

Carbon – Rhodium – Uranium

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Introduction

Phase relations in multicomponent systems containing actinide carbides are of great interest in nuclear technology. In particular, the failure of metallic fuels to meet the requirements of nuclear power reactors is gradually leading to their replacement by ceramic fuels. A great deal of interest has consequently been centred on uranium carbide and its solid solutions. During fission, the composition of the fuel will change, with the result that at about 10% burn-up approximately 16% of the atoms will be foreign atoms [1974Nar]. A knowledge of the behavior of these fission products is obviously very important. Consequently, phase relation and thermodynamic studies of C-U fission product systems are necessary to ascertain the stability of the fuel during operating conditions. Among the metals presenting metallic fission products, rhodium has been identified. Results of experimental studies of the C-Rh-U system are presented as isothermal sections at 1300°C [1973Hol, 1975Hol, 1984Hol1, 1984Hol2] and 700°C ([1973Wes], quoted by [1974Nar]). The crystal structure of the U_2RhC_2 phase and its stability were studied by [1964Far, 1968Hol, 1969Hai, 1985Ara]. A brief survey of the literature on the C-Rh-U system has been presented by [1975Hol, 1984Hol1, 1984Hol2].

Experimentally determined thermodynamic properties were obtained by [1973Hol, 1974Nar, 1978Gup]. The thermal conductivity of the U_2RhC_2 phase was determined by [1985Ara]. Magnetic properties of the U_2RhC_2 phase were studied by [1996Ebe]. The experimental methods employed along with the temperature and composition ranges studied are presented in Table 1. Information about phase equilibria in the C-Rh-U system is incomplete. In particular, experimental data relating to phase equilibria in the U rich region need further refinement. Information about the conditions of crystallization of the alloys is lacking. Future investigations of phase relations in the C-Rh-U system should be concentrated on the continuation of the study of alloy properties in the equilibrium state at different temperatures.

Binary Systems

Data relating to the forming binary C-Rh, C-U and Rh-U systems are accepted from [Mas2].

Solid Phases

Crystallographic data of the known unary and binary phases as well as the ternary τ phase are compiled in Table 2. The ternary compound was discovered as a product of the solid-state reaction between uranium carbide and rhodium at a temperature of about 1700°C, and was subsequently prepared by arc melting together the three elements [1964Far, 1967Kri]. The ternary τ phase was supposed to form by a peritectic reaction at a temperature above 1700°C [1967Kri]. According to [1973Hol], the binary URh_3 compound dissolved up to 5 at.% C in a cast alloy and 3 at.% C after annealing at 1300°C. [1968Far] noted that UC reacts with a small amount of rhodium to form the τ phase, and the reaction of UC with more than the stoichiometric amount of rhodium required to form this ternary phase causes the formation of graphite and the ϵ phase.

Isothermal Sections

Isothermal sections for the whole composition range at temperatures of 1300°C and 700°C are shown in Figs. 1 and 2, respectively. The section at 1300°C is presented based on the data of [1973Hol] reproduced in [1975Hol, 1984Hol1, 1984Hol2]. Some corrections have been made to ensure agreement with the accepted boundary binary systems. Thus, the extension of the liquid phase field in the uranium corner is increased due to a higher extension in the boundary C-U system (up to 6 at.% C). The (Rh) single-phase range is drawn due to the existence of solubilities at 1300°C of uranium (about 1.5 at.%) and carbon (about

0.5 at.%) in rhodium. The position of the τ phase is shown in Fig. 1 at the stoichiometric composition without a homogeneity range because there is no information on its composition range given in [1973Hol]. The isothermal section at 700°C (Fig. 2) is shown following the data of [1973Wes], presented in [1974Nar], with corrections in accordance with the accepted binary systems. Because the ϵ and ζ phases do not take part in equilibria at 700°C in the C–U binary system, the corresponding fields are replaced by the $\delta + (C) + \tau$ field. As the U_2Rh and URh phases do not exist in the binary Rh–U system their phase fields are replaced by corresponding fields including the αU_4Rh_3 phase. The U rich corner of the section is speculative, taking into account the γ and β modifications of U.

