY ON ThC_2 AND $(\text{U,Th})\text{C}_2$ ALLOYS

Composition	Rate	Heating						Rate	Cooling					
		Lower Transformation ^a			Upper Transformation ^a				Upper Transformation ^a			Lower Transformation ^a		
		Temperature, ^b °C			Temperature, ^b °C				Temperature, ^b °C			Temperature, ^b °C		
		T _s	T _f	T _f - T _s	T _s	T _f	T _f - T _s		T _s	T _f	T _s - T _f	T _s	T _f	T _s - T _f
(mole % ThC ₂)	(°C/min)	(°C/min)												
100	3.8	1428	1443	15	1505	1522	17	3.4	1493	1473	20	1436	1419	17
100	2.2	1425	1441	16	1502	1520	18	2.0	1494	1476	18	1436	1421	15
87.7	3.2	1280	1318	38	1431	1450	19	1.7	1413	1394	19	1310	1276	34
87.7	1.5	1293	1330	37	1433	1455	22	1.1	1415	1395	30	1311	1282	29
75.1	2.4	1020	1180	160	1324	1348	23	2.1	1328	1310	18	1170	1002	168
75.1	1.9	1054	1167	113	1337	1361	24	1.7	1326	1313	13	1138	974	164

^aTwo transformations, designated as upper and lower, were observed in these materials.^b T_s and T_f signify the temperatures where the indicated transformation started and ended, respectively, as determined from inflections in the dilation versus time curves.

Figure 38. Tracing of the Dilation Versus Time Curve for Specimen M-6. This curve is typical of those obtained for most of the (U,Th)C₂ alloys.

Figure 39. Tracing of the Dilation Versus Time Curve for ThC_2 . This curve is similar to those obtained for the thorium-rich $(\text{U,Th})\text{C}_2$ alloys that have two-phase transformations.

TABLE XII

AVERAGE TEMPERATURES DETERMINED FROM COOLING CURVE ANALYSIS FOR SOLID-STATE TRANSFORMATIONS IN (U,Th)₂ ALLOYS

Composition (mole % ThC ₂)	Sample Designation	Transformations Observed for Heating				Transformations Observed for Cooling				Temperature Difference between Heating and Cooling	
		Rate	Lower Temperature, ^a T _{LH} (°C)	Upper Temperature, ^b T _{UH} (°C)	T _{UH} - T _{LH} (°C)	Rate	Upper Temperature, ^c T _{UC} (°C)	Lower Temperature, ^d T _{UL} (°C)	T _{UC} - T _{LC} (°C)		
		(°C/min)				(°C/min)				(°C)	(°C)
62.3	T-21	250	1230	1251	20	280	1130	1115	15	115	121
51.1	T-20	240	1100	1130	30	260	1060	1030	30	70	70
45.5	T-12	220	1085	1107	22	250	1035	1015	20	70	70
40.7	T-8	230	1080	1120	40	410	1065	1055	10	35	55
37.6	T-14	220	1155	1168	13	240	1115	1105	10	50	53
35.0	T-5	500	1172	1190	18	660	1145	1120	25	52	45
29.3	T-9	250	1297	1312	15	270	1247	1225	22	72	65
29.3	T-9-20-118 ^e	260	1297	1315	18	280	1240	1215	25	72	75
25.4	M-6	250	1370	1382	12	280	1325	1305	20	65	57
f	T-23	260	1772	1787	15	280	1762	1750	12	22	25

^aThe lower transformation temperature observed on heating.^bThe upper transformation temperature observed on heating.^cThe upper transformation temperature observed on cooling.^dThe lower transformation temperature observed on cooling.^eAnnealed 118 hours at 2000°C prior to examination.^fContains UC₂ plus excess graphite.

TABLE XIII

TEMPERATURES OF PHASE TRANSFORMATIONS IN ThC₂ DETERMINED FROM COOLING CURVE ANALYSIS

Composition	Rate	Heating						Rate	Cooling					
		Lower			Upper				Upper			Lower		
		Transformation ^a Temperature, ^b °C			Transformation ^a Temperature, ^b °C				Transformation ^a Temperature, ^b °C			Transformation ^a Temperature, ^b °C		
		T _S	T _f	T _f - T _S	T _S	T _f	T _f - T _S		T _S	T _f	T _S - T _f	T _S	T _f	T _S - T _f
(mole % ThC ₂)	(°C/min)							(°C/min)						
100	40	1430	1450	20	1490	1520	30	50	1480	1450	30	1425	1375	50
100	120	1425	1470	45	1505	1530	25	125	1470	1445	25	1375	1330	45
100	240	1420	1440	20	1505	1530	25	-	-	-	-	-	-	-

^aTwo transformations, designated as upper and lower, were observed in these materials.

^bT_s and T_f signify the temperatures where the indicated transformation started and ended, respectively, as determined from inflections in the temperature versus time curves, as indicated by the derivative, dT/dt.

Figure 40. Heating and Cooling Rate Curves for UC_2 . Note that the derivative, dT/dt , is almost constant during a major portion of transformation.

Figure 41. Heating and Cooling Rate Curves for ThC_2 . Note that the derivative, dT/dt , for the transformation is characteristic of a shear transformation. (a) Heating. (b) Cooling.

Figure 42. Heating and Cooling Rate Curves for a (U,Th) C_2 Alloy Containing 29.3 Mole Per Cent ThC_2 . Heating rate $350^{\circ}C$ per minute. Cooling rate $210^{\circ}C$ per minute.

Figure 43. Recorder Plot of Temperature Versus the Derivative of Temperature, dT/dt , for a $(U,Th)C_2$ Alloy Containing 29.3 Mole Per Cent ThC_2 . The heating and cooling rates are 50°C per minute and 60°C per minute, respectively, for the curves on the left. The heating and cooling rates are 80°C per minute and 100°C per minute, respectively, for the curves on the right.

Figure 44. Heating Rate (500°C Per Minute) and Cooling Rate (660°C Per Minute) Curves for a (U,Th)C₂ Alloy Containing 35 Mole Per Cent ThC₂.

Figure 45. Cooling Curves at Various Rates for a (U,Th)C₂ Alloy Containing 35.0 Mole Per Cent ThC₂. Curve No. 1 cooled at 44°C per minute. Curve No. 2 cooled at 68°C per minute. Curve No. 3 cooled at 90°C per minute. Curve No. 4 cooled at 180°C per minute. Curve No. 5 cooled at 360°C per minute.

Figure 46. Heating and Cooling Rate Curves for a $(U,Th)C_2$ Alloy Containing 37.6 Mole Per Cent ThC_2 . (a) Heating. (b) Cooling.

Figure 47. Summary of the Dilatometry and Thermal Analysis Data and the Room-Temperature X-Ray Diffraction Results.

obtained, by dilatometry, on heating at rates on the order of 1°C per minute were higher than the temperatures obtained by thermal analysis at rates on the order of 300°C per minute. The transformation temperatures obtained, by both methods, on cooling generally did not differ by more than the experimental accuracy of the measuring techniques, that is, $\pm 30^{\circ}\text{C}$.

The difference in the data generated by the above two techniques could not be explained. With the standards described in Chapter III, both systems were checked for errors in temperature measurement. In addition, the thermocouples used in the dilatometer were checked against a standard thermocouple by placing the tip of the standard and test thermocouples in a massive alumina block and inserting this assembly into a furnace. The temperature difference was obtained at several different positions in the furnace which imposed different thermal gradients along the length of the couples. The temperatures agreed within $\pm 3^{\circ}\text{C}$. A further check on the thermal analysis data is the close agreement with Wallace (9) on the transformation temperature of the fcc to bct transformation in UC_2 . Wallace reported a temperature of $(1785 \pm 20)^{\circ}\text{C}$, which is the arithmetic mean for the temperatures observed on heating and cooling. After making a similar average of the data given in Table XII, page 98, the transformation temperature was $(1768 \pm 25)^{\circ}\text{C}$.

No attempt was made to correct for emissivity in the thermal analysis system since the crucible was designed to approach blackbody conditions and since emissivity data for the $(\text{U,Th})\text{C}_2$ alloys at the

transformation temperatures were not available. This would appear to be a safe deletion in view of the melting points obtained for copper (1080 ± 25)°C and nickel (1460 ± 25)°C.

Limits of accuracy on temperatures determined using thermocouples (estimated to be $\pm 5^\circ\text{C}$) are generally better than those for an optical brightness pyrometer (estimated to be $\pm 25^\circ\text{C}$), and therefore, the dilatometry data were given more weight for this investigation. From the data summarized in Figure 47, two tentative phase diagrams, which are consistent with the results, are presented in Figures 48 and 49. The heating and cooling transformation temperatures were forced to agree by arithmetically averaging them, except in the region around 45 mole per cent ThC_2 ; these data were weighted in favor of the higher temperature. Another plausible explanation is discussed in Recommendation 4, page 123. Both transformations in ThC_2 always occurred over a temperature range and not isothermally as would be predicted by Figure 2, page 7. An average value for each temperature range was used to construct a conventional pseudobinary diagram.

Two solid-state transformations were observed for ThC_2 (specimen T-22) by thermal analysis and dilatometry. The typical dilatometry curve, Figure 39, page 97, showed that, on heating or cooling, the rate of expansion or contraction for the lower transformation [monoclinic to tetragonal according to Langer et al. (15)] was 20 to 30 per cent lower than the upper transformation to the fcc structure. Length changes for the lower transformation were 25 to 40 per cent larger than those for the upper transformation. This difference in length change was much

Figure 48. Tentative Phase Diagram of the ThC₂-UC₂ Pseudo-binary System in the Presence of Excess Graphite.

Figure 49. Tentative Phase Diagram of the ThC₂-UC₂ Pseudo-binary System in the Presence of Excess Graphite.

more apparent on a dilation versus temperature plot. Transformation temperatures for ThC₂, determined by dilatometry, are given in Table XI, page 95. Average values for these data place the upper and lower transformations at $(1500 \pm 25)^{\circ}\text{C}$ and $(1430 \pm 10)^{\circ}\text{C}$, respectively. The limits on each temperature include the differences observed for heating and cooling and the temperature range for each transformation. For the heating and cooling rates examined by dilatometry (2.0 to 3.8°C per minute), there was no change in the transformation temperature of either phase transition.

Thermal analysis data given in Table XIII, page 99, show good agreement with the above transformation temperatures for heating but are generally lower for cooling. The hysteresis for the lower transformation increases approximately 50°C as the cooling rate increased from 50 to 125°C per minute, and for faster cooling rates it was nearly impossible to determine the lower transformation temperatures, as shown in Figure 41, page 101. Within experimental error, the hysteresis was constant for the upper transformation. The rate curves dT/dt versus t for heating [Figure 41(a)] showed that the upper transformation absorbed the larger amount of heat. The shape of the rate curve associated with the upper transition is characteristic of a shear or diffusionless transformation. Similar appearing rate curves were obtained by Engel (46) for the austenite to martensite transformation. The appearance (that is, jagged nature of this region of the rate curves), particularly on cooling, suggests the burst of diffusionless transformation in lithium-magnesium alloys, which causes audible clicks

also twinning in the tin, which is accompanied by clicks.

Langer et al. (15) suggested that both transformations are diffusionless. The evidence presented here has strongly supported the view that the upper transformation is diffusionless.

The transformation temperatures for ThC_2 are in excellent agreement with the results of Langer et al. (15), who reported the transformation temperatures as $(1481 \pm 28)^\circ\text{C}$ and $(1427 \pm 21)^\circ\text{C}$. Recently, Dalton and Griffin (47) reported the transformations as 1476 and 1410°C on the basis of dilatometry results.

As discussed previously, the transformation temperatures for UC_2 bct to fcc structure agreed within the limits of experimental error with those reported by Wallace (9). The heating and cooling curves for UC_2 are given in Figure 40, page 100. The transformation in UC_2 , as in ThC_2 , was not isothermal but occurred over a temperature range of approximately 15°C . Engel (48) showed that the derivative of temperature with respect to time, dT/dt , for a diffusionless transformation has the form of that shown in Figure 40 for UC_2 . The rate, dT/dt , is essentially constant during the course of the transformation but changes rapidly at the start and termination of the phase change. This type of response would be unusual for a diffusional transformation. Figure 42, page 102, and Figures 44 through 46, pages 104 through 106, illustrate similar shaped rate curves for the $(\text{U,Th})\text{C}_2$ alloys, particularly on cooling. Jagged-appearing curves, which were obtained for ThC_2 specimens, were not observed for UC_2 or the $(\text{U,Th})\text{C}_2$ alloys. The absence of these fluctuations was probably due to a "damping" effect caused by the

large amount of free graphite in the specimens. Such an effect of the free graphite was suggested by an experiment on ThC_2 . Thermal analysis on the ThC_2 shown in Figure 28(b), page 57, exhibited the most pronounced thermal arrest witnessed during this study, and jagged rate curves were obtained during the transformation on heating and cooling. The same material was melted in the presence of graphite, and the rate curves for heating and cooling were determined. The two transformations occurred smoothly and without any apparent burst of transformations; that is, the rate curves did not have jagged regions. Subsequent examination of the microstructure showed a large amount of free graphite similar to the photomicrographs shown in Figures 21 through 26, pages 49 through 54. The temperatures of the transformations for this sample before and after melting were not significantly different. A similar observation was made by Langer et al. (23), who reported thermal arrests to be sharper for melted samples than for sintered samples.

