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SOME EXPERIMENTAL RESULTS ON THE SORPTION OF CAESIUM BY TUNGSTEN AND GRAPHITE

by

G.F. DE BENI, O. GAUTSCH and H. WAHL

1970



Joint Nuclear Research Centre Ispra Establishment - Italy

Physical Chemistry

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Commission of the European Communities Joint Nuclear Research Centre - Ispra Establishment (Italy) Physical Chemistry

Luxembourg, November 1970 - 22 Pages - 7 Figures - B. Fr. 40.-

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The Cs saturation loadings on the W-powder ranged at different temperature and pressure conditions, from 1.0 - 1.4 mgCs/gW. The sorbed Cs was strongly fixed to the powder and to obtain at a given constant temperature a release of 0.1 mgCs/gW pressure decreases of more than $20 \,^{0}/_{0}$ were required. For a loading of 1.2 mg/g vapour pressures of 1-10 mm Hg were obtained in the temperature range of 600 to 800°C. The low loadings and their small changes may result in difficulties for a practical use in a converter.

The graphite samples attained saturation loadings of up to 1500 mg/g and the loading isotherms showed transition stages corresponding to compounds between $C_{24}C_S$ and $C_{10}C_S$ roughly. In the temperature range of 700-800°C these compounds had vapour pressures between 1 and 10 mm Hg. For a pressure decrease of 20 % Cs amounts of 10 to 200 mg/g were released. The pressure, temperature and loading characteristics of the Cs-graphite systems are more suitable for a practical use in a converter than those of the tested W-powder.

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ABSTRACT

A submicron W-powder, a nuclear graphite and a pyrolytic graphite sample were loaded with Cs at sample temperatures between 600 and 800°C and Csvapour pressures from about 0.01 to 30 mm Hg to see whether such systems could be used as Cs-reservoirs in thermionic converters. The amounts of Cs sorbed by the samples at a given temperature as a function of time, were measured by a radioactive tracer method using Cs¹³⁴. From these sorption curves the saturation loadings and the corresponding equilibrium pressures of the Cs-vapour were determined.

The Cs saturation loadings on the W-powder ranged at different temperature and pressure conditions, from 1.0 - 1.4 mgCs/gW. The sorbed Cs was strongly

KEYWORDS

CAESIUM TUNGSTEN DIFFUSION PYROLYTIC GRAPHITE SAMPLING THERMIONIC CONVERSION TEMPERATURE VAPOUR PRESSURE POWDER METALLURGY RESERVOIRS

1) Introduction *)

The purpose of this work was to measure the quantity of Cs sorbed by W and graphite and the Cs-vapour pressure of such systems, to be possibly used as Cs- reservoirs in thermionic converters (1) (2). Such reservoirs should at given operating temperatures maintain an automatically self-regulating Cs pressure of 1-10 mm Hg, to assure optimum converter operation without external control. They also must supply sufficient Cs to the converter volume to compensate for Cs losses which occur as a result of reaction with or adsorption on internal surfaces of the converter.

Earlier work (1) (2) (3) had identified W and graphite as interesting materials. Therefore for the present experiments a submicron W-powder of high specific surface and graphite, which forms interlamelar compounds with Cs, were selected. The Csloadings at saturation and the corresponding equilibrium Cs-vapour pressures were determined for sample temperatures of 600 to 800° C and Cs pressures of about 0.01 - 30 mm Hg by adsorption and desorption measurements.

2) Apparatus and procedure

The sorption apparatus is shown in fig. 1 and was in principle similar to those used by others during earlier work (1) (2) (3). The SS-flange was sealed with a Ni-gasket and contained a Nb-capsule, filled with about 400 mg of purified Cs and closed by cold welding. Each of the two Cu-tubes (fig. 1) had several holes into which thermocouples were inserted. The Ni-tubes outside the furnaces were wound with thermocoax heating wires, to keep their temperature during the measurements above that of the Cs. A spacer bolt (fig. 1) was applied to fix the position of the sample chamber with respect to a gamma ray scintillation counter. A high vacuum installation was used to evacuate the apparatus, which was placed under a vacuum bell jar $(10^{-2} - 10^{-3} \text{ mm Hg})$.

