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Critical review of carbon monoxide pressure measurements in the uranium–carbon–oxygen ternary system

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Abstract

For high temperature reactors (HTR), the high level of fuel operating temperature in normal and accidental conditions requires to predict the possible chemical interactions between the fuel components. Among the concerns of the TRISO fuel particle thermomechanical behavior, it is necessary to better understand the carbon monoxide formation due to chemical interactions at the UO_2 kernel and graphite buffer's interface. In a first step, the thermodynamic properties of the U–C–O system have to be assessed. The experimental data from literature on the equilibrium CO gas pressure measurements in the UO_2–UC_2–C ternary section of the U–C–O system are critically reviewed. Discrepancies between the different determinations can be explained – (i) by the different gaseous flow regimes in the experiments and – (ii) by the location of the measuring pressure gauge away from the reaction site. Experimental values are corrected – (i) from the gaseous flow type (molecular, transition or viscous) defined by the Knudsen number and – (ii) from the thermomolecular effect due to the temperature gradient inside the experimental vessels. Taking account of the selected and corrected values improves greatly the consistency of the original set of measurements.

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1. Introduction

A considered fuel for the Generation IV high temperature nuclear reactors is the TRISO particle. The kernel of this particle is made of UO_2 surrounded by an a priori inert matrix which is constituted of a first layer made of pyrolytic graphite buffer. The high temperature interaction at the interface between UO_2 and the graphite under direct physical contact or via the gaseous phase is taken into account in order to predict the fuel behavior in nominal and accidental conditions. Even if kinetics may play an important role in the gas formation reaction, the knowledge of the thermodynamic data and moreover of the pressure laws in the U–C–O system is necessary to understand the thermomechanical strength of the particle since this thermodynamic limit is the highest pressure that can be reached after a while.

In thermodynamic studies of complex systems, the total equilibrium pressures can be determined by static manometric measurements if the gaseous

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species are not condensable. In case of the U–C–O ternary system, the main gaseous species are CO(g) and CO_2(g) and pressure measurements are very often performed at the housing wall under static conditions.

The present work consists in a new critical review of manometric measurements of CO pressure [1], mainly performed in the 1960s and 1970s. Indeed, some discrepancies exist between the different studies. The gauges allow to measure static pressures which can be described by the gas kinetic theory. The gaseous flow – defined by the Knudsen number – and the temperature differential between the heated sample and the manometric measurement point can generate a bias on the values, due to – (i) the nature of the flow (molecular, transitional or viscous) and – (ii) the degree of applying the so-called thermomolecular effect. The knowledge of the experimental vessel geometries allows defining the nature of the flow regime under stationary conditions of the gaseous phase and then to correct the original literature values. In the following, corrections of CO(g) pressures for the monovariant equilibrium $[UO_2 + UC_2 + C]$ are applied to take into account this thermomolecular effect phenomenon.

2. Static pressure measurements on the U–C–O system

In general, the experimental devices are designed to avoid temperature gradients, at least within the sample. Nevertheless, temperature conditions and reactor geometries do not allow installing the gauge in the vicinity of the furnace. Actually, pressures are measured on the housing and not near by the furnace, as shown in the case of Piazza and Sinnott apparatus [2].

Pressures are measured with McLeod gauges, with Hg columns and cathetometers [3–5] or with thermocouples or Pirani gauges calibrated with McLeod gauges [2,6]. More sensitive devices, like capacitance manometers, have also been used later [7,8]. However, several stages of calibration have to be performed with these thermal gauges. Indeed, Pirani gauges need to be calibrated with Hg columns and the same gas because their response depends on the nature of the gas. For electronic reasons, the same procedure must be applied to capacitance gauges which are also calibrated with classical manometers.

These several kinds of gauges used to measure equilibrium CO pressures in the U–C–O system have their advantages and drawbacks. McLeod gauges do not detect condensed vapours because these gases do not reach the McLeod gauge and cannot give a continuous reading of pressure values. Furthermore, because of the gaseous diffusion phenomenon linked to thermal conditions, pressures are not really stable below 10^{-2}–10^{-3} Pa. Thermal gauges (Pirani, thermocouple) also have great disadvantages. Reliability of these gauges, especially in the low pressure range, can become impaired by surface condition changes of the hot wire due to oxidation, gas adsorption or back-streaming of vacuum pump oil. It is difficult to maintain a stable 'zero pressure level'; this is the reason why these gauges are unable to accurately measure pressures below 10^{-2} Pa.