Chang (39) first suggested that the UC_2 cubic to tetragonal structural change was a shear transformation. Langer et al. (15,23) gave evidence for this type of transformation in ThC_2 and the $(\text{U,Th})\text{C}_2$ alloys. The present observations on the shape of the thermal arrest curves adds strength to this argument for UC_2 and the $(\text{U,Th})\text{C}_2$ alloys. Another indication of this type of transformation is the fact that the temperatures of transformation for the $(\text{U,Th})\text{C}_2$ alloys did not change significantly as the heating or cooling rates were varied. Figure 45, page 105, clearly shows that the transformation temperature for a 35 mole per cent ThC_2 alloy did not change as the rate varied from

44 to 360°C per minute. Figure 43, page 103, a plot of temperature versus the derivative of temperature with respect to time, also shows no effect of cooling or heating rate. Examination of the dilatometry data of Appendix G, page 181, did not reveal a significant change in the transformation temperatures for a given specimen using rates from 0.3 to 6.5°C per minute. The high-temperature, fcc structure of UC_2 , ThC_2 , or the solid solution could not be retained even at cooling rates of 800°C per minute. This has been experienced previously by Hill and Cavin (21) and Langer et al. (23). These behaviors are characteristic of, but not restricted to, a diffusionless transformation.

To determine whether the hysteresis shown in Tables X through XIII, pages 94, 95, 98, and 99, respectively, could be decreased or eliminated, a $(U,Th)C_2$ alloy containing 29.7 mole per cent ThC_2 was isothermally annealed several times in the dilatometer. The specimen (T-10A) was initially heated and cooled at 2.4 and 1.8°C per minute, respectively, to determine the transformation temperatures, which are shown in Figure 38, page 96. For heating, the lower transformation temperature, T_{LH} , was $(1320 \pm 5)^\circ C$ and the upper transformation temperature, T_{UH} , was $(1349 \pm 5)^\circ C$. For cooling, the lower transformation temperature, T_{LC} , was $(1254 \pm 5)^\circ C$ and the upper transformation temperature, T_{UC} , was $(1279 \pm 5)^\circ C$. This represents a hysteresis of 66 to 70°C. After the above cycle, the sample was heated to $(1284 \pm 2)^\circ C$ and held for 15 hours. On subsequent cooling, a phase change did not occur. Next, the sample was held for twelve hours at $(1305 \pm 5)^\circ C$, and on

cooling, no transformation was noted. The next isothermal holding was for twenty-two hours at $(1315 \pm 5)^{\circ}\text{C}$, and again no transformation was observed on cooling. When the temperature was increased to $(1320 \pm 5)^{\circ}\text{C}$ and held for sixteen hours, a small amount of transformation was noted on cooling. This experiment was repeated on cooling. Sample T-10A was heated until it had completely transformed, and it was then cooled to 1285°C , a temperature just above the upper transformation temperature on cooling. It was held for sixteen hours and reheated slowly to 1500°C . A transformation did not occur, but on cooling, the transformation started at $(1280 \pm 5)^{\circ}\text{C}$ and ended at $(1261 \pm 5)^{\circ}\text{C}$. These experiments show that isothermal holding for fifteen to twenty hours within 5°C of the transformation temperature on heating or cooling will not initiate the transformation. This result could be due to:

1. The kinetics of the reaction, that is, diffusion at these temperatures, is slow for the large uranium and thorium atoms.
2. Shear or diffusionless transformation, as discussed previously.

It was shown by dilatometry and thermal analysis that the transformations from a tetragonal to a cubic structure or from a cubic to a tetragonal structure for these materials stopped when the temperature was held constant and restarted when the temperature was increased. The transformation would also stop if the increasing temperature were decreased, and after approximately the same amount of hysteresis, the reverse transformation would occur. For example, if the transformation

were stopped at 50 per cent of completion, a 50 per cent reverse transformation occurred after sufficient amount of cooling. The temperature for the start of the transformation on heating or cooling was apparently unaffected by the percentage of transformation that occurred.

Three dilatometry samples (T-12A, T-5, and T-9) were examined for phase changes in the as-cast condition and also after annealing for 118 hours at $(2000 \pm 25)^{\circ}\text{C}$. The results are summarized in Table X, page 94, and Figure 47, page 107, and show that the lower transformation temperature for heating and cooling was slightly higher after annealing. The upper transformation temperatures and the hysteresis were not significantly changed by the anneal. After annealing, the microstructure was an equiaxed grain structure similar to those of Figures 32 and 33, pages 64 and 67, respectively. Samples were also annealed at $(1700 \pm 25)^{\circ}\text{C}$ for 114 hours. They showed similar, but not as extensive, changes in the microstructure. However, both the upper and lower transformation temperatures on heating and cooling were lowered slightly or unchanged as a result of the heat treatment. This is the exact opposite of the results for the higher heat treatment (2000°C). An explanation was not readily apparent for this behavior. Several factors, such as the free-graphite morphology, free-graphite content, microstructure of the carbide, and oxygen content probably affect the transformation temperature. The lowering of the martensite start temperature by increasing the carbon content of a plain carbon steel has been reported (49). Also, addition of certain alloying

elements, such as aluminum, manganese, and chromium, are known to lower the martensite start temperature for steels (49).

The transformation temperatures given in Table X, page 94, were obtained from dilatometry and are slightly higher than those obtained by Hill and Cavin (21), who used differential thermal analysis. Data in Tables X through XIII, pages 94, 95, 98, and 99, respectively, agree reasonably well with results of Langer et al. (23). The thermal analysis data, Tables XII and XIII, agreed more closely than the dilatometry data. The minimum transformation temperature occurred between 40 and 50 mole per cent ThC_2 according to the data reported by Hill and Cavin (21) and Langer et al. (23), but the tentative diagrams resulting from those investigations showed the minimum between 36 and 38 mole per cent ThC_2 . The results presented in Figure 47, page 107, show the temperature minimum in the transformation occurring between 44 and 48 mole per cent ThC_2 ; this agrees closely with the data, but not the tentative diagrams, of the previous investigators.

The transition from a monoclinic to a bct structure reported by Langer et al. (23) for ThC_2 -rich alloys containing up to approximately 80 mole per cent ThC_2 was confirmed. Using the dilatometry data of Table XI, page 95, it was possible to delineate the boundaries of the two-phase region separating the two structures. These results are combined with the previous findings for this investigation, and two tentative phase diagrams are proposed. These phase diagrams, Figures 48 and 49, pages 110 and 111, are consistent with data presented

and apparently agree with most of the previous results on the $\text{UC}_2\text{-ThC}_2$ pseudobinary system.

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

This investigation was concerned with providing an improved understanding of the phase relationships for the UC_2 - ThC_2 pseudo-binary system and with the measurement of the thermal expansion of $(\text{U,Th})\text{C}_2$ alloys. Major conclusions are as follows:

1. The mean coefficient of expansion between room temperature and 1000°C for the $(\text{U,Th})\text{C}_2$ alloys increases uniformly with decreasing ThC_2 content from ThC_2 to UC_2 .
2. The expansion as a function of temperature is generally described by a quadratic equation.
3. The microstructure of the $(\text{U,Th})\text{C}_2$ alloys that contain large amounts of free graphite show little response to heat treatments below 1900°C .
4. Microhardness tends to increase slowly with annealing time at approximately 1900°C but shows no change below 1500°C .
5. Dilatometry and thermal analysis clearly show two polymorphic phase transformations for ThC_2 [$(1500 \pm 25)^\circ\text{C}$ and $(1430 \pm 10)^\circ\text{C}$].
6. Two solid-state transformations also occur for the ThC_2 -rich alloys containing from 0 to 25 mole per cent UC_2 .
7. Only one solid-state transformation occurs for the $(\text{U,Th})\text{C}_2$ alloys between 25 and 65 mole per cent ThC_2 , and

decomposition of the low-temperature phase for these alloys did not occur after extensive annealing.

8. A phase change occurs in UC_2 at $(1768 \pm 25)^\circ\text{C}$, but the low-temperature UC_2 bct structure decomposes to cubic U_2C_3 after heat treatment.
9. The minimum transformation temperature for the fcc alloys is approximately 1100°C and occurred between 44 and 48 mole per cent ThC_2 .
10. The results indicate that the fcc to bct phase transitions in UC_2 , ThC_2 , and the $(\text{U,Th})\text{C}_2$ alloys are diffusionless.
11. X-ray diffraction on as-cast and annealed specimens shows a two-phase region between 35 and 42 mole per cent ThC_2 composed of two bct structures. Two carbide phases could not be observed metallographically.
12. At least two phase diagrams are consistent with the data.

It is recommended that further studies of the uranium-thorium-carbon system be directed at increasing the understanding of the mechanism of the observed phase transformations. Studies are needed to determine the effect of carbon content on the mechanism and temperature of these solid-state transformations. Specific recommendations are:

1. Alloys containing between 55 and 75 mole per cent ThC_2 should be examined by dilatometry to completely define the phase boundaries of the transition from a ThC_2 monoclinic to a bct structure.

2. Anneals terminated by slow cooling and quenching should also be carried out on the above alloys to determine whether the lower transformation can be suppressed.
3. Quantitative metallography should show whether the carbon solubility changes significantly with temperature. For example, interpretation of the x-ray data in Table IX on samples T-6 and T-7 would be clearer if one assumes that, on annealing, the samples were reacting with graphite and if one further assumes that the bct ThC_2 phase contains more bound carbon than the bct UC_2 phase, with which it is in equilibrium.
4. Critical experiments should be carried out to determine more closely the phase relationships for the UC_2 - ThC_2 pseudo-binary system. Because the bct to fcc transitions closely approximate the martensitic, shear-type transformation, heavier weight has been given to interpreting the hysteresis effect in this light. Another possibility exists: the system is not binary, but ternary and the hysteresis is, in reality, an indication of this. One could imagine that the fcc phase contains more carbon than the bct phase. On transformation, the fcc phase actually undergoes peritectoid decomposition into the bct phase plus free carbon. (The carbon could be in the form of submicroscopic particles.) On reheating, the bct phase transforms at a

different (higher) temperature than the fcc phase did on cooling. It is necessary to assume that, for some reason, the carbon (in whatever form) is more mobile in the fcc phase.

5. Alloys that have carbon contents varying from a monocarbide to dicarbide plus excess carbon should be studied by dilatometry and thermal analysis for changes in the solid-state transformation temperature.
6. The above alloys are needed to relate the UC_2 - ThC_2 pseudo-binary system to the uranium-thorium-carbon ternary system.
7. Different etching techniques and anodizing should be tried to determine whether the two carbide phases can be seen metallographically.
8. High-temperature metallography should be used to determine if a topography change is associated with these solid-state transformations. This will add much to the understanding of the mechanism of the phase transformation.

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APPENDIX A

TEMPERATURE CORRECTIONS FOR SIGHT GLASSES AND PRISMS

TABLE XIV
TEMPERATURE CORRECTION FOR VARIOUS SIGHT GLASSES

Brightness Pyrometer Scale Reading		Type Sight Glass				
(°C)	1/8-in.-thick Quartz	Right-Angle Coated and Silvered Prisms		Quartz Quick Connector	6-in.-diam Pyrex Bell Jar	
		24 x 33 x 24 mm	28 x 54 x 38 mm			
		Temperature Correction, ^a °C				
846				-4		-4
901	3	14	9			
1001				-6		-6
1048	4	20	13			
1201	6	25	15			
1203				-9		-9
1400				-11		-11
1410	9	35	23			
1549				-14		-16
1560	10	42	25			
1700				-18		-16
1709	11	47	29			
1897				-22		-22
2091				-22		-26
2109	19	66	46			
2220	21	71	49			
2279				-28		-26

^aThe precision of this calibration was ±0.2 per cent of the readings.

Figure 50. Calibration Data for Optical Pyrometer.