The temperature of the furnaces was controlled by a power regulation system, based on the compensation of the thermocouple current, by means of an external mV-source. The calibrated thermocouples for the regulation and measurement of temperatures had maximum deviations of $\pm 2^{\circ}$ C, producing a maximum average error of ± 6 % on the Cs-vapour pressures.

The quantity of Cs stored on the samples was measured by counting the gamma radiation of Cs¹³⁴. Therefore the Cs in the Nb-capsules and the Cs¹³⁴-reference sources were irradiated for 2 hours with a thermal neutron flux of 1,8. 10^{13} n/cm²sec ± 3 % (taking into account the flux depression in the capsule walls). Two gamma ray counters, shielded by 10 cm of lead, viewed either sample or the Cspool through a collimator hole of 10 mm dia, on which the scintillators were centered top-on, to record the Cs¹³⁴ radiation above 0.45 MeV integrally. The Cs¹³⁴-reference sources, which served to transform the counting rate of Cs^{134} obtained from the sample into mg of Cs, were prepared with known amounts of CsNO3. They had the same geometrical shape as the samples and self-absorption was accounted for. From their counting rate (same position as the samples with respect to the scintillator) their specific activity S_a in cpm/mg at the end of irradiation was determined after correction for background radiation and decay. The maximum error from several measurements generally amounted to ± 11 %.

The counting rate of Cs^{134} from the sample, which corresponded to the amount of sorbed Cs, was corrected for background, selfabsorption in the sample and for the decay. After dividing the corrected counting rate by the value of S_a and by the weight of the sample the Cs-loadings in mgCs/g sample were obtained.

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Sample's of known weight were filled into the sample chamber and covered with pieces of a fine-mesh Ni-grid to prevent losses due to a possible blow-up during evacuation. Then the chamber was welded to the rest of the sorption apparatus (fig. 1) and the whole system was outgassed for 48 hours at 450° C, with exception of the sample which was at 850° C and the brazing zone, which was kept at 650° C during the last hour. The final dynamic vacuum was always about $1 \cdot 10^{6}$ mm Hg, improving to $1 \cdot 10^{-8}$ mm Hg upon cooling the system to room temperature. The pre-pinch was then pinched together and the brazing material above it (fig. 1) melted and cooled. After that the vacuum line was closed by means of a UHV-valve and the sample chamber heated to 850° C. Then a leak test was made in static conditions to check the brazing. The resulting leak rates were always about $1 \cdot 10^{-9}$ Torr 1/sec.

The Nb-capsule was opened by counterpinching on the Ni-tube below the SS-flange (fig. 1) and the Cs was distilled into the Cs-pool. Radiation counting and radiographies taken before and after distillation, confirmed that practically all the Cs had moved into the Cs-pool.

The measurements started by heating the sample to $800^{\circ}C$ and the Cs to the desired temperature, and by recording the Cs¹³⁴ - activity on the sample as a function of time. When the Cs-loading reached a saturation value, the temperature of the Cs was either increased (adsorption) or decreased to an other value (desorption). The temperature changes of the sample and the Cs could always be accomplished within 20 minutes at maximum. When enough measurements of saturation loadings were obtained, the sample temperature was decreased and a next sorption-desorption cycle started. The relation between the Cs-vapour pressure and Cs-temperature was taken from the best available data (4).

3) <u>Submicron</u> W-powder

The powder (CIBA) had a specific surface of 5 $m^2/g \pm 20 \%$ which after a heat treatment of 2 hours at 1300°C in a vacuum of about 10^{-5} mm Hg decreased to roughly 0.26 m²/g. The sample, 15.4 g, completely filled the volume of the sample chamber, and was heated to 800°C. The Cs was brought to 260°C, while the rest of the apparatus was kept at 450°C. After having obtained the saturation loading at these conditions other adsorption stages were run through up to Cs-temperatures of 400°C, corresponding to a vapour pressure of 17,7 mm Hg. At the end of the 400°C-step, the desorption stages were started by decreasing in steps the Cs-temperatures and measuring for each temperature the corresponding saturation loadings in cpm/15.4 g. Generally saturation was reached in about 30 minutes. From these results the loadings in mgCs/g were obtained by means of the specific activity, which from several measurements had been determined to $S_{a} = 245 \text{ cpm/mgCs}$ ± 11 %. The same type of measurements was carried out also for sample temperatures of 700°C and 600°C. The results are shown in fig. 2.