Some of these problems can be solved by appropriate calibration [9–11], but the high uncertainty levels in the low pressure operating range make the choice of a device a critical point. Indeed, the degree of accuracy of the gauge defines the reliability of the thermodynamic data obtained with these instruments. The pressure ranges and the inherent uncertainty of measurements of different manometers used to study equilibrium pressure of the U–C–O system are represented in Table 1. These values are independent of the reactor device and furnace geometries.

Experimental thermodynamic studies are usually performed in such a manner that equilibrium gas pressures upon a chemical system are well established. They involve a physical interaction between a detector and the gaseous phase via the molecular

Table 1
Validity range and inherent uncertainty of measurements of different manometers

Gauge	Pressure working range (Pa)	Detection limit (Pa)	Uncertainty (Pa)
Hg column and cathetometer	$2.6–1.3 \times 10^5$	2.6	1.3×10^{-1}
Mac Leod	$1.3 \times 10^{-3}–1.3 \times 10^2$	1.3×10^{-3}	1.3×10^{-2}
Thermocouple	$1.3 \times 10^{-1}–1.3 \times 10^2$	1.3×10^{-1}	1.3×10^{-1}
Pirani	$1.3 \times 10^{-2}–1.3 \times 10^3$	1.3×10^{-2}	1.3×10^{-2}

motion described by the gas kinetic theory. These measurements need a good control of temperature and pressure conditions which must stay homogeneous inside the whole measured volume. In case of temperature gradients between the pressure source and the measuring point, the thermomolecular effect has to be considered.

3. Thermomolecular effect

The molecular flow regime occurs for a Knudsen number (Kn) larger than 1/3 [12]. Kn is defined as the ratio of the mean free path of gaseous molecules (λ) to the tube diameter (d) in which the flow occurs (1):

$$Kn = \frac{\lambda}{d} = \frac{k_B T}{p\pi\sigma^2 d\sqrt{2}}, \tag{1}$$

where k_B is the Boltzmann's constant and σ the collision cross-section of the molecules.

For an equilibrated (steady-state) chemical system under molecular flow regime, a relation based on the Knudsen effusion principle determines the pressure deviation between two vessels at different temperatures and separated by an orifice. Furthermore, in case of a transition state ($3 \leqslant 1/Kn \leqslant 80$) the same correction can be applied by introducing a proportionality coefficient between molecular and viscous (laminar) flows [13,14]. For lower values of the Knudsen number, the viscous state is not considered as turbulent because ingoing and outgoing flows result from thermal diffusion and both are equal and small and hence mechanical equilibrium of total pressures is established. However, in case of a mixture of species in the gas, a composition gradient will exist. This phenomenon due to vapour phase thermochemical diffusion can be taken into account [2,15].

3.1. Case of the molecular flow regime

In the case of measurements performed with vessels built like effusion cells in a vacuum housing under static and stationary conditions, the Hertz–Knudsen relation for the gaseous carbon monoxide (CO) flow (F) in one direction from the sample through the orifice into the vessel can be applied (2):

$$F = \frac{C \cdot p}{\sqrt{2\pi MRT}}. \tag{2}$$

In this relation (Eq. (2)), C is the orifice conductance, M the molar mass of the gaseous species, p its pressure above the sample, R the gas constant. Indeed, in case of a stationary gaseous flow (F_s) setting out of the heated sample at temperature T to the housing in vacuum is equal to the reverse flow (F_r) coming from the measuring point (or housing) at room temperature ($T_0 = 300$ K) [16]. This equality between the flows is expressed by (3) which is the so-called thermomolecular relation:

$$\frac{C \cdot p_{CO}(T)}{\sqrt{2\pi MRT}} = \frac{C \cdot p_{CO}(T_0)}{\sqrt{2\pi MRT_0}}. \tag{3}$$

Here, C is the total intermediary conductance between the sample and the housing.

