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MEASUREMENTS OF THE THERMAL EXPANSION COEFFICIENT OF QUASI MONO-CRYSTAL PYROLITIC GRAPHITE SAMPLES

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Abstract

Kholodenko A.G., Heinzinger K., Moser H.-G. Measurement of the Thermal Expansion Coefficient of Quasi Mono-Crystal Pyrolitic Graphite Samples: IHEP Preprint 2001–32. – Protvino, 2001. – p. 9, figs. 12, refs.: 7.

Thermal expansion coefficients of TPG (Thermal Pyrolytic Graphite) were measured using TPG samples manufactured by Advanced Ceramics[1] and ATOMGRAPH[2]. Thermal expansion coefficients of TPG longitudinal ($\alpha_{in-plane}$) and transverse ($\alpha_{out-of-plane}$) were measured. Based on these measurements, the temperature dependence of the longitudinal thermal expansion coefficient ($\alpha_{in-plane}(T)$) of TPG is estimated. The transverse thermal expansion of PG (Pyrolytic Graphite) and HOPG (High Oriented Pyrolytic Graphite, graphite X-ray monochromator) (both samples from ATOMGRAPH) are presented, too. The results of these measurements are in good agreement with the literature.

Аннотация

Холоденко А.Г., Хайзингер К., Мозер Г.-Г. Измерение коэффициента теплового расширения квазимонокристаллических образцов пиролитического графита: Препринт ИФВЭ 2001–32. – Протвино, 2001. – 9 с., 12 рис., библиогр.: 7.

Измерены продольный и поперечный коэффициенты теплового расширения образцов термопиролитического графита производства Advanced Ceramics и Атомграф. Оценена величина температурной зависимости продольного коэффициента теплового расширения. Результаты измерений согласуются с опубликованными значениями.

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[2] Atomgraph, 2 Electrodnaya Str.111524 Moscow, Russia

Introduction

The ATLAS-SCT detector modules [1], have a sandwich-like structure including different kinds of materials such as Silicon, glue, AlN–ceramics etc. To assist the power dissipation in ATLAS-SCT modules, high-thermal-conductive materials like TPG are used. This quasi mono-crystal pure-carbon material is characterized by a high level of anisotropy in its mechanical, electrical, magnetic, thermal etc. properties [2]. The required thickness tolerance of the modules is as low as $2\mu m$. Hence it is important to know the mechanical, thermal and thermo-mechanical properties of the materials used in these modules. Following this way, we studied the in-plane and out-of-plane thermal conductivity coefficient of TPG, PG and HOPG [3], the correlation of the in-plane thermal and electrical conductivities and the transverse pull strength of various Pyrolytic graphite materials [4]. Here we describe the method and main results of the measurement the thermal expansion property of two types of TPG materials, manufactured by Advanced Ceramics and ATOMGRAPH.

Measurement Setup

The simplest setup for measuring of the thermal expansion is shown in Fig.1. Preliminary tests with this setup using copper samples, as reference, show an accuracy of $\sim 6\%$. However, for mechanically soft carbon material such as TPG, this configuration gives an un-repeatable results if the long flexible samples (length ~ 100 mm, thickness 0.5 mm) are measured. Also, for measurements of short samples the sensitivity of the mechanical micrometer is too low. When we tried to estimate the OUT-OF-PLANE thermal expansion coefficient of a PG sample the results had an error of $\sim 100\%$ despite of the using a special hard insertion between micrometer and surface of the sample. The results of the reference measurement with copper and measurements of a PG sample are plotted in Fig.1.

A new, improved setup overcomes most of these difficulties. The scheme of this setup is shown in Fig.2. One end of the test sample is clamped to the support. The opposite end has a direct contact to a rotating table which holds a mirror. A narrow laser beam is projected to the screen, using a two mirror system (one of them is rotating). The beam spot on the screen is traced online by a digital camera. The total distance from the rotating mirror to the screen is more than 5 m, the lever arm of the rotation table is about 50 mm, hence the magnification factor of the optical system is more than 100.