The sample was then loaded and unloaded at several constant Cs-pressures for sample temperatures of 600, 700, and 800°C. The results are plotted in fig. 3. A radiography made at the end of the experiment showed a homogeneous distribution of Cs throughout the sample.

It can be deduced from fig. 2 and 3 that there are only small differences in the loadings for the various sample temperatures and Cs-pressures. Possibly the specific surface of the sample had further decreased during the long experimental runs between 800 and 600° C, leading to relatively diminished loadings at 600° C. The highest measured Cs-loading was about 1.4 mg/g, at 800° C and 8 mm Hg of Cs-pressure, corresponding with S = 0.26 m²/g and C = 28 Å²/Cs atom (1)(2) to 5.4 mg Cs/m² of W, or very roughly to 7 monolayers. Also other authors (1)(2) already observed such relatively high loadings even on W-samples with 0.02 m²/g, i.e. less than 9 mg/m² at 800° C and 10 mm Hg of Cs-pressure.

Generally, however, a coverage of 1-2 monolayers was found, therefore the above higher loadings cannot be due to adsorption only. Possibly the oxygen content of the submicron powder (several thousands ppm) is responsible for the relatively high quantity of sorbed Cs.

4) Nuclear Graphite

The sample (Carbone Lorraine) consisted of several cylinders, 2 - 4 mm long, 2 mm in diameter, thermally annealed for 2 hours at 1700° C in vacuum. After this treatment it contained about 50 ppm 0₂ and less than 40 ppm of other elements. An X-ray analysis showed that the nuclear graphite was isotropic and that the degree of Crystallisation, defined as peak width at half-height of the 002 peak, was 0.65° as compared to the 0.40° of a natural graphite. The total weight of the sample amounted to 184.2 mg.

The Cs¹³⁴-reference source had a diameter of 12 mm and a height of 2 mm, comparable to the geometry of the sample, given the distance of 155 mm between the sample and the NaJ-crystal. The specific activity was $S_{g} = 352 \text{ cpm/mg} \pm 11 \%$.

The outgassing of the sample at 850° C required several days to obtain a dynamic vacuum of $1 \cdot 10^{-6}$ mm Hg, which at room temperature improved to $1,5 \cdot 10^{-8}$ mm Hg. After brazing and after the leak test, the Vb-capsule was opened. An activity measurement showed that the graphite had, at room temperature, sorbed already 2000 cpm of Cs. When the sample had reached the first working temperature of 800° C this activity decreased to 600 cpm approximately, and the sorption-desorption cycle was started. During the desorption stages the Cs-pool was cooled with compressed air and water in order to attain the desired lower Cs- temperatures within some minutes, since the times to obtain saturation were also of this order.

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The results are shown in fig. 4. The transition stages at the different sample temperatures of 600 to 800°C are more or less horizontal, indicating the formation of a compound. No hysteresis of the saturation loadings during the desorption stages was observed.

At the end of the experiment a final desorption of Cs was made leaving on the sample a Cs-loading of 60 mg/g \pm 20 %. Then the sample chamber was opened and the graphite was found to have crumbled into smaller pieces. A radiometric analysis of the Cs¹³⁴-content in the sample yielded a value of 46 mg/g \pm 25 %, showing that the reference source method was reliable within \pm 20 %.

Each loading isotherm in fig. 4 has only one transition stage which begins at about 600 mg/g and finishes at roughly 1000 mg/g. These loadings correspond to the transition C_{24} Cs- C_{10} Cs (5) for which the theoretical values are 465 - 1120 mg/g. A comparison of the loadings and Cs-pressures at the end of the transition stages with the results of reference (1)(2) is given in table 1. There is a certain disagreement of the data, due probably to differences of the graphite samples used.

