

Dispersed materials & problems of homogenisation

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We have obtained the physical features of an isotropic and homogeneous environment on both theoretical and experimental levels, in particular when thermal features are linked through the $\lambda = a \rho C_p$ relation. As far as dispersed samples are particularly concerned, this largely used correlation is not confirmed in out-of balance non-homogeneous samples (transitional rate of flow /regime). So, we have developed, by electronic analogy a theoretical model that allows us to study the thermal performance and to discuss conditions of homogenization in such material.

KEY WORDS

Thermal characterization, dispersed materials, thermal conductivity, thermal diffusivity and homogenization.

I. INTRODUCTION

A dispersed material is composed of a rigid solid matrix (environment 1) in which a random or an ordered assemblage of solid bodies with varied shapes and dimensions is distributed (environment 2). The relative concentration of these constituents allows to attribute a very interesting mechanical, thermal and electrical properties to these materials [2]. These dispersed materials are particularly contributive in specific industrial applications. Both environments can particularly have a very different thermal performance : it depends on whether the materials are good or bad conductors.

It is essential to describe and modelise the heat transfer processes in such complex environments. During these processes, We have to understand the thermo physical performance in every environment and to deal with the problem of homogenization and interface between the processes different phases.

In this work, we propose a theoretical model to determine the thermal features of dispersed materials according to the properties of each phase. We will also study the degree of validity of such a model and as a result, we will try to find to which extent we can liken such a complex environment to an equivalent environment with real properties.

II. A HOMOGENEOUS THEORETICAL MODEL

Let's study a cylindrical sample with a circular basis (R is for Radius, and e for thickness in figure1), which is subject to its axis of revolution, and in the same time to an impulse incidental thermal flow. We assume that the materials thermal features remain invariable during the impulse interval which should be shorter than the time the sample temperature goes up after reception of the pulse [1 et 3].

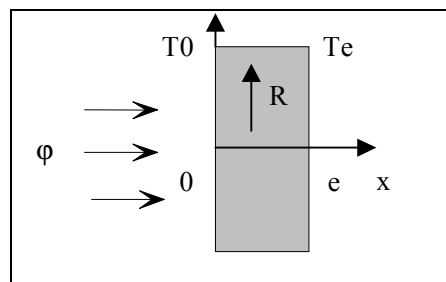


FIG1 : Model of a homogeneous sample

In this model, we choose a small ratio of thickness by the sample radius, to consider the heat diffusion process as one-dimensional. In this way, we ignore lateral losses. Finally, we consider that the heat time and impulse interval start at the starting point $x = 0$.

As far as the source incidental signal was concerned, preliminary tests have pointed out that the thermal impulse at the initial instant $t = 0$, can be described through a temperature area $T(x,t)$ as follows :

$$T(x,t) = Tamb \left\{ 1 + \frac{t}{20\alpha_f^2} \exp\left(-\frac{t}{t_f}\right) + \frac{t}{t+10} \exp(-0.0075) \right\}$$

The FOURIER's law of heat propagation through conduction allows to link the density heat flow denseness ϕ existing at a point in a material with a constant thermal conductivity λ , to the temperature pressure that exists in the material. This will be possible through :

$$\phi = -kgradT \quad (1)$$

Therefore, if we consider an unchanging environment, in the absence of a heat source and of chemical reaction and if thermal features were independent from temperature, the equation (1) will be one-dimensional :

$$\text{For } 0 < x < e : \frac{\partial T}{\partial t} = a \frac{\partial^2 T}{\partial x^2} \quad (2)$$

With : $a = \kappa / \rho C_p$, κ , ρ , C_p and a represent thermal conductivity ($\text{W/m}^\circ\text{C}$), voluminal mass (Kg/m^3), Latent heat ($\text{J/kg}^\circ\text{C}$) and thermal diffusivity (m^2/s) respectively.