APPENDIX B

COMPUTER PROGRAM FOR PROCESSING

THERMAL EXPANSION DATA

TABLE XV

COMPUTER PROGRAM FOR PROCESSING THERMAL EXPANSION DATA

```

PROGRAM COOK
  DIMENSION CM(20),FL(5),T(100,5),ND(5),D(100),CE(100,5),CEC(100,5),
  1TCE(100,5),TCEC(100,5),TEM(100,5),TEMC(100,5),TEI(100),TAC(100),
  2TCES(5),TCEL(5),TCECS(5),TCECL(5),TTEMCS(5),TTEMCL(5),
  3AC(100,5),TTEM(100),TTEMC(100,5),ARR(1500),DELTAT(100),TM(5)
  DIMENSION NRA(5),B(4),FT(2),ET(2),TA(100)
  COMMON M,MT C1,MT CN,N,I,NS,NR,S1,S2,S3,DT,T2,T22,T21,T12,P1,P2,P3,P
  14,P5,CM,T,FL,ND,D,CE,CEC,TEM,TEMC,TEI,AC,TCE,TCEC,TTEM,TTEMC,TAC,
  2Y1,DY,AS,AL,DELTAT,
  3TCES,TCEL,TCECS,TCECL,TTEMS,TTEML,TTEMCS,TTEMCL
  COMMON ILS,IW,FT,ET,B,IG,IA,ID
  1 FORMAT(71THERMAL EXPANSION SAMPLE 73A87 RUN NO 711//
  14H L1=F7.5,7 MEASURING SYSTEM=711,
  27 MEAN TEMP COEFF=(DN-D1)/((TN-T1)*L1)7//)
  CALL PLOTS(ARR,1500,6)
  9 READ(10,2)M,MT C1,MT CN,DT
  IF(M)5,6,5
  6 CALL EXIT
  5 N=((MT CN-MT C1)/100)+1
  READ(10,3)(CM(I),I=1,N)
  4 FORMAT(11)
  3 FORMAT(10E8.2)
  7 READ(10,4)NS
  IF(NS)8,9,8
  10 FORMAT(3A8)
  8 READ(10,10)S1,S2,S3
  13 FORMAT(18,F8.5)
  2 FORMAT(318,F8)
  11 READ(10,4)NR
  IF(NR)12,98,12
  12 READ(10,13)ND(NR),FL(NR)
  14 FORMAT(10F8)
  15 FORMAT(10F8.6)
  NDR=ND(NR)
  READ(10,14)(T(I,NR),I=1,NDR)
  READ(10,15)(D(I),I=1,NDR)
  DO16 J=1,NDR
  DELTAT(J)=T(J,NR)-T(1,NR)
  TCE(J,NR)=(D(J)-D(1))/FL(NR)
  IF(DELTAT(J))17,18,17
  17 TTEM(J)=TCE(J,NR)/DELTAT(J)

```

TABLE XV (continued)

```

18 I=T(J,NR)/100.0
   TI=I*100
   I=I+1
   TAC(J)=((TI+100-T(J,NR))*CM(I)+(T(J,NR)-TI)*CM(I+1))/100.0
   TCEC(J,NR)=TCE(J,NR)+TAC(J)*DELTAT(J)
16 TTEM(J,NR)=TTEM(J)+TAC(J)
   TTEM(1)=TTEM(2)
   TTEM(J,NR)=TTEM(1)+TAC(1)
19 FORMAT(7 TEMPERATURE TN-T1      DIAL      CUMULATIVE      MEAN
1      CORRECTION      CUMULATIVE      MEAN7/
27      DEGREES      READING      EXPANSION      TEMP COEFF
3 DEGREES      EXPANSION      TEMP COEFF7/
47      C      INCHES      UNCORRECTED      UNCORRECTED
5      1/C      CORRECTED      CORRECTED7/
67      IN/IN      DEGREES 1/C
7      IN/IN      DEGREES 1/C7)
   WRITE(9,1)S1,S2,S3,NR,FL(NR),M
   WRITE(9,19)
21 FORMAT(F9.1,3XF7.1,3XF9.5,2XE13.5,4E14.5)
   DO20J=1,NDR
20 WRITE(9,21)T(J,NR),DELTAT(J),D(J),TCE(J,NR),TTEM(J),TAC(J),TCEC(J,
1NR),TTEM(J,NR)
   TCES(NR)=FMIN(TCE,NDR,NR)
   TCEL(NR)=FMAX(TCE,NDR,NR)
   TCECS(NR)=FMIN(TCEC,NDR,NR)
   TCECL(NR)=FMAX(TCEC,NDR,NR)
   TTEMS=FMIN(TTEM,NDR,1)
   TTEML=FMAX(TTEM,NDR,1)
   TTEMCS(NR)=FMIN(TTEM,NDR,NR)
   TTEMCL(NR)=FMAX(TTEM,NDR,NR)
   TM(NR)=FMAX(T,NDR,NR)
   IF(TM(NR)-1000.0)46,47,47
46 TS=50.0
   GOTO 48
47 TS=100.0
48 L=(TM(NR)/TS)+2.0
22 FORMAT(7 TEMPERATURE      CORRECTION      CUMULATIVE      EXPANSION      MEA
1N TEMPERATURE COEFFICIENT7/
27 DEGREES C      DEGREES 1/C      IN/IN      DEGR
3EES 1/C7/
47      UNCORRECTED      CORRECTED      UNCORRE
5CTED CORRECTED7)
   WRITE(9,1)S1,S2,S3,NR,FL(NR),M
   WRITE(9,22)

```

TABLE XV (continued)

```

23 FORMAT(F9.1,4X,E13.5,1X,2E13.5,2XE13.5,2XE13.5)
   IS=1
   T2=DT
   NDR1=NDR-1
   DO27 J=1, NDR1
26 IF (T2-T(J+1, NR)) 24, 24, 27
24 T22=T2-T(J+1, NR)
   T21=T2-T(J, NR)
   T12=T(J, NR)-T(J+1, NR)
   AC(IS, NR)=(T22*TAC(J)-T21*TAC(J+1))/T12
   CEC(IS, NR)=(T22*TCEC(J, NR)-T21*TCEC(J+1, NR))/T12
   CE(IS, NR)=(T22*TCE(J, NR)-T21*TCE(J+1, NR))/T12
   TEM(IS, NR)=(T22*TTEM(J, NR)-T21*TTEM(J+1, NR))/T12
   TEMC(IS, NR)=(T22*TTEMC(J, NR)-T21*TTEMC(J+1, NR))/T12
   WRITE(9, 23) T2, AC(IS, NR), CE(IS, NR), CEC(IS, NR), TEM(IS, NR), TEMC(IS, NR)
1)
   IS=IS+1
   T2=T2+DT
   GO TO 26
27 CONTINUE
   FL1=FL(NR)
28 FORMAT(5I1)
   READ(10, 28) P1, P2, P3, P4, P5
   IF(P1) 29, 30, 29
30 IF(P2) 31, 32, 31
32 IF(P3) 33, 34, 33
34 IF(P4) 35, 36, 35
36 IF(P5) 37, 38, 37
29 AS=TCES(NR)
   AL=TCEL(NR)
   CALL LABEL(L, S1, S2, S3, NR, FL1, M, TS)
   CALL YSCALE(AS, AL, Y1, DY)
   CALL SYMBOL(-1.25, 3.0, 0.14, 34HCUMULATIVE EXPANSION (UNCORRECTED), 9
10.0, 34)
   CALL PLOT(0, 0, 3)
   X=0
   DO43 J=1, NDR
   X2=X
   X=T(J, NR)/TS
   Y=(TCE(J, NR)-Y1)/DY
   IF(X2-X) 44, 44, 45
44 CALL SYMBOL(X, Y, 0.08, 3, 0, -1)
   GOTO 43
45 CALL SYMBOL(X, Y, 0.08, 1, 0, -1)

```

TABLE XV (continued)

```
43 CONTINUE
  XL=L+3
  CALL PLOT(XL,0,-3)
  GO TO 30
31 AS=TCECS(NR)
  AL=TCECL(NR)
  CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
  CALL YSCALE(AS,AL,Y1,DY)
  CALL SYMBOL(-1.25,3.0,0.14,32HCUMULATIVE EXPANSION (CORRECTED),90.
10,32)
  CALL PLOT(0,0,3)
  X=0
  DO50J=1,NDR
  X2=X
  X=T(J,NR)/TS
  Y=(TCEC(J,NR)-Y1)/DY
  IF(X2-X)51,51,52
51 CALL SYMBOL(X,Y,0.08,3,0,-1)
  GOTO 50
52 CALL SYMBOL(X,Y,0.08,1,0,-1)
50 CONTINUE
  XL=L+3
  CALL PLOT(XL,0,-3)
  GO TO 32
33 AS=TTEMS
  AL=TTEML
  CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
  CALL YSCALE(AS,AL,Y1,DY)
  CALL SYMBOL(-1.25,3.0,0.14,29HMEAN TEMP COEFF (UNCORRECTED),90.0,2
19)
  CALL PLOT(0,0,3)
  X=0
  DO57J=1,NDR
  X2=X
  X=T(J,NR)/TS
  Y=(TTEM(J)-Y1)/DY
  IF(X2-X)130,130,131
130 CALL SYMBOL(X,Y,0.08,3,0,-1)
  GO TO57
131 CALL SYMBOL(X,Y,0.08,1,0,-1)
57 CONTINUE
  XL=L+3
  CALL PLOT(XL,0,-3)
  GO TO 34
```

TABLE XV (continued)

```
35 AS=TTEMCS(NR)
   AL=TTEMCL(NR)
   CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
   CALL YSCALE(AS,AL,Y1,DY)
   CALL SYMBOL(-1.25,3.0,0.14,27HMEAN TEMP COEFF (CORRECTED),90.0,27)
   CALL PLOT(0,0,3)
   X=0
   DO64J=1,NDR
   X2=X
   X=T(J,NR)/TS
   Y=(TTEMCS(J,NR)-Y1)/DY
   IF(X2-X)132,132,133
132 CALL SYMBOL(X,Y,0.08,3,0,-1)
   GO TO 64
133 CALL SYMBOL(X,Y,0.08,1,0,-1)
64 CONTINUE
   XL=L+3
   CALL PLOT(XL,0,-3)
   GO TO 36
37 AS=TCES(NR)
   AL=TCEL(NR)
   A=1.0
   IF(AS)67,68,67
68 I=0
   GO TO 71
67 I=AS*A
   IF(I)71,70,71
70 A=A*10.0
   GO TO 67
71 AI=I
   Y1=AI/A
69 J=AL*A
   IF(J)77,72,77
72 A=A*10.0
   GO TO 69
77 DY=1.0/(A*10.0)
   DY1=DY
78 IF(10.0*DY+Y1-ABSF(AL))79,75,75
79 DY=DY+DY1
   GO TO 78
75 DY=DY/2.0
   DO80J=1,11
   X=(J-1)*2
80 CALL NUMBER(X-.8,-0.2,0.14,Y1+X*DY,0,4HE9.1)
```

TABLE XV (continued)

```

CALL PLOT(20.0,0,3)
CALL PLOT(0,0,2)
CALL SYMBOL(3.0,-0.4,0.14,34HCUMULATIVE EXPANSION (UNCORRECTED),0,
134)
CALL PLOT(20.0,0,3)
CALL PLOT(20.0,10.0,2)
CALL PLOT(0,10.0,1)
CALL SYMBOL(0.0,10.06,0.21,17HTHERMAL EXPANSION,0,17)
CALL SYMBOL(3.5,10.06,0.14,6HSAMPLE,0,6)
CALL SYMBOL(4.5,10.06,0.14,S1,0,8)
CALL SYMBOL(5.5,10.06,0.14,S2,0,8)
CALL SYMBOL(6.5,10.06,0.14,S3,0,8)
CALL SYMBOL(8.0,10.06,0.14,3HRUN,0,3)
CALL NUMBER(8.5,10.06,0.14,NR,0,2H11)
CALL SYMBOL(9.0,10.06,0.14,3HL1=,0,3)
CALL NUMBER(9.5,10.06,0.14,FL1,0,4HF7.5)
CALL SYMBOL(11.0,10.06,0.14,16HMEASURING SYSTEM,0,16)
CALL NUMBER(13.0,10.06,0.14,M,0,2H11)
CALL PLOT(0,10.0,3)
CALL PLOT(0,0,2)
I=(TM(NR)/100.0)+1.0
DX=I*10
IF(DX-100.0)81,82,82
81 DX=100.0
GOTO84
82 IF(DX*10.0-TM(NR))83,84,84
83 DX=DX+10.0
GO T082
84 D085K=1,11
Y=K-1
85 CALL NUMBER(-.52,Y-0.07,0.14,Y*DX,0,2HF4)
CALL SYMBOL(-1.0,3.0,0.14,32HTEMPERATURE DEGREES CENTIGRADE,90.0
1,32)
CALL PLOT(0,0,3)
Y=0
D086J=1,NDR
X2=Y
X=(TCE(J,NR)-Y1)/DY
Y=T(J,NR)/DX
IF(X2-Y)65,65,66
65 CALL SYMBOL(X,Y,0.08,3,0,-1)
GO TO 86
66 CALL SYMBOL(X,Y,0.08,1,0,-1)
86 CONTINUE

```

TABLE XV (continued)

```

      CALL PLOT(24.0,0,-3)
40  FORMAT(2I8,4F8,I2)
38  READ(10,40) ILS, IW, FT(1), ET(1), FT(2), ET(2), ID
      IF( ILS) 41, 11, 41
41  IA=1
      NRA(1)=NR
      CALL PLS( IW, FT, ET, T, TTEMC, TCEC, NRA, NDR, TS, S1, S2, S3, FL1, M, IG, IA, B)
49  IF( ID) 97, 11, 97
92  FORMAT(7 TEMPERATURE MEAN TEMP COEFF INSTANTANEOUS7/
17  DEGREES C CORRECTED TEMP COEFF7/
27  DEGREES 1/C DEGREES 1/C7/)
97  WRITE(9,1) S1, S2, S3, NR, FL(NR), M
      WRITE(9, 92)
93  FORMAT(F9.1, 5XE13.5, 2XE13.5)
      T2=DT
      IS=TM(NR)/DT
      T1=T(1, NR)
      DO90N=1, IS
      IG1J=IG+1
      TEMP=0.0
      DO91J=1, IG
      TEMP=TEMP*(T2-T1)+B(IG1J)*(IG1J-1)
91  IG1J=IG1J-1
      WRITE(9, 93) T2, TEMC(N, NR), TEMP
90  T2=T2+DT
94  FORMAT(70PARAMETERS OF POLYNOMIAL7)
95  FORMAT(7 B(7I1,7)=7E12.4)
      WRITE(9, 94)
      IG1=IG+1
      DO96J=1, IG1
96  WRITE(9, 95) J, B(J)
      GO TO 11
98  READ(10,4) IA
      IF( IA) 99, 100, 99
99  READ(10, 102) (NRA(J), J=1, IA)
102 FORMAT(5I1)
101 FORMAT(71AVERAGE THERMAL EXPANSION SAMPLE 73A8//
17  MEASURING SYSTEM=7I1,
27  MEAN TEMP COEFF=(DN-D1)/((TN-T1)*L1)7//)
      WRITE(9, 101) S1, S2, S3, M
103 FORMAT(7 RUN 7I1,7 L1(RUN7I1,7)=7F7.5)
      J=NRA(1)
      TML=TM(J)
      DO104J=1, IA

