Carbon – Iron – Uranium

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Introduction

The system was studied to provide a background for the description of the interaction between carbide fuels and steel canning materials.

Most attention has been paid to the vertical sections. The quasibinary section UC-Fe was studied by [1961Bar, 1963Bri1, 1963Bri2, 1963Nic, 1971Guh] and the results of [1963Bri2, 1963Nic, 1971Guh] are in good agreement. The UC-UFe_2 quasibinary section was studied by [1961Bar] and [1963Nic]. It was found that the section was of a simple eutectic type, but the eutectic temperature presented by [1961Bar] and the eutectic temperature presented by [1963Nic] differed by more than 120°C. The discrepancies in the results may have arisen from differences in the techniques used. [1961Bar] used metallographic observation of melting of powder mixtures of Fe and UC which had been annealed at various temperatures, whereas [1963Nic] used thermal analysis studies of alloys of higher purity which had been prepared by arc melting under an argon atmosphere on a water cooled copper hearth using a tungsten electrode.

The phase equilibria along the Fe-UC_2 tie line were studied by [1962Bal, 1963Bri2, 1963Nic]. [1962Bal] found that the $UFeC_2$ ternary carbide was formed peritectically from UC_2 and liquid [1963Bri2]. The existence of a quasibinary eutectic between Fe and $UFeC_2$ was established by [1963Bri2, 1963Nic]. The UC_2 - Fe section cannot be considered as truly quasibinary because of the eutectoid decomposition of UC_2 at 1516°C in the C-U binary system.

In addition to these sections, thermal analysis studies by [1963Nic] led to the suggestion of a number of invariant four-phase equilibria, but these are very uncertain and in need of further experimental investigation. They also presented a scheme of solid state tie lines for “low temperatures”. Based on these data, [1984Hol1, 1984Hol2] constructed schematic isothermal sections for 1000°C.

Recently, [1990Ale, 1992Ale] investigated the system with special attention to the UC-UC_2-$UFeC_2$ composition region through the study of approximately 10 alloys.

Based on the results of X-ray analysis of annealed and quenched alloys, [1990Ale] reported the existence of two new ternary carbides, namely $U_2Fe_2C_3$ and $U_3Fe_2C_5$. Later, [1995Wac] reported that compound called “$U_2Fe_2C_3$” by [1990Ale] is isotypic with $Th_{11}Fe_{12}C_{18}$. [1986Ger] found the $UFeC_2$ compound to be isostructural with $UCoC_2$.

[1992Ale] presented the liquidus projection, partial isothermal section at 1400°C and isothermal section at 1150°C. In addition to $UFeC_2$, three more ternary phases were found. The solubility of Fe in UC_{1+x} was studied; for compositions with $x \approx 0$ (near UC composition). The homogeneity region stretches towards a carbon content of ≥ 50 at.%, so this result does not contradict the absence of Fe solubility in UC found in the Fe-UC and UFe_2-UC sections by earlier investigators. For compositions with $x \approx 1$ (αUC_2), an island phase field of UC_{1+x} is found to exist at both temperatures. [1992Ale] also attempted to make a qualitative description of all topologically different isothermal sections where the liquid phase exists. These results were presented in the review of [2002Rag].

Thermodynamic data exist only for the $UFeC_2$ phase. Its Gibbs energy of formation was obtained by [1973Tan] using emf measurement of U activity in a $UFeC_2$+(C)+(Fe) mixture at 722 to 811°C. CaF_2 was used as solid electrolyte with a U+UF_3 mixture as reference electrode.

The reaction of UC and UC_2 powders with Fe as well as stainless steel at 1000°C was studied by [1962Kat] and [1963Nic]. [1974Mat] studied the influence of Fe on the self-diffusion of C in UC. [1988Jon] determined the products of acid hydrolysis of $UFeC_2$ and some other carbides as a tool for determining whether C atoms form chains.

Experimental investigations of phase relations, crystal structure and thermodynamics are reviewed in Table 1.

Binary Systems

The three binary systems are taken from [Mas2]. In the C-U binary system, the UC_{1+x} phase ($x \approx 1$) in equilibrium with graphite should be denoted as the βUC_2 phase with a crystal structure of the CaF_2 type.