Table 1

Composition and pressure at the end of the transition stages

Temperature of the graphite, ^o C	600		700		800	
reference	(1)(2)	fig. 4	(1)(2)	Fig. 4	(1)(2)fig. 4
mm Hg of Cs	0.8	0.2	4.0	1	10	5
mg Cs/g	630	1100	600	1000	450	980
approximate composition	° ₁₈ °s	° ₁₀ °s	° ₁₈ °s	^C 11 ^{Cs}	c ₂₄ c	s C ₁₂ Cs

- 8 -

The p-T relations for a given compound were obtained from fig. 4 by plotting the Cs-vapour pressures at constant loadings vs the corresponding 1/T.(fig. 5). From the slope of the 800 mg/g - isostere a heat of formation of approximately -36 Kcal/mole was estimated, which agrees fairly well with the value of -38 Kcal/mole, given in reference (1) (2). A comparison of the p-T data for the loadings in the theoretical $C_{24}C - C_{10}Cs$ range with those of other authors is given in table 2.

Table 2

Temperature of graphite, C	ref(1)(2), fig. 8 475 mg/g	ref(6), fig. 6 and 9	500 mg/g	Fig. 5 800 mg/g	1200 mg/g
600	0,8	0,7	0,04	0,2	0,6
650	2	1,5	0,14	0,4	1,8
700	4	3,5	0,4	0,9	4,8
800	10	14	2,2	3,5	28

p-T relations for compounds in the C₂₄Cs-C₁₀Cs range

The results for the Cs-pressures of ref(1)(2) and ref(6) show a relatively good agreement with each other and with the 1200 mg/g - isostere of fig. 5. The isosteres corresponding to loadings of 500 and 1200 mg/g do not fall within the actually measured transition stage shown in fig. 4 (600 - 1000 mg/g). They were listed in table 2 for the sake of comparison. The 800 - mg/g isostere represents the measured C_{24} Cs - C_{10} Cs stage, but the Cs pressure values are systematically by a factor of about 4 lower than those of ref(1)(2) and ref (6).

5) Pyrolytic graphite

The sample (Carbone Lorraine) consisted of serveral small blocks with linear dimensions of 2 x 2 x 4 mm, having Coriented structure. Thermal annealing was performed in Argon by heating to 2950° C for 5 minutes, 2700° C for 10 minutes and cooling to 1000° C in 20 minutes. Then the HF-furnace was spent. The weight of the sample was 194.6 mg. The experimental procedure was the same as for the nuclear graphite (section 4). The specific activity was 366 cpm/mg \pm 11 %. Also the saturation times were practically the same as for nuclear graphite.

The saturation loadings and the corresponding Cs-vapour pressures are plotted in fig. 6. Also in this case only one transition stage was found for each of the sample temperatures, extending from about 500 to 800 mg/g. No hysteresis was observed.

At the end of the experiment, during final cooling, a compound with 960 mg/g, equivalent roughly to C_{12} Cs had formed. On inspection the sample turned out to be dark blue, but not crumbled. A comparison of the loadings and Cs-pressures at the end of the transition stages with the results of ref(1)(2), fig. 7, is given in table 3.

Table 3

Composition and pressure at the end of the transition stages

Temperature of the graphite, C	600		700		800	
reference	(1)(2)	Fig.6	(1)(2)	Fig.6	(1)(2)	Fig.6
mm Hg	0,8	0,3	4,0	1,5	10	8
mgCs/g	1000	800	900	800	830	800
approx.composition	e ₁₁ Cs	^C 14 ^{Cs}	° ₁₂ °s	^C 14 ^{Cs}	^C 13 ^{Cs}	^C 14 ^{Cs}

- 10 -

As in the case of nuclear graphite, there is a certain disagreement in the Cs-pressures. The p-T relations for different loadings are shown in fig. 7. From the slope of the 800 mg/gisostere the heat of formation resulted to roughly -32 Kcal/mole, that of the 900 mg/g - isostere yielded - 38 Kcal/mole. Other authors (5) reported values between -31 and -36 Kcal/mole. A comparison of the p-T data for the loadings in the theoretical C_{24} Cs-C₁₀Cs range with those of other authors is given in Table 4.

Table 4

p-T relations for compounds in the $C_{24}Cs-C_{10}Cs$ range

Temperature of graphite, C	ref(5) fig.3	ref(1)(2) fig.8 475 mg/g	500 mg/g	fig. 7 800 mg/g	900 mg/g
600	0,5	0,8	0,2	0,3	0,4
650	1,3	2	0,5	0,7	1,2
700	3,0	4	1,2	1,6	3,4
800	-	10	5,5	8,5	22
		L			

Also here the Cs-pressures are lower than those reported previously (1)(2)(5) for the $C_{24}Cs - C_{10}Cs$ transition.