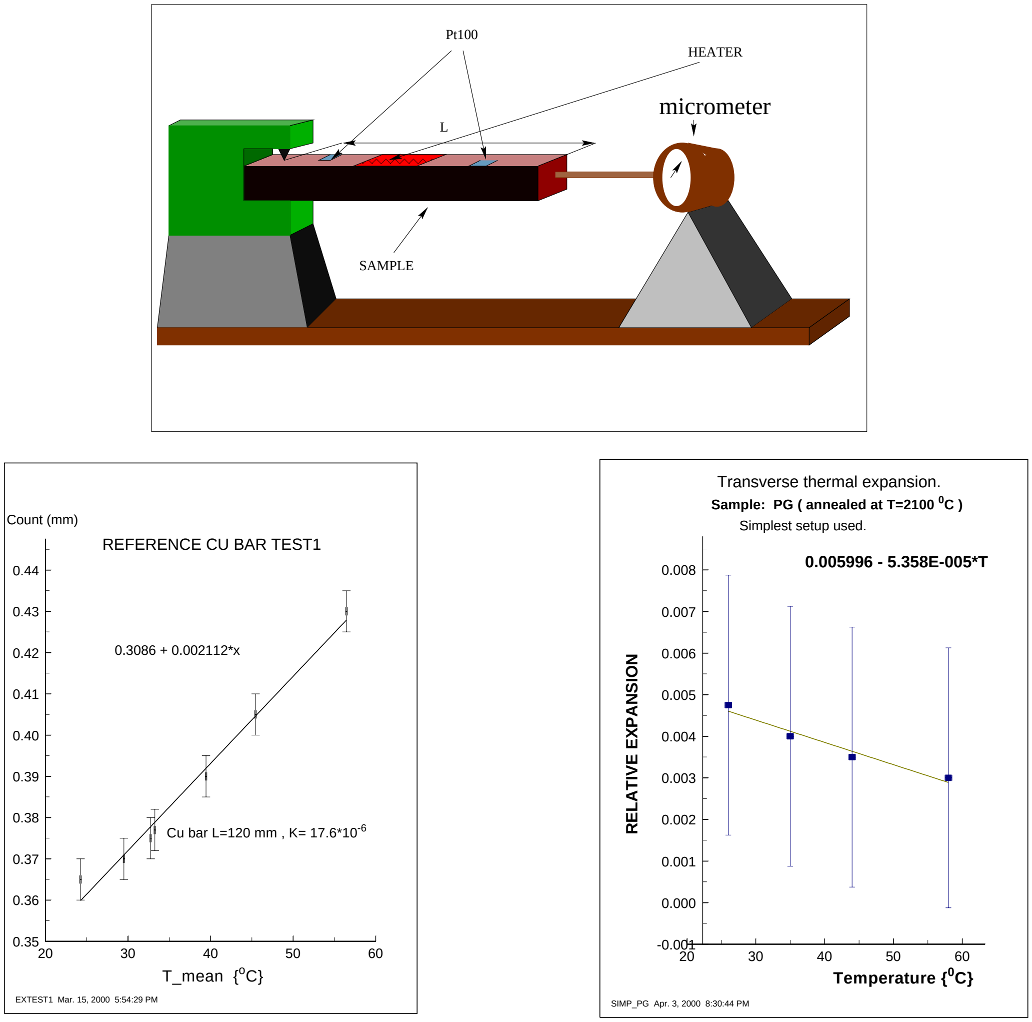


Fig. 1. Drawing of the simplest measurement setup. Thermal expansion of a reference copper bar and transverse expansion of a PG sample measured with this setup.

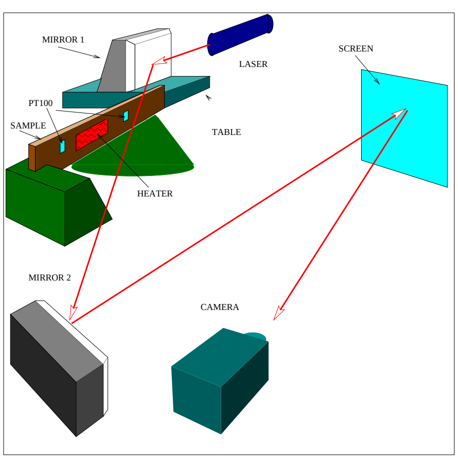


Fig. 2. Drawing of the setup used for the thermal expansion measurements.