Solid Phases

In the C-U binary phase UC_{1+x} at temperatures between 2104 and 1769°C, there exists two separate regions with $x \approx 0$ and at $x \approx 1$ which are often considered as separate carbides, UC and βUC_2 [2001Che]. At 1400°C, the phase dissolves about 10 at.% Fe near the "UC" composition and also exists as a separate region at Fe contents of about 4 to 6 at.% and C contents of about 62 to 65 at.%, *i.e.* close to the "UC_2" binary composition [1992Ale]. At 1150°C, the Fe solubility is smaller, but both solid solutions near "UC" and a small separate region near "UC_2" still exist (see below Isothermal Sections).
Four ternary phases are known, denoted here τ_1 to τ_4. The τ_1 phase with the composition $UFeC_2$ is formed by a peritectic reaction in the section "UC_2"-Fe [1962Bal, 1963Bri1]. [1986Ger] found it to be isostructural with $UCoC_2$. Phase τ_2 was found by [1990Ale] to form peritectoidally at 1500 ± 100°C; a composition close to $U_3Fe_2C_5$ was claimed. Later, [1992Ale] shifted its composition from the section UC-Fe to a higher C content $U_{\sim 32}Fe_{\sim 17}C_{\sim 51}$ (see isothermal section at 1400°C below); this shift is accepted here. X-ray diffraction of this phase suggests it to be a distorted tetragonal solid solution of Fe in UC_{1+x} [1990Ale, 2002Rag]; though no structural studies were performed. The phase τ_3 exists at 1400°C but not at 1150°C in an alloy with a gross composition of $U_{30.6}Fe_{8.6}C_{60.8}$, taken from figure 2 of [1992Ale]; no structural data exist. The phase τ_4 is formed by peritectoid reaction at 1197°C [1992Ale] with a composition close to $U_2Fe_2C_3$; the stoichiometry $U_{11}Fe_{12}C_{18}$ following from its crystal structure was established by [1995Wac]. All of the phases are listed in Table 2.

Quasibinary Systems

The sections between UC-Fe and UC-UFe_2 are quasibinary with simple eutectics [1961Bar, 1963Bri1, 1963Bri2, 1963Nic, 1971Guh]. No solid solubilities were found in either. All data for the eutectic temperature and composition for UC-Fe section are in good agreement. The UC-Fe section is given in Fig. 1. A eutectic temperature of 1117°C was accepted from [1963Nic], where it was measured using TA. The UC-UFe_2 section is presented in Fig. 2 after [1961Bar, 1963Bri1, 1963Bri2, 1963Nic]. A eutectic melting temperature of 1201°C is accepted from [1963Nic]. This value is preferred as the method of observation of melting used by [1961Bar, 1963Bri1] may be too sensitive to admixtures.
The solubility of Fe in UC which was found by [1992Ale] does not lie in either of these sections (see Isothermal Sections below) and so does not contradict these findings.
The UC_2-Fe section, as presented by [1963Bri2], may be regarded as quasibinary but in the Fe-$UFeC_2$ composition region only, since the eutectoid decomposition of αUC_2 at 1516°C takes place in the C-U binary system. The peritectic formation of the τ_1 phase $UFeC_2$ occurs at 1615°C. The region between the τ_1 phase and βUC_2 is unclear. The Fe solid solution (about 3 mass%) is stabilized at low temperatures, and the βUC_2 phase and the τ_3 phase should exist at least around 1400°C according to [1992Ale]. But these results are in contradiction with those presented by [1963Bri2], who reported the decomposition of βUC_2 at temperatures lower than 1516°C. Since additional experimental data in this composition region are absent and the phase equilibria promise to be very complicated and would include the phase relations between solid phases existing at these temperature and composition intervals, the composition region between $UFeC_2$ and UC_2 was deleted from the section and it was bordered by $UFeC_2$ compound. The quasibinary section $UFeC_2$-Fe is presented in Fig. 3.

Invariant Equilibria

The invariant equilibria are listed in Table 3. They are almost confined to those in the quasibinary sections UC-Fe, UC-UFe_2 and the vertical section Fe-"UC_2" (see below).
Also, according to [1963Nic], two eutectic reactions: E_1: $L \rightleftharpoons UC + (Fe) + UFe_2$ at 1037°C and E_2: $L \rightleftharpoons UC + UFe_2 + U_6Fe$ with a melting point of 720°C exist in the system. A series of invariant reactions

proposed in [1992Ale] was omitted since they have not any experimental confirmation and are only speculative constructions.