```

TABLE XV (continued)

```

      NR=NRA(J)
      TML=MIN1F(TML,TM(NR))
104 WRITE(9,103)NR,NR,FL(NR)
      WRITE(9,22)
      T2=DT
      IS=TML/DT
      DIA=IA
      DO105J=1,IS
      TEM1=0.0
      TEM2=0.0
      TEM3=0.0
      TEM4=0.0
      TEM5=0.0
      DO106K=1,IA
      NR=NRA(K)
      TEM5=AC(J,NR)+TEM5
      TEM1=CEC(J,NR)+TEM1
      TEM2=CE(J,NR)+TEM2
      TEM3=TEM(J,NR)+TEM3
106 TEM4=TEMC(J,NR)+TEM4
      AC(J,1)=TEM5/DIA
      CEC(J,1)=TEM1/DIA
      CE(J,1)=TEM2/DIA
      TEM(J,1)=TEM3/DIA
      TEMC(J,1)=TEM4/DIA
      WRITE(9,23)T2,AC(J,1),CE(J,1),CEC(J,1),TEM(J,1),TEMC(J,1)
      TA(J)=T2
105 T2=T2+DT
      READ(10,40)ILS,IW,FT(1),ET(1),FT(2),ET(2),ID
      IF(ILS)107,100,107
107 CALL PLS(IW,FT,ET,TA,TEMC,CEC,NRA,IS,TS,S1,S2,S3,FL1,M,IG,IA,B)
      IF(ID)108,100,108
108 WRITE(9,101)S1,S2,S3,M
      DO109J=1,IA
      NR=NRA(J)
109 WRITE(9,103)NR,NR,FL(NR)
      WRITE(9,92)
      T2=DT
      T1=TA(1)
      DO110N=1,IS
      IG1J=IG+1
      TEMP=0.0
      DO111J=1,IG
      TEMP=TEMP*(T2-T1)+B(IG1J)*(IG1J-1)

```

TABLE XV (continued)

```

111 IG1J=IG1J-1
    WRITE(9,93)T2,TEMC(N,1),TEMP
110 T2=T2+DT
    WRITE(9,94)
    IG1=IG+1
    DO112J=1,IG1
112 WRITE(9,95)J,B(J)
100 READ(10,120)IA,(NRA(J),J=1,5)
120 FORMAT(611)
    IF(IA)121,7,121
121 CALL MM(IA,TCEC,TCEL,AS,AL,NRA)
    CALL MM(IA,TM,TM,TMS,TMM,NRA)
    IF(TMM-1000.0)122,122,123
122 TS=50.0
    GO TO124
123 TS=100.0
124 L=(TMM/TS)+2.0
    NR=0
    CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
    CALL YSCALE(AS,AL,Y1,DY)
    CALL SYMBOL(-1.25,3.0,0.14,34HCUMULATIVE EXPANSION (UNCORRECTED),9
10.0,34)
    CALL SP(IA,NRA,T,TCEC,TS,Y1,DY,ND)
    CALL MM(IA,TCECS,TCECL,AS,AL,NRA)
    NR=0
    CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
    CALL YSCALE(AS,AL,Y1,DY)
    CALL SYMBOL(-1.25,3.0,0.14,32HCUMULATIVE EXPANSION (CORRECTED),90.
10,32)
    CALL SP(IA,NRA,T,TCEC,TS,Y1,DY,ND)
    CALL MM(IA,TTEMCS,TTEMCL,AS,AL,NRA)
    NR=0
    CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
    CALL YSCALE(AS,AL,Y1,DY)
    CALL SYMBOL(-1.25,3.0,0.14,27HMEAN TEMP COEFF (CORRECTED),90.0,27)
    CALL SP(IA,NRA,T,TTEMC,TS,Y1,DY,ND)
    GO TO 7
END
SUBROUTINE YSCALE(AS,AL,Y1,DY)
    A=1.0
    IF(AS)3,12,3
12 I=0
    GO TO4
3 I=AS*A

```

TABLE XV (continued)

```

      IF(I)4,1,4
1  A=A*10.0
   GO TO 3
4  AI=I
   Y1=AI/A
2  J=AL*A
   IF(J)19,5,19
5  A=A*10.0
   GO TO 2
19 DY=1.0/(A*10.0)
   DY1=DY
15 IF(10.0*DY+Y1-ABSF(AL))14,14,8
14 DY=DY+DY1
   GO TO 15
8  DO10K=1,11
   Y=K-1
10~ CALL NUMBER(-1.1,Y-0.07,0.14,Y1+Y*DY,0,4HE9.1)
   END
   SUBROUTINE SP(IA,NRA,T,B,TS,Y1,DY,ND)
   DIMENSION NRA(5),T(100,5),B(100,5),ND(5)
   Y2=9.75
   LA=1
   DO1L=1,IA
   NR=NRA(L)
   NDR=ND(NR)
   CALL SYMBOL(0.1,Y2,0.08,LA,0,-1)
   CALL SYMBOL(0.2,Y2,0.14,3HRUN,0,3)
   CALL NUMBER(0.7,Y2,0.14,NR,0,2H11)
   DO2J=1,NDR
   X=T(J,NR)/TS
   Y=(B(J,NR)-Y1)/DY
2  CALL SYMBOL(X,Y,0.08,LA,0,-1)
   Y2=Y2-0.25
1  LA=LA+2
   CALL PLOT(24.0,0,-3)
   END
   FUNCTION FMIN(ARRAY,NDR,NR)
   DIMENSION ARRAY(100,5)
   AS=ARRAY(1,NR)
   DO1J=1,NDR
   AJ=ARRAY(J,NR)
1  AS=MIN1F(AS,AJ)
   FMIN=AS
   END

```

TABLE XV (continued)

```

FUNCTION FMAX(ARRAY,NDR,NR)
  DIMENSION ARRAY(100,5)
  AL=ARRAY(NDR,NR)
  DO1 J=1,NDR
    AJ=ARRAY(J,NR)
1  AL=MAX1F(AL,AJ)
  FMAX=AL
  END
  SUBROUTINE MM(IA,ABS,ABL,AS,AL,NRA)
    DIMENSION ABS(5),ABL(5),NRA(5)
    J=NRA(1)
    AS=ABS(J)
    AL=ABL(J)
    DO1 L=1,IA
      J=NRA(L)
      AS=MIN1F(AS,ABS(J))
1  AL=MAX1F(AL,ABL(J))
  END
  SUBROUTINE LABEL(L,S1,S2,S3,NR,FL1,M,TS)
    XLABEL=0.0
    DO1 J=1,L
      X=J-1
      CALL NUMBER(X,-.22,-0.2,0.14,XLABEL,0,2HF4)
1  XLABEL=XLABEL+TS
    X=L
    CALL PLOT(X,0,3)
    CALL PLOT(0,0,2)
    CALL SYMBOL(3.0,-0.4,0.14,32HTEMPERATURE DEGREES CENTIGRADE,0,32
1)
    CALL PLOT(X,0,3)
    CALL PLOT(X,10.0,2)
    CALL PLOT(0,10.0,1)
    CALLSYMBOL(0.0,10.06,0.21,17HTHERMAL EXPANSION,0,17)
    CALL SYMBOL(3.5,10.06,0.14,6HSAMPLE,0,6)
    CALL SYMBOL(4.5,10.06,0.14,S1,0,8)
    CALL SYMBOL(5.5,10.06,0.14,S2,0,8)
    CALL SYMBOL(6.5,10.06,0.14,S3,0,8)
    IF(NR)2,3,2
2  CALL SYMBOL(7.0,10.06,0.14,3HRUN,0,3)
    CALL NUMBER(7.5,10.06,0.14,NR,0,2H11)
    CALL SYMBOL(8.0,10.06,0.14,3HL1=,0,3)
    CALL NUMBER(8.5,10.06,0.14,FL1,0,4HF7.5)
3  CALL SYMBOL(10.0,10.06,0.14,16HMEASURING SYSTEM,0,16)
    CALL NUMBER(12.0,10.06,0.14,M,0,2H11)

```

TABLE XV (continued)

```

      CALL PLOT(0,10.0,3)
      CALL PLOT(0,0,2)
      END
      SUBROUTINE PLS(IW,FT,ET,T,A1,A2,NRA,NDR,TS,S1,S2,S3,FL1,M,IG,IA,B)
      DIMENSION T(100,5),A1(100,5),A2(100,5),W(100),YA(100),YB(100),X(10
10,5)YCALC(100),STDEB(5),DIF(100),SIG(5),NRA(5),B(5),FT(2),ET(2)
      DIMENSION W1(100)
1  FORMAT(10F8)
      JDT=0
      IF(IW)25,26,25
26  DO27J=1,NDR
27  W1(J)=1.0
      GOTO 24
25  READ(10,1)(W1(J),J=1,NDR)
24  IA1=IA-1
      IF(IA1)20,21,20
21  NR=NRA(1)
      GO TO 22
20  NR=1
22  I=0
      DO2J=1,NDR
      TJ=T(J,NR)
      IF(TJ-FT(1))2,3,3
3  IF(TJ-ET(1))4,4,2
4  I=I+1
      W(1)=W1(J)
      X(1,1)=1.0
      X(1,2)=TJ
      X(1,3)=TJ*TJ
      X(1,4)=TJ*TJ*TJ
      X(1,5)=X(1,3)*X(1,3)
      YA(1)=A1(J,NR)
2  CONTINUE
      NDP=I
      AS=FMIN(YA,NDP,1)
      AL=FMAX(YA,NDP,1)
      L=(ET(1)/TS)+2.0
      IF(IA1)23,28,23
23  NR=0
28  DO5N=1,3
      NP=N+1
      CALL MULREG(W,X,YA,B,VARY,STDEB,YCALC,NP,NDP,DIF)
      SIG(N)=VARY
      N1=N-1

```

TABLE XV (continued)

```

      IF(N1)8,5,8
8  D09J=1,N1
      IF(SIG(N)-SIG(J)*0.5)9,9,80
9  CONTINUE
5  CONTINUE
      IG=N1+1
      GO TO 10
80  IG=N1
10  N=N1+1
      NP=IG+1
11  FORMAT(7H SIGMA(11,3H )=E12.4)
      D012J=1,N
12  WRITE(9,11)J,SIG(J)
      CALL MULREG(W,X,YA,B,VARY,STDEB,YCALC,NP,NDP,DIF)
      AJ=FMIN(YCALC,NDP,1)
      AS=MIN1F(AS,AJ)
      AJ=FMAX(YCALC,NDP,1)
      AL=MAX1F(AL,AJ)
      CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
      CALL YSCALE(AS,AL,Y1,DY)
      IF(IA1)33,34,33
34  CALL SYMBOL(-1.25,3.0,0.14,27HMEAN TEMP COEFF (CORRECTED),90.0,27)
      GO TO 35
33  CALL SYMBOL(-1.25,2.5,0.14,35HAVERAGE MEAN TEMP COEFF (CORRECTED),
190.0,35)
      YL=9.75
      D039J=1,IA
      NR=NRA(J)
      CALL SYMBOL(4.0,YL,0.14,3HRUN,0,3)
      CALL NUMBER(4.5,YL,0.14,NR,0,2H11)
      CALL SYMBOL(5.0,YL,0.14,3HL1=,0,3)
      CALL NUMBER(5.5,YL,0.14,FL1,0,4HF7.5)
39  YL=YL-0.25
35  CALL PLOT(0,0,3)
      D06J=1,NDP
      X1=X(J,2)/TS
      Y2=(YA(J)-Y1)/DY
6  CALL SYMBOL(X1,Y2,0.08,3,0,-1)
      CALL SYMBOL(0.1,9.5,0.14,21HDEGREE OF POLYNOMIAL=,0,21)
      CALL NUMBER(2.7,9.5,0.14,IG,0,2H11)
      CALL SYMBOL(0.1,9.0,0.14,10HPARAMETERS,0,10)
      N1=IG+1
      YL=9.0
      D030J=1,N1

```

TABLE XV (continued)

```

YL=YL-0.4
30 CALL NUMBER(0.1,YL,0.14,B(J),0,5HE12.4)
   X1=X(1,2)/TS
   Y2=(YCALC(1)-Y1)/DY
   CALL PLOT(X1,Y2,3)
   D07J=1,NDP
   X1=X(J,2)/TS
   Y2=(YCALC(J)-Y1)/DY
7  CALL SYMBOL(X1,Y2,0.08,1,0,-2)
   XL=L+3
   CALL PLOT(XL,0,-3)
   IF(IA1)70,71,70
70 NR=1
71 I=0
   D072J=1,NDR
   TJ=T(J,NR)
   IF(TJ-ET(2))72,73,73
73 IF(TJ-ET(2))74,74,72
74 I=I+1
   W(I)=W1(J)
   X(I,1)=1,0
   X(I,2)=TJ
   X(I,3)=TJ*TJ
   X(I,4)=TJ*TJ*TJ
   YB(I)=A2(J,NR)
72 CONTINUE
   NDP=I
   L=(ET(2)/TS)+2.0
92 IF(IA1)75,46,75
75 NR=0
46 D031N=1,3
   NP=N+1
   CALL MULREG(W,X,YB,B,VARY,STDEB,YCALC,NP,NDP,DIF)
   SIG(N)=VARY
   N1=N-1
   IF(N1)38,31,38
38 D059J=1,N1
   IF(SIG(N)-SIG(J)*0.5)59,59,81
59 CONTINUE
31 CONTINUE
   IG=N1+1
   GO TO 60
81 IG=N1
60 N=N1+1