A film heater heats the test sample. Two Pt100 sensors measure the temperature of its surface. The image of the laser spot is read by a PC. The x-coordinate of the center of the spot on the screen is calculated for five cross sections shown in Fig.3 and averaged. A typical image of the spot is shown in the same Figure. The stability in time of the center of spot position on the screen is illustrated in Fig.4. The distribution of the center position for 33 pictures taken during one hour is plotted. The standard deviation of this distribution is less than one pixel (0.86 pixels).

We calibrated our setup using the reference measurements of copper bars of different length. Out-of-plane measurements (the region of sample length is 2.5–20 mm), and longitudinal thermal expansion measurements (the sample length is 35–150 mm) were calibrated separately. The reference value of the copper thermal expansion coefficient is taken as $16.8 * 10^{-6}$, according to [5]. The temperature dependence of this coefficient was ignored. According to [5] the relative extension of copper is:

$$\frac{\Delta L}{L} = \alpha \times \Delta t + \beta \times \Delta t^2 + \gamma \times \Delta t^3,$$

where $\alpha = 15.89 \times 10^{-6}$, $\beta = 4.492 \times 10^{-9}$,$\gamma = 3.888 \times 10^{-12}$, in the temperature range from $t = 20$ to 825^0C.

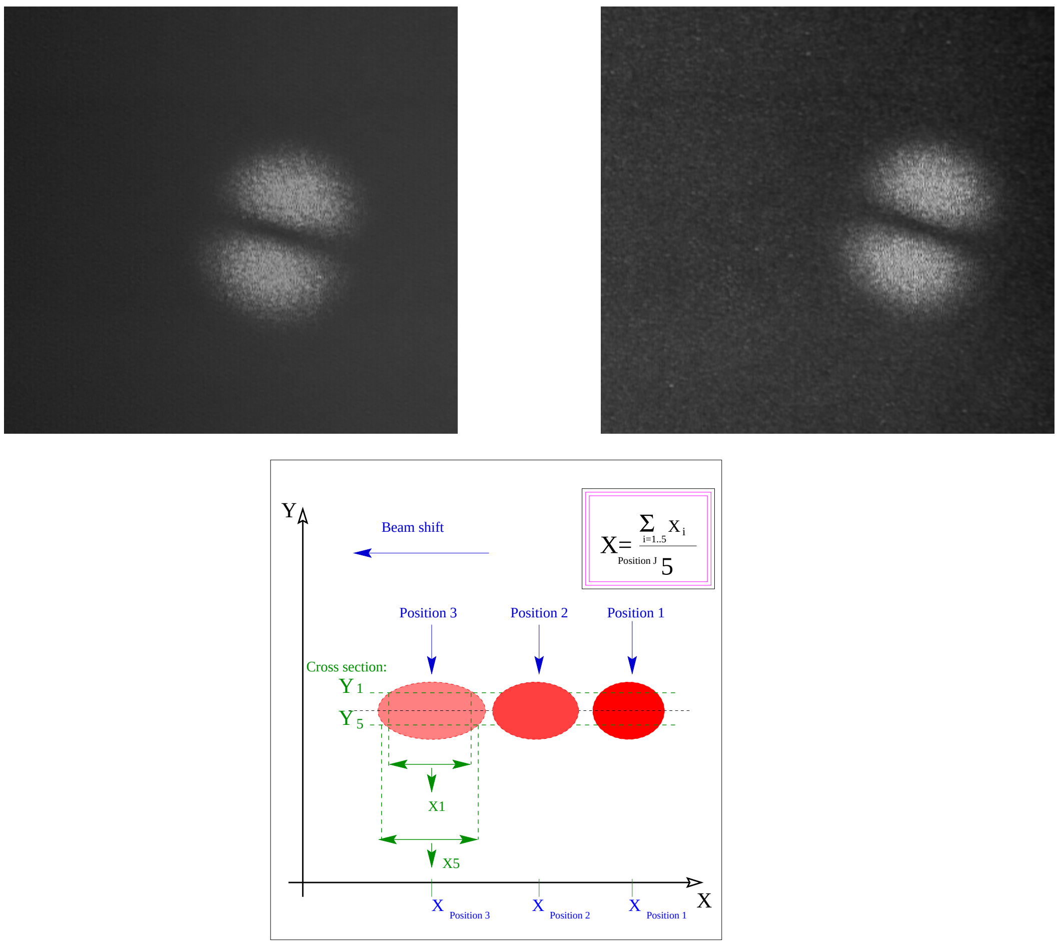


Fig. 3. Typical view of the beam spot before (right) and after (left) background subtraction. The lower figure explains the calculation of X-coordinate of the spot center.

