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Graphite melting temperature

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METHOD AND EXPERIMENTAL SETUP

The experimental method used for metals was fast heating (1-5 μs) in ambient air atmosphere by unitary pulse of electrical current, using a 20 kJ storage capacitor bank of low inductance facilities. Test specimens included foils of ~ 20-50 μm thickness for creating a blackbody model and temperature measurements. It is not necessary to take into account the heat losses and chemical reactions for the short duration of the experiment (several microseconds). Owing to short time of the experiment an electrical discharge did not appear (up to ~ 6000 K) along the specimen in air ambient atmosphere (even with tungsten investigation). For graphite investigations the upper limit of temperature measurements was ~ 12000 K under elevated pressure (for specimen covered in solid medium).

Diagnostic equipment included a digital 4-channel oscilloscope Tektronix TDS-754C for registration of signals from voltage and current transducers, and a fast pyrometer.

TEMPERATURE MEASUREMENT AND A BLACKBODY MODEL

The most reliable method to measure true temperature is to develop a blackbody model with emissivity close to 1. It is required to develop such a blackbody model that would exist not only in solid state but also in the liquid state.

Temperature measurements were made with a composite two-strip wedge-shaped blackbody model. The model was constructed specially for metals, hard steels and carbon investigations. The model's novelty is that its elements are made of separate flat foils. It consists of two metal strips (thickness 20-50 μm), 1.5-2 mm wide and 30 mm in length. Two sides of these strips are compressed together along the length, while the rest two sides have a tiny gap (about 60-100 microns) between them. This side gap is used to insert a light guide inside the model (by 0.3 mm). To improve the efficiency of the model the metal strips were cambered. Practically both strips were folded with cambers outside (see Fig. 1). A flexible glass-fiber light guide is used with some 10-50 fibers of 50 microns diameter each. The fibers are distributed in one layer along the side gap to make a flat system and slightly inserted inside the blackbody cavity. The tips of the fibers light guide (2-3 mm) were cut away after each experiment by diamond knife to make a light guide ready for next experiment.

The blackbody model serves as a usual testing specimen, undergoing temperature measurement and heating up to destruction in the experiments. In the experiments we used L/D = 20 for our two-strip blackbody model, where L - a width of the strip, D - the distance between two slightly "opened" strips (Fig. 1 and Fig. 2). A previous investigated wedge-shaped model [8,9], has two weak places: at the edges and in the centre. These two places were characterised by a small number of reflections. Our new model of a blackbody has improved form and properties (see Fig. 2). Estimation of the apparent emittance of this blackbody model gives the value 0.99, provided that the reflection is specular.

FAST PYROMETER

The basic components of the pyrometer are the silicon PIN-photodiode, interference light filter with the half-width of 16 nm at 855 nm wavelength, two lenses, diaphragm, transimpedance amplifier and glass-fiber light guide (Fig. 2). Settling time of the pyrometer with an accuracy of 1% is 12 ns. The pyrometer dynamic range was 800 at 0 – 50 MHz.