Liquidus, Solidus and Solvus Surfaces

The liquidus projection presented by [1992Ale] was omitted since the amount of experimental data associated with its construction was not sufficient.

Isothermal Sections

A partial isothermal section for 1400°C is presented in Fig. 4. It is taken mainly from [1992Ale], but redrawn slightly to bring it into agreement with the accepted binary systems.
The UC phase dissolves up to about 10 at.% Fe. This unary region is directed along and somewhat above the line at 50 at.% C. This is the reason for it being missing in early works where the sections studied were directed from UC to Fe and UFe_2. Another stability region for that phase is near the UC_2 composition.
The isothermal section at 1050°C is presented in Fig. 5. It is also based on the data of [1992Ale]. The composition of the τ_4 phase has been changed slightly to bring it into accordance with the accepted stoichiometry $U_{11}Fe_{12}C_{18}$. Two regions of solid solubility of Fe in UC and βUC_2 still exist at this temperature though with narrower homogeneity ranges. Two three-phase fields with a seemingly identical phase composition of $L+UC+UFe_2$ are separated by the quasibinary section $UC\text{-}UFe_2$ with a maximum on the monovariant line and so differ in the composition of the participating liquid phases.

Thermodynamics

The Gibbs energy of formation of the τ_1 phase is presented in Table 4 as obtained by emf studies [1973Tan].

Notes on Materials Properties and Applications

Uranium monocarbide has received a good deal of attention as a reactor fuel, particularly for fast reactor applications, since it has the advantage over the dioxide UO_2 of possessing a high thermal conductivity and is, therefore, capable of being used at higher ratings with lower centre temperatures [1961Bar]. To elucidate the interaction between UC-base nuclear fuel and canning materials, the underlying phase equilibrium relationships in the system involved have been studied.
[1962Kat] studied the interaction of UC and UC_2 powders with Fe in samples held at 1000°C for 500 h. At these conditions, UC reacted with Fe; however, the UC_2 showed no signs of interaction. A parabolic time dependence was found for the reaction with UC.
[1963Nic] noticed that UC gives a “eutectic” (*i.e.* begins to form liquid) with stainless steel 100°C lower than with pure Fe.
Both UC and UC_2 quickly react with stainless steel after an induction period of 24 h [1962Kat]. [1966Far] noted that in the three-phase region $UC\text{-}UFe_2\text{-}Fe$, a UC-Fe mixture containing less than 50 at.% carbon did not react with type 316 stainless steel after 1500 h at 750°C, whereas unalloyed UC containing less than 50 at.% carbon reacted with stainless steel in 24 h at 750°C.

Miscellaneous

[1988Jon] found that the products of acid hydrolysis of $UFeC_2$ contain only CH_4 and concluded that no C-C bonds exist in crystals of that phase.
The self-diffusion of uranium in stoichiometric UC which was doped with Fe is increased as compared to undoped UC at all temperatures studied (1380 to 2200°C). The increase was most pronounced (by more than a factor of 100) at low temperatures [1974Mat]. Simultaneously, pronounced grain-boundary penetration was observed at low temperatures.

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

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1961Bar]	Metallography, XRD	Up to 1800°C, sections UC-Fe and UC-UFe_2,
[1962Bal]	Metallography, chemical analysis of single-phase specimen, density and microhardness measurements, thermal analysis, XRD	$UFeC_2$ and close compositions
[1963Bri1]	Metallographical and X-ray investigation of annealed mixtures of powders of UC and UC_2 with Fe	Up to 1800°C, sections UC-Fe and UC-UFe_2
[1963Bri2]	Metallographical and X-ray investigation of annealed mixtures of powders of UC and UC_2 with Fe	Up to 1950°C, sections UC-Fe, UC-UFe_2 and UC_2-Fe
[1963Nic]	Thermal analysis, metallography	Up to 1700°C, U_6Fe-UC-UC_2-Fe composition region
[1971Guh]	Metallography, XRD	Section UC-Fe
[1973Tan]	emf	$UFeC_2$ compound, 722 to 811°C
[1986Ger]	Powder XRD	Crystal structure of $UFeC_2$
[1990Ale]	XRD	X-ray data and temperatures of formation of "$U_2Fe_2C_3$" and "$U_3Fe_2C_5$"
[1992Ale]	Metallography, XRD, EPMA	UC-UC_2-Fe region: UC-UC_2-$UFeC_2$ at 1400°C, other at 1050°C
[1995Wac]	XRD	Crystal structure of "$U_2Fe_2C_3$" phase