Thermodynamics

The Gibbs energies of formation of the “ URh_3C_x ” and U_2RhC_2 compounds were determined by [1973Hol] and [1974Nar], respectively, by means of solid-state electrolytic cells, using CaF_2 as the electrolyte. The temperature dependence of ΔG_T° “ URh_3C_x ” for the temperature range 597–827°C is expressed as ΔG_T° (“ URh_3C_x ”) = $-239000 - 23 \cdot T \pm 1676$ (J, mol, K). At 827°C, the values of the thermodynamic properties of the $(URh_3)_{1-x}C_x$ compound are: $\Delta G^\circ = -264389 \pm 1676$ J·mol⁻¹; $\Delta H^\circ = -238830 \pm 18855$ J·mol⁻¹; $\Delta S^\circ = -23045 \pm 16760$ J·K⁻¹·mol⁻¹. All of these data correspond to the ϵ phase in equilibrium with (Rh) and (C). [1973Hol] noted that the stability of the ϵ phase in the C–Rh–U ternary system is only negligibly higher than in the binary Rh–U system. The Gibbs energy of formation of the U_2RhC_2 compound at 827°C was estimated in [1973Hol] as being between -528000 and -268000 J·mol⁻¹. According to the experimental results of [1974Nar], the value of this parameter at 827°C is similar to the limiting value in the presented interval being equal to -267190 J·mol⁻¹. The temperature dependence of the Gibbs energy of formation of the U_2RhC_2 compound for the temperature range 727–927°C is as follows: ΔG_T° (U_2RhC_2) = $-303380 - 32.9 \cdot T$ (J, mol, K) [1974Nar]. These results were determined using a $U_{43}Rh_{22}C_{35}$ alloy containing the τ phase together with the δ and U_3Rh_5 phases. [1978Gup] observed the gaseous molecule $URhC_2$ in the Knudsen-cell effusate from a C–Rh–Th–U alloy at high temperatures. The thermodynamic properties of $URhC_2$ were evaluated from the various reaction enthalpies determined by the third law method, $\Delta H_{298}^\circ = T \cdot \{-R \cdot \ln K_p - \Delta((G_T^\circ - H_{298}^\circ)/T)\}$. The free energy functions $(G_T^\circ - H_{298}^\circ)/T$ were taken from the literature when available or calculated from known or estimated molecular parameters through standard statistical thermodynamic expressions. The calculated values of $-(G_T^\circ - H_{298}^\circ)/T$ and the heat content functions, $H_T^\circ - H_{298}^\circ$, for the $URhC_2$ molecule at various temperatures are listed in Table 3. The values of the $H_{298}^\circ - H_0^\circ$ heat content functions were obtained as 15378 J·mol⁻¹ for the Rh–U–C–C structure and 13700 J·mol⁻¹ for the Rh–C–C–U structure. The average value of the atomization energy, $\Delta H_{at, 298}^\circ$ ($URhC_2$) = 1757 ± 50 kJ·mol⁻¹, which is closer to the value of 1775 ± 46 kJ·mol⁻¹ estimated for the structure Rh–U–C–C than the corresponding value, $\Delta H_{at, 298}^\circ$ ($URhC_2$) = 1789 ± 50 kJ·mol⁻¹ to the value of 1704 ± 50 kJ·mol⁻¹ estimated for the Rh–C–C–U structure from bond additivity considerations. The authors proposed that the linear structure Rh–U–C–C represents the molecule $URhC_2$. The atomization energies of gaseous $URhC_2$ were calculated to be 1757 ± 50 kJ·mol⁻¹ and 1746 ± 50 kJ·mol⁻¹ for temperatures of 25°C and -273.15°C , respectively. A summary of the third law reaction enthalpies and the derived atomization energies for $URhC_2$ are given in Table 4.