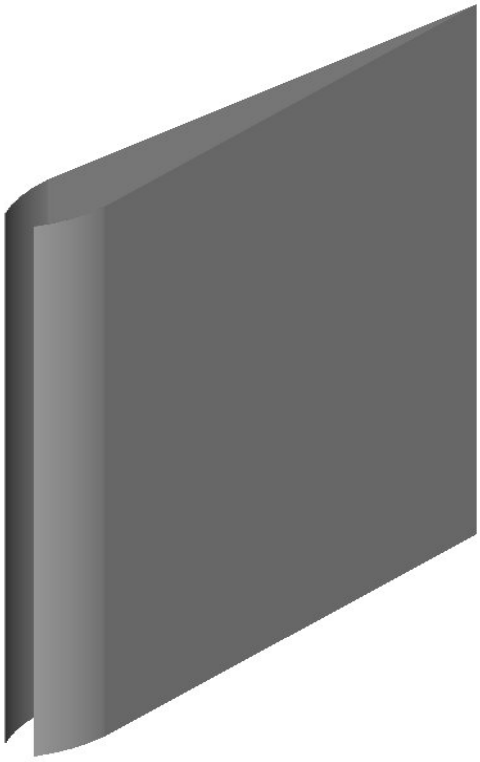

FIGURE 1. The wedge-shaped blackbody model made of two specularly polished metal foils (a middle section of the specimen). Width is 1.5 mm.
The current is going through the foils from the top down or from the bottom to top (the whole length is 30 mm). The end of the light guide is placed on the side of the blackbody model, just to the gap between two foils.

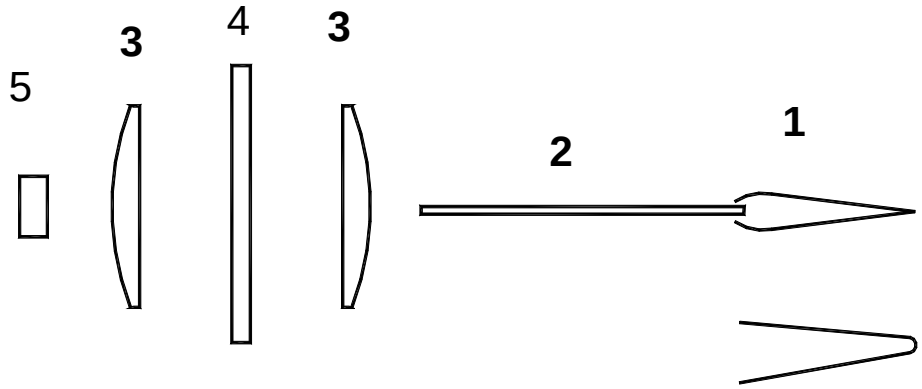

FIGURE 2. A scheme for measurement of two-strip blackbody radiation.

1- Two-strip blackbody model (cross-section), the both strips were cambered outside. Lower - the previous model, investigated in [1, 2].
2- Light guide consists of 10-50 fibers, 50 microns diameter each. On the right side the fibers are distributed in one layer along the side gap of the blackbody cavity. Left end of the light guide looks like a circle.
3- Lenses. 4 - Interference filter. 5 - PIN-photodiode.

One and the same pyrometer signal was sent to two digital oscilloscope channels with different sensitivity. The sensitivity of one of the channel was selected in such a way that it would be possible to record the temperature plateau with high resolution at metal (Zr) melting. The signal of this channel was off-scale at temperature of T ~ 2300 K. The second channel (low sensitivity) recorded spectral brightness at temperatures of 4000 K and higher. "Sewing" of the signals gives one smooth curve with high dynamic range. The linear dependence (up to 1800 mV) of photodiode output versus radiance was obtained. The next step was to calibrate this photodiode signal with the temperatures.

The start of melting plateau was used as a calibration point for temperature measurement. Plank's equation was used for temperature calculations. The combined uncertainty for temperature measurements is ~ 1.5 % at 4000 K.

LIQUID CARBON PROPERTIES

We fulfilled preliminary experiment with the graphite blackbody model, which was made of the two strips (highly oriented graphite). Calibration point we used was a plateau of tungsten melting, recorded in the previous shot for tungsten blackbody model (filled with epoxy glue) with the same light guide. Pyrometer recorded the melting point of the tungsten blackbody at the known melting temperature (3690 K). Cavity of the graphite blackbody was filled also with the epoxy glue. As it turned out in our experiment, the melting temperature of the graphite is 4800 K ± 200 K. This value corresponds to other known experimental data 4600-5000 K (pressure ≥ 0.2 kbar).