Longitudinal (In-Plane) Thermal Expansion

Thermal expansion coefficient

The calibration of our setup for this measurements is shown in Fig.5. The longitudinal thermal expansion of Advanced Ceramics and ATOMGRAPH TPG is plotted in Fig.6. From this the thermal expansion coefficient of both kinds of TPG can be calculated.

Our results for the In-Plane thermal expansion coefficients are:

- the coefficient is **negative** for both materials;
- $\alpha_{in-plane} = -1.17^{\pm 0.15} \times 10^{-6}\ [^0C^{-1}]$ for Ad.Cer.;
- $\alpha_{in-plane} = -1.04^{\pm 0.11} \times 10^{-6}\ [^0C^{-1}]$ for ATOMGRAPH.

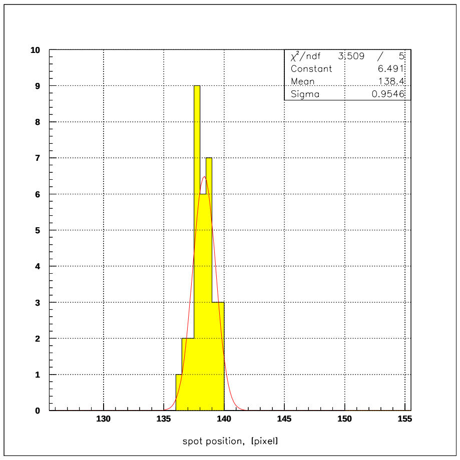


Fig. 4. Position distribution (in pixels) for 33 spots taken during 1 hour. Estimation of the X-coordinate accuracy.

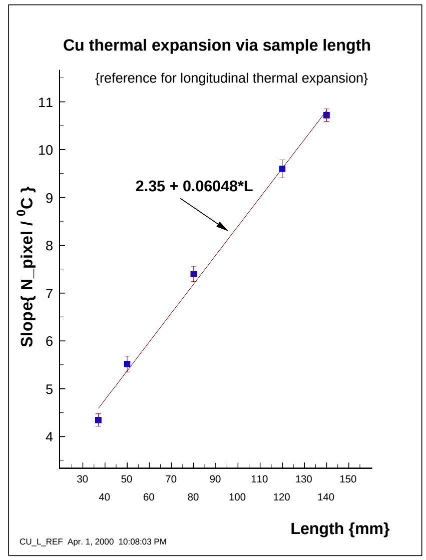


Fig. 5. Thermal expansion of the copper samples used as reference for the IN-PLANE measurements.