6) Discussion

For the W-Cs system saturation loadings of about 1.0 - 1.4 mgCs/gwere obtained for sample temperatures between 600 and 800° C. The corresponding Cs-vapour pressures were in the range of 0,5-18 mm Hg. The variation of the loadings with changing p-T conditions was very low (fig. 2). This inertness was possibly caused by a continuous decrease of the specific surface of the W-powder during the experiments, due to sintering. The presence of O_2 is known to increase the sintering rate of W, at relatively low temperatures, especially if the powder contains a substantial proportion of ultra-fine particles (8).

An idea on the mobility of the Cs can be obtained from fig. 3. At constant Cs pressures of 2.4 - 18 mm Hg only about 0,1 mgCs/g were released when the sample temperature was increased from 600 to 800° C. To obtain at constant sample temperatures a release of 0.1 mgCs/g, practically untolerable pressure changes are needed(fig. 2). Also the relative low loadings may result in difficulties for a practical use of this type of a Cs-reservoir in a converter.

The nuclear graphite formed interlamellar compounds in the range $C_{24}Cs - C_{10}Cs$. The p-T data show a certain disagreement with those of other authors, but differences of this kind were already observed previously for different graphite materials (9). There seems to be a correlation of the different sorption characteristics with graphite density and crystallite size (9).

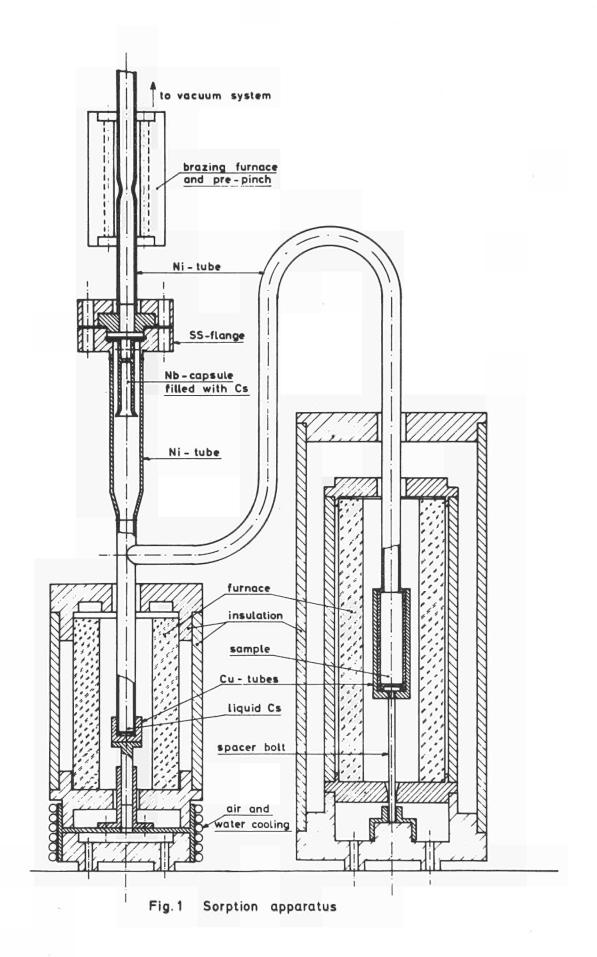
From the isotherms in fig. 4 it can be deduced that between 20 and 200 mgCs/g are released in the range of 1-10 mmHg, when the Cs- vapour pressure decreases by 20 %. Also the variations of the Cs-vapour pressures with temperatures can be estimated from fig. 4. The end of the transition stages of all isotherms is approximately at a loading of 1000 mg/g, and at these points the Cs pressure changes by a factor of about 2 for each temperature interval of 50°C. If additional isotherms are constructed in the range of interest (1 - 10 mm Hg, 700 - 800°C), it can be seen that temperature variations of, e.g., ± 25°C produce, at a loading of 1000mg/g, pressure variations of about ± 20 % around the 800° C and 750° C isotherm, and ± 40 % around the 700°C isotherm. Orientative values, obtained from a p-T plott of the 1000 mg/g-isostere are roughly 0,02, 0,04, and 0,2 Torr/°C for working temperatures around 700, 750, and 800°C respectively.