The first results of fast heating (by electrical pulse current) of highly oriented pyrolitic graphite specimens (in the form of flat foils) are presented in [3, 4]. Some problems of graphite melting were solved in the experiment: the specimen in the form of strip was placed in solid transparent medium (Canadian balsam), between two glass plates. It prevents sublimation of graphite under high temperatures (3000-10000 K) Temperature measurement was based on measuring the radiance from the graphite surface (**a**-plane) at the wavelength 900 ± 8 nm. Temperatures were calculated using Plank's equation. Calibration of the pyrometer signal was made at the imparted energy 9.1 $kJ \cdot g^{-1}$, where the temperature of graphite equals to 4500 K according to [5]. Fast heating of the graphite specimen in solid cover provides high pressure. Modelling by A.D. Rakhel gives 5-15 kbar for liquid region (Fig. 3).

We eliminate the contradiction in measuring of imparted energy in [4]: the start of melting measured at **a**-plane (12 $kJ \cdot g^{-1}$) differs from that of **c**-plane (10.5 $kJ \cdot g^{-1}$). Heat losses in the thin (~ 1μ) graphite surface layer are estimated during short period of time contact between graphite strip and medium. It gives 10-12% correction to the calculated energy axis T(E). As a result a satisfactory coincidence takes place for kinks on voltage signal and temperature signal during graphite melting (Fig. 3).

There is a well-established imparted energy at the start of fast melting E_s = 10.5 $kJ \cdot g^{-1}$ (pressure ~ 10 kbar). It is in agreement with slow pulse heating result 10.45 $kJ \cdot g^{-1}$ (10-30 kbar) [6], and with microseconds pulse heating datum 10.4 $kJ \cdot g^{-1}$ (0.2 kbar) [7].

Enthalpy of liquid state under melting: E_l = 20.5 $kJ \cdot g^{-1}$. Heat of graphite melting: ΔE = 10 $kJ \cdot g^{-1}$.

Heat capacity C_p of the solid graphite before melting is equal to 3.2 $J \cdot g^{-1} \cdot K^{-1}$, which is near to published steady state data. Preliminary datum on specific heat capacity of liquid carbon in this experiment is presented (as dE/dT according to Fig. 4). It is equal ~ 4.2 $J \cdot g^{-1} \cdot K^{-1}$ for T from 6000 K up to 12000 K (but blackbody model was not used in the results shown in Fig. 3, 4).

Initial density of the graphite under investigation 2.26 g·cm^{-3}. Our experiments show that under heating the resistivity of the liquid carbon (at the density ~ 1.8 g·cm^{-3}) is equal 730μΩ·cm (for pressure 10-20 kbar and temperatures 5000 - 7000 K). To our opinion (based