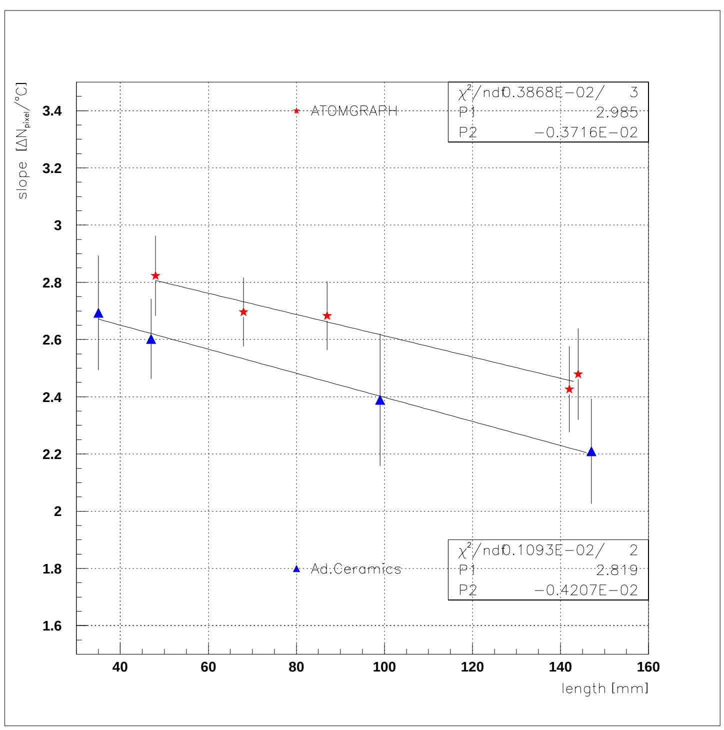


Fig. 6. The IN-PLANE thermal expansion for the samples of Advanced Ceramics and ATOMGRAPH TPG .

The temperature dependence of the thermal expansion coefficient

The expansion of the TPG sample is plotted in Fig.7. The discrepancy of the first points in this plot is caused by the mechanical slackness of our setup, and these points are ignored in the evaluation.

A polynomial (order is two) fit to this data gives the first derivative of the coefficient:

$$\frac{\Delta\alpha_{in-plane}}{\Delta t} = 6.33^{\pm 4.2} \times 10^{-9}\ [{}^{0}C^{-2}]$$

in a temperature range of 30–70^{0}C.

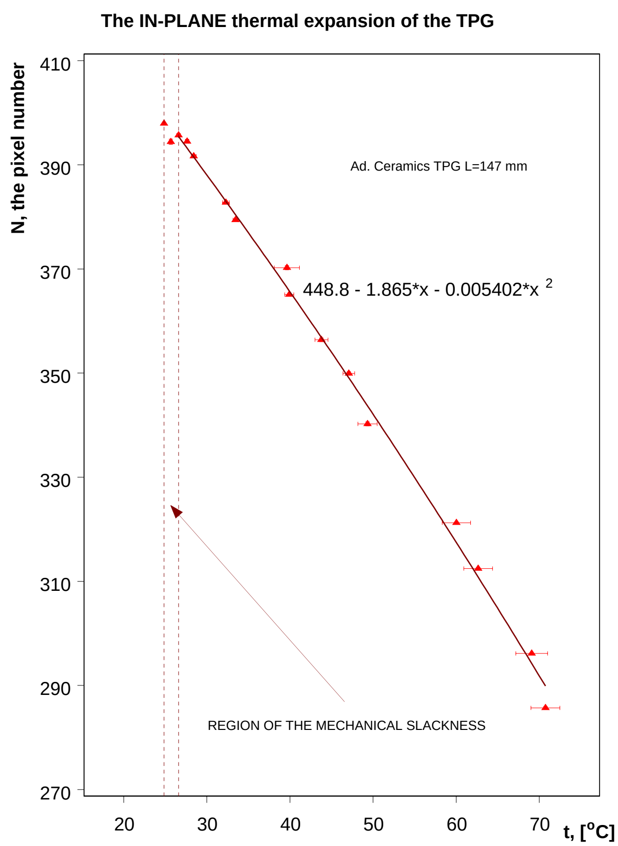


Fig. 7. Thermal expansion of the TPG sample. The temperature regions for the estimation of the temperature dependence of the coefficient of thermal expansion are indicated.

Transverse (OUT-OF-PLANE) Thermal Expansion

A series of measurements of the transverse thermal expansion coefficient was performed. The setup used is very close to that used for longitudinal expansion measurements. The only difference is the way the samples are fixed in the setup. Two samples of TPG (thickness is 2.8 mm and 3.2 mm), a sample of raw PG (thickness is $8mm$) and a sample of material used for X-ray graphite monochromators (thickness is 7.8 mm) were measured. The results are shown in Fig.8. The expansion of each sample is compared to the equivalent expansion of the reference copper samples. For regions between the reference points we made the interpolation as shown in Fig.9. Using these data we determine the out-of plane thermal expansion coefficients to:

- TPG (averaged) : $\alpha_{out-of-plane} = 26.84^{\pm 0.4} \times 10^{-6}$ $[^0C^{-1}]$
- PG (source) : $\alpha_{out-of-plane} = 26.47^{\pm 0.4} \times 10^{-6}$ $[^0C^{-1}]$
- Monochromator : $\alpha_{out-of-plane} = 19.96^{\pm 0.4} \times 10^{-6}$ $[^0C^{-1}]$

The values apply in a temperature range of 25-60^oC.