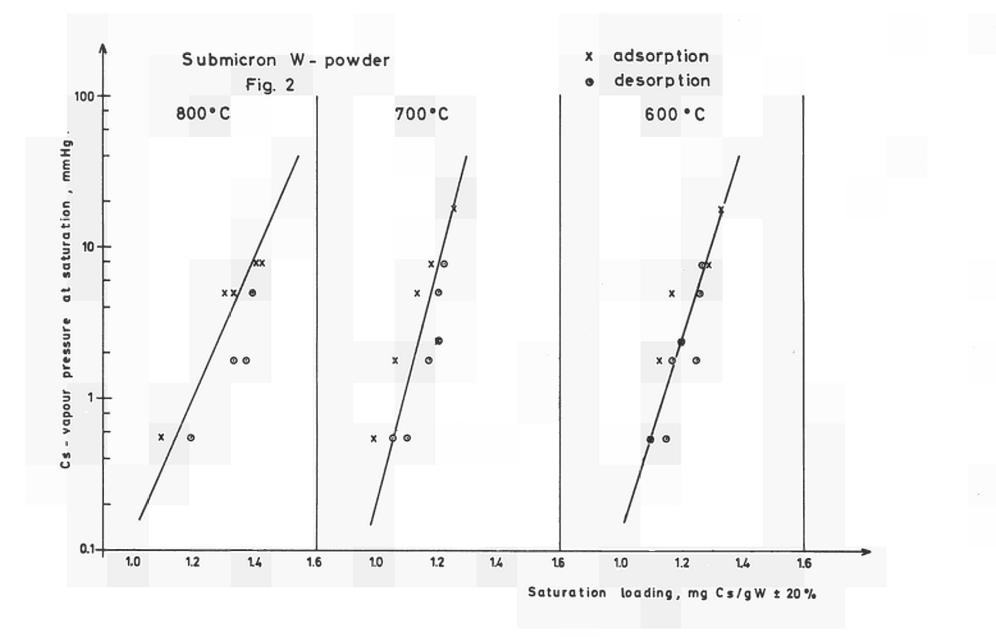
Also the pyrolytic graphite formed compounds in the range of compositions C_{24} Cs - C_{10} Cs. From the isotherms in fig. 6 it follows that between 10 and 170 mgCs/g are released in the pressure range of 1 - 10 mm Hg, when the Cs pressure decreases by 20 %. The end of the transition stages of all isotherms is at about 800 mgCs/g. If additional isotherms are constructed in the pressure range of 1-10 mmHg and temperature range of 700 - 800° C, it can be seen that, at 800 mg/g, temperature variations of $\pm 25^{\circ}$ C produce changes in pressure of $-\pm 30$ to ± 45 %. Orientative values from a p-T plott of the 800 mg/g-isostere resulted in 0.2, 0.06, and 0.03 Torr/ $^{\circ}$ C for temperatures near 800, 750 and 700 $^{\circ}$ C respectively.

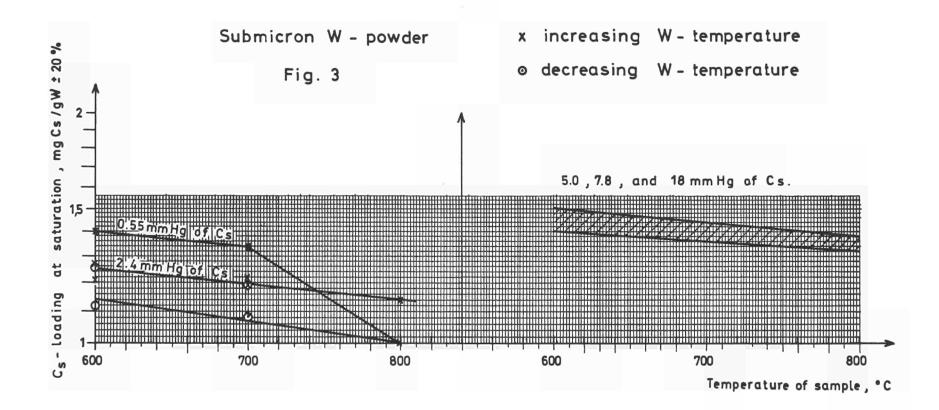
It can, from the above discussion, be concluded that graphite reservoirs have much more suitable pressure, temperature and loading characteristics than the tested W-powder. However, these characteristics may be quite different and unpredictable for different graphite materials. It seems therefore necessary to determine experimentally the pressure-temperature-loading data for each type of graphite to be used as a reservoir.