This conductance is flow direction independent and unaffected by the temperature distribution provided that the gaseous species do not decompose. The previous equation can be reduced to the following expression (4) which refers to the impact of the thermomolecular effect on a pressure measurement as it is shown in Fig. 1

$$p_{CO}(T) = p_{CO}(T_0)\sqrt{\frac{T}{T_0}}. \tag{4}$$

Each specific Knudsen number related to an experimental geometry has to be calculated to determine the flow regime limits (molecular, transitional or viscous). So, the characteristic dimensions of

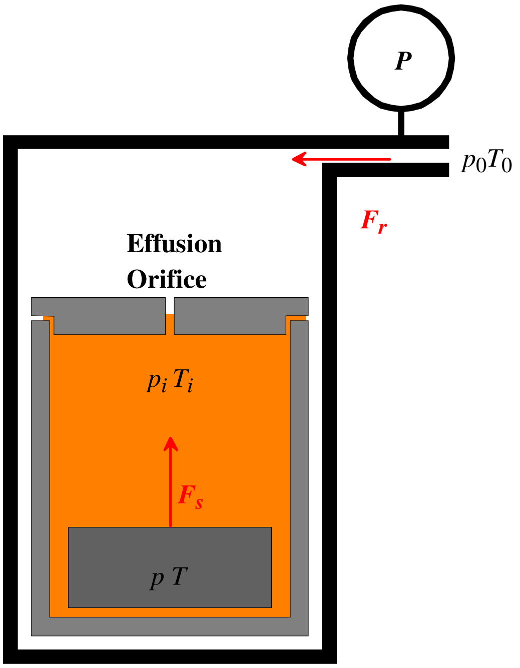

Fig. 1. Thermomolecular effect principle for an intermediary orifice (p_i, T_i) between a sample (p, T) at high temperature and a manometric gauge at room temperature (p_0, T_0).

tubes whose conductance governs the difference over the sample and that in the pressure gauge need to be evaluated. The pertinent design data is either directly taken from the examined papers when sufficiently detailed descriptions are given or estimated from plans of the experimental setups. In case of a gaseous flow only constituted of carbon monoxide, whose molecular diameter is very close to that of air [12], the mean free path (L_m in m) is expressed by the following relation (5):

$$L_m = \frac{2.213 \times 10^{-5} T}{p}, \tag{5}$$

with L_m in m, and p in Pa.

3.2. Case of the transition regime

If ($3 \leqslant 1/Kn \leqslant 80$), the transition flow regime can be treated as the combination of a molecular (F_{mol}) and a viscous (F_{vis}) flow. The θ coefficient is the contribution of the molecular fraction of the flow. For a given effusion orifice, the transition flow is expressed by Santeler's relation which is also valid for a tube geometry [13]. The values of θ are simply calculated using the expression proposed by De Muth and Watson [14].

This empirical model predicts accurately the flow behavior through an orifice and has been evaluated by volumetric measurements performed on ambient air. It relates the θ coefficient to the Knudsen number (6):

$$\theta = 1.05^{-(1/Kn)}. \tag{6}$$

In case of a conventional flow through a tube with a fixed upstream pressure and a pump regulated downstream pressure the total flow in the transitional regime (F_{trans}) can be considered as the contribution of two parallel flows (7). The thermomolecular effect which expresses a thermal force can be compared to a pressure force where the gradient is fixed by pumping

$$F_{trans} = F_{vis} \times (1 - \theta) + \theta \times F_{mol}. \tag{7}$$

From the measuring point at p_0 pressure and T_0 temperature a viscous/diffusional flow transport a fraction of the gaseous molecules to the source (under p and T conditions) with a zero pressure difference due to mechanical equilibrium. This viscous flow generates a p_0 pressure but only for the $(1-\theta)$ part. The thermomolecular effect leads the θ molecular part of the flow and generates a pressure proportional to the square root of the temperature ratio between the two points. The total pressure (p) over the sample is expressed by the sum of these two contributions and the obtained relation will be used to correct the experimental pressure measurements $p(T_0)$ (8):

$$p(T) = (1 - \theta) \times p(T_0) + \theta \times p(T_0)\sqrt{\frac{T}{T_0}}. \tag{8}$$

4. Corrections of carbon monoxide pressures measured for the [UO_{2-x} + UC_2 + C] three-phase domain

This section presents the results for the thermomolecular effect corrections on experimental CO pressure measurements in the U–C–O ternary system. In the Generation IV – high temperature reactor (HTR), the thermomechanical behavior of the TRISO fuel particle is directly affected by the CO pressure values. Indeed, the [$UO_{2-x} + UC_2 + C$] monovariant equilibrium directly illustrates the high temperature interaction between the $UO_{2\pm x}$ fuel kernel and the pyrolitic graphite layer of the TRISO fuel particle since CO created in the gas phase let the uranium oxide become hypo-stoichiometric [17].