```

TABLE XV (continued)

```

NP=IG+1
CALL MULREG(W,X,YB,B,VARY,STDEB,YCALC,NP,NDP,DIF)
AS=FMIN(YB,NDP,1)
AL=FMAX(YB,NDP,1)
AJ=FMIN(YCALC,NDP,1)
AS=MIN1F(AS,AJ)
AJ=FMAX(YCALC,NDP,1)
AL=MAX1F(AL,AJ)
DO42J=1,N
42 WRITE(9,11)J,SIG(J)
CALL LABEL(L,S1,S2,S3,NR,FL1,M,TS)
CALL YSCALE(AS,AL,Y1,DY)
IF(IA1)13,14,13
14 CALL SYMBOL(-1.25,3.0,0.14,32HCUMULATIVE EXPANSION (CORRECTED),90.
10,32)
GO TO 15
13 CALL SYMBOL(-1.25,2.5,0.14,40HAVERAGE CUMULATIVE EXPANSION (CORREC
1TED),90.0,40)
YL=9.75
DO29J=1,IA
NR=NRA(J)
CALL SYMBOL(4.0,YL,0.14,3HRUN,0,3)
CALL NUMBER(4.5,YL,0.14,NR,0,2H11)
CALL SYMBOL(5.0,YL,0.14,3HL1=,0,3)
CALL NUMBER(5.5,YL,0.14,FL1,0,4HF7.5)
29 YL=YL-0.25
15 CALL PLOT(0,0,3)
DO36J=1,NDP
X1=X(J,2)/TS
Y2=(YB(J)-Y1)/DY
36 CALL SYMBOL(X1,Y2,0.08,3,0,-1)
CALL SYMBOL(0.1,9.5,0.14,21HDEGREE OF POLYNOMIAL=,0,21)
CALL NUMBER(2.7,9.5,0.14,IG,0,2H11)
CALL SYMBOL(0.1,9.0,0.14,10HPARAMETERS,0,10)
N1=IG+1
YL=9.0
DO40J=1,N1
YL=YL-0.4
40 CALL NUMBER(0.1,YL,0.14,B(J),0,5HE12.4)
X1=X(1,2)/TS
Y2=(YCALC(1)-Y1)/DY
CALL PLOT(X1,Y2,3)
DO37J=1,NDP
X1=X(J,2)/TS

```

TABLE XV (continued)

```
      Y2=(YCALC(J)-Y1)/DY
37 CALL SYMBOL(X1,Y2,0.08,1,0,-2)
      IF(JDT)49,50,49
49 CALL SYMBOL(2.0,-0.4,0.14,7H(TN-T1),0,7)
50 XL=L+3
      CALL PLOT(XL,0,-3)
      IF(IA1)90,91,90
90 NR=1
91 IF(JDT)48,47,48
47 T1=T(1,NR)
      DO45J=1,NDP
      X(J,2)=X(J,2)-T1
      X(J,3)=X(J,2)*X(J,2)
45 X(J,4)=X(J,2)*X(J,3)
      JDT=1
      GO TO 92
48 END
```

APPENDIX C

TYPICAL COMPUTER OUTPUT

TABLE XVI

TYPICAL COMPUTER OUTPUT OF THERMAL EXPANSION DATA FOR ONE DETERMINATION

THERMAL EXPANSION SAMPLE (THU)C2 T6B SILICA RUN NO 2
 LI=1.08428 MEASURING SYSTEM=1 MEAN TEMP COEFF=(DN-DI)/((TN-TI)*LI)

TEMPERATURE DEGREES C	TN-TI	DIAL READING INCHES	CUMULATIVE EXPANSION UNCORRECTED IN/IN	MEAN TEMP COEFF UNCORRECTED DEGREES 1/C	CORRECTION DEGREES 1/C	CUMULATIVE EXPANSION CORRECTED IN/IN	MEAN TEMP COEFF CORRECTED DEGREES 1/C
52.0	0	.19612	0	3.65728E-06	1.20720E-06	0	4.86448E-06
110.0	58.0	.19635	2.12122E-04	3.65728E-06	1.12400E-06	2.77314E-04	4.78128E-06
166.0	114.0	.19698	7.93153E-04	6.95748E-06	1.03440E-06	9.11075E-04	7.99188E-06
230.0	178.0	.19762	1.38341E-03	7.77195E-06	9.32000E-07	1.54930E-03	8.70395E-06
289.0	237.0	.19833	2.03822E-03	8.60008E-06	8.37600E-07	2.23673E-03	9.43768E-06
358.0	306.0	.19899	2.64692E-03	8.65006E-06	6.75000E-07	2.85347E-03	9.32506E-06
427.0	375.0	.19980	3.39396E-03	9.05055E-06	5.51100E-07	3.60062E-03	9.60165E-06
501.0	449.0	.20093	4.43612E-03	9.88001E-06	4.99400E-07	4.66035E-03	1.03794E-05
591.0	539.0	.20220	5.60741E-03	1.04034E-05	4.45400E-07	5.84748E-03	1.08488E-05
666.0	614.0	.20321	6.53890E-03	1.06497E-05	3.93800E-07	6.78069E-03	1.10435E-05
765.0	713.0	.20468	7.89464E-03	1.10724E-05	3.11500E-07	8.11674E-03	1.13839E-05
788.0	736.0	.20506	8.24510E-03	1.12026E-05	2.90800E-07	8.45913E-03	1.14934E-05
856.0	804.0	.20612	9.22271E-03	1.14710E-05	2.35200E-07	9.41181E-03	1.17062E-05
922.0	870.0	.20719	1.02095E-02	1.17351E-05	1.91200E-07	1.03759E-02	1.19263E-05
980.0	928.0	.20818	1.11226E-02	1.19855E-05	1.68000E-07	1.12785E-02	1.21535E-05
1010.0	958.0	.20870	1.16022E-02	1.21108E-05	1.54000E-07	1.17497E-02	1.22648E-05

TABLE XVII

TYPICAL COMPUTER OUTPUT OF INTERPOLATED THERMAL
EXPANSION DATA FOR ONE DETERMINATION

THERMAL EXPANSION SAMPLE (THU)C2 T6B SILICA RUN NO 2
LI=1.08428 MEASURING SYSTEM=1 MEAN TEMP COEFF=(DN-DI)/((TN-TI)*LI)

TEMPERATURE DEGREES C	CORRECTION DEGREES 1/C	CUMULATIVE EXPANSION IN/IN		MEAN TEMPERATURE COEFFICIENT DEGREES 1/C	
		UNCORRECTED	CORRECTED	UNCORRECTED	CORRECTED
50.0	1.21007E-06	-7.31456E-06	-9.56256E-06	3.65728E-06	4.86735E-06
100.0	1.13834E-06	1.75550E-04	2.29502E-04	3.65728E-06	4.79563E-06
150.0	1.06000E-06	6.27144E-04	7.30000E-04	5.01457E-06	7.07457E-06
200.0	9.80000E-07	1.10673E-03	1.25013E-03	7.39017E-06	8.37017E-06
250.0	9.00000E-07	1.60538E-03	1.78233E-03	8.05267E-06	8.95267E-06
300.0	8.11678E-07	2.13526E-03	2.33505E-03	8.60805E-06	9.41973E-06
350.0	6.93852E-07	2.57634E-03	2.78196E-03	8.64426E-06	9.33812E-06
400.0	5.99583E-07	3.10164E-03	3.30826E-03	8.89384E-06	9.49342E-06
450.0	5.35031E-07	3.71787E-03	3.93000E-03	9.30836E-06	9.84339E-06
500.0	5.00099E-07	4.42204E-03	4.64603E-03	9.86880E-06	1.03689E-05
550.0	4.70000E-07	5.07382E-03	5.30668E-03	1.01649E-05	1.06349E-05
600.0	4.39208E-07	5.71919E-03	5.95946E-03	1.04329E-05	1.08721E-05
650.0	4.04808E-07	6.34018E-03	6.58161E-03	1.05971E-05	1.10019E-05
700.0	3.65535E-07	7.00451E-03	7.23954E-03	1.07949E-05	1.11604E-05
750.0	3.23970E-07	7.68922E-03	7.91431E-03	1.10084E-05	1.13323E-05
800.0	2.80988E-07	8.41762E-03	8.62725E-03	1.12500E-05	1.15309E-05
850.0	2.40106E-07	9.13645E-03	9.32775E-03	1.14473E-05	1.16875E-05
900.0	2.05867E-07	9.88060E-03	1.00545E-02	1.16471E-05	1.18529E-05
950.0	1.80000E-07	1.06503E-02	1.08116E-02	1.18560E-05	1.20360E-05
1000.0	1.58667E-07	1.14423E-02	1.15926E-02	1.20691E-05	1.22277E-05
SIGMA(1) = 9.8546E-13					
SIGMA(2) = 4.4369E-13					
SIGMA(3) = 2.8010E-13					
SIGMA(1) = 9.5132E-08					
SIGMA(2) = 6.1231E-09					
SIGMA(3) = 4.8955E-09					
SIGMA(1) = 9.5132E-08					
SIGMA(2) = 6.1231E-09					
SIGMA(3) = 4.8955E-09					

TABLE XVIII

TYPICAL COMPUTER OUTPUT OF INSTANTANEOUS AND MEAN TEMPERATURE

COEFFICIENTS OF EXPANSION FOR ONE DETERMINATION

THERMAL EXPANSION SAMPLE (THU)C2 T6B SILICA RUN NO 2
 L1=1.08428 MEASURING SYSTEM=1 MEAN TEMP COEFF=(DN-D1)/((TN-T1)*L1)

TEMPERATURE DEGREES C	MEAN TEMP COEFF CORRECTED DEGREES 1/C	INSTANTANEOUS TEMP COEFF DEGREES 1/C
50.0	4.86735E-06	8.98331E-06
100.0	4.79563E-06	9.34278E-06
150.0	7.07457E-06	9.70224E-06
200.0	8.37017E-06	1.00617E-05
250.0	8.95267E-06	1.04212E-05
300.0	9.41973E-06	1.07806E-05
350.0	9.33812E-06	1.11401E-05
400.0	9.49342E-06	1.14996E-05
450.0	9.84339E-06	1.18590E-05
500.0	1.03689E-05	1.22185E-05
550.0	1.06349E-05	1.25780E-05
600.0	1.08721E-05	1.29374E-05
650.0	1.10019E-05	1.32969E-05
700.0	1.11604E-05	1.36564E-05
750.0	1.13323E-05	1.40158E-05
800.0	1.15309E-05	1.43753E-05
850.0	1.16875E-05	1.47347E-05
900.0	1.18529E-05	1.50942E-05
950.0	1.20360E-05	1.54537E-05
1000.0	1.22277E-05	1.58131E-05

PARAMETERS OF POLYNOMIAL

B(1)= -1.4616E-04

B(2)= 8.9977E-06

B(3)= 3.5946E-09

TABLE XIX

TYPICAL COMPUTER OUTPUT OF THE AVERAGE THERMAL EXPANSION
DATA FOR THREE DETERMINATIONS

AVERAGE THERMAL EXPANSION SAMPLE (THU)C2 T6B SILICA

MEASURING SYSTEM=1 MEAN TEMP COEFF=(DN-DI)/((TN-TI)*LI)

RUN 1 LI(RUN1)=1.08428
RUN 2 LI(RUN2)=1.08428
RUN 3 LI(RUN3)=1.08428

TEMPERATURE DEGREES C	CORRECTION DEGREES 1/C	CUMULATIVE EXPANSION IN/IN		MEAN TEMPERATURE COEFFICIENT DEGREES 1/C	
		UNCORRECTED	CORRECTED	UNCORRECTED	CORRECTED
50.0	1.21002E-06	-1.77082E-05	-1.92375E-05	6.54576E-06	7.75577E-06
100.0	1.13874E-06	2.98363E-04	3.53225E-04	6.43359E-06	7.57233E-06
150.0	1.05996E-06	7.59746E-04	8.63539E-04	7.52519E-06	8.58515E-06
200.0	9.80000E-07	1.26538E-03	1.40943E-03	8.44761E-06	9.42761E-06
250.0	8.99581E-07	1.76238E-03	1.93947E-03	8.86380E-06	9.76338E-06
300.0	8.13009E-07	2.26553E-03	2.46651E-03	9.11046E-06	9.92347E-06
350.0	6.94226E-07	2.75288E-03	2.95831E-03	9.21397E-06	9.90819E-06
400.0	5.99401E-07	3.27188E-03	3.47901E-03	9.37528E-06	9.97468E-06
450.0	5.35749E-07	3.83637E-03	4.04932E-03	9.61095E-06	1.01467E-05
500.0	5.00978E-07	4.49549E-03	4.71975E-03	1.00074E-05	1.05084E-05
550.0	4.69973E-07	5.15144E-03	5.38494E-03	1.03156E-05	1.07856E-05
600.0	4.39167E-07	5.80393E-03	6.04447E-03	1.05733E-05	1.10425E-05
650.0	4.04885E-07	6.45891E-03	6.70068E-03	1.07830E-05	1.11879E-05
700.0	3.66361E-07	7.13388E-03	7.37028E-03	1.09882E-05	1.13546E-05
750.0	3.24657E-07	7.82019E-03	8.04636E-03	1.11890E-05	1.15137E-05
800.0	2.80771E-07	8.52088E-03	8.73064E-03	1.13790E-05	1.16598E-05
850.0	2.40035E-07	9.23185E-03	9.42285E-03	1.15553E-05	1.17953E-05
900.0	2.05700E-07	9.97250E-03	1.01465E-02	1.17466E-05	1.19523E-05
950.0	1.79929E-07	1.07243E-02	1.08858E-02	1.19316E-05	1.21115E-05
1000.0	1.58842E-07	1.14796E-02	1.16302E-02	1.20997E-05	1.22586E-05
SIGMA(1)=	1.2150E-13				
SIGMA(2)=	7.5873E-14				
SIGMA(1)=	5.1792E-08				
SIGMA(2)=	2.0163E-09				
SIGMA(3)=	1.7541E-09				
SIGMA(1)=	5.1792E-08				
SIGMA(2)=	2.0163E-09				
SIGMA(3)=	1.7541E-09				

TABLE XX

TYPICAL COMPUTER OUTPUT OF THE AVERAGE INSTANTANEOUS AND MEAN
TEMPERATURE COEFFICIENTS FOR THREE DETERMINATIONS

AVERAGE THERMAL EXPANSION SAMPLE (THU)C2 T6B SILICA
MEASURING SYSTEM=1 MEAN TEMP COEFF=(DN-DI)/((TN-TI)*LI).

