

Carbon – Thorium – Uranium

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Introduction

Thorium carbide is one of the most favorable chemical forms of fuel for nuclear reactors because of its high thermal conductivity and high thorium density, while the mixture with uranium carbide is considered to be a practical chemical form for use in a reactor [1987Nam]. Therefore, the study of the phase relations in the corresponding ternary system C–Th–U is of great interest. Information about phase equilibria in this system is presented in the literature via a quasibinary section ThC–UC [1982Ogo], isothermal sections at 1800°C [1975Hol, 1984Hol], 1700°C [1961Ben], 1330°C [1984Hol] and 1000°C [1961Ben, 1984Hol] as well as by a temperature-composition section for ThC₂–UC₂ [1966Hen]. Phase content of the alloys and crystal structures of the intermediate phases were studied by [1957Now, 1958Cir, 1958Iva, 1958Lau, 1959Now, 1960Bre, 1961Ben, 1961Iva1, 1961Iva2, 1962Kat, 1966Hen, 1966Lan, 1971Pet, 1987Jon]. Data on thermodynamic properties were obtained experimentally by [1962Kat, 1989Koy, 1991Yam]. Electrical properties of the thorium carbide–uranium carbide solid solutions were reported by [1969Aus]. The morphology of the C–Th–U alloys was studied by [1964Keg]. [1965Kel, 1965Pet, 1965Wym] studied aspects of the application of sol–gel processing to the production of carbon–thorium–uranium particles. The synthesis of thorium–uranium carbides using the method of carbothermic reduction was carried out by [1987Nam]. Experimental methods used in the above works are presented in Table 1. Short assessments of the literature information related to the C–Th–U system were published by [1966Bar, 1975Hol, 1984Hol]. However, knowledge of the phase equilibria in the C–Th–U system is incomplete. In particular, the conditions of the invariant four-phase equilibria need to be established. Information about the constitution of the liquidus, solidus and solvus surfaces is lacking. Future investigations of phase relations in the C–Th–U system should be concentrated on the continuation of studies of alloy properties in the equilibrium state at different temperatures.

Binary Systems

Data related to the boundary C–Th and Th–U systems are accepted from [Mas2]. The constitution of the C–U system is accepted from [2001Che], who presents essentially the same phase diagram as [Mas2].

Solid Phases

Crystallographic data relating to the known unary and binary phases are listed in Table 2. No ternary phases having crystal structures different from those inherent in the unary and binary phases have been found. At high temperatures in the C–Th boundary binary system, a continuous series of solid solutions between (α Th), the δ ThC phase and the γ ThC₂ phase are found. All of these phases possess a cubic structure but of different space groups and prototypes. In the ternary system, a continuous series of solid solutions exists between these phases and the normal pressure modification of the δ UC and δ UC₂ phase (labeled as λ).

Quasibinary Systems

The crystal structure of the alloys along the ThC–UC section have been studied by [1957Now, 1958Cir, 1958Lau, 1959Now, 1961Iva1, 1961Iva2] revealing the presence of a continuous series of solid solutions between the ThC and UC phases. The interaction parameters and the energies of mixing in the quasibinary ThC–UC system have been calculated by [1982Ogo] from the atomization energies, melting points and lattice parameters. From the energies of mixing thus obtained, the liquidus, solidus and solid solution decomposition lines have been calculated applying the regular solution approximation. The formation of a continuous series of solid solutions is confirmed. It was calculated that the liquidus and solidus curves in the quasibinary section possess a minimum at 2195°C and 26.19 at.% U, and decomposition of the λ solid solution takes place at temperatures lower than 1593°C. This section is presented in Fig. 1. Some further

experimental studies are needed because the melting temperature accepted by [1982Ogo] is higher than that accepted by [Mas2] (2670°C and 2500°C, respectively).

Isothermal Sections

Several isothermal sections have been determined over the temperature range 1800–1000°C [1961Ben, 1975Hol, 1984Hol]. The isothermal section reported at 1800°C is based on experimental results obtained by [1966Hen] and [1971Pet]. In order to maintain consistency with the accepted binary systems, the isothermal sections at 1800°C (Fig. 2), 1700°C (Fig. 3), 1330°C (Fig. 4) and 1000°C (Fig. 5) were redrawn. The presence of a miscibility gap in the λ solid solution has been also taken into account at 1330 and 1000°C along the quasibinary system ThC–UC. The corresponding phase equilibria associated with this miscibility gap along the binary edge have been presented as dashed lines. The partial isothermal section for 1000°C proposed by [1961Ben] for the carbon-poor region was not retained in this assessment since the C–Th binary system proposed by [1961Ben] does not reflect the formation of a continuous series of solid solutions between the (α Th) and the δ ThC phases. All the presented isothermal sections are in need of further experimental verification.