The critical analysis of literature data is performed on mass-spectrometric measurements [18] and on manometric pressure determinations either by applying the thermomolecular effect correction for data measured in the molecular flow regime or using our formula (8) for those performed in the transition flow regime [2,3,5,19–21]. For all the studies considered (see Tables 2 and 3, Figs. 2(a)–(f) and 3), our corrections are justified by analysis of the experimental device and of the conditions for measurements.

As a general rule, the gas phase composition was either analysed directly by mass spectrometry [18] or following the mass spectrometric analysis of gas. Volumes sampled during experiments [2,7] was mainly composed by CO molecules and traces of other components (H_2, CO_2, CH_4) when identified were attributed to the furnace degassing.

4.1. Khromov et al. determinations

This study deals with interactions between a $UO_{1.9995}$ kernel and a porous pyrocarbon coating deposited by precipitation from the gas phase in order to provide a plenum storage volume preventing a too high pressure increase inside the fuel particle. CO pressure measurements were performed

Table 2
CO pressure laws after correction of experimental values for the UO_2 (s) + 4C (s) ↔ UC_2 (s) + 2CO (g) three-phase equilibrium

Authors	Temperature range (K)	Geometry and orifice diameter (mm)	$1/Kn$ range	Flow regime	Equilibrium CO pressure (Pa)
Khromov	1500–1800	Long tube 2.0	11–1614	Transitional Viscous	$\log p_{CO} = 14.732 - \frac{19\,980}{T}$
Besmann and Lindemer	1473–1823	Long tube 2.6	4×10^{-4}–67	Molecular Transitional	$\log p_{CO} = 14.266 - \frac{19\,575}{T}$
Piazza and Sinnott	1714–1953	Effusion cell 2.0	21–351	Transitional Viscous	$\log p_{CO} = 13.082 - \frac{17\,710}{T}$
Hennecke and Scherff	1623–1773	Effusion cell 2.0	5–24	Transitional	$\log p_{CO} = 12.564 - \frac{16\,714}{T}$
Heiss and Dodé	1673–2063	Open crucible 2.0	7–2126	Transitional Viscous	$\log p_{CO} = 14.383 - \frac{19\,946}{T}$
Heiss	1703–2140	Open crucible 2.0	14–1891	Transitional Viscous	$\log p_{CO} = 13.501 - \frac{18\,599}{T}$
Lorenz, Scherff and Toussaint	1550–1730	Effusion cell 2.0	1–28	Molecular Transitional	$\log p_{CO} = 12.626 - \frac{16\,824}{T}$
All authors except Khromov	1473–2140	–	–	Molecular Transitional Viscous	$\log p_{CO} = 13.528 - \frac{18\,525}{T}$

Table 3
Pressure range and proposed uncertainties of the present analysed works

Authors	Gauge	Pressure range (Pa)	Authors uncertainty	Retained uncertainty
Khromov	Mass spectrometry	0.4–10^3	±35%	±35%
Piazza and Sinnot	Mac Leod thermocouple	499–8.37×10^3	±13.33 Pa	±3%
Hennecke and Schreff	Hg manometer	94.5–434	±2%	±3%
Heiss	Hg manometer	270–69.5×10^3	±1%	±3%
Heiss and Dodé	Hg manometer	133–4.9×10^4	±13.33 Pa	±3%
Besmann and Lindemer	Capacitance	4.5×10^{-3}–10^3	±0.03%	±2%
Lorenz	Mac Leod	30–550	–	±3%

The retained uncertainty corresponds to the values used to calculate the linear fits of CO pressure.

in the 1423–1723 K range with an excess of carbon according to the global reaction $UO_2 + 4C \leftrightarrow UC_2 + 2CO$.

Using of graphite coated particles as samples provides conditions closer to reality than pressed pellets made of a mixture of the two powders. However, the author observed after the experiments that the graphite coatings broke down in approximately one-third of the particles. The samples are loaded in a 25 mm height and a 15 mm diameter Knudsen cell. The effusion orifice was replaced by a long tube, at the end of which an Omegatron type mass-spectrometer was placed [22] in an own housing. As stated by the authors, the analysis of the gas composition was carried out with a pressure uncertainty of ±35% due to the calibration procedure. The spectrometer was previously calibrated by using the decomposition pressure of titanium nitride (TiN) into gaseous nitrogen (N_2) as a standard. As pressure values are close to those obtained in the U–C–O system, the thermomolecular effect due to the gas transfer through a 2 mm diameter tube was taken into account in the calibration function of the spectrometer. Thus we do not need to apply any correction to these data.