Notes on Materials Properties and Applications

The C–Rh–U alloys are the prospective materials for applications in nuclear technology as the alloys contain uranium carbide and the transition metal rhodium. The temperature dependence of the thermal diffusivity of the U_2RhC_2 phase measured by [1985Ara] over the temperature range from 477 to 1227°C is shown in Fig. 3. The temperature dependence of the thermal conductivity obtained for the U_2RhC_2 phase normalized to a 100% theoretical density (12.7 g·cm⁻³) was measured over the temperature range from 477 to 1227°C and is presented in Fig. 4. The magnetic susceptibility of the U_2RhC_2 phase was investigated by [1996Ebe] using a SQUID magnetometer and magnetic flux densities of up to 5.5 T over the temperature range from 2 and 300 K (-271°C to 27°C). A temperature dependent paramagnetism was shown (Fig. 5). This property is compatible with one unpaired electron per transition metal site. The U_2RhC_2 phase also exhibits complex

magnetic behavior below a Néel temperature of $T_N = 18 \pm 1$ K (the inset of Fig. 5). At still lower temperature, the reciprocal susceptibility of U_2RhC_2 decreases suggesting ferromagnetic ordering with a Curie temperature of $T_C = 13 \pm 2$ K. This is confirmed by the magnetization measurements (Fig. 6) which clearly show a hysteresis in the plot recorded at 5 K. Between the different measuring cycles, the sample was heated to above 50 K (-223°C) and cooled to the temperatures indicated in Fig. 6 in a zero magnetic field.

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

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1964Far] as quoted by [1969Hai]	X-ray studies	U_2RhC_2
[1968Far]	Studies of UC (I) interaction with rhodium	The $\text{U}_2\text{RhC}_2 + (\text{C}) + \text{URh}_3$ phase region
[1968Hol]	X-ray Debye-Scherrer and Guinier studies	1300-1600°C, $\sim \text{U}_2\text{RhC}_2$
[1969Hai]	X-ray Debye-Scherrer studies	U_2RhC_2
[1973Hol]	X-ray Guinier studies, metallography, emf measurements	598-877°C, 1300°C, 1500°C, whole range of compositions
[1974Nar]	Metallography, EMPA, emf measurements	700, 727-927°C, whole range of compositions
[1975Hol]	Experimental techniques	1300°C, whole range of compositions
[1978Gup]	High temperature mass spectrometry	U_2RhC_2
[1984Hol1]	Experimental techniques	1300°C, whole range of compositions
[1984Hol2]	Experimental techniques	1300°C, whole range of compositions
[1985Ara]	X-ray diffraction, chemical analysis, bulk density and thermal conductivity measurements	477-1227°C, 1550°C, U_2RhC_2
[1996Ebe]	X-ray Guinier studies, SQUID magnetic susceptibility measurements	2-300 K (–271-27°C), U_2RhC_2