Temperature – Composition Sections

The results of X-ray diffraction and microstructural studies of the alloys located along the Th–UC and ThC₂–UC₂ sections are presented in [1958Iva, 1961Iva1] and [1960Bre, 1966Lan], respectively. The reaction of thorium with the UC and UC₂ compounds at 1000°C were studied by [1962Kat] using the diffusion couple method. The formation of the λ phase and uranium was noted. The temperature-composition section constructed as a result of an investigation of alloys with about 66.67 at.% C in the presence of about 1 mass% free carbon was reported by [1966Hen]. The excess carbon was added to reduce the presence of oxide impurities in the uranium and thorium powder starting materials. This section needs further experimental study, in particular, at the UC₂ side because at the temperatures below ~1500°C, the UC₂ phase does not exist in the C–U boundary binary system, according to the accepted data of [2001Che].

Thermodynamics

Calculations of the Gibbs energy of formation ΔG of the λ phase in three-phase equilibria involving the participation of metal-rich phases were carried out by [1961Ben]. Good agreement with the literature was noted. [1962Kat] carried out a comparison between the observed reactivity of the UC–Th and UC₂–Th systems and thermodynamic predictions based on simple displacement reactions whose products were uranium and a metal carbide. Thermodynamic predictions were based on the corresponding standard state reactions and values of free energies of formation of the carbides. According to the results, both phases must react with thorium, as is observed experimentally. Vapor pressures over the λ phase were measured by [1989Koy, 1991Yam] using Knudsen effusion mass spectrometry at temperatures ranging from about 2000 to 2200 K (1727 to 1927°C) (Table 3). The activities of thorium and carbon in the ThC_{1±x} phase were derived from the measured vapor pressures of thorium. Based on the vapor pressure measurement over the λ phase, the Gibbs energy of formation of the phase was derived as a function of uranium fraction. The values obtained are plotted in Fig. 6 as white circles. The $\Delta_f G^\circ$ of the λ phase decreases continuously with decreasing uranium content and was smoothly extrapolated to the values of $\Delta_f G^\circ$ of the δ phase previously obtained by M. Yamawaki *et al.* These values are shown in Fig. 6 by filled circles.

Notes on Materials Properties and Applications

Carbides of thorium can serve as alternative fissile materials for nuclear breeder reactor systems. The monocarbide is particularly interesting on account of its high metal-to-carbon atomic ratio, high thermal conductivity and wide compositional homogeneity range. The uranium-thorium mixed monocarbide (U,Th)C, according to the opinion of [1991Yam], may be a better choice than pure thorium carbide. [1969Aus] have investigated alloys of the ThC-UC quasibinary system with uranium contents of 3, 8 and 15 at.%. Plates were pressed and annealed under vacuum at 1800°C for 6 to 10 h. The compositional dependence of absolute thermoelectric power, Hall coefficient and electrical resistivity are shown in Figs. 7, 8 and 9, respectively. An estimation of the chemical state of various fission products in irradiated uranium-thorium mixed monocarbide fuel pins was performed by [1991Yam] using the SOLGASMIX computer code. In carbide fuel pins, more fission product-containing secondary phases were predicted to form than in oxide fuel pins. Thus, the chemical interaction between fuel and cladding will be much less in thorium carbide fuel pins as compared to oxide fuel pins.

Miscellaneous

[1964Keg] prepared metallographic specimens consisting of pyrolytic carbon coated as-cast alloys with a carbon content of about 66.67 at.% and different contents of thorium and uranium. Both vibratory and mechanical polishing techniques were used. The etchant comprised a solution of HNO₃ and H₂O in a volume ratio of 1:1. [1965Wym, 1966Bar] proposed the application of sol-gel processing for the production of (U,Th)C spheres. An analogous process for the preparation of the (U,Th)C₂ spheroidal particles has been developed by [1965Kel, 1965Wym, 1966Bar]. This process consists of four steps: preparation of the oxide sol; incorporation of carbon in the sol; formation of gel spheroids; firing of the spheroids. [1965Pet, 1966Bar] prepared dense particles of (U,Th)C₂ from dense sol-gel ThO₂ or (U,Th)O₂ microspheres by dispersing the spheres in graphite flour or lampblack and heating the mixture in a graphite crucible. They also have developed a process for coating (U,Th)C₂ particles with carbon films to protect the carbide against attack by water. [1969Aus] proposed a scheme of changes in the band structure of thorium monocarbide after the addition of uranium. The result seems to be a lower Fermi level and an increase in the ratio of holes to electrons. Carbothermic reduction of mechanically mixed ThO₂ + UO₂ + C compacts to (U,Th)C has been studied by [1987Nam] over the temperature range 1470 and 1770°C with an emphasis on the study of reaction kinetics. The rate-limiting step of this reaction was attributed to the diffusion of CO gas in the outermost layer of the reaction products. ThO₂ and UO₂ were found to react with graphite to produce two nearly separate dicarbide phases, both of which then reacted with residual ThO₂ to form a monocarbide phase. An apparent activation energy of about 320 kJ·mol⁻¹ was obtained for this carbothermic reduction.