The adopted resolution method is founded on the identification of thermo physical features of both environments that respectively stand for the dispersed material and the equivalent homogeneous environment if there is one. Since LAPLACE transformed the equation (2), it becomes possible to formulate the outflow parameters $T(e, p)$ and $\phi(e, p)$ according to inflow parameters $T(0, p)$ et $\phi(0, p)$. Then we can express the temperature area $T(x, t)$ and flow $\phi(x, t)$ in this matrix form:

$$\begin{pmatrix} T(e, p) \\ \phi(e, p) \end{pmatrix} = \begin{pmatrix} A & B \\ C & D \end{pmatrix} \begin{pmatrix} T(0, p) \\ \phi(0, p) \end{pmatrix} \quad (3)$$

$$\text{with : } \begin{pmatrix} A & B \\ C & D \end{pmatrix} = M = \begin{pmatrix} ch\alpha e & -\frac{1}{k\alpha} sh\alpha e \\ -k\alpha sh\alpha e & ch\alpha e \end{pmatrix}$$

and $\alpha = \sqrt{\frac{p}{a}}$; p being LAPLACE's variable.

It should be noted that the matrix M has a determinant equal to the unit ; i.e.: $ch^2\alpha e - \frac{k\alpha}{k\alpha} sh^2 = 1$ (4), and both

parameters A and D are the same (this property is generally verified in a homogeneous environment).

This method allows, then, to link the flow and the temperature of two surfaces of a sample in a transitional rate of flow /regime.

III. APPLICATION TO DISPERSED MATERIALS

For symmetry reasons and to simplify calculations, we regard the basic cell as a cylinder with R_1 for radius and e for thickness, in the center of which there is a cylindrical cell whose thickness is $(e_2 - e_1)$ and radius is $R_2 < R_1$ (figure 2a).

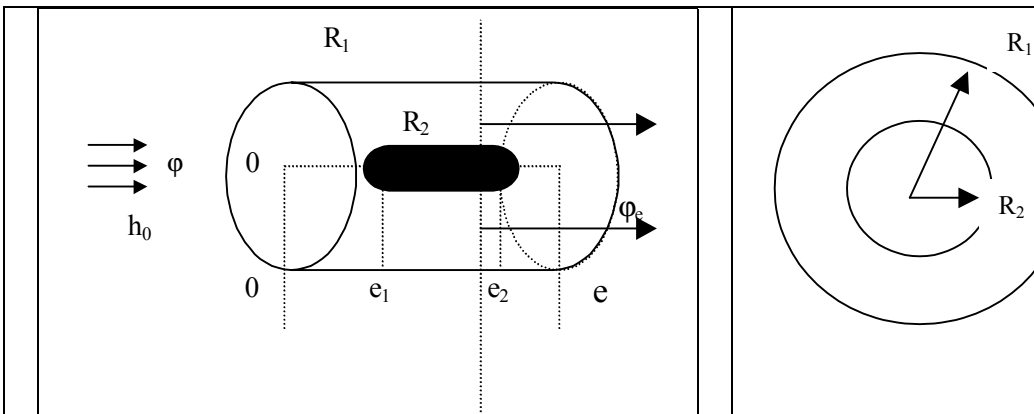


FIG 2a : Basic cell of a dispersed material

While adopting the same hypotheses than previously, we consider the cell plane section (figure 2b) made up of two different environments or milieus (milieu 1 and milieu 2) represented as three layers (L_1 , L_2 and L_3) (figure 2b).

Layers (L_1) and (L_3), have a same thermal conductivity κ_1 , a same thermal diffusivity a_1 and thicknesses respectively e_1 and e_3 . Similarly, the Layer (L_2) has thermal conductivity κ' , thermal diffusivity a' and thickness e_2 .