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(C) (graphite) < 3827	*hP*4 $P6_3/mmc$ C (graphite)	a = 246.12 c = 670.90	at 25°C [Mas2] sublimation point
(δFe) 1538 - 1394	*cI*2 $Im\bar{3}m$ W	a = 293.15	[Mas2]
(γFe) 1394 - 912	*cF*4 $Fm\bar{3}m$ Cu	a = 364.67	at 915°C [V-C2, Mas2]
(αFe) < 912	*cI*2 $Im\bar{3}m$ W	a = 286.65	at 25°C [Mas2]
(γU) 1135 - 776	*cI*2 $Im\bar{3}m$ W	a = 352.4	[Mas2]
(βU) 776 - 668	*tP*30 $P4_2/mnm$ βU	a = 1075.9 c = 565.6	[Mas2]
(αU) < 668	*oC*4 *Cmcm* αU	a = 285.37 b = 586.95 c = 495.48	at 25°C [Mas2]
UC_{1+x} < 2530 (at $x \approx 0$) 2585 - ~2100 (at $x \approx 1$)	*cF*8 $Fm\bar{3}m$ NaCl	a = 496.1 a = 507.5 a = 550.4	x = 0, room temperature [V-C] x = 0, at 2100°C x = 0.9 at 2100°C [1972Ben]
βUC_2 ~2100 - 1768	*cF*12 $Fm\bar{3}m$ CaF_2?	a = 547.5	[2001Che] actually, "βUC_2" phase represents the UC phase in equilibrium with graphite [2001Che]
αUC_2 1793 - 1516	*tI*6 *I*4/*mmm* CaC_2	a = 355.2 c = 598.8	[V-C2] may be stabilized by alloying with Fe up to 1050°C [1992Ale]
U_2C_3 1823 - 850	*cI*40 $I\bar{4}3d$ Pu_2C_3	a = 807.4	[V-C2]
UFe_{2-x} < 1228	*cF*24 $Fd\bar{3}m$ $MgCu_2$	a = 707.2 a = 706.3	x = –0.137 x = 0.048, [V-C2]
U_6Fe < 795	*tI*28 *I*4/*mcm* Mn_6U	a = 1028.63 c = 524.10	[V-C2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* τ_1, $UFeC_2$ < 1615 ± 10	*tP*8 *P*4/*mmm* $UCoC_2$	*a* = 349.44 ± 0.06 *c* = 738.9	[V-C2]
* τ_2, $U_{\sim 32}Fe_{\sim 17}C_{\sim 51}$ < 1500 ± 100	*t***	*a* = 500.7 *c* = 508.4	stoichiometry $U_3Fe_2C_5$ [1990Ale] stoichiometry $U_{\sim 32}Fe_{\sim 17}C_{\sim 51}$ [1992Ale]
* τ_3, $U_{30.6}Fe_{8.6}C_{60.8}$ (≥1400) - (>1150)	-	-	[1992Ale]
* τ_4 $U_{11}Fe_{12}C_{18}$ < 1197	*cI*82 $I\bar{4}3m$ $Th_{11}Ru_{12}C_{18}$	*a* = 1006.8	[1990Ale], [1995Wac]

Table 3: Invariant Equilibria

Reaction	*T* [°C]	Type	Phase	Composition (at.%)		
				C	Fe	U
l + UC_2[a)] ⇌ $UFeC_2$	1615±10	p_1	L	48.8	26.8	24.4
			$UFeC_2$	50	25	25
l ⇌ UC + UFe_2	1201	e_1 (max)	L	3.8	61.7	34.5
			UC	~ 50	~ 0	~ 50
			UFe_2	~ 0	~ 66.7	~ 33.3
l ⇌ $UFeC_2$ + (γFe)	1160	e_2 (max)	L	14.8	70.4	14.8
			$UFeC_2$	50	25	25
			(γFe)	~ 0	~ 100	~ 0
l ⇌ UC + (γFe)	1117	e_3 (max)	L	48.8	26.8	24.4
			UC	~ 50	~ 0	~ 50
			(γFe)	~ 0	~ 100	~ 0
L ⇌ UC + (γFe) + UFe_2	1037	E_1	UC	~ 50	~ 0	~ 50
			(γFe)	~ 0	100	~ 0
			UFe_2	~ 0	~ 66.7	~33.3
L ⇌ UC + UFe_2 + U_6Fe	720	E_2	UC	~ 50	~0	~ 50
			UFe_2	~ 0	~ 66.7	~ 33.3
			U_6Fe	~ 0	~ 7.7	92.3