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Table 1: Investigations of the C-Th-U Phase Relations, Structures and Thermodynamics

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1957Now]	X-ray diffraction studies	1900°C, 50 at.% C
[1958Cir] as quoted by [1961Ben]	X-ray diffraction studies	50 at.% C
[1958Iva]	X-ray diffraction studies, microstructural investigations	1000°C, 700°C, the Th-UC section
[1958Lau] as quoted by [1959Now]	X-ray diffraction studies	1800°C, 50 at.% C
[1959Now]	X-ray diffraction studies, microstructural investigations	1700-1800°C, 50 at.% C
[1960Bre]	X-ray Debye-Scherrer powder diffraction studies, microstructural investigation, stability tests (moisture, air, oxygen)	1800°C, 66.67 at.% C

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1961Ben]	X-ray powder diffraction studies	1900 - 1000°C, whole composition range
[1961Iva1]	X-ray diffraction studies, microstructural investigations	1000°C, 700°C, the Th-UC and ThC-UC sections
[1961Iva2]	X-ray powder diffraction studies, microstructural analysis	50 at.% C
[1962Kat]	Metallography, X-ray diffraction, diffusion couples	1000°C, the Th-UC and Th-UC ₂ sections
[1964Keg]	Optical microscopy	Pyrolytic carbon coated with as-cast ThC ₂ or (U,Th)C ₂
[1965Kel] as quoted by [1966Bar]	Sol-gel preparation of particles	66.67 at.% C
[1965Pet] as quoted by [1966Bar]	Sol-gel preparation of particles, carbon films coating	66.67 at.% C
[1965Wym] as quoted by [1966Bar]	Sol-gel preparation of particles	66.67 at.% C
[1966Hen]	Thermal analysis, high-temperature X-ray diffraction	> 500°C, 66.67 at.% C in the presence of about 1 mass% free carbon
[1966Lan] as quoted by [1984Hol]	X-ray diffraction studies	66.67 at.% C
[1969Aus]	X-ray diffraction, conventional dc methods, density measurements, chemical analysis	50 at.% C
[1971Pet] as quoted by [1975Hol]	X-ray diffraction studies	66.67 at.% C
[1984Hol]	Phase relations investigation	1800°C, whole range of compositions
[1987Jon]	Neutron powder diffraction	66.67 at.% C
[1987Nam]	X-ray diffraction, carbothermic reduction	1470 - 1770°C
[1989Koy]	Knudsen effusion mass spectrometry	1723 - 1923°C, about 50 at.% C
[1991Yam]	Knudsen effusion mass spectrometry	About 50 at.% C