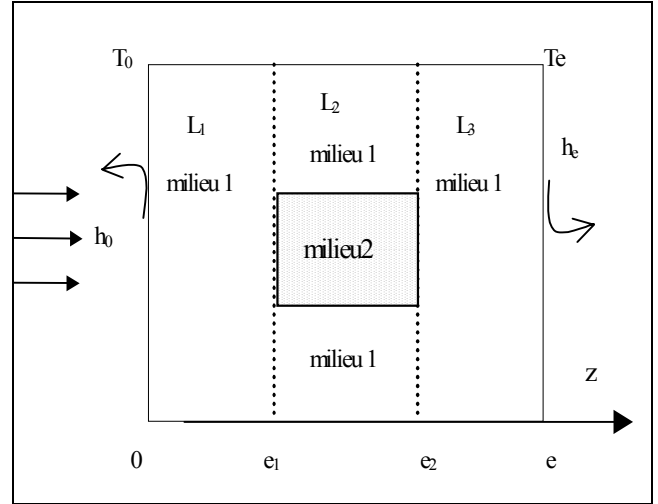


FIG 2b : A cell projection of a dispersed sample model

If we apply the same previously used techniques that LAPLACE employed to transform equations, we will be able to determine the outflow parameters of the model $\bar{T}(e, p)$ and the model $\bar{\phi}(e, p)$ according to inflow parameters $\bar{T}(0, p)$ and $\bar{\phi}(0, p)$. As a matrix formula, we have :

$$\begin{pmatrix} \bar{T}(e, p) \\ \bar{\phi}(e, p) \end{pmatrix} = \begin{pmatrix} A_3 & B_3 \\ C_3 & A_3 \end{pmatrix} \begin{pmatrix} A' & B' \\ C' & D' \end{pmatrix} \begin{pmatrix} A_1 & B_1 \\ C_1 & A_1 \end{pmatrix} \begin{pmatrix} \bar{T}(0, p) \\ \bar{\phi}(0, p) \end{pmatrix} \quad (5)$$

A continuous flow between Layers (L_1) - (L_2), and Layers (L_2) - (L_3), enables us to write down this :

$$\begin{pmatrix} A' & B' \\ C' & D' \end{pmatrix} = \begin{pmatrix} \frac{\gamma K_2 A_2 + (1-\gamma) K_1 A_1}{\gamma K_2 + (1-\gamma) K_1} & \frac{\gamma K_2 B_2 + (1-\gamma) K_1 B_1}{\gamma K_2 + (1-\gamma) K_1} \\ \frac{\gamma C_2 + (1-\gamma) C_1}{\gamma A_2 + (1-\gamma) A_1} & \frac{\gamma D_2 + (1-\gamma) D_1}{\gamma A_2 + (1-\gamma) A_1} \end{pmatrix}$$

The equivalent environment to the model in figure 2b, if there is one, may be described through the following matrix :

$$\begin{pmatrix} \bar{T}(e, p) \\ \bar{\varphi}(e, p) \end{pmatrix} = \begin{pmatrix} A_{eq} & B_{eq} \\ C_{eq} & D_{eq} \end{pmatrix} \begin{pmatrix} \bar{T}(0, p) \\ \bar{\varphi}(0, p) \end{pmatrix} \quad (6)$$

Condition (3) in this situation is written this way :

$$A_{eq} D_{eq} - B_{eq} C_{eq} = 1 \quad (7)$$

The identification between matrix (6) and matrix (7) can be represented as :

$$\begin{pmatrix} A_{eq} & B_{eq} \\ C_{eq} & D_{eq} \end{pmatrix} = \begin{pmatrix} A_3 & B_3 \\ C_3 & D_3 \end{pmatrix} \begin{pmatrix} A' & B' \\ C' & D' \end{pmatrix} \begin{pmatrix} A_1 & B_1 \\ C_1 & D_1 \end{pmatrix} \quad (8)$$

$$A_i = ch(\alpha_i e_i); B_i = -Sh(\alpha_i e_i)/\kappa_i \alpha_i; D_i = ch(\alpha_i e_i);$$

$$C_i = \kappa_i sh(\alpha_i e_i); \alpha_i = \sqrt{\frac{p}{a_i}} \text{ with}$$

γ representing ratio ($l_2 = \gamma l_1$) and the width of environment 1 and environment 2 respectively.