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 (sublimation point), 1.013 bar	<i>hP4</i> <i>P6₃/mmc</i> C (graphite)	$a = 246.12$ $c = 670.9$	at 25°C [Mas2]
(C) (II) > 60.78 bar	<i>cF8</i> <i>Fd$\bar{3}m$</i> C (diamond)	$a = 356.69$	at 25°C [Mas2]
(Rh) < 1963	<i>cF4</i> <i>Fm$\bar{3}m$</i> Cu	$a = 380.32$	dissolves 1.5 at.% C at 1694°C and 2 at.% U at 1393°C [Mas2] at 25°C [Mas2]
$U_xRh_{1-x-y}C_y$			$x = 0, 0 \leq y \leq 0.015, 1694 \pm 17^\circ\text{C}$ [Mas2] $x = 0, 0 \leq y \leq 0.005, 1300^\circ\text{C}$ [Mas2] $y = 0, 0 \leq x \leq 0.02, 1393^\circ\text{C}$ [Mas2] $y = 0, 0 \leq x \leq 0.015, 1300^\circ\text{C}$ [Mas2]
(γ U) (h_2) 1135 - 776	<i>cF2</i> <i>Im$\bar{3}m$</i> W	$a = 352.4$	dissolves ~9 at.% Rh at 855°C and 0.22 to 0.37 at.% C at 1119°C [Mas2] [Mas2]
$U_{1-x-y}Rh_xC_y$			$x = 0, 0 \leq y \leq 0.0022$ to 0.0037, 1119 ± 1°C [Mas2] $y = 0, 0 \leq x \leq 0.01, 855^\circ\text{C}$ [Mas2]
(β U) (h_1) 776 - 668	<i>tP30</i> <i>P4₂/mnm</i> β U	$a = 1075.9$ $c = 565.6$	dissolves ~2 at.% Rh at 683°C [Mas2] [Mas2]
$U_{1-x-y}Rh_xC_y$			$x = 0, 0 \leq y \leq 0.0002, 772^\circ\text{C}$ [Mas2] $y = 0, 0 \leq x \leq 0.02, 683^\circ\text{C}$ [Mas2]
(α U) (r) < 668	<i>oC4</i> <i>Cmcm</i> α U	$a = 285.37$ $b = 586.95$ $c = 495.48$	dissolves about 1 at.% Rh at 625°C [Mas2] at 25°C [Mas2]
$U_{1-x-y}Rh_xC_y$			$x = 0, 0 \leq y \leq 6 \cdot 10^{-5}, 660^\circ\text{C}$ [Mas2] $y = 0, 0 \leq x \leq 0.01, 625^\circ\text{C}$ [Mas2]
αU_4Rh_3 (h) 1155 - 720	-	-	43 at.% Rh [V-C2]
βU_4Rh_3 (r) 720 - at least < 400	-	-	43 at.% Rh [V-C2]
U_3Rh_4 1450 - at least < 400	-	-	57 at.% Rh [V-C2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
U ₃ Rh ₅ 1550 - at least < 400	-	-	~ 63 at.% Rh [V-C2]
URh ₃ < 1700	<i>cP4</i> <i>Pm$\bar{3}m$</i> AuCu ₃	<i>a</i> = 399.2 <i>a</i> = 399.1 <i>a</i> = 401.5 <i>a</i> = 400.8 <i>a</i> = 399.0 <i>a</i> = 399.3 <i>a</i> = 399.1	75 at.% Rh [V-C2] Labelled as “URh ₃ C _{0.1} ” [1975Hol, 1984Hol1, 1984Hol2] or “URh ₃ C _x [1973Hol] dissolves up to 5 at.% C in cast alloy and 3 at.% C at 1300°C [1973Hol] [E] [1961Dwi] for URh ₃ in ternary cast alloy [1973Hol] for URh ₃ in equilibrium with τ and (C) at 1300°C [1973Hol] for URh ₃ in equilibrium with (Rh) at 1300°C [1973Hol] for URh ₃ in equilibrium with (Rh) and (C) at 1300°C [1973Hol] for URh ₃ in equilibrium with (Rh) and (C) at 800°C [1973Hol]
δ , UC (I) 2585 - 1119 1.013 bar	<i>cF8</i> <i>Fm$\bar{3}m$</i> NaCl	<i>a</i> = 495.98 <i>a</i> = 495.63 <i>a</i> = 495.1 <i>a</i> = 496 to 496.2 <i>a</i> = 507 <i>a</i> = 495.6 <i>a</i> = 496.05	47 to 66 at.% C [Mas2] [E] at 48 at.% C [S] [1957Kie] [1961Ben] at 2100°C [1972Ben] at 1400°C [S] at 25°C [1972Ben]
UC (II) > 2.7·10 ⁵ bar	rhombic	-	[1987Ben]
ζ , U ₂ C ₃ 1823 - ~ 850	<i>cI40</i> <i>I$\bar{4}3d$</i> Pu ₂ C ₃	<i>a</i> = 808.8 <i>a</i> = 808.90 <i>a</i> = 808.89	60 at.% C [Mas2] [H] [S] [1972Ben]
ϵ , UC ₂ 1793 - 1516	<i>tI6</i> <i>I4/mmm</i> CaC ₂	<i>a</i> = 351.7 <i>c</i> = 598.7 <i>a</i> = 352.4 <i>c</i> = 599.9 <i>a</i> = 351.9 to 352.41 <i>c</i> = 597.87 to 599.62 <i>a</i> = 352.7 <i>c</i> = 598	62 to 62.5 at.% C [Mas2] [E] [H] [S] [1961Ben]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
UC ₂ 2434 - 1762	<i>cF12</i> <i>Fm$\bar{3}m$</i> CaF ₂ ?	<i>a</i> = 545.0	actually, “UC ₂ ” phase represents the δ ,UC phase in equilibrium with graphite [2001Che]
* τ , U ₂ RhC ₂ ≤ 1717	<i>tI10l</i> <i>I4/mmm</i> U ₂ IrC ₂	<i>a</i> = 346.6 <i>c</i> = 1251.2	[1964Far]
		<i>a</i> = 346.4 <i>c</i> = 1251.3	at 1300°C [1968Hol, 1973Hol]
		<i>a</i> = 346.6 <i>c</i> = 1251.5	in the single-phase alloy annealed at 1500°C [1969Hai]
		<i>a</i> = 347 <i>c</i> = 1253	sintered at 1550°C, with traces of UC [1985Ara]