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(C) (I) < 3827 ± 50	<i>hP4</i> <i>P6₃/mmc</i> C (graphite)	<i>a</i> = 246.12 <i>c</i> = 670.9	at 25°C [Mas2] sublimation point at 1.013 bar
(C) (II) > 60.78 bar	<i>cF8</i> <i>Fd$\bar{3}m$</i> C (diamond)	<i>a</i> = 356.69	at 25°C [Mas2] high pressure phase (> 60.78 bar)
(βTh) (h) 1755 - 1360	<i>cI2</i> <i>Im$\bar{3}m$</i> W	<i>a</i> = 411	[Mas2] dissolves ~9 at.% C at 1707°C [Mas2] dissolves 12.2 at.% U at 1375°C [Mas2]
(γU) (h ₂) 1135 - 776	<i>cI2</i> <i>Im$\bar{3}m$</i> W	<i>a</i> = 352.4	[Mas2] dissolves ~1 at.% Th at 1100°C [Mas2] dissolves 0.22 to 0.37 at.% C at 1119 ± 1°C, 1 at.% C at 1100°C, 0.3 at.% C at 900°C [Mas2]
(βU) (h ₁) 776 - 668	<i>tP30</i> <i>P4₂/mnm</i> βU	<i>a</i> = 1075.9 <i>c</i> = 565.6	[Mas2] dissolves 0.02 at.% C at 772°C and 0.05 at.% C at 700°C [Mas2]
(αU) (r) < 668	<i>oC4</i> <i>Cmcm</i> αU	<i>a</i> = 285.37 <i>b</i> = 586.95 <i>c</i> = 495.48	at 25°C [Mas2] dissolves ~6·10 ⁻³ at.% C at 660°C [Mas2]
βThC ₂ (h ₁) 1495 - 1255	<i>tP6</i> <i>P4₂/mmc</i> βThC ₂	<i>a</i> = 423.5 <i>c</i> = 540.8	63 to 66 at.% C [Mas2]
αThC ₂ (r) < 1440	<i>mC12</i> <i>C2/c</i> αThC ₂	<i>a</i> = 669.2 <i>b</i> = 422.3 <i>c</i> = 674.4 β = 103.0°	66 at.% C [Mas2]
ωTh ₂ C ₃	-	<i>a</i> = 856.09 to 865.13	metastable [1996Vel] high pressure phase (at 1325°C, 3.5·10 ⁻⁴ bar)
ζ, U ₂ C ₃ < 1833	<i>cI40</i> <i>I$\bar{4}3d$</i> Pu ₂ C ₃	<i>a</i> = 808.9	60 at.% C [2001Che]
εUC ₂ 1780 - 1478	<i>tI6</i> <i>I4/mmm</i> CaC ₂	<i>a</i> = 351.90 <i>c</i> = 597.87	62 to 62.5 at.% C [2001Che] U rich [2001Che]
		<i>a</i> = 352.41 <i>c</i> = 599.62	C rich [2001Che]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
δUC_2 2435 - 1763	$cF12$ $Fm\bar{3}m$ $\text{CaF}_2?$	$a = 545.0$	[2001Che] actually, “ δUC_2 ” phase represents the UC phase in equilibrium with graphite [2001Che]
δUC (II)	o^{**}	-	high pressure phase ($> 2.7 \cdot 10^5$ bar)
λ , (U,Th)C			solid solution
(αTh) (r) < 1360	$cF4$ $Fm\bar{3}m$ Cu	$a = 508.42$	at 25°C [Mas2] dissolves 6.8 at.% U at 1270°C [Mas2]
γThC_2 (h_2) 2610 - 1470	$cP12$ $Pa\bar{3}$ FeS_2	$a = 580.8$	at 1500°C [1964Hil]
ThC < 2500	$cF8$ $Fm\bar{3}m$ NaCl	$a = 534.6$ $a = 533.8$	$x = 0$ [E] $x = 0$ [H]
UC (I) < 2515		$a = 495.97$	from 47 to 66 at.% C, miscibility gap (critical point at 2050°C, 43.8 at.% C) [2001Che]

Table 3: Partial Vapor Pressures Measurements of U (gaseous) and Th (gaseous) over the λ Phase

Phase(s)	Temperature [K (°C)]	$\log(p \text{ [Pa]})$	Comments
$\text{U}_{0.1}\text{Th}_{0.9}\text{C}_{0.855}$ U (g)	1960 - 2250 (1687 - 1977)	$-(28290 \pm 445) / T$ $+ (9.4793 \pm 0.0093)$	[1989Koy, 1991Yam] Knudsen effusion
Th (g)	2020 - 2250 (1747 - 1977)	$-(30875 \pm 400) / T$ $+ (10.3382 \pm 0.0085)$	
$\text{U}_{0.2}\text{Th}_{0.8}\text{C}_{0.973}$ U (g)	1960 - 2270 (1687 - 1997)	$-(25879 \pm 339) / T$ $+ (8.3584 \pm 0.0073)$	
Th (g)	2080 - 2270 (1807 - 1997)	$-(30174 \pm 239) / T$ $+ (9.6639 \pm 0.0052)$	
$\text{U}_{0.4}\text{Th}_{0.6}\text{C}_{0.973}$ U (g)	1950 - 2170 (1677 - 1897)	$-(24346 \pm 237) / T$ $+ (7.7685 \pm 0.0064)$	
Th (g)	1950 - 2170 (1677 - 1897)	$-(28932 \pm 312) / T$ $+ (8.9489 \pm 0.0064)$	