a) note: contains some (not determined exactly, probably about 3 mass%) Fe

Table 4: Thermodynamic Data of Reaction or Transformation

Reaction or Transformation	Temperature [°C]	Quantity, per mol [kJ, mol, K]	Comments
(αFe) + (βU) + 2C(gr) ⇌ $UFeC_2$	722-778	$\Delta G = -139.03 + 0.01448T$	emf [1973Tan]
(αFe) + (γU) + 2C(gr) ⇌ $UFeC_2$	778-811	$\Delta G = -146.65 + 0.02172T$	emf [1973Tan]

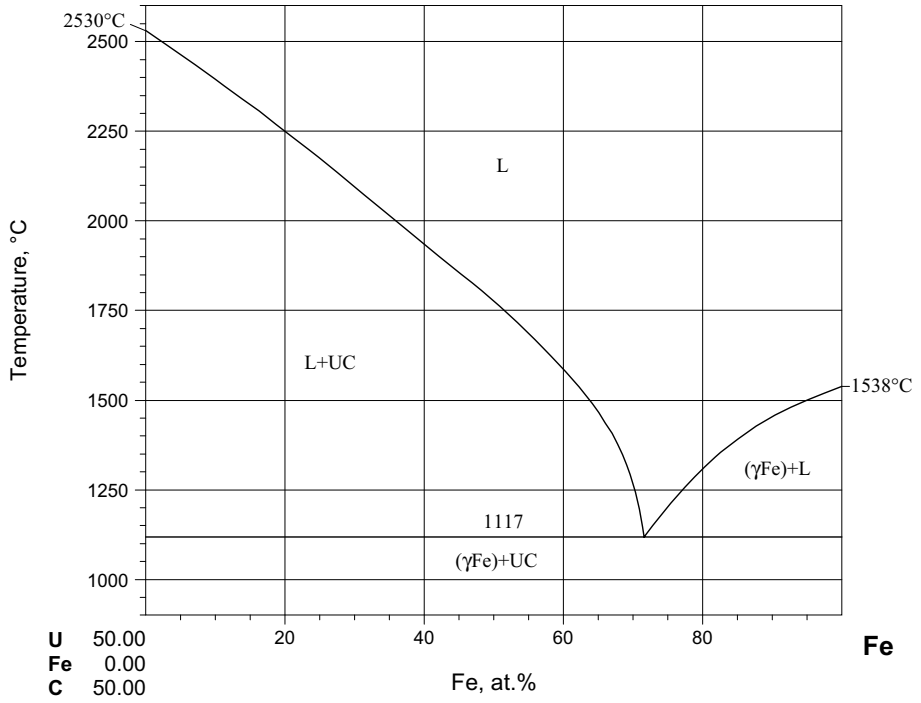

Fig. 1: C-Fe-U. The UC - Fe quasibinary section

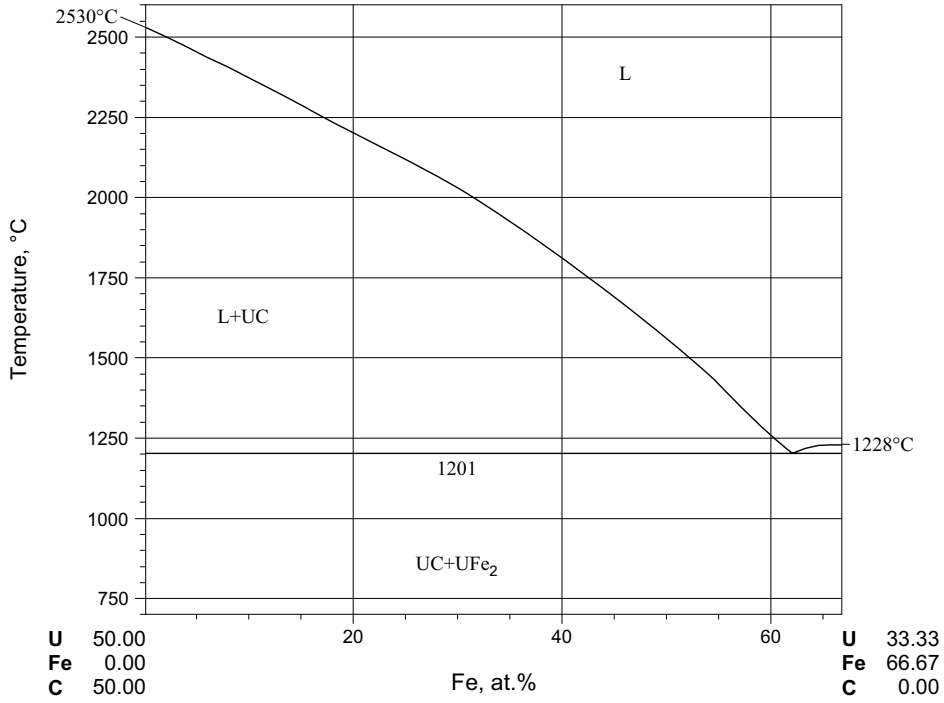

Fig. 2: C-Fe-U. The UC - UFe_2 quasibinary section