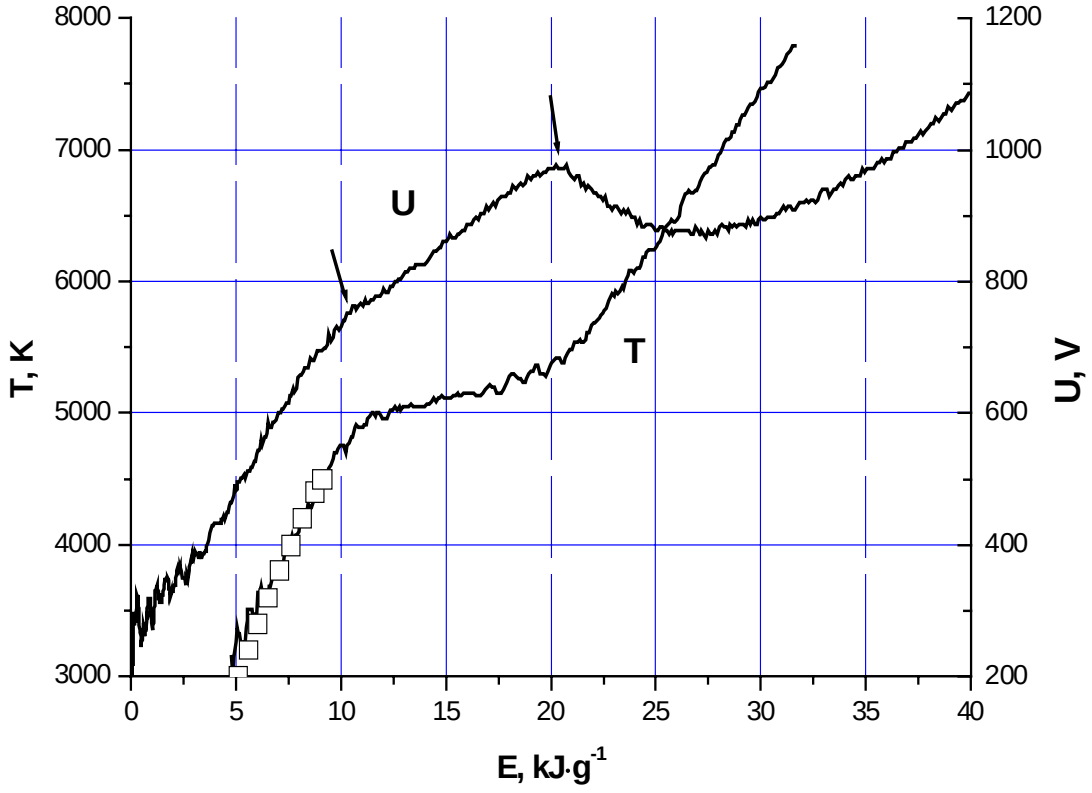

FIGURE 3. Melting of a highly oriented pyrolitic graphite.
The whole time of heating - 1.5 μs. Graphite strip (1mm ×10mm, thickness 0.3 mm) is between two thick glasses. The start and the end of melting are shown with the arrows on the voltage curve U.
Experimental data [5] are shown with open squares. Temperature (**a**-plane) calibration: imparted energy E = 9.1 kJ/g equals to 4500 K as it was published in [5].

on the experiments) liquid carbon resistivity is consistent with the resistivity of expanded (by four times) liquid refractory metals (W, Ta, Mo) [3].
To cite the equation of Clapeyron-Klausius in the next form for graphite melting point, provided that the equation is valid for unisotropic substance (graphite):

$$\mathbf{V_{liquid} / V_{solid} = 1 + \Delta H_{melting} / V_{solid} (T_{melting} \times dP/dT),}$$

where V_{liquid} - volume of the liquid phase at melting point; V_{solid} - volume of the solid phase at melting point (~ $1.2V_0$ - literature datum); $\Delta H_{melting}$ - heat of melting (10 kJ·g^{-1} - our measurement); $T_{melting}$ - melting temperature (4800 K - our measurement); dP/dT = 54 bar·K^{-1} [8].
Then we have: V_{liquid} / V_{solid} ~ 1.7
As it turned out the graphite expands significantly, the expansion consists of ~ 70 % during melting. For the expansion of the substance during melting [9] and value of resistivity in liquid state the melted graphite retains covalent bonds, which usually characterise solid graphite. Obviously liquid carbon has a complicated molecular structure.
Remind one more feature: liquid carbon specific heat capacity (C_p) according to our measurements has very high value ~ 4.2 J·g^{-1}·K^{-1} (11.5 cal·mol^{-1}·K^{-1}). It is near to heat capacity of liquid refractory metals.

In the next Table there are experimental and calculated data on melting temperature of carbon. Really it is the history of carbon investigations since 1963 year, when Francis Bundy published his outstanding work on carbon melting under high pressure.