Table 3: Thermodynamic Properties of the URhC₂ Molecule in a Gaseous State [1978Gup]

Structure of the Molecule	Temperature [°C]	Free Energy Function, $-(G_T^\circ - H_{298}^\circ)/T$ [J·mol ⁻¹ ·K ⁻¹]	Heat Content Function, $H_T^\circ - H_{298}^\circ$ [J·mol ⁻¹]
Rh-U-C-C	25	310.60	0.00
	1827	394.61	147330
	1927	397.89	155940
	2027	401.06	164550
	2127	404.12	173180
	2227	407.08	181810
	2327	409.94	190450
	2427	412.71	199100
Rh-C-C-U	25	302.66	0.00
	1827	382.96	144080
	1927	386.17	152640
	2027	389.27	161220
	2127	392.27	169810
	2227	395.17	178410
	2327	397.98	187020
	2427	400.71	195640

Fig. 1: C-Rh-U.
Isothermal section at
1300°C

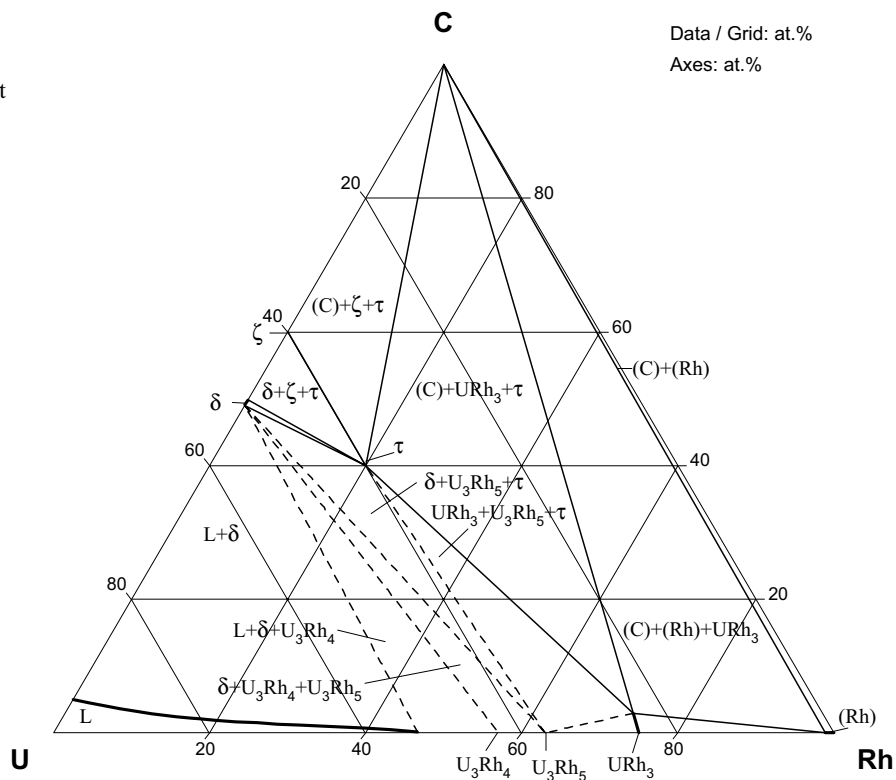


Fig. 2: C-Rh-U.
Isothermal section at
700°C

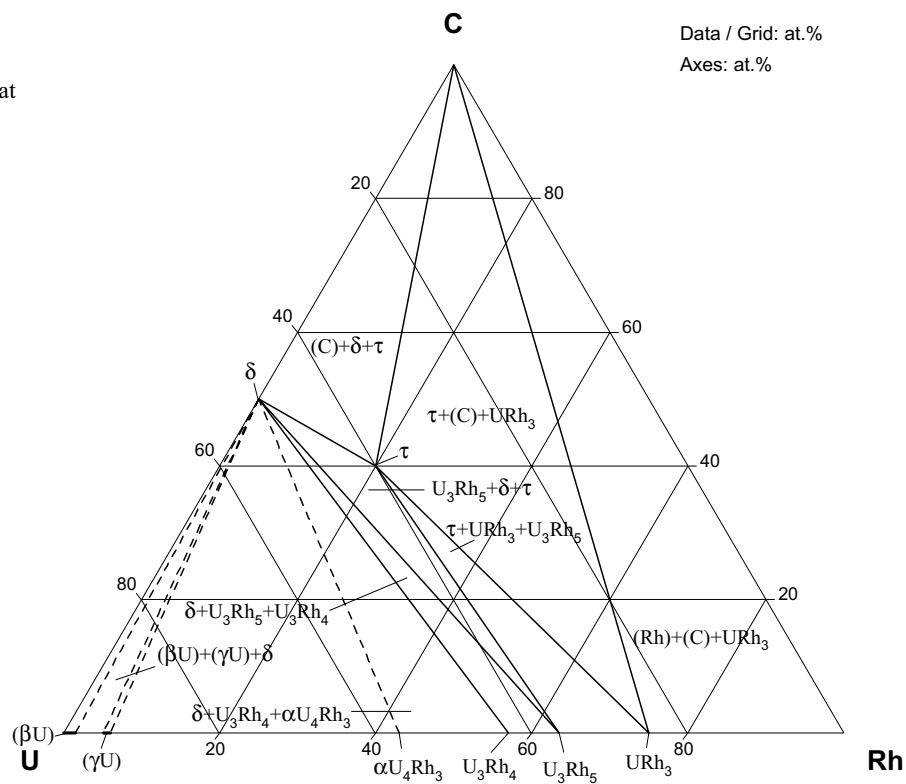


Fig. 3: C-Rh-U.
Thermal diffusivity of the U_2RhC_2 phase measured over the temperature range from 477 to 1227°C

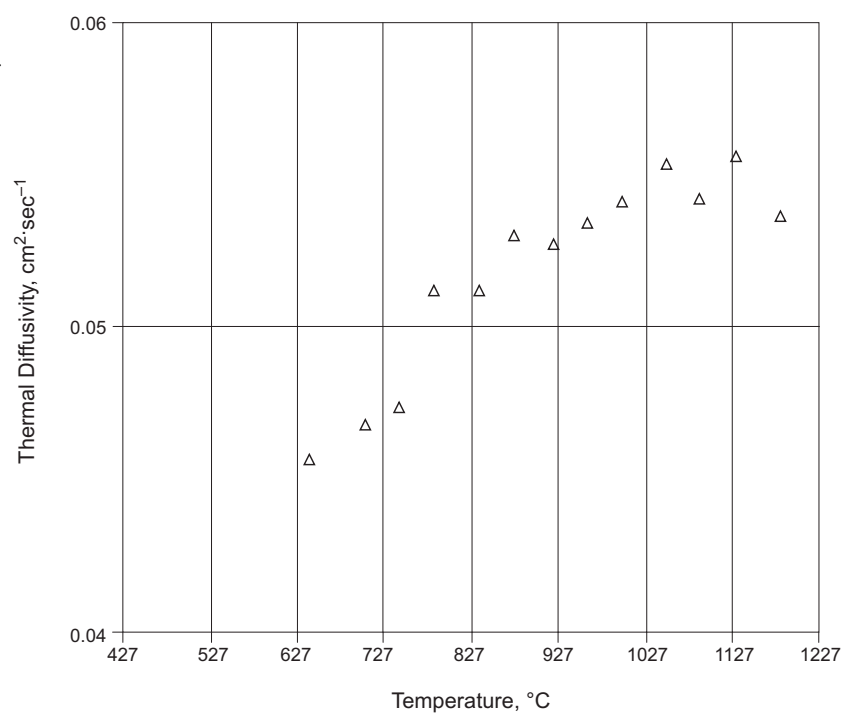


Fig. 4: C-Rh-U.
Thermal conductivity of the U_2RhC_2 phase normalized to 100% theoretical density measured over the temperature range from 477 to 1227°C