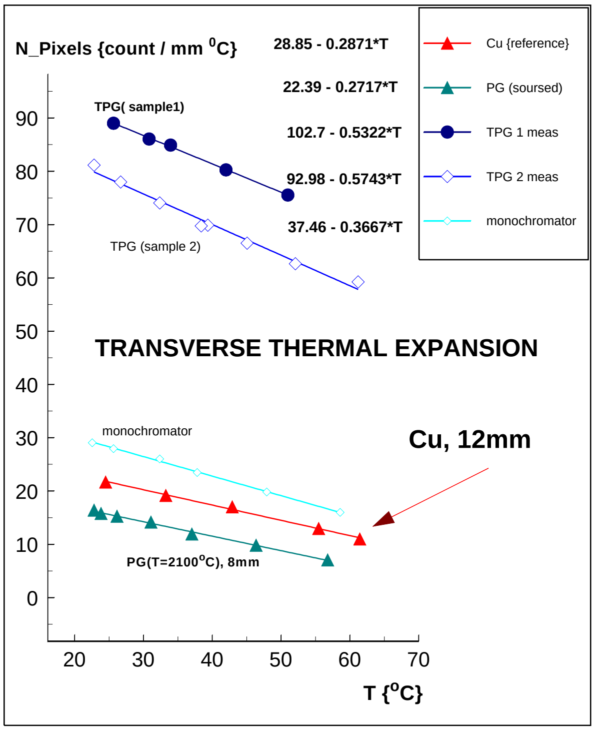


Fig. 8. The OUT-OF-PLANE thermal expansion for the samples ATOMGRAPH TPG, monochromator and raw PG .

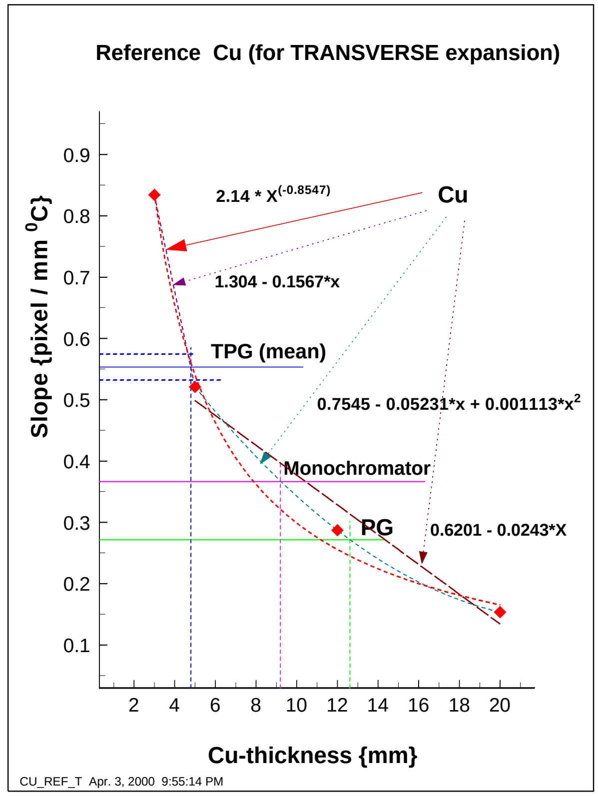


Fig. 9. Thermal expansion of Cu used as reference for measurements of the out-of-plane thermal expansion coefficient. The OUT-OF-PLANE thermal expansion of ATOMGRAPH TPG , monochromator and PG are plotted as horizontal lines.