4.2. Piazza and Sinnot determinations

This work establishes phase relations in the U–C–O system involving graphite, uranium dioxide and the carbides [2]. The graphite reaction vessel is heated by induction and pellet samples are contained in a graphite crucible. These pellets are made of finely ground and mixed uranium dioxide and graphite powders pressed with a binder made of paraffin in toluene. Prior to the experiments, the pellet was heated slowly to about 1473 K under vacuum to volatilize the binder and adsorbed gases.

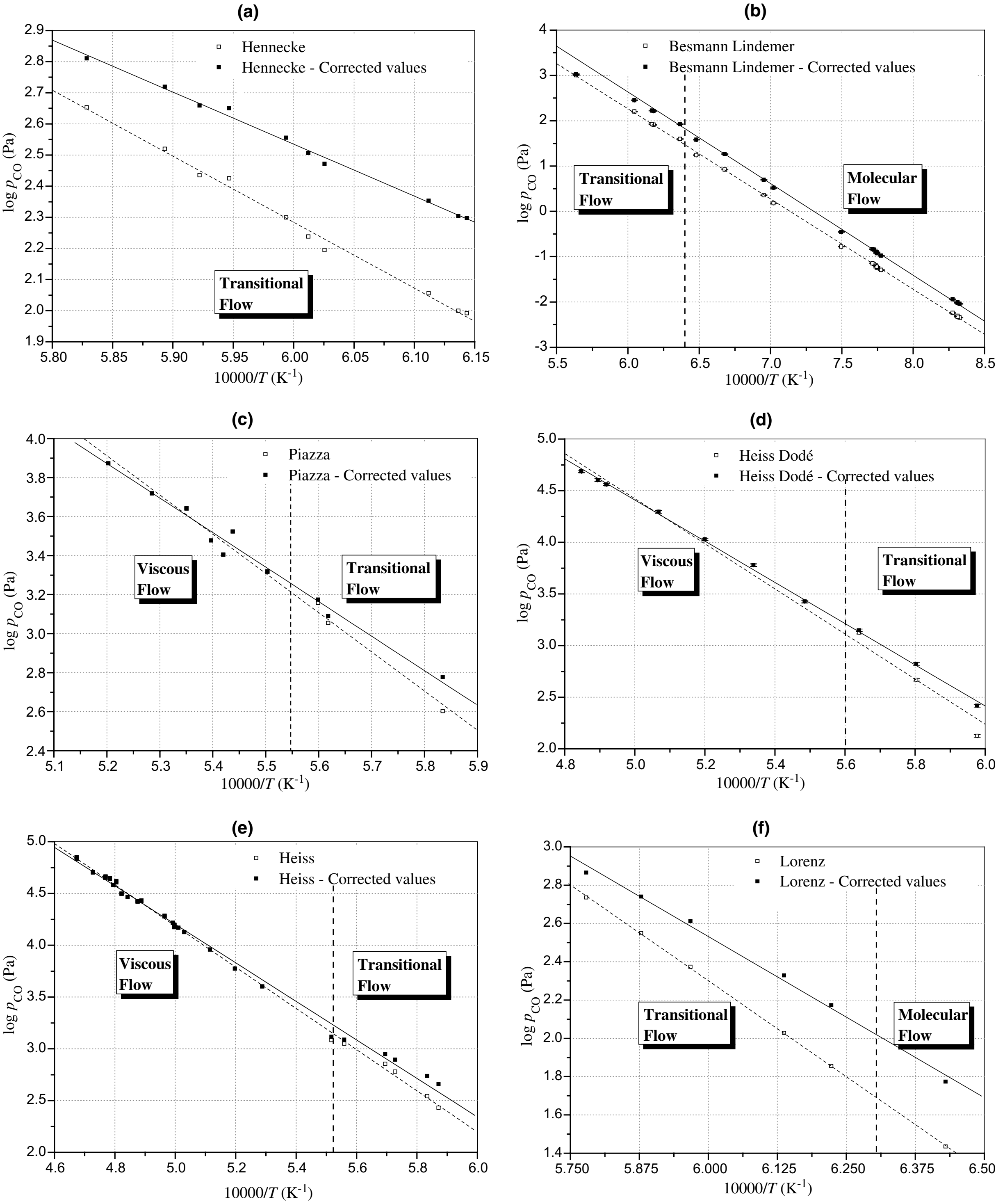

Fig. 2. CO pressure values for the monovariant equilibrium [UO_{2-x} + UC_2 + C] for each analysed study. □, Original CO pressure values – ■, corrected CO pressure values: (a) Hennecke, (b) Besmann Lindemer, (c) Piazza, (d) Heiss Dodé, (e) Heiss, and (f) Lorenz.

However, this preliminary heating can already induce a reaction between UO_2 and C.

Like in a few other studies, pressure measurements are performed with both thermocouple or

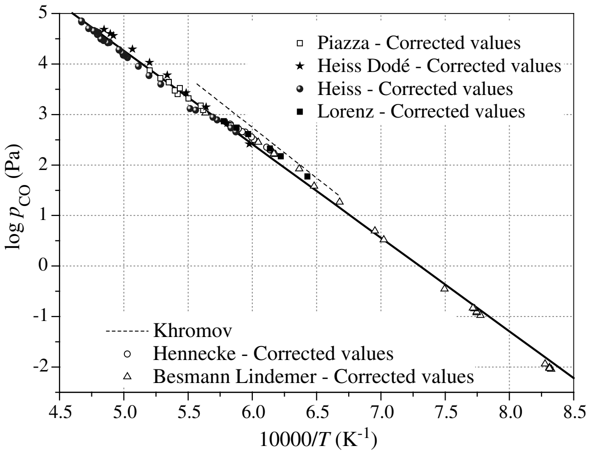

Fig. 3. Corrected CO pressure values for the monovariant equilibrium $[UO_{2-x} + UC_2 + C]$ and pressure law obtained by fitting all retained experimental data (except Khromov et al.) in the 1473–2140 K temperature range.

McLeod gauges with cold traps inserted in line according to the considered pressure range. In this last case, using of a cold trap can generate a ≈25% error on the measurements mainly at lower pressures which has to be considered in the uncertainties. This bias is due to thermal and molecular diffusion of the mercury vapour toward the cold trap from its storage vessel in the McLeod gauge through the gas to be measured, carrying away some of this gas from the measuring bulb of the gauge [10,11]. Furthermore, mass spectrometric analysis of the gaseous phase samples had revealed some impurities (H_2, O_2, CO_2, Ar, N_2). As a result, Piazza and Sinnott applied a correction mainly due to hydrogen vapour because of the gas concentration gradient caused by thermal diffusion. Indeed, this steady-state diffusion process concentrates light and volatile species (H_2) in the high temperature part of the system close to the sample.