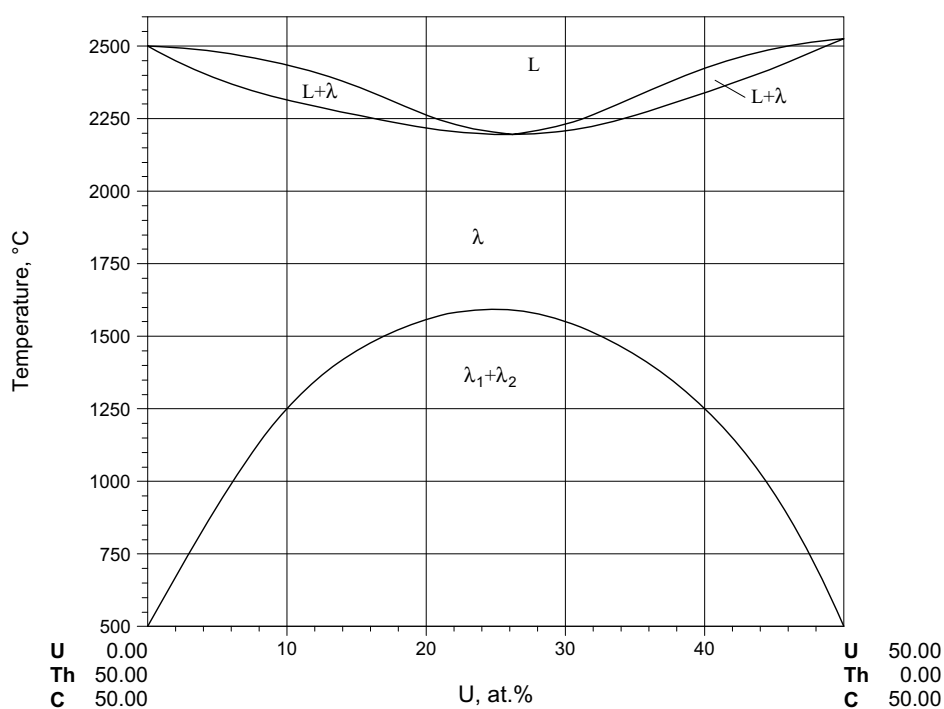
Fig. 1: C–Th–U.
The ThC–UC
quasibinary section

Fig. 2: C-Th-U.
Isothermal section at
1800°C

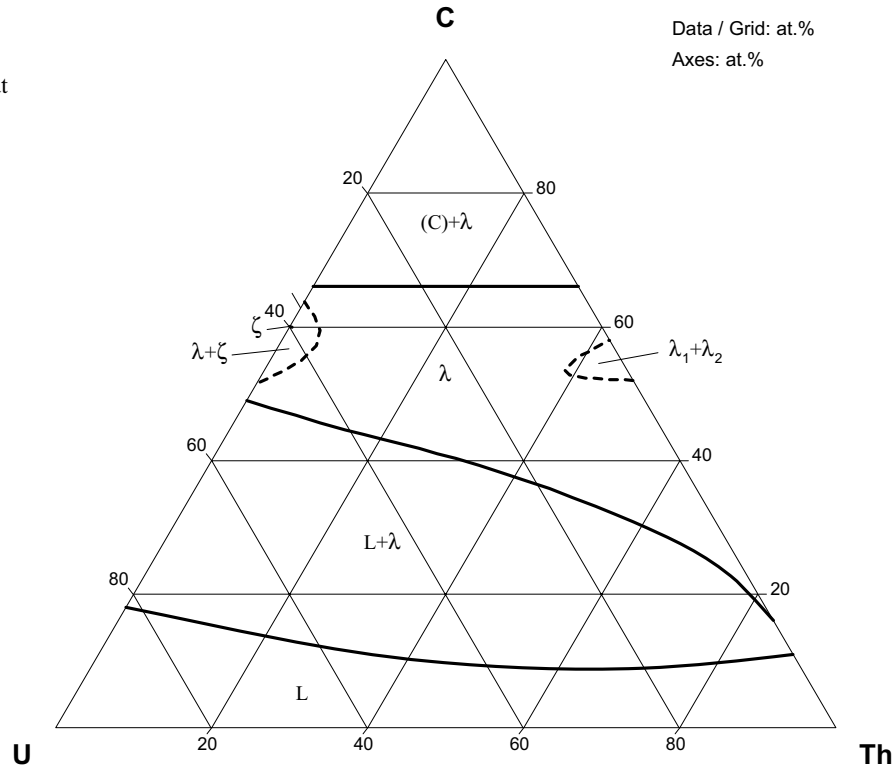


Fig. 3: C-Th-U.
Isothermal section at
1700°C in the range
of compositions
50-100 at.% C