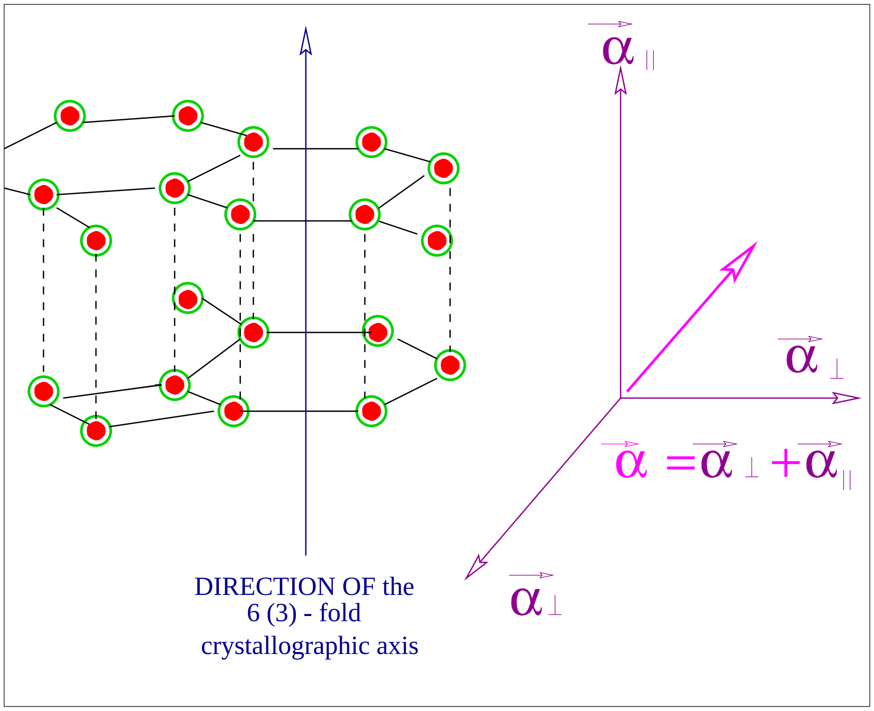
Discussion of the results

The properties of different types of carbon structures depend on many factors. For example, the out-of-plane thermal expansion coefficient of various graphites given in [5], varies within $(2.9^{*}\text{– } 26.7^{\dagger}) \times 10^{-6} [^0C^{-1}]$, the in-plane thermal expansion coefficient varies within $(1.6^{\ddagger}\text{– } 6.7^{\dagger}) \times 10^{-6} [^0C^{-1}]$, in a temperature range of 20–40^0C.

All coefficients are **positive**, whilst according to our measurements the in-plane expansion coefficient of TPG is **negative**. This can be explained as follows. The values given in [5] is for materials with **non-ideal crystal structures**. The family of HOPG (Highly Oriented

* heighly pressed, artificial
† ceylon-graphite
‡ reactor-graphite

Pyrolytic Graphite) like TPG and PG has a structure very close to the ideal graphite crystal. In Fig.10 more recent data for crystalline graphite taken from [6] are shown. These data are in good agreement with our measurements. The different behavior of in-plane expansion HOPG and non-ideal graphite crystal is shown as picture in Fig.10.



TEMPERATURE ^{o}K	Direction of 3 (6)-fold crystallografic axis	
	$\alpha_{\parallel} \ast 10^{6}$	$\alpha_{\perp} \ast 10^{6}$
200	24.9	− 1.33
300	26.7	− 1.22
400	27.5	− 0.91
500	28.1	

Fig. 10. The graphite structure, crystallographic axis and coefficients of thermal expansion according to [6].

Our sample labeled "monochromator" is raw PG additionally annealed under high pressure. In this structure additional interlayer crystalline connections are created. This can explain the different values of the out-of-plane thermal expansion coefficient of our measurements for TPG (PG) and monochromator material. In Fig.11 and Fig.12 the temperature dependence of the in-plane and out-of-plane thermal expansion coefficients is shown for ideal graphite crystals [7]. Our measurements which are in good agreement with these data are indicated as well.