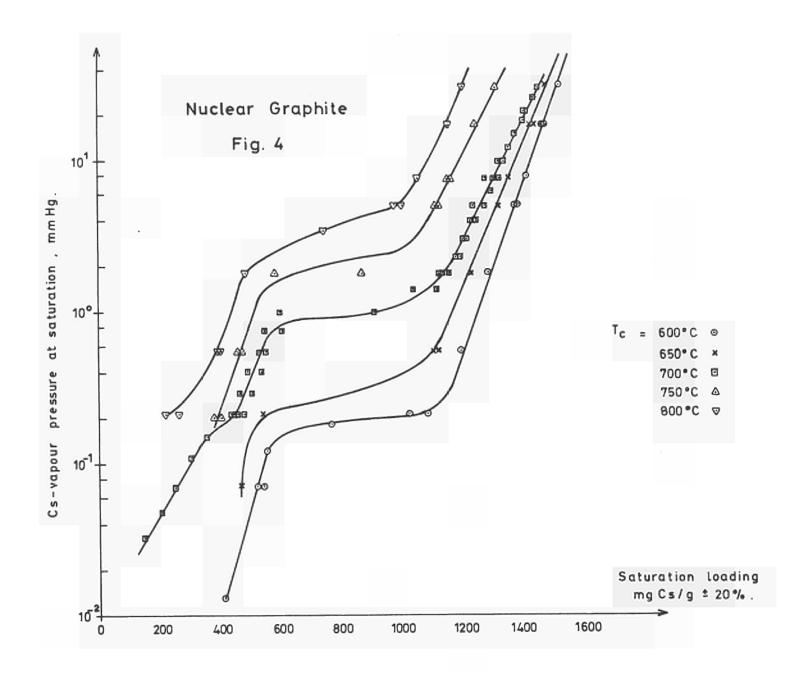
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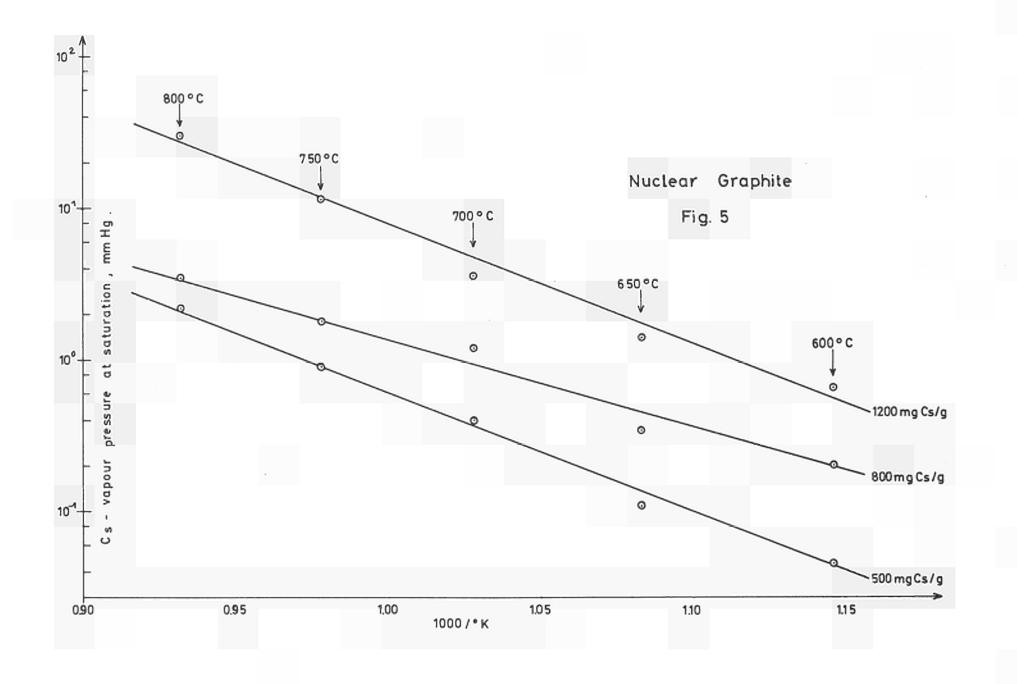
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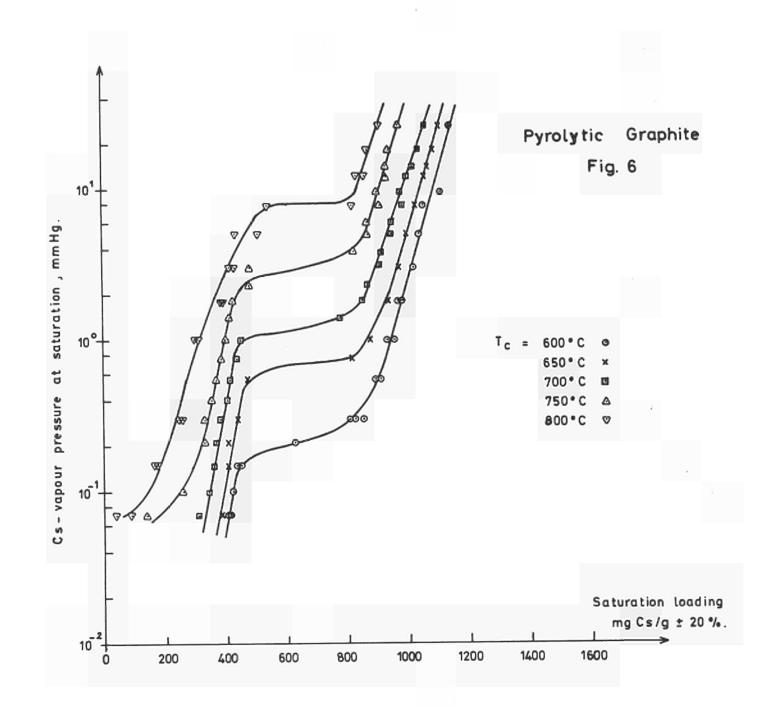