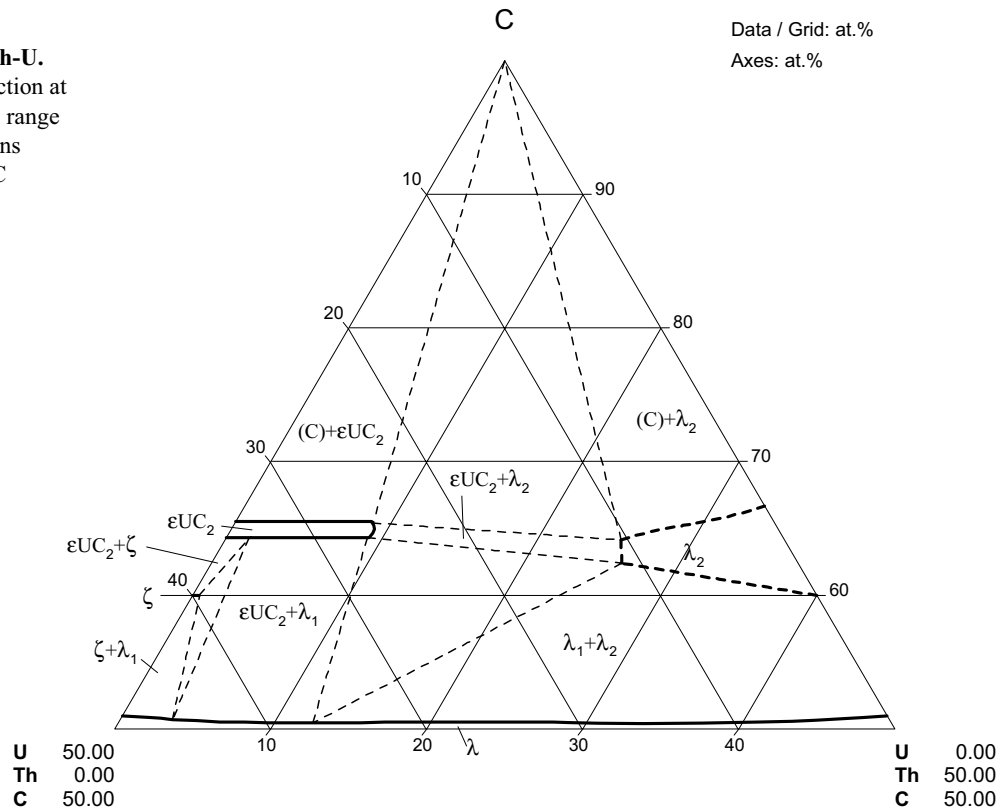


Fig. 4: C-Th-U.
Isothermal section at
1327°C

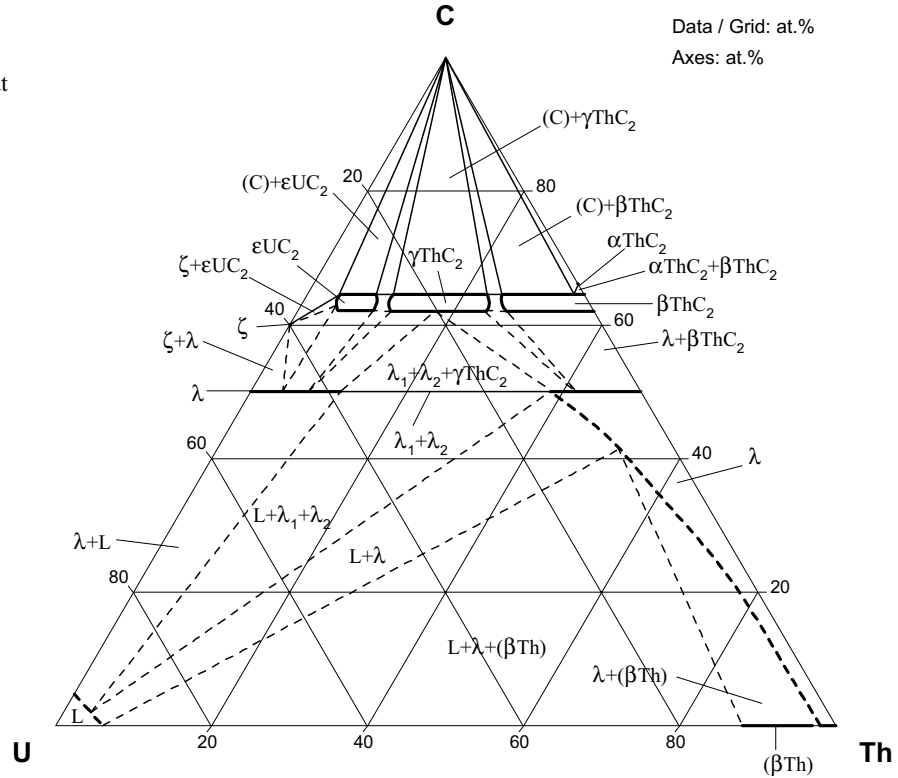


Fig. 5: C-Th-U.
Isothermal section at