RUN 1	LI(RUN1)=1.08428	
RUN 2	LI(RUN2)=1.08428	
RUN 3	LI(RUN3)=1.08428	
TEMPERATURE	MEAN TEMP COEFF	INSTANTANEOUS
DEGREES C	CORRECTED	TEMP COEFF
	DEGREES 1/C	DEGREES 1/C
50.0	7.75577E-06	9.38814E-06
100.0	7.57233E-06	9.70443E-06
150.0	8.58515E-06	1.00207E-05
200.0	9.42761E-06	1.03370E-05
250.0	9.76338E-06	1.06533E-05
300.0	9.92347E-06	1.09696E-05
350.0	9.90819E-06	1.12859E-05
400.0	9.97468E-06	1.16022E-05
450.0	1.01467E-05	1.19185E-05
500.0	1.05084E-05	1.22347E-05
550.0	1.07856E-05	1.25510E-05
600.0	1.10125E-05	1.28673E-05
650.0	1.11879E-05	1.31836E-05
700.0	1.13546E-05	1.34999E-05
750.0	1.15137E-05	1.38162E-05
800.0	1.16598E-05	1.41325E-05
850.0	1.17953E-05	1.44488E-05
900.0	1.19523E-05	1.47651E-05
950.0	1.21115E-05	1.50813E-05
1000.0	1.22586E-05	1.53976E-05

PARAMETERS OF POLYNOMIAL

B(1)= -1.1142E-04

B(2)= 9.3881E-06

B(3)= 3.1629E-09

Figure 51. Typical Computer Plot of Cumulative Expansion Versus Temperature for One Determination on a 41.4 Mole Per Cent ThC_2 Alloy.

Figure 52. Typical Computer Plot of Cumulative Expansion Versus Temperature for Three Determinations on a 41.4 Mole Per Cent Alloy.

Figure 53. Typical Computer Plot of Mean Temperature Coefficient Versus Temperature for Three Determinations on a 41.4 Mole Per Cent ThC₂ Alloy.

Figure 54 Typical Computer Plot of the Average Cumulative Expansion Versus Temperature for Three Determinations on a 41.4 Mole Per Cent ThC₂ Alloy.

Figure 55. Typical Computer Plot of the Average Cumulative Expansion Versus Temperature Above the Datum Level for Three Determinations on a 41.4 Mole Per Cent ThC_2 Alloy.

APPENDIX D

TEMPERATURE CORRECTION FOR THERMAL ANALYZER

TABLE XXI
TEMPERATURE CORRECTION FOR THERMAL ANALYZER

Brightness Pyrometer Scale Reading (°C)	Temperature Correction ^a (°C)
900	100
988	112
1063	137
1145	155
1226	174
1307	193
1382	218
1460	240
1540	260
1614	286
1691	309
1765	335
1795	345

^aThis correction includes errors due to the optical-beam splitter, two 1/8-in.-thick quartz sight glasses, and the pyrometer (serial No. M-5418). An NBS standard lamp No. G25664b was used.

Figure 56 Calibration for Optical Pyrometer Serial No. M-5418 Using the Supplementary Objective Lens Marked "B" and Sighting on the Optical Beam Splitter Drawing No. SK-GWA-105, and Two One-Eighth-Inch-Thick Quartz Sight Glasses.

APPENDIX E

MICROHARDNESS FOR ANNEALED $(U,Th)C_2$ ALLOYS

TABLE XXII
MICROHARDNESS FOR ANNEALED (U,Th)C₂ ALLOYS

Specimen Designation	Composition (mole % ThC ₂)	Annealing Treatment ^a		Knoop Hardness	
		Temperature (°C)	Time (hr)	Range (KHN)	Average (KHN)
T-22	100	As cast		359-495	439
ThC ₂ -8C	100	As cast		583-631	609
ThC ₂ -8C-13-218	100	1300	218	423-541	477
M-3	87.7	As cast		484-907	749
M-3-14-80	87.7	1400	80	127-171	154
T-12	45.5	As cast		317-386	347
T-12-12-446	45.5	1200	446	398-647	538
T-12-15-455	45.5	1500	455	484-578	513
T-12-20-118	45.5	2000	118	488-732	613
T-11	45.2	As cast		295-612	508
T-11-10-190	45.2	1000	190	402-578	505
T-11-10-453	45.2	1000	453	446-574	495
T-11-10-950	45.2	1000	950	351-528	431
T-11-12-15-446	45.2	1200	446	495-588	538
T-11-15-445	45.2	1500	445	384-607	513
T-11-19-350	45.2	1900	350	583-618	602
T-11-19-1190	45.2	1900	1190	788-907	858
T-7	41.7	As cast		435-549	497
T-7-10-190	41.7	1000	190	455-567	519
T-7-10-453	41.7	1000	453	342-432	394
T-7-10-950	41.7	1000	950	545-607	580
T-7-12-150	41.7	1200	150	391-588	517
T-7-12-450	41.7	1200	450	462-583	545

TABLE XXII (continued)

Specimen Designation	Composition (mole % ThC ₂)	Annealing Treatment ^a		Knoop Hardness	
		Temperature (°C)	Time (hr)	Range (KHN)	Average (KHN)
T-7-12-744	41.7	1200	744	446-647	559
T-7-12-907	41.7	1200	907	391-488	437
T-7-12-1243	41.7	1200	1243	423-625	460
T-7-15-150	41.7	1500	150	361-703	526
T-7-15-425	41.7	1500	425	311-658	505
T-7-15-1165	41.7	1500	1165	368-664	538
T-7-19-150	41.7	1900	150	574-742	675
T-7-19-500	41.7	1900	500	321-618	512
T-7-19-1350	41.7	1900	1350	585-872	709
T-6	41.4	As cast		492-547	523
T-6-10-190	41.4	1000	190	356-558	446
T-6-10-453	41.4	1000	453	286-499	390
T-6-10-950	41.4	1000	950	426-618	527
T-6-12-150	41.4	1200	150	528-607	583
T-6-12-450	41.4	1200	450	703-818	765
T-6-12-744	41.4	1200	744	481-583	554
T-6-12-907	41.4	1200	907	435-670	560
T-6-12-1243	41.4	1200	1243	377-636	487
T-6-15-150	41.4	1500	150	373-583	475
T-6-15-425	41.4	1500	425	441-524	488
T-6-15-1165	41.4	1500	1165	647-735	693
T-6-19-150	41.4	1900	150	578-715	653
T-6-19-500	41.4	1900	500	438-722	600
T-6-19-1350	41.4	1900	1350	618-818	749
T-8	40.7	As cast		268-384	317
T-8-10-190	40.7	1000	190	484-620	552

TABLE XXII (continued)

Specimen Designation	Composition (mole % ThC ₂)	Annealing Treatment ^a		Knoop Hardness	
		Temperature (°C)	Time (hr)	Range (KHN)	Average (KHN)
T-8-10-453	40.7	1000	453	262-429	374
T-8-10-950	40.7	1000	950	241-578	394
T-8-12-446	40.7	1200	446	536-670	611
T-8-15-445	40.7	1500	445	532-690	600
T-8-19-350	40.7	1900	350	377-703	569
T-8-19-1190	40.7	1900	1190	696-898	792
T-3	35.3	As cast		429-488	461
T-3-10-190	35.3	1000	150	435-583	528
T-3-10-743	35.3	1000	743	386-506	450
T-3-10-950	35.3	1000	950	358-541	455
T-3-12-150	35.3	1200	150	435-583	528
T-3-12-450	35.3	1200	450	536-612	608
T-3-12-740	35.3	1200	740	381-558	465
T-3-12-907	35.3	1200	907	465-625	518
T-3-12-1243	35.3	1200	1243	484-631	582
T-3-15-150	35.3	1500	150	484-675	569
T-3-15-925	35.3	1500	925	455-625	529
T-3-15-1165	35.3	1500	1165	532-703	646
T-3-19-150	35.3	1900	150	598-742	664
T-3-19-500	35.3	1900	500	452-722	588
T-3-19-990	35.3	1900	990	652-795	745
T-3-19-1350	35.3	1900	1350	625-766	706
T-2	35.0	As cast		481-547	498
T-2-11-150	35.0	1100	150	429-549	488
T-2-11-465	35.0	1100	465	506-696	595
T-2-11-750	35.0	1100	750	435-569	538

TABLE XXII (continued)

Specimen Designation	Composition (mole % ThC ₂)	Annealing Treatment ^a		Knoop Hardness	
		Temperature (°C)	Time (hr)	Range (KHN)	Average (KHN)
T-2-11-1200	35.0	1100	1200	405-607	540
T-2-13-150	35.0	1300	150	386-696	538
T-2-13-221	35.0	1300	221	536-612	573
T-2-19-500	35.0	1900	500	735-554	653
T-2-19-990	35.0	1900	990	652-755	726
T-2-19-1350	35.0	1900	1350	690-728	708
T-5	35.0	As cast		484-675	545
T-5-10-190	35.0	1000	190	351-536	471
T-5-10-453	35.0	1000	453	417-528	470
T-5-10-743	35.0	1000	743	558-641	591
T-5-10-950	35.0	1000	950	323-658	502
T-5-12-150	35.0	1200	150	583-722	673
T-5-12-450	35.0	1200	450	602-728	671
T-5-12-744	35.0	1200	744	452-598	542
T-5-12-907	35.0	1200	907	359-492	435
T-5-12-1243	35.0	1200	1243	469-553	510
T-5-15-150	35.0	1500	150	612-684	645
T-5-15-425	35.0	1500	425	365-696	539
T-5-15-665	35.0	1500	665	545-612	571
T-5-15-925	35.0	1500	925	414-675	541
T-5-15-1165	35.0	1500	1165	545-670	625
T-5-19-150	35.0	1900	150	722-818	791
T-5-19-500	35.0	1900	500	488-696	550
T-5-19-667	35.0	1900	667	773-810	795
T-5-19-990	35.0	1900	990	620-748	708
T-5-19-1350	35.0	1900	1350	438-742	616

TABLE XXII (continued)

Specimen Designation	Composition (mole % ThC ₂)	Annealing Treatment ^a		Knoop Hardness	
		Temperature (°C)	Time (hr)	Range (KHN)	Average (KHN)
T-5-20-118	35.0	2000	118	495-826	676
T-10	29.7	As cast		389-528	470
T-10-10-190	29.7	1000	190	220-602	407
T-10-10-453	29.7	1000	453	348-652	479
T-10-10-950	29.7	1000	950	459-528	503
T-10-12-446	29.7	1200	446	455-631	566
T-10-15-445	29.7	1500	445	583-675	603
T-10-19-350	29.7	1900	350	540-755	607
T-10-19-1190	29.7	1900	1190	625-728	689
T-9	29.3	As cast		472-558	512
T-9-10-150	29.3	1000	150	459-595	539
T-9-10-190	29.3	1000	190	377-636	515
T-9-10-453	29.3	1000	453	238-549	398
T-9-12-446	29.3	1200	446	488-690	593
T-9-15-445	29.3	1500	445	449-690	594
T-9-19-350	29.3	1900	350	449-780	639
T-9-19-1190	29.3	1900	1190	625-863	720
T-9-20-118	29.3	2000	118	549-722	664
T-23	b	As cast		429-589	520

^aAll specimens were cooled at an average rate of between 5 and 140°C per minute.

^b100 per cent UC₂.