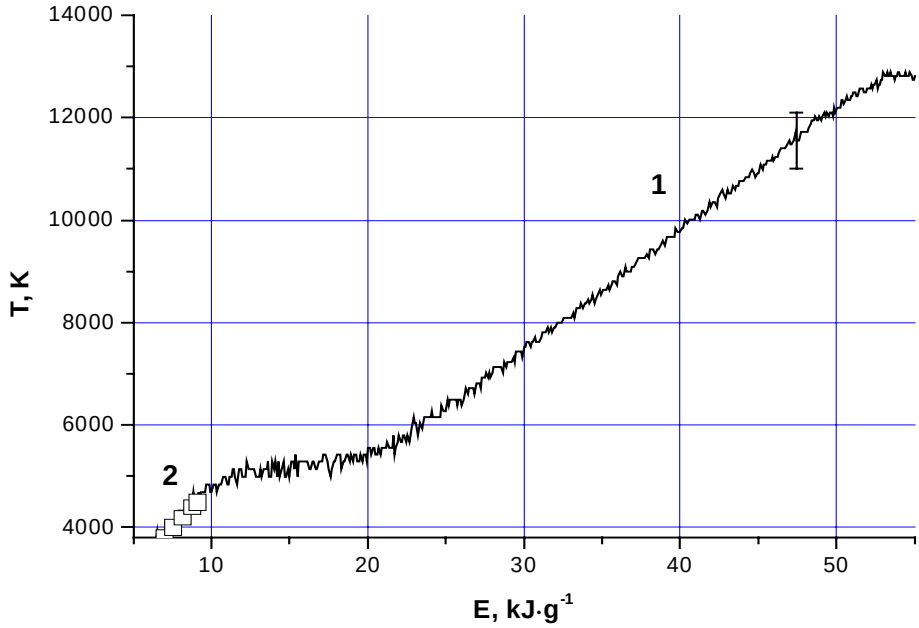

FIGURE 4. Temperature of the solid and liquid carbon versus imparted energy.
1 – Our measurements (error of the temperature measurement is shown above 10000 K).
Specific heat capacity of the liquid carbon (according to this figure) ~ 4.2 $J \cdot g^{-1} \cdot K^{-1}$.
2 - Open squares, experimental data of pulse heating [5] (tens of microseconds).

The authors of the article noted in the Table the main results of every investigation, that gives an interesting view on the problem as a whole and on measuring of graphite melting temperature. The main disadvantage is in measuring melting temperature of the graphite: ~ 4000 K or ~ 5000 K ?

In 1996 year Bundy F.P. et al. presented a new phase diagram for carbon with melting temperature equals to 5000 K as the most probable value for triple point (see Table). It was the most thorough review for the last years and many of investigations were considered. Just after this publication another opinion on triple point parameters appeared.

The data of Asinovski E.I. et al. 1997-1999, obtained by slow (up to 0.5-1 hour) heating of graphite at 1 bar pressure, are marked with cursive text in the Table. These data are not consistent with many other investigations. The mentioned authors consider (see for example [10]) that measured temperature of graphite depends on velocity of heating, the faster heating - the higher temperature. We don't find it right, analysing all the data published in the Table and in [10]. Just on the contrary: the lower heating is, the lower melting temperature has been obtained (as in the works of mentioned authors). This is connected with several reasons.

The first reason concerns particularly with the different expansions of the graphite on different axes (**a** and **c)**, and destruction of the graphite specimens before melting. The second reason should be underlined: under slow heating the recording of the light emission of condensed vapour, but not the graphite surface itself (at the level near 4000 K) may takes place. This problem was solved by Basharin A.Yu. in his experimental investigations (1999-2001) [11,12] with laser pulse heating.

Musella M. et al. in 1998 gives 4800±150 K by pulse current and Basharin A.Yu. et al., in 2000-2001 [11, 12]) gives 4750 ±150 K by laser pulse heating, as a melting graphite temperature. Moreover there was declared by Sheindlin M.A. and Senchenko V.N. in 1988 year (see Table): "At 4700 K graphite does not melted yet (5 experiments under 100 bar)". In

our experiments (fulfilled with the help of a blackbody model made of the two graphite strips) there was obtained 4800±200 K with calibration plateau under the melting of the specimen made of tungsten strips as a blackbody model.

Call your attention to the fact that the start of the graphite melting was repeatedly shown in the Table as first value 10.45 $kJ \cdot g^{-1}$ (Bundy F. in 1963); as the second value 10.4 $kJ \cdot g^{-1}$ (Baitin A.V. et al., in 1988); and as the third value 10.5 $kJ \cdot g^{-1}$ (Korobenko V.N. et al. in 2001). One may consider this value (10.5 $kJ \cdot g^{-1}$) to be the well-established datum. Then assuming known Cp for solid state at high temperature we may calculate the melting temperature, which should be higher than 4600 K.