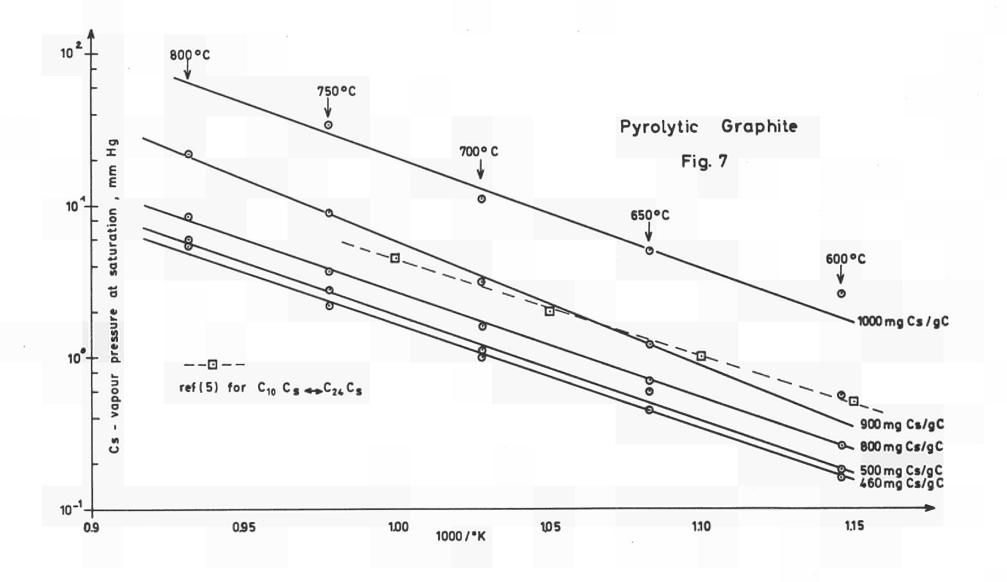












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