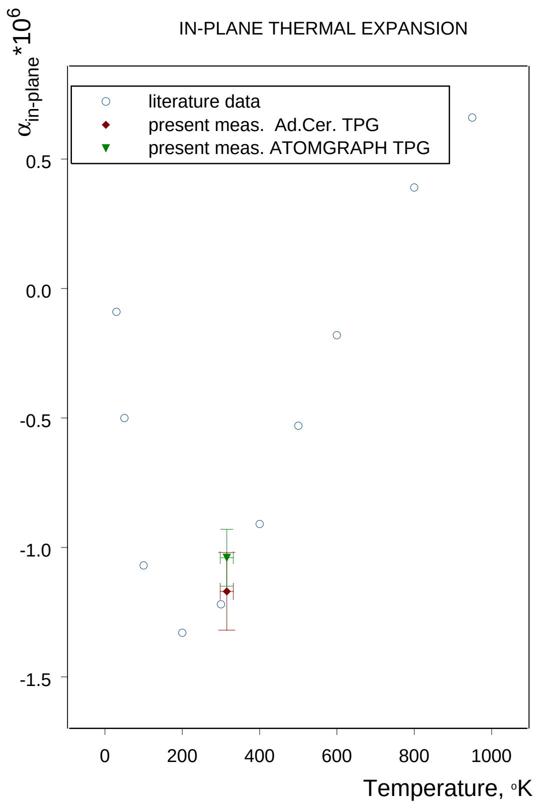


Fig. 11. The IN-PLANE thermal expansion coefficinet of crystal graphite and its temperatures dependence according to [7] and our data.

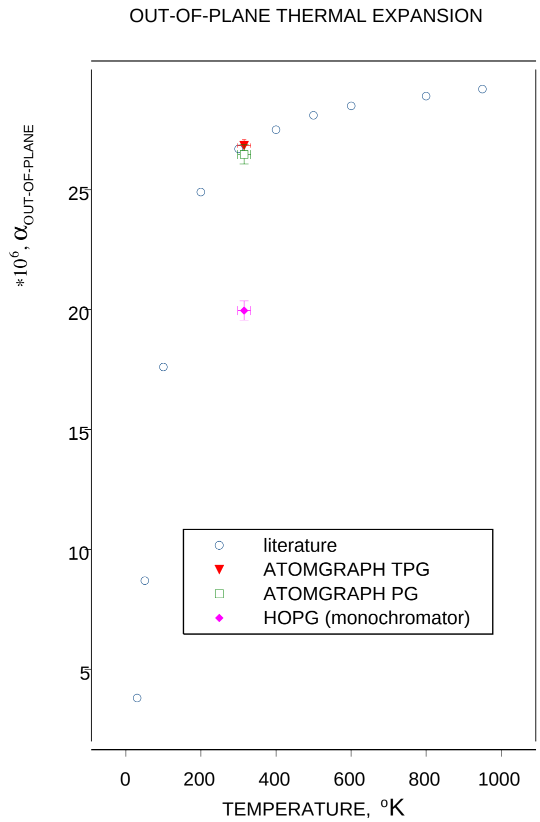


Fig. 12. The OUT-OF-PLANE thermal expansion coefficient of crystal graphite and its temperature dependence according to [7] and our data.

Conclusion

According to our measurements:

- The measured thermal expansion coefficients of TPG are in good agreement with the data for graphite crystals given in [5].
- The measured temperature dependence of $\alpha_{in-plane}$ for the Advanced Ceramics sample is close to the dependence given in [5].
- The in-plane thermal expansion coefficient of TPG (at $t = 25^0C$–60^0C) is :
  - $\alpha_{in-plane} = -1.17^{\pm 0.15} \times 10^{-6}$ $[{}^0C^{-1}]$ for Advanced Ceramics material;
  - $\alpha_{in-plane} = -1.04^{\pm 0.11} \times 10^{-6}$ $[{}^0C^{-1}]$ for ATOMGRAPH material.
- The temperature dependence of $\alpha_{in-plane}$ is:

$$\frac{\Delta\alpha_{in-plane}}{\Delta t} = 6.3^{\pm 4.2} \times 10^{-9}\ [{}^0C^{-2}]$$

  in a temperature range of 30–70^0C.
- The OUT-OF-PLANE thermal expansion coefficient in the temperature region of 25–60^0C is :

TPG (averaged) : $\alpha_{out-of-plane} = 26.84^{\pm 0.4} \times 10^{-6}$ $[{}^0C^{-1}]$
PG (raw) : $\alpha_{out-of-plane} = 26.47^{\pm 0.4} \times 10^{-6}$ $[{}^0C^{-1}]$
Monochromator : $\alpha_{out-of-plane} = 19.96^{\pm 0.4} \times 10^{-6}$ $[{}^0C^{-1}]$

Acknowledgments

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