One more feature in correlating graphite properties under melting: resistivity of liquid carbon equal to 30 μOhm·cm at 1 bar pressure, obtained by Heremans J. et al. in 1988 (a shunting discharge may appeared in the experiment); equal to 625 μOhm·cm (pulse laser heating by Downer M.S. et al. in 1993); and equal to 730 μOhm·cm at pressures more than 1 kbar (our measurements). The last value has repeatedly proved in many experiments (see Table) give 600-900 μOhm·cm under different pressures more than 100 bar. We suppose as the most authors (see Table), that 1 bar pressure has not allowed measuring specific properties of melted carbon.

Remind one of the arguments, that the action of an electrical arc in 1 bar ambient atmosphere gives solidificated drops of liquid carbon [13]. These drops were found also under discharges between graphite electrodes in vacuum [14]. There were established in [14, 15] that the action of an electrical discharge between graphite electrodes leads to appearance of pressure higher than 100 bar (up to 400 bar) in the vicinity of cathode spot. Estimation of temperature gives [14] 5500 K in the cathode zone. Evidently liquid carbon phase may appear during the experiment under the initial low pressure of ambient atmosphere.

Why is it so important to establish the true graphite melting temperature? The main goal - to external temperature measurements up to higher values, and reach plasma state measurements (10000-30000 K) with the help of optical device under fast heating of the specimens. Graphite melting temperature will be a fixed point for future high temperature measurements.

CONCLUSION

A simple wedge-shaped model for one foil (folded along length) was examined in [1, 2]. Note considerable positive distinctions between our model and first proposed. Two foils are pressed together along one side that did not give dark stroke in the centre of the slit [1, 2]. The both foils were cambered outside formed a cavity. The tip of a light guide placed inside the cavity. 10-50 fibers distributed in one layer along the narrow gap.

A new two-strip blackbody model will be useful for investigation of metals and liquid carbon properties [3, 4]. Especially it will be useful for specimens made of hardened steels and graphite, which are difficult to roll as a blackbody cavity in the shape of a tube. Moreover, the rod grown of pyrolitic graphite will be destructed out of different thermal expansion on different axes even in solid state, before the graphite melting.

Blackbody models made of two-strip foils will be useful for design and investigation of high temperature fixed points, based on metal-carbon compounds. For example, the highest melting temperatures for carbides HfC equal to 4220 K, for TaC - 4270 K. The possibility exists of creating an experimental calibration scale measuring blackbody radiation of the models made of different metals (Zr, Hf, Mo, Ta, Re) and carbides. As carbides have a rising electrical resistivity with rising temperature, it gives a homogeneous electrical fast heating of these substances. A solvable task is only to manufacture carbide thin foils. It gives an opportunity to refine on carbon melting temperature and to determine those for different carbide eutectics.

Two-strip blackbody model (under electrical fast heating) is a simple and convenient instrument to achieve and to measure the true high temperatures.