The value-to-value identification of the two members of identity (7), gives a four-equation system. The development of every equation is limited to order 2, which permits to establish the two following identities, identity [2,4] :

$$\frac{e^2}{a_{eq}} = \frac{e_1^2}{a_1} + \frac{e_3^2}{a_1} + \frac{e_2^2}{(1-\gamma)k_1 + \gamma k_2} \left(\frac{(1-\gamma)k_1}{a_1} + \frac{\gamma k_2}{a_2} \right) + \frac{2e_1 e_3}{a_1} + \frac{2e_2 e_3}{(1-\gamma)k_1 + \gamma k_2} \left(\frac{k_1}{a_1} \right) + 2e_1 e_2 \left(\frac{(1-\gamma)}{a_1} + \frac{k_2}{k_1 a_2} \right) \quad (9)$$

$$\frac{e^2}{a_{eq}} = \frac{e_1^2}{a_1} + \frac{e_3^2}{a_1} + \left(\frac{(1-\gamma)}{a_1} + \frac{\gamma}{a_2} \right) e_2^2 + \frac{2e_1 e_3}{a_1} + \frac{2e_1 e_2}{(1-\gamma)k_1 + \gamma k_2} \left(\frac{k_1}{a_1} \right) + \frac{2e_2 e_3}{(1-\gamma)k_1 + \gamma k_2} \left(\frac{(1-\gamma)}{a_1} + \frac{k_2}{k_1 a_2} \right) \quad (10)$$

$$\frac{e}{k_{eq}} = \frac{e_1}{k_1} + \frac{e_3}{k_1} + \frac{e_2}{(1-\gamma)k_1 + \gamma k_2} \quad (11)$$

$$\frac{e k_{eq}}{a_{eq}} = \frac{e_1 k_1}{a_1} + \frac{e_3 k_1}{a_1} + \left(\frac{(1-\gamma)k_1}{a_1} + \frac{\gamma k_2}{a_2} \right) e_2 \quad (12)$$

This mathematical processing has a main advantage: it generates relations between the characteristic magnitudes of every environment such as thermal thickness and conductivity and it has a representing equation system which is independent from the layout space and from the surrounding environment.

* Relations (9) and (10) indicate the thermal diffusivity of the equivalent environment whereas Relation (11) expresses its thermal conductivity. As for Relation (12), it confirms the mixing law of the equivalent environment.

The sample is considered as homogeneous if equation (9) and (10) are identical. To this end and if $e_1 = e_3$, We must absolutely verify the following relations :

$$\left(\frac{(1-\gamma)}{a_1} + \frac{\gamma}{a_2} \right) \left((1-\gamma)k_1 + \gamma k_2 \right) = \left(\frac{(1-\gamma)k_1}{a_1} + \frac{\gamma k_2}{a_2} \right)$$

$$\text{and} \left(\frac{k_1}{a_1((1-\gamma)k_1 + \gamma k_2)} \right) = \left(\frac{(1-\gamma)}{a_1} + \frac{\gamma k_2}{a_2 k_1} \right) \quad (13)$$

These necessary conditions enable us to use the relation : $a_{eq} = \kappa_{eq}/(p C_p)_{eq}$

IV. MEANS OF DIAGNOSIS AND EXPERIMENTAL METHODS

We have designed an experimental assembly, whose synoptic diagram is represented in figure 3. It allows us to measure the thermal diffusivity of an environment by using the Parker's method [1].

The principle measurement consists of exposing a sample plane surface to a short thermal impulse; the impulse generates an increase in temperature in the opposite face according to time. We obtain this information in a thermogramme form (Figure 4), from an electrical signal given by a thermocouple (Chrome-Nickel/Chrome Alloy). While studying the thermogramme, we have been able to come back to the visible value of thermal diffusivity.