Knowledge of the geometry of the vessel allows us to take into account the correction of the thermomolecular effect (Table 2, Fig. 2(c)) for temperatures lower than 1800 K according to an effusion orifice of 2 mm diameter that produces a transitional flow regime. Corrected pressure values lead to new parameters of the CO(g) pressure law.

4.3. Hennecke and Scherff determinations

This carbothermic reduction reaction study [19] is similar to earlier CO pressures measurements of Piazza and Sinnot [2] since the experimental vessel and the manometric devices are based on this previous work. So, the thermomolecular effect is corrected accordingly. The Knudsen number defines a transition regime for a more extended temperature and pressure range not studied previously by Piazza and Sinnot. After correction, we can observe a higher impact of the thermomolecular effect for pressure values measured in the transition regime (Table 2) and thus the results of both studies are in good agreement (Fig. 2(a)).

4.4. Heiss and Dodé and Heiss determinations

Several studies of these authors [3,5] deal with the chemical composition evolution of high temperature UO_2 and graphite powder mixtures. Indeed, a thorough understanding of the U–C–O system was necessary in view of establishing the best synthesis route of uranium carbide or oxycarbide by the carbothermic reduction reaction. The experiments were performed in tubular furnaces. The graphite crucible had a 10 mm diameter and 25 mm height, the size of the effusion orifice was estimated from the scale plans of the experimental apparatus to have a size of 2 mm. In both studies, the flow regime is first transitional then viscous for temperatures above 1800 K (Fig. 2(d) and (e)).