TABLE

Year	Method (duration)	Authors (reference)	Imparted energy E, T melting, E_{solid}, E_{liquid}, heat capacity Cp		Expansion, resestivity ρ
1963	Electrical pulse heating under high pressure (milliseconds)	Bundy F. J. of Chemical Physics, v.38, #3, p. 631, 1963	E_{solid} = **10.45 kJ·g^{-1}** (for 9-31 kbar и 97 kbar) E_{solid} = 12.2 kJ·g^{-1} (for 67 kbar) Heat of melting = (7-10.45) kJ·g^{-1} (estimation) E_{solid} = 13.2 kJ·g^{-1} (for 48 kbar), data obtained with grinding the specimens after the experiment		
1973	Estimations	Leider H. et al. Carbon, v.11, p.555, 1973	Heat of melting = 8.7 kJ·g^{-1} 4765 K		
1974	Electrical pulse heating (microseconds)	Gathers G.R. et al. UCRL-51644, Livermor, 1974	4000-5000 K Graphite of low initial density	E_{solid} ≈ 14.5 kJ·g^{-1} E_{liquid} ≈ 20 kJ·g^{-1}	
1978	*Laser heating (4 sec)*	*WhittakerA.G.et al. Science, V.200, 763, 1978 Nature, V.276, 695, 1978*	*3800 K*	*A new interpretation of the triple point parameters for carbon is declared. At the triple point: (P = 1 bar, T = 3800 K)*	
1984	Steady-state heating	Buchnev L.M. et al. Doklady AN USSR v.278.# 5, P. 1109. 1984	4890 K (estimation**)**	Cp(solid) = 4.1 J·g^{-1}·K^{-1} (estimation)	"Steady-state heating up to 3818 K did not change the main parameters of the structure"
1985	Steady-state laser heating (seconds)	Kirillin A.V. et al. TVT, v. 23, #4, p. 699, 1985	5000±200 K (estimation)	Convection vapor flows hindered temperature measurements**.** The gap (between specimen and the pyrometer) is diminished from 50 mm up to 2-3 mm	
1986	Electrical pulse heating (10 μs)	Lebedev S.V. and Savvatimskiy A.I. TVT, v.24, #5, p.892, 1986	Analysis of the homogeneous pulse heating of highly oriented graphite	Expansion V/Vo = 1.68 for imparted energy E = 18 kJ·g^{-1}	For liquid carbon ρ ~ 730 μΩ·cm
1988	Electrical pulse heating (several μs up to tens of μs)	Heremans J. et al. Phys. Rev. Letters, v. 60, #5, p. 452, 1988	(1 bar pressure !) ~ 4450 K (estimation)	Electrical discharge may appear along the graphite specimens during heating	Resistivity of liquid carbon: ρ = 30 ± 8 μΩ·cm (under 1 bar pressure !)
1988	Electrical pulse heating (2 ms)	Sheindlin M.A. and Senchenko V.N. DAN, v.298, #6, p. 1383, 1988	At 4700 K graphite does not melted yet (5 experiments under 100 bar)	9.1 kJ·g^{-1} – 4500 K	
1990	Electrical pulse heating (15 ms)	Cezairliyan A. and Miiller A.P. Int. J. of Therm., v.11, # 4, p.643, 1990	4530 ± 150 K (140 - 200 bar)	Only the start of melting plateau was observed. O_2 injected to a chamber to convert carbon vapor to CO_2.	
1990	Electrical pulse heating (tens of μs)	Baitin A.V. et al. High Temp.-High. Press. V.21, p.157, 1990	5080 ± 70 K Only the start of a temperature plateau was visible	The start of the graphite melting at the imparted energy E_{solid} = **10.4 kJ·g^{-1}**	
1993	Electrical pulse heating (ns - μs)	Pottlacher G. et al. Thermochimica Acta, v. 218, p. 183, 1993	4900±200 K (3 kbar) The whole temperature plateau was fixed	Authors: "Graphite of low density (1.55 g·cm^{-3}) will not do for liquid phase investigation".	V/Vo= 1.69 or 2.47 (two exp.) at 8.5 kJ·g^{-1} (before melting !) Inhomogeneous heating !