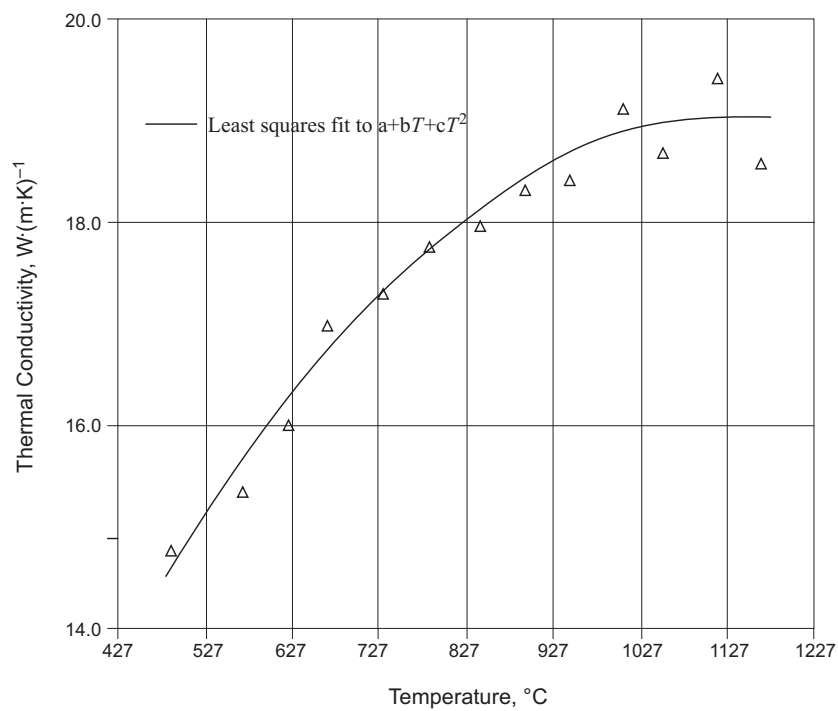


Fig. 5: C-Rh-U.
Temperature dependence of the reciprocal susceptibility of U_2RhC_2 . Low temperature behaviour of the reciprocal susceptibility in magnetic fields of 1 T and 5 T (the inset)

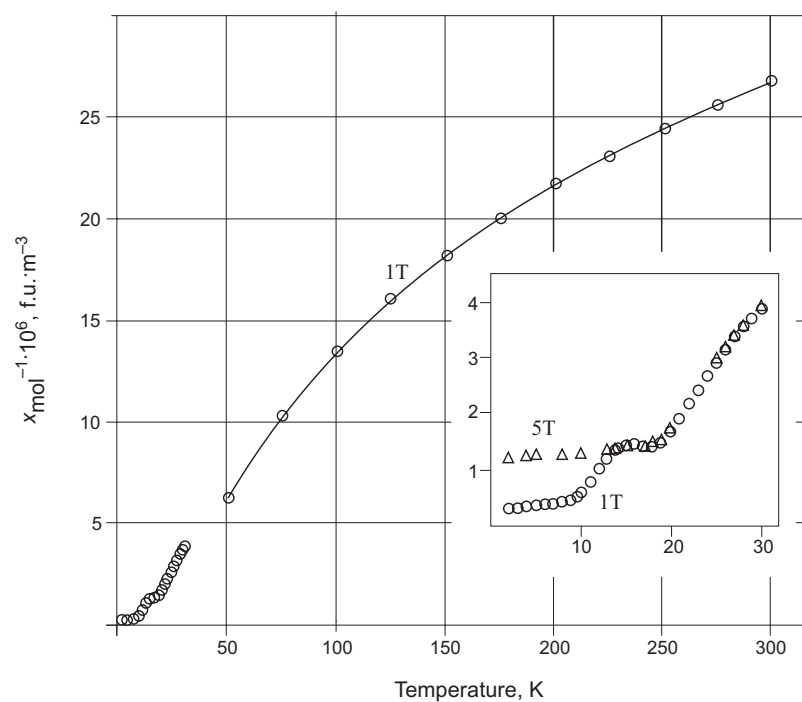


Fig. 6: C-Rh-U.
Magnetisation vs magnetic flux densities B of U_2RhC_2 at different temperatures