1000°C

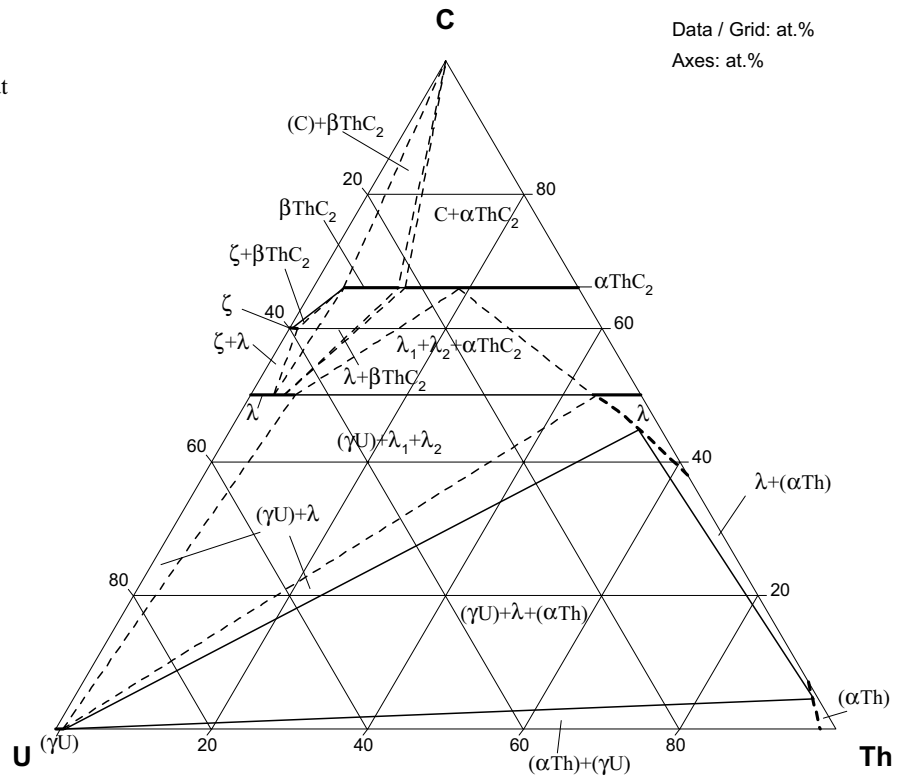


Fig. 6: C-Th-U.
Compositional
dependence of Gibbs
energy of formation
of the λ , $U_xTh_{1-x}C$
phase ($x = 0$ to 0.5)