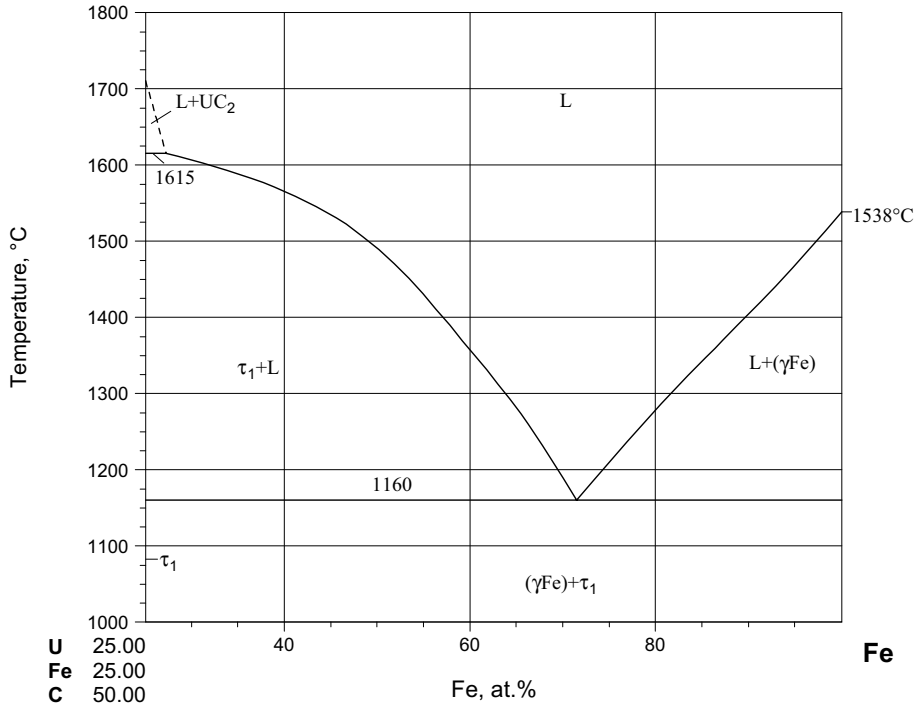

Fig. 3: C-Fe-U. The $UFeC_2$ - Fe quasibinary section

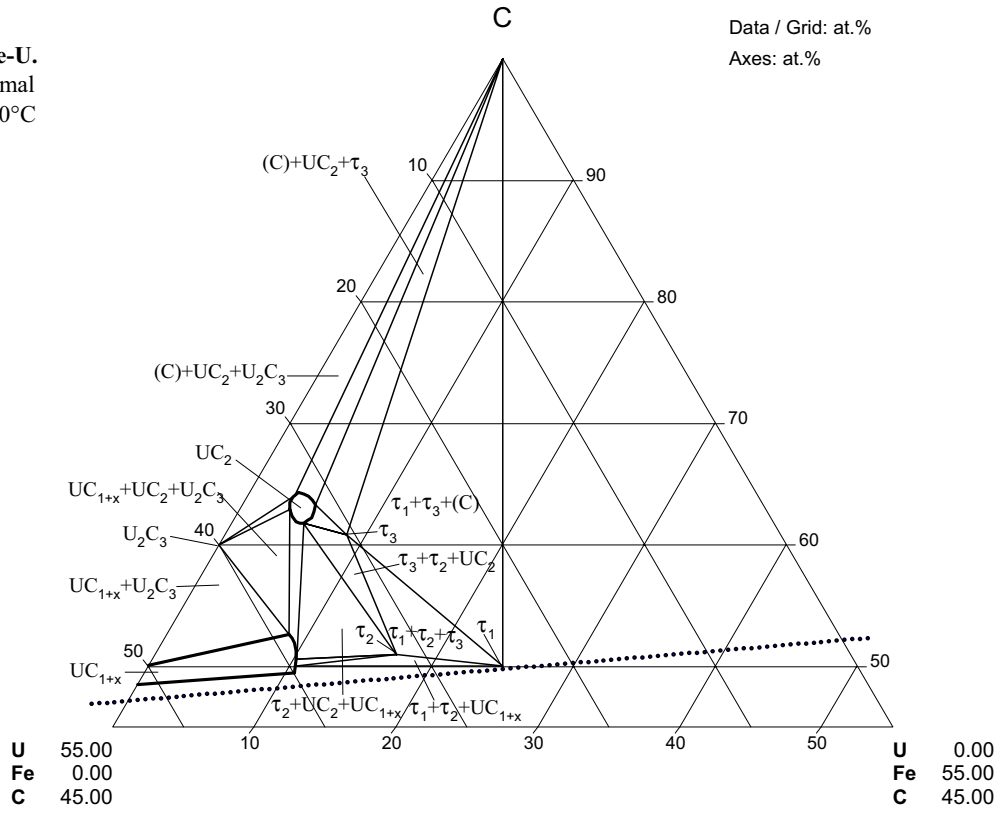

Fig. 4: C-Fe-U. Partial isothermal section at 1400°C

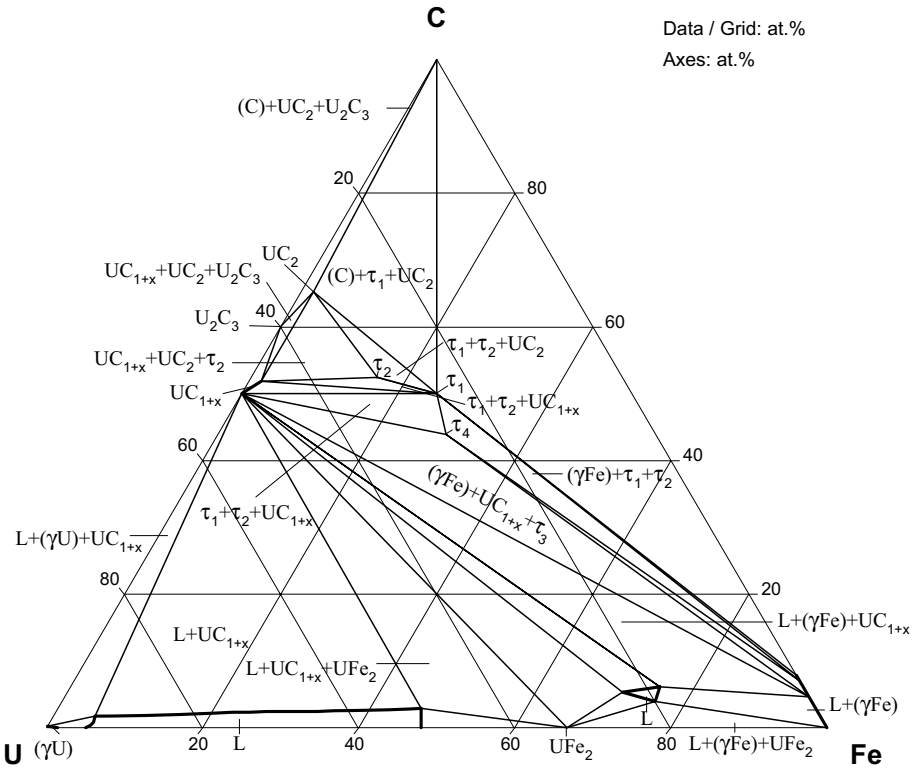

Fig. 5: C-Fe-U.
Isothermal section at 1050°C