In each case, the equilibrium pressures are easily obtained in both direct and reverse directions of the reaction $UO_2 + 4C \leftrightarrow UC_2 + 2CO$ by CO pressure increments as long as graphite still exists in the mixture. But, as soon as the whole carbon is consumed into UC_2 and the pressure deviates from the plateau, it becomes impossible to go in the reverse direction even if an excess additional CO pressure is imposed to the system. These findings are in accordance with Besmann's conclusions who pointed out the high kinetic influence and the necessity of adding 10% of H_2 in CO in order to obtain fast chemical rates starting from a pure phase [8]. Furthermore, none strictly horizontal plateaus of CO pressures are observed when thick pellets (10 mm) were used. This phenomenon is due to diffusion related effects in the pellets and was not observed for thinner ones (5 mm).

4.5. Besmann and Lindemer determinations

This study deals with the carbothermic reduction reaction [7]. During this synthesis route of actinide carbides, the first formed three-phase domain contains the slightly hypo-stoichiometric dicarbide

$UC_{1.91}$, uranium dioxide UO_2, and graphite. The samples were made of a mixture of packed powders of UO_2 and graphite or partially reacted mixtures containing $UC_{1.91}$, UO_2, and graphite. Specimens were initially heated under vacuum to approximately 1700 K to remove residual moisture in the sample and any hyper-stoichiometry in the dioxide.

The experimental vessel is a 300 cm^3 high vacuum housing. The pellets are charged into a 20 mm long with a 3.2 mm outer diameter graphite tube. To calculate the flow regime, the inner part of the tube diameter is estimated to be 2.6 mm based on a scale plan of the furnace. The flow is molecular except for the main part of the analysed pressure range. The flow requires a correction due to a transition regime for temperatures higher than 1550 K (Fig. 2(b)).

4.6. Lorenz et al. determinations

Though this work deals with the carbothermic reduction of protactinium oxide, some additional measurements were also performed on the U–C–O system [21]. A pelletized mixture of U_3O_8 and graphite was used as starting material. First of all, it was necessary to reduce U_3O_8 into UO_2 by a temperature step performed in the 973–1273 K temperature range under vacuum. The reaction was carried out in a graphite crucible contained in a heated vacuum furnace which was closed during the measurements, this apparatus being identical to the device used by Hennecke [19]. The corrections are thus calculated taking into account the same vessel geometry. For lower temperatures, the Knudsen number first defines a molecular regime. Then, for temperatures higher than 1585 K, the gaseous flow becomes transitional (Fig. 2(f)).

These CO pressures measurements obtained by Lorenz are close to the values of Hennecke who have worked in nearly the same temperature range (Table 2). Furthermore, these results approach the extrapolated line calculated from Piazza and Sinnott results at higher temperatures (Fig. 3).

5. Conclusion

The whole set of analysed CO pressure measurements in the U–C–O system is listed in Table 3. Knowledge of the devices used to perform the individual CO pressure measurements allows specifying for each study the working range and its associated uncertainty. Moreover, taking into account the nature of the gas and the flow regime inside the experimental vessel makes it possible to correct the measured CO pressure values with established formulae. These corrections are based on the kinetic theory of gas. The applied corrections account for – (i) the thermomolecular effect at very low pressures and – (ii) the transition flow regime at higher pressures, both effects being induced by material steady-state flow between the hot source and the measuring point. The present critical review of the CO equilibrium pressure data over the U–C–O system shows a considerable influence of the corrections on the pressure measurements. The proposed new set of data presents much smaller discrepancies than the original one and enables us to establish new CO pressure values for the $[UO_{2-x} + UC_2 + C]$ three-phase equilibrium over a wide temperature range (Fig. 3). The law obtained by fitting all the data is reliable not only because it is established from a large number of experimental data points but also because the applied corrections have improved the consistency among the original data whose validity has been essentially confirmed (Table 2). The present work improves noticeably the thermodynamic description of the U–C–O system at high temperature. The new set of data can also be used as a reference for the study of the thermomechanical interaction of the UO_2 kernel and the pyrocarbon buffer in the TRISO fuel particle for high temperature Generation IV nuclear plants. Indeed, even if the analysed experiments were performed to establish a thermodynamic description of the $UO_2 + 4\ C \leftrightarrow UC_2 + 2CO$ gas phase reaction, some kinetic aspects were highlighted [3,5,8] and are to be considered in further applications to the kernel fuel behavior.

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