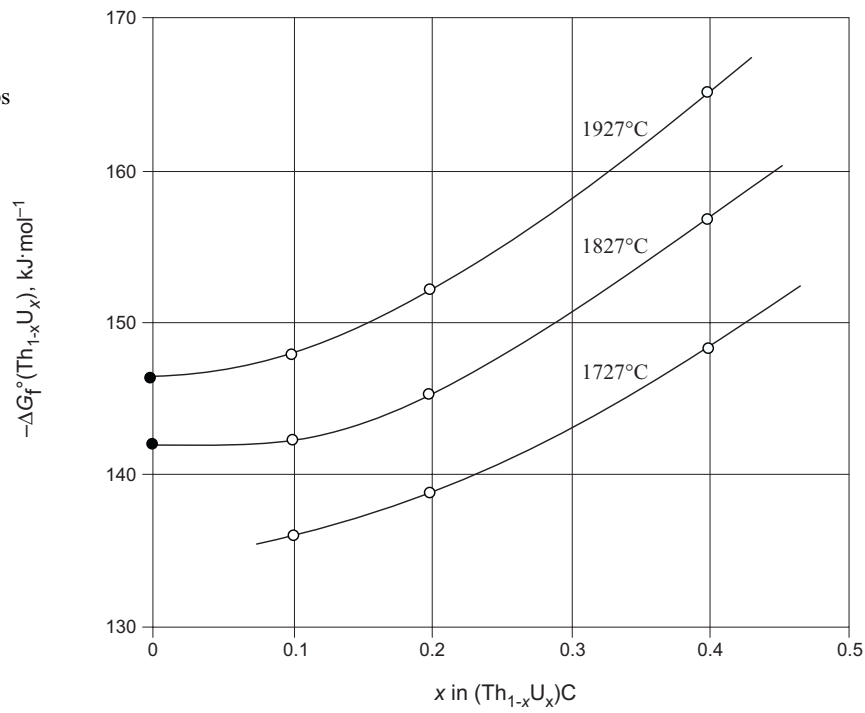


Fig. 7: C-Th-U.
Absolute
thermoelectric power
of alloys of the
ThC-UC section

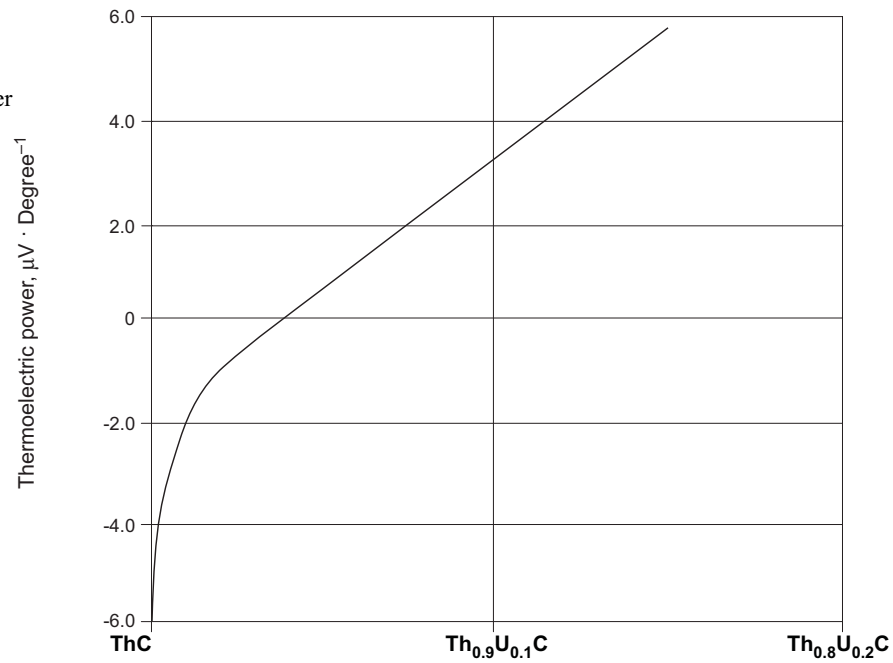


Fig. 8: C-Th-U.
Hall coefficient of
alloys of the ThC-UC
section

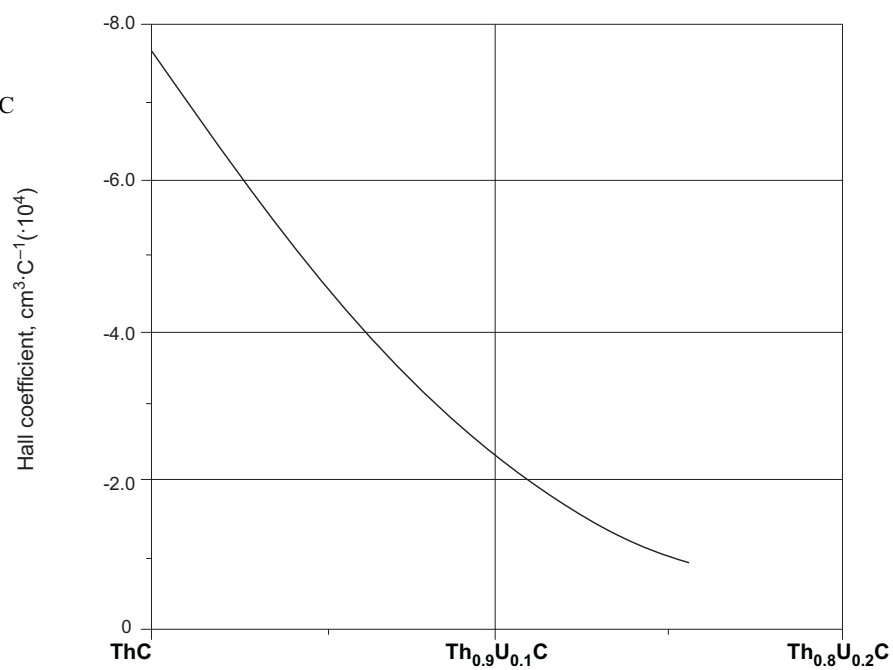


Fig. 9: C-Th-U.
Electrical resistivity
of alloys of the
ThC-UC section