APPENDIX F

THERMAL EXPANSION DATA FOR $(\text{U,Th})\text{C}_2$ ALLOYS

TABLE XXIII
MEAN TEMPERATURE COEFFICIENT OF EXPANSION FOR ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Specimen Designation	Mean Temperature Coefficient of Expansion ^a ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) for the Given Temperature Range ^b						
		40 to 400°C	40 to 500°C	40 to 600°C	40 to 700°C	40 to 800°C	40 to 900°C	40 to 1000°C
100	M-2	7.33	7.56	8.15	8.26	8.35	8.52	8.70
100	T-22A	7.08	7.42	7.52	7.60	7.69	7.85	8.03
100	T-22B	7.38	7.46	7.52	7.61	7.70	7.97	8.43
87.7	M-3	8.49	8.35	8.31	8.31	8.49	8.66	8.92
75.1	M-4	8.59	8.73	8.94	9.17	9.34	9.57	9.99
62.3	T-21A	9.00	9.24	9.60	10.01	10.42	10.82	11.21
62.3	T-21B	9.11	9.14	9.58	10.04	10.55	11.02	12.96
54.4	T-24B	9.22	9.57	10.12	10.62	10.57	11.55	12.05
51.1	T-20A	9.06	9.47	10.00	10.47	10.87	11.30	11.94
45.5	T-12A	9.47	9.89	10.50	11.05	11.58	12.07	12.52
45.5	T-12B	8.86	9.34	9.91	10.46	11.06	11.63	12.23
45.5	T-12A-20-118	9.47	9.72	10.03	10.48	10.89	11.24	11.48
45.2	T-11A	9.81	9.80	10.12	10.72	11.22	11.54	11.97
45.2	T-11B	9.38	9.84	10.40	10.95	11.47	11.97	12.44
43.2	T-18A	9.53	9.99	10.48	10.96	11.49	12.06	12.44
43.2	T-18B	9.06	9.60	10.24	10.92	11.58	12.24	12.70
41.7	T-7A	10.01	10.51	10.99	11.43	11.87	12.25	12.76
41.7	T-7B	9.86	10.24	10.72	11.19	11.56	11.98	12.39
41.7	T-7A-17-114	10.07	10.32	10.72	11.08	11.47	11.74	12.07
41.4	T-6A	9.22	9.62	10.19	10.71	11.24	11.76	12.28
41.4	T-6B	9.97	10.51	11.01	11.35	11.66	11.95	12.26
40.7	T-8A	9.69	10.11	10.61	11.02	11.42	11.84	12.15
40.7	T-8B	9.82	10.28	10.73	11.32	11.86	12.26	12.61
40.1	T-16A	9.93	10.25	10.78	11.32	11.72	12.40	12.95
40.1	T-16B	9.58	10.04	10.59	11.12	11.58	12.02	12.25
35.0	T-2B	9.88	10.60	11.16	11.56	11.99	12.36	13.01
35.0	T-2B-17-114	10.42	10.66	11.06	11.43	11.77	12.07	12.33
37.6	T-14B	9.41	9.80	10.44	10.99	11.49	12.04	12.56

TABLE XXIII (continued)

Composition (mole % ThC ₂)	Specimen Designation	Mean Temperature Coefficient of Expansion ^a ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) for the Given Temperature Range ^b						
		40 to 400°C	40 to 500°C	40 to 600°C	40 to 700°C	40 to 800°C	40 to 900°C	40 to 1000°C
36.6	T-15A	9.71	10.11	10.56	11.05	11.40	11.87	12.36
36.6	T-15B	9.51	9.75	10.24	10.98	11.31	11.56	12.11
35.0	T-5	9.82	10.13	10.70	11.22	11.69	12.21	12.88
35.0	T-5-20-118	11.16	10.92	10.91	11.25	11.57	11.85	12.09
34.0	T-17A	10.16	10.52	10.95	11.38	11.87	12.37	12.73
34.0	T-17B	10.39	10.64	11.17	11.66	12.14	12.66	13.23
32.6	T-19B	9.45	10.39	10.91	11.40	11.85	12.42	12.59
29.7	T-10A	10.26	10.79	11.19	11.53	11.99	12.39	12.77
29.7	T-10B	11.29	11.77	12.07	12.89	13.32	13.12	13.00
29.7	T-10A-17-114	10.54	10.69	11.14	11.55	11.94	12.32	12.64
29.3	T-9	10.41	10.86	11.45	11.90	12.38	12.80	13.25
29.3	T-9-20-118	11.25	11.28	11.55	11.85	12.24	12.46	12.69
25.4	M-6	11.79	11.98	12.27	12.41	12.56	12.90	13.23
c	T-23B	12.66	12.98	12.88	12.32	12.63	13.04	13.51

^a Each of the values reported is the average of at least three determinations on the same specimen.

^b The temperature of the specimen at the start of each run, ranged from 30 to 50°C. Actual temperatures were used in the calculations.

^c 100 per cent UC₂.

TABLE XXIV

INSTANTANEOUS TEMPERATURE COEFFICIENT OF EXPANSION FOR ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Specimen Designation	Instantaneous Coefficient of Expansion ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) at the Given Temperatures ^a						
		400 °C	500 °C	600 °C	700 °C	800 °C	900 °C	1000 °C
100	M-2	8.37	8.71	9.05	9.39	9.73	10.07	10.24
100	T-22A	7.74	7.98	8.23	8.40	8.73	8.97	9.22
100	T-22B	7.31	7.47	7.88	8.55	9.47	10.64	12.07
87.7	M-3	8.17	8.20	8.46	8.94	9.64	10.57	11.72
75.1	M-4	9.16	9.51	9.94	10.44	11.02	11.68	12.13
62.3	T-21A	9.98	10.79	11.61	12.42	13.23	14.05	14.86
62.3	T-21B	10.03	10.92	11.80	12.69	13.58	14.47	15.36
54.4	T-24B	10.69	11.69	12.69	13.68	14.68	15.68	16.68
51.1	T-20A	10.53	11.46	12.40	13.34	14.28	15.21	16.15
45.5	T-12A	11.23	12.24	13.26	14.27	15.28	16.29	17.30
45.5	T-12B	10.37	11.59	12.81	14.03	15.25	16.47	17.09
45.5	T-12A-20-118	10.60	11.32	12.03	12.75	13.46	14.18	14.90
45.2	T-11A	10.81	11.66	12.51	13.36	14.21	15.06	15.91
45.2	T-11B	11.08	12.12	13.16	14.21	15.25	16.29	17.33
43.2	T-18A	11.24	12.21	13.18	14.15	15.12	16.09	17.07
43.2	T-18B	11.23	12.37	13.52	14.67	15.82	16.97	18.11
41.7	T-7A	11.83	12.61	13.39	14.18	14.96	15.74	16.80
41.7	T-7B	11.45	12.24	13.04	13.84	14.64	15.44	16.32
41.7	T-7A-17-114	11.20	11.87	12.54	13.22	13.89	14.56	15.24
41.4	T-6A	11.03	12.03	13.02	14.02	15.01	16.0	17.46
41.4	T-6B	11.60	12.23	12.87	13.50	14.13	14.77	15.40
40.7	T-8A	11.24	12.06	12.88	13.70	14.52	15.34	15.87
40.7	T-8B	11.60	12.51	13.41	14.32	15.22	16.13	17.03
40.1	T-16A	10.88	12.03	13.18	14.33	15.47	16.62	17.77
40.1	T-16B	11.33	12.28	13.22	14.17	15.11	16.06	16.39
35.0	T-2B	12.04	12.82	13.61	14.39	15.18	15.96	16.8
35.0	T-2B-17-114	11.78	12.37	12.95	13.54	14.12	14.71	15.27
37.6	T-14B	11.02	12.13	13.24	14.35	15.46	16.57	17.67

TABLE XXIV (continued)

Composition (mole % ThC ₂)	Specimen Designation	Instantaneous Coefficient of Expansion ($\times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) at the Given Temperatures ^a						
		400°C	500°C	600°C	700°C	800°C	900°C	1000°C
36.6	T-15A	11.22	12.08	12.95	13.81	14.67	15.53	16.40
36.6	T-15B	11.02	11.87	12.73	13.59	14.44	15.30	16.32
35.0	T-5	11.44	12.41	13.38	14.35	15.32	16.29	17.26
35.0	T-5-20-118	11.26	11.83	12.41	12.98	13.56	14.13	14.71
34.0	T-17A	11.51	12.44	13.37	14.29	15.22	16.15	17.08
34.0	T-17B	11.39	12.45	13.51	14.56	15.62	16.68	17.74
32.6	T-19B	11.54	12.60	13.64	14.69	15.74	16.79	17.84
29.7	T-10A	11.91	12.67	13.44	14.20	14.97	15.73	16.49
29.7	T-10B	13.67	13.67	13.67	13.67	13.67	13.67	13.67
29.7	T-10A-17-114	11.73	12.48	13.22	13.96	14.70	15.44	16.19
29.3	T-9	12.34	13.18	14.03	14.88	15.72	16.57	17.41
29.3	T-9-20-118	11.82	12.40	12.99	13.58	14.16	14.75	15.34
25.4	M-6	12.75	13.06	13.36	13.66	13.96	14.27	14.5
b	T-23B	13.49	13.49	13.49	13.49	13.49	13.49	13.49

^aDetermined from the equations obtained from the data in Table

^b100 per cent UC₂.

TABLE XXV
CUMULATIVE EXPANSION FOR ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Specimen Designation	Total Cumulative Expansion ^a ($\times 10^{-3}$ in./in.) for the Given Temperature Range ^b						
		40 to 400°C	40 to 500°C	40 to 600°C	40 to 700°C	40 to 800°C	40 to 900°C	40 to 1000°C
100	M-2	2.58	3.42	4.51	5.39	6.28	7.27	8.42
100	T-22A	2.57	3.37	4.17	5.00	5.80	6.71	7.66
100	T-22B	2.53	3.31	4.09	4.90	5.72	6.74	7.95
87.7	M-3	2.92	3.70	4.55	5.40	6.37	7.36	8.47
75.1	M-4	2.98	3.90	4.89	5.94	6.98	8.11	9.31
62.3	T-21A	2.87	3.87	4.99	6.20	7.51	8.87	10.29
62.3	T-21B	3.07	4.01	5.15	6.41	7.79	9.22	10.57
54.4	T-24B	3.24	4.31	5.58	6.90	8.29	9.81	11.52
51.1	T-20A	3.06	4.14	5.41	6.70	8.01	9.46	11.19
45.5	T-12A	3.26	4.42	5.74	7.13	8.62	10.19	11.81
45.5	T-12B	3.04	4.14	5.38	6.72	8.20	9.79	11.64
45.5	T-12A-20-118	3.21	4.26	5.42	6.70	8.05	9.43	10.77
45.2	T-11A	3.50	4.48	5.62	7.06	8.50	8.88	11.47
45.2	T-11B	3.31	4.46	5.76	7.15	8.63	10.21	11.85
43.2	T-18A	3.41	4.55	5.78	7.13	8.64	10.26	11.81
43.2	T-18B	3.09	4.21	5.55	6.96	8.55	10.25	11.90
41.7	T-7A	3.54	4.77	6.09	7.47	8.94	10.46	12.12
41.7	T-7B	3.47	4.62	5.92	7.29	8.69	10.20	11.81
41.7	T-7A-17-114	3.43	4.55	5.80	7.1	8.49	9.86	11.35
41.4	T-6A	3.23	4.34	5.61	6.97	8.44	10.0	11.85
41.4	T-6B	3.48	4.72	6.04	7.37	8.73	10.15	11.63
40.7	T-8A	3.40	4.56	5.85	7.18	8.58	10.08	11.52
40.7	T-8B	3.44	4.62	5.90	7.36	8.89	10.42	11.98
40.1	T-16A	2.86	4.01	5.29	6.69	8.08	10.11	11.0
40.1	T-16B	3.42	4.60	4.90	7.30	8.76	10.29	11.68
37.6	T-14B	3.25	4.37	5.70	7.09	8.56	10.18	11.86
35.0	T-2B	3.5	4.82	6.18	7.56	9.04	10.56	12.34
35.0	T-2B-17-114	3.6	4.74	6.04	7.37	8.77	10.19	11.72
36.6	T-15A	3.37	4.50	5.77	7.13	8.50	10.03	11.68

TABLE XXV (continued)

Composition (mole % ThC ₂)	Specimen Designation	Total Cumulative Expansion ^a ($\times 10^{-3}$ in./in.) for the Given Temperature Range ^b						
		40 to 400°C	40 to 500°C	40 to 600°C	40 to 700°C	40 to 800°C	40 to 900°C	40 to 1000°C
36.6	T-15B	3.36	4.39	5.66	7.16	8.50	9.84	11.33
35.0	T-5	3.48	4.60	4.93	7.34	8.82	10.43	12.30
35.0	T-5-20-118	3.67	4.78	5.95	7.27	8.62	10.00	11.42
34.0	T-17A	3.49	4.67	6.63	7.32	8.82	10.44	12.00
34.0	T-17B	2.95	4.12	5.44	6.84	8.32	9.94	11.71
32.6	T-19B	3.19	4.58	5.89	7.29	8.75	10.41	11.75
29.7	T-10A	3.53	4.78	6.08	7.41	8.90	10.44	12.03
29.7	T-10B	3.99	5.33	6.68	8.43	10.03	11.17	12.38
29.7	T-10A-17-114	3.76	4.89	6.21	7.58	9.03	10.55	12.08
29.3	T-9	3.64	4.89	6.30	7.73	9.28	10.88	12.58
29.3	T-9-20-118	3.60	4.77	6.04	7.38	8.84	10.24	11.69
25.4	M-6	4.12	5.39	6.71	8.03	9.39	10.94	12.49
c	T-23B	4.33	5.71	6.95	7.89	9.37	10.96	12.71

^aEach of the values reported is the average of at least three determinations on the same specimen.

^bAt the start of each run, specimen temperatures ranged from 30 to 50°C. Actual temperatures were used in the calculations.

^c100 per cent UC₂.