	Method (Duration)	Authors	T melting	E_{solid}, E_{liquid}, , heat capacity Cp	Expansion, resestivity ρ
1993	Laser pulse heating (femtoseconds)	Downer M.S. et al. Int. J. of Therm. V.14, 3#, p.361, 1993	Measurements of the optical constants	Resistivity of liquid carbon: 625 ± 75 μΩ·cm (calculation)	
1996	Review Article	Bundy F.P. et al. Carbon, V.34, #2, P.141-153, 1996	P-T phase diagram is constructed with T melting = 5000 K	Carbon liquid is metallic.	
1997	Electrical pulse heating (milliseconds)	Togaya M. Phys. Rev. Letters, v. 79, #13, p.2474, 1997	4650 K (14 kbar) 4790 K (56 kbar) 4640 K (94 kbar)	dP/dT = 300 bar/K , P < 56 kbar dP/dT = - 258 bar/K (> 56 kbar) $E_{liquid} - E_{solid}$ = (9,2 -9,5) kJ·g^{-1} ρ = 600-900 μΩ·cm (P = 94 -14 kbar)	
1997	*Steady-state laser heating (10 s)*	*Asinovskiy E.I. et al. TVT, v.35, #5, p. 716, 1997*	*4000 K (under P more than 220 bar)*	*A "new" interpretation (after the Witthaker, 1978) of the triple point parameters for carbon is declared. At the triple point: (P = 1 bar, T = 4000 K)*	
1998	*Steady-state electrical heating (0.5 – 1 hour)*	*Asinovskiy E.I. et al. TVT, v. 36, #5, p. 740, 1998*	*3700 ± 150 K (2.5 bar)*	*The destruction of the specimen is identified with the melting*	
1998	Electrical pulse heating (20 ms)	Musella M. et al. J. of Appl. Physics, v. 84, #5, p. 2530, 1998	4800 ± 150 K (110-2500 bar)	It was found the diminishing of the heat conductivity from 290 up to 5 W/m·K under graphite melting	
1998	Electrical pulse heating (1 μs)	Korobenko V.N. et al. Int. J. of Therm., v.20, #4, p. 1247, 1998	5500 ± 500 K Calibration through E under 4500 K; Temperature plateau under melting was observed	E_{solid} = 12±1 kJ·g^{-1}, E_{liquid} = 23±1 kJ·g^{-1}, Cp (liquid) ~ 4 J·g^{-1}·K^{-1}	Resistivity of liquid carbon: ρ = 730 μΩ·cm (under high pressure in solid ambient medium) P ≥ 1 kbar
1999	*Steady-state electrical arc heating*	*Asinovskiy E.I. et al. DAN, v. 369, #5, p. 614, 1999*	*~ 4000 K (estimation) (1 bar)*		
2000	Laser pulse heating (0.7 ms)	Basharin A.Yu. et al. (IHED) [27, 28], 2000	4750±150 K (P = 150 bar)	It was found, that graphite vapor shield the specimen, condense and radiate under 3400-4200 K. The marks of the melting are lacking under P < 100 bar.	
2000	Electrical pulse heating (5 μs)	Ivanov V.V. and Paranin S.N. RFBR-grant, # 98-02-16278 (Ekaterinburg)	Pulse pressure was measured for graphite specimen, placed in a thick-walled tube	Resistivity of liquid carbon: ρ ~ 700 μΩ·cm under P ≈ 20 kbar (measured)	
2001	Electrical pulse heating (1 μs)	Korobenko V.N. (Ph.D. thesis) and Savvatimskiy A.I. (this work, IHED)	4800 ± 200 K (graphite blackbody model was melted in the experiment, P≥1 kbar. Calibration through T melting = 3690 K for W blackbody	Enthalpy of solid state under melting (E_s = **10.5 kJ·g^{-1}**), measuring for both surfaces "a" and "c". Enthalpy of liquid state under melting (E_l = 20.5 kJ·g^{-1}). Heat of melting ΔE = $E_l - E_s$ = 10 kJ·g^{-1}. Cp for liquid carbon = 4.12 J·g^{-1}·K^{-1} (T = 5000 - 12 000 K). Liquid carbon is a molecular liquid ?	

2002	*Slow heating by electrical and laser methods*	*Asinovskiy E.I. et al. Usp. Fiz. Nauk, V.172, #8, P.931-944*	*4000 K. It is a collection of the works fulfilled by the authors earlier.*	*Authors: "Triple point parameters are predicted: P = 1bar; T = 4000 K". Really this prediction was established in the works of WhittakerA.G.et al. in 1978 (see Table). The new theme: the problem of carbine, which may appears before melting.*

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