TABLE XXVI

EQUATION CONSTANTS FOR THERMAL EXPANSION AS A FUNCTION OF
TEMPERATURE FOR ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Specimen Designation	Equation Constants ^a			
		A	B	C	D
		$\times 10^{-4}$	$\times 10^{-6}$	$\times 10^{-9}$	$\times 10^{-12}$
100	M-2	-4.1	7.0	1.7	
100	T-22A	-3.4	6.7	1.2	
100	T-22B	-6.2	9.2	-4.9	4.2
87.7	M-3	-6.6	10.3	-4.9	3.7
75.1	M-4	-5.0	8.5	0.05	1.3
62.3	T-21A	-4.9	6.7	4.1	
62.3	T-21B	-2.7	6.5	4.4	
54.4	T-24B	-2.5	6.7	5.0	
51.1	T-20A	-3.8	6.8	4.7	
45.5	T-12A	-3.9	7.2	5.1	
45.5	T-12B	-1.0	5.5	6.1	
45.5	T-12A-20-118	-4.7	7.7	3.6	
45.2	T-11A	-2.1	7.4	4.2	
45.2	T-11B	-2.5	6.9	5.2	
43.2	T-18A	-3.5	7.4	4.9	
43.2	T-18B	-4.8	6.6	5.7	
41.7	T-7A	-5.4	8.7	3.9	
41.7	T-7B	-4.6	8.3	4.0	
41.7	T-7A-17-114	-5.1	8.5	3.4	
41.4	T-6A	-3.9	7.1	5.0	
41.4	T-6B	-5.7	9.1	3.2	
40.7	T-8A	-4.0	7.9	4.1	
40.7	T-8B	-4.6	8.0	4.5	
40.1	T-16A	-7.4	6.31	5.7	
40.1	T-16B	-3.3	7.6	4.7	
35.0	T-2B	-6.1	8.9	3.9	
35.0	T-2B-17-114	-6.7	9.4	2.9	
37.6	T-14B	-2.6	9.6	5.5	
36.6	T-15A	-4.5	7.8	4.3	
36.6	T-15B	-3.8	7.4	4.7	
35.0	T-5	-3.4	7.6	4.9	
35.0	T-5-20-118	-5.0	9.0	2.9	
34.0	T-17A	-3.8	7.8	4.6	
34.0	T-17B	-7.6	7.2	5.3	

TABLE XXVI (continued)

Composition (mole % ThC ₂)	Specimen Designation	Equation Constants ^a			
		A	B	C	D
		$\times 10^{-4}$	$\times 10^{-6}$	$\times 10^{-9}$	$\times 10^{-12}$
32.6	T-19B	-4.5	7.3	5.3	
29.7	T-10A	-6.3	8.9	3.8	
29.7	T-10B	-12.3	13.7		
29.7	T-10A-17-114	-3.7	8.8	3.7	
29.3	T-9	-6.1	9.0	4.2	
29.3	T-9-20-118	6.7	9.5	2.9	
25.4	M-6	-7.5	11.5	1.5	
b	T-23B	-11.9	13.5		

^aBy the method of least squares, the data for the total cumulative expansion, expressed as a function of temperature, were fitted to a first-, second-, or third-order polynomial as required. The form of the equation is:

$$\text{Expansion (in./in.)} = A + BT + CT^2 + DT^3,$$

where T is expressed in °C.

^b100 per cent UC₂.

TABLE XXVII

EQUATION CONSTANTS FOR THERMAL EXPANSION AS A FUNCTION
OF TEMPERATURE ABOVE THE DATUM LEVEL FOR
ARC-MELTED (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Specimen Designation	Equation Constants ^a			
		A	B	C	D
		$\times 10^{-4}$	$\times 10^{-6}$	$\times 10^{-9}$	$\times 10^{-12}$
100	M-2	-0.5	7.2	1.7	
100	T-22A	-0.01	6.9	1.2	
100	T-22B	-1.7	8.7	-4.3	4.2
87.7	M-3	-1.6	9.8	-4.3	3.7
75.1	M-4	-7.8	8.5	0.2	1.3
62.3	T-21A	-1.5	7.1	4.1	
62.3	T-21B	0.7	6.9	4.4	
54.4	T-24B	1.0	7.2	5.0	
51.1	T-20A	-0.3	7.2	4.7	
45.5	T-12A	-0.2	7.7	5.1	
45.5	T-12B	1.9	6.1	6.1	
45.5	T-12A-20-118	-0.8	8.1	3.6	
45.2	T-11A	1.7	7.8	4.2	
45.2	T-11B	1.1	7.4	5.2	
43.2	T-18A	0.3	7.8	4.9	
43.2	T-18B	-1.3	7.2	5.7	
41.7	T-7A	-0.9	9.1	3.9	
41.7	T-7B	-0.3	8.7	4.0	
41.7	T-7A-17-114	-7.2	8.8	3.4	
41.4	T-6A	-2.2	7.5	5.0	
41.4	T-6B	-1.1	9.4	3.2	
40.7	T-8A	0.05	8.4	4.1	
40.7	T-8B	0.5	8.4	4.5	
40.1	T-16A	-4.0	6.9	5.7	
40.1	T-16B	0.6	8.0	4.7	
35.0	T-2B	-1.5	9.3	3.9	
35.0	T-2B-17-114	-1.9	9.7	2.9	
37.6	T-14B	0.9	7.1	5.5	
36.6	T-15A	-0.5	8.2	4.3	
36.6	T-15B	0.1	8.0	4.3	
35.0	T-5	0.5	8.0	4.9	
35.0	T-5-20-118	0.5	9.2	2.9	
34.0	T-17A	0.2	8.3	4.6	

TABLE XXVII (continued)

Composition (mole % ThC ₂)	Specimen Designation	Equation Constants ^a			
		A	B	C	D
		$\times 10^{-4}$	$\times 10^{-6}$	$\times 10^{-9}$	$\times 10^{-12}$
34.0	T-17B	-3.9	7.7	5.3	
32.6	T-19B	-0.7	7.9	5.3	
29.7	T-10A	-1.8	9.2	3.8	
29.7	T-10B	-5.5	13.7		
29.7	T-10A-17-114	-0.08	9.1	3.7	
29.3	T-9	-1.5	9.4	4.2	
29.3	T-9-20-118	-1.9	9.8	2.9	
25.4	M-6	-17.6	11.7	1.5	
b	T-23B	-5.1	1.3		

^aBy the method of least squares, the data for the total cumulative expansion, expressed as a function of temperature above the initial temperature, were fitted to a first-, second-, and third-order polynomial as required. The form of the equation is:

$$\text{Expansion (in./in.)} = A + B(T - T_I) + C(T - T_I)^2 + D(T - T_I)^3 ,$$

where T is the temperature for any expansion and T_I is the initial temperature.

^b100 per cent UC₂.

APPENDIX G

TEMPERATURES FOR SOLID-STATE TRANSFORMATIONS DETERMINED
BY DILATOMETRY ON (U,Th) C_2 ALLOYS

TABLE XXVIII
TEMPERATURES FOR SOLID-STATE TRANSFORMATIONS DETERMINED BY DILATOMETRY ON (U,Th)C₂ ALLOYS

Composition (mole % ThC ₂)	Sample Designation	Heating				Cooling				Temperature Difference between Heating and Cooling	
		Rate (°C/min)	Lower Temperature, ^a T _{LH} (°C)	Upper Temperature, ^b T _{UH} (°C)	T _{UH} - T _{LH} (°C)	Rate (°C/min)	Upper Temperature, ^c T _{UC} (°C)	Lower Temperature, ^d T _{UL} (°C)	T _{UC} - T _{UL} (°C)	T _{LH} - T _{LC} (°C)	T _{UH} - T _{UC} (°C)
62.3	T-21A	3.5	1254	1282	28	2.0	1204	1182	22	72	78
54.4	T-24B	1.5	1234	1274	40	1.5	1201	1183	18	51	73
		3.7	1183	1217	34	2.4	1176	1135	41	48	41
51.1	T-20A	1.4	1209	1230	21	1.3	1193	1180	13	29	37
		3.0	1179	1217	38	2.4	1047	1007	40	172	172
45.5	T-12A	1.3	1178	1214	36	1.5	1047	995	52	183	167
		4.1	1169	1196	27	2.3	1002	978	24	191	194
		2.1	1174	1209	35	6.4	1018	996	22	178	191
		1.8	1166	1190	24	2.1	996	982	14	184	194
		1.3	1167	1190	23	1.4	998	975	23	192	192
45.5	T-12A-20-118	0.4	1165	1189	24	0.4	983	962	21	203	206
		4.1	1176	1206	30	-	-	-	-	-	-
		2.8	1174	1194	20	-	-	-	-	-	-
45.2	T-11B	3.5	1189	1218	29	-	-	-	-	-	-
		1.7	1178	1202	24	-	-	-	-	-	-
41.7	T-7A	1.8	1190	1208	18	1.4	1058	1041	17	149	150
		1.1	1188	1201	13	0.6	1056	1036	20	152	145
		0.6	1183	1196	13	0.7	1051	1035	16	150	153
41.7	T-7A-17-114	3.3	1175	1194	19	2.5	1056	1026	30	149	138
		1.5	1174	1192	18	1.6	1054	1034	20	140	138
41.4	T-6B	3.3	1190	1214	24	3.6	1060	1041	19	149	154
		2.7	1192	1210	18	2.3	1063	1046	17	146	147
		2.1	1193	1212	19	1.6	1060	1044	16	149	152
		0.6	1190	1210	20	0.7	1060	1039	21	151	150
40.1	T-16B	3.6	1173	1223	50	-	-	-	-	-	-
		3.5	1188	1225	37	2.3	1083	1055	28	133	142
		1.5	1181	1212	31	1.2	1082	1056	26	125	130
		1.5	1162	1204	42	1.0	1073	1052	21	110	131

TABLE XXVIII (continued)

Composition	Sample Designation	Heating				Cooling				Temperature Difference between Heating and Cooling	
		Rate	Lower	Upper	$T_{UH} - T_{LH}$	Rate	Upper	Lower	$T_{UC} - T_{LC}$	$T_{LH} - T_{LC}$	$T_{UH} - T_{UC}$
			T_{LH}	T_{UH}			T_{UC}	T_{UL}			
(mole % ThC ₂)		(°C/min)	(°C)	(°C)	(°C)	(°C/min)	(°C)	(°C)	(°C)	(°C)	(°C)
37.6	T-14B	3.4	1241	1259	18	2.3	1136	1127	9	114	123
		1.5	1233	1243	10	1.4	1133	1122	11	111	110
35.0	T-5	6.5	1242	1266	24	3.2	1171	1151	20	91	95
		2.1	1250	1273	23	1.7	1177	1153	24	97	120
		2.0	1242	1281	39	1.8	1174	1151	23	91	107
		1.9	1249	1272	23	1.4	1177	1155	22	94	95
		1.9	1245	1271	26	1.6	1189	1163	26	82	82
		0.6	1228	1266	38	0.6	1172	1150	22	78	94
35.0	T-5-20-118	0.3	1241	1264	23	0.3	1168	1139	29	102	96
		3.3	1251	1267	16	2.3	1177	1162	15	89	90
		1.4	1253	1270	17	1.3	1174	1161	13	92	96
		1.6	1245	1263	18	1.4	1180	1165	15	80	83
35.0	T-2A	1.6	1248	1270	22	1.3	1181	1171	10	77	89
		4.8	1246	1280	34	2.6	1189	1165	24	81	91
35.0	T-2B	4.3	1242	1273	31	3.3	1183	1165	18	77	90
		1.9	1250	1274	24	1.7	1186	1163	23	87	88
		0.3	1254	1267	13	0.4	1187	1161	26	93	80
		3.3	1244	1271	27	2.7	1188	1130	58	114	83
35.0	T-2B-17-114	2.0	1240	1273	33	1.6	1189	1138	51	102	84
		0.3	1235	1276	41	-	-	-	-	-	-
		3.2	1271	1287	16	2.1	1199	1180	19	91	88
34.0	T-17B	1.7	1270	1285	15	1.5	1200	1183	17	87	85
		3.2	1281	1300	19	2.1	1221	1200	21	81	79
32.6	T-19B	1.5	1274	1296	22	1.2	1219	1200	19	74	77
		3.2	1336	1359	23	2.0	1280	1252	28	84	79
29.7	T-10A	2.4	1320	1349	29	1.8	1279	1245	25	75	70
		0.8	1334	1359	25	0.8	1278	1250	28	84	81
		0.4	1330	1356	26	0.5	1285	1256	29	74	71
0.4		0.4	1317	1356	39	0.4	1280	1261	19	56	76
		0.3	1319	1335	16	0.4	1281	1261	20	58	54

TABLE XXVIII (continued)

Composition	Sample Designation	Heating				Cooling				Temperature Difference between Heating and Cooling	
		Rate	Lower Temperature, ^a	Upper Temperature, ^b	$T_{UH} - T_{LH}$	Rate	Upper Temperature, ^c	Lower Temperature, ^d	$T_{UC} - T_{LC}$		
			T_{LH}	T_{UH}			T_{UC}	T_{UL}		$T_{LH} - T_{LC}$	$T_{UH} - T_{UC}$
(mole % ThC ₂)		(°C/min)	(°C)	(°C)	(°C)	(°C/min)	(°C)	(°C)	(°C)	(°C)	(°C)
29.7	T-10A-17-114	5.0	1308	1342	34	3.5	1273	1244	29	64	69
		2.2	1298	1334	36	1.6	1276	1250	26	48	40
		1.1	1308	1344	36	1.1	1276	29	29	61	68
		0.3	1300	1342	42	0.4	1280	1250	30	50	62
29.3	T-9	2.9	1308	1344	36	3.0	1272	1245	27	63	72
		2.8	1303	1337	34	1.6	1267	1247	20	56	70
		1.6	1307	1341	34	1.2	1268	1248	20	59	73
		0.5	1316	1340	24	0.6	1268	1251	17	65	72
29.3	T-9-20-118	4.0	1326	1343	17	1.9	1281	1261	20	65	62
		1.3	1327	1344	17	1.2	1280	1265	15	62	64
25.4	M-6	2.8	1391	1417	26	1.8	1338	1315	23	76	79
		1.4	1390	1410	20	1.3	1348	1327	21	63	62

^aThe lower transformation temperature observed on heating, schematically shown in Figure 38.

^bThe upper transformation temperature observed on heating, schematically shown in Figure 38.

^cThe upper transformation temperature observed on cooling, schematically shown in Figure 38.

^dThe lower transformation temperature observed on cooling, schematically shown in Figure 38.

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