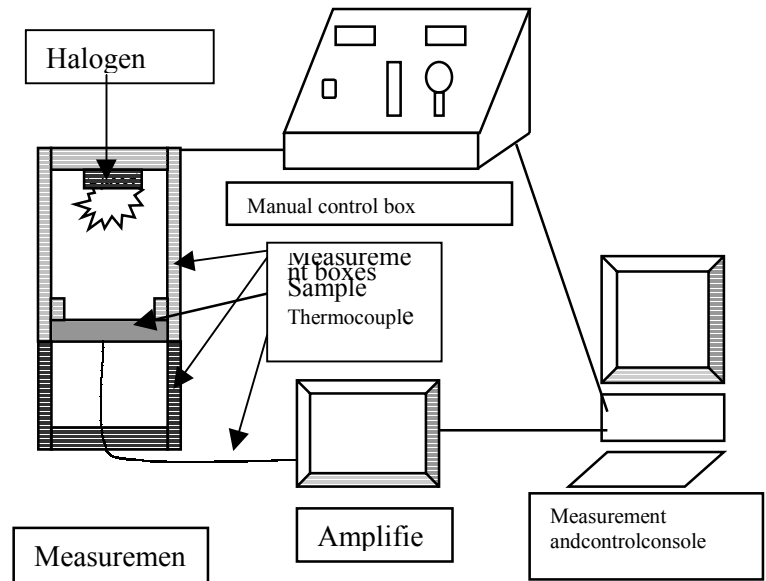


FIG 3: Assembly for thermal diffusivity measurement

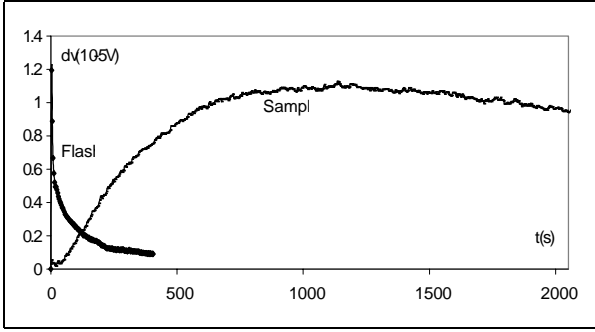


FIG 4: Example of the incidental flux (flash) and of the thermogramme produced by the sample

Samples	$e_1/(e_2-e_1)/(e-e_2)(Cm)$	a_{exp} $10^{-7}m^2/s$	a_{eq1} $10^{-7}m^2/s$	a_{eq2} $10^{-7}m^2/s$	a_{eq} $10^{-7}m^2/s$	Δa (exp/analy)	$\Delta H =$ $\Delta a_{acq1/acq2}$	Δeff
P/C	0.7/1.4/0.7	6.95	5.7	4.7	5	28%	17%	44%
P/Z	0.7/1.4/0.7	6	6.16	5.76	5.95	0.8%	6.7	60%
P/Bs	0.7/1.4/0.7	6.95	6.38	4.82	5.6	19%	24%	26%
P/G	0.7/1.4/0.7	8.5	12.5	17.7	15	43%	29%	93%
C/P	0.65/1.4/0.65	4.15	3.8	3.4	3.6	13.5%	9%	44%
C/Z	0.7/1.4/0.7	3.85	3.84	3.76	3.76	2.3%	4%	29%
C/B	0.65/1.4/0.65	4.1	3.44	4.8%	3.1	24%	14%	59%
C/G	0.65/1.4/0.65	6.1	7.39	8.57	8	23%	13.7%	88%

Table I : Comparisons of analytical and experimental results

From results in Table I, we can see :

- The average relative error between thermal diffusivity values obtained by experimental and analytical calculation does not exceed 3% when the relative difference between the analytical values a_{eq1} and a_{eq2} is low and it is all the more important in the opposite case. It varies of 0.8 % when the homogenization condition ΔH tends towards zero at 43% if ΔH is important.

- Environment 1 (matrix) imposes its thermal diffusivity to both environments, considered as a whole. It is interesting to observe that when environment (1) is a bad thermal conductor in comparison with environment (2), the real thermal response of the sample made by both environments is conditioned by thermal diffusivity of environment (1). For example, for a variation in thermal diffusivity between Plaster ($a_1=7.3 \cdot 10^{-7}m^2/s$) and Graphite ($a_2=600 \cdot 10^{-7}m^2/s$), the visible experimental value of the equivalent thermal diffusivity is $a_{exp} \sim 8.5 \cdot 10^{-7}m^2/s$ (See Tableau I).

V. SEARCH FOR THE EQUIVALENT ENVIRONMENT, HOMOGENIZATION

The Mathematical model (13) shows that the dispersed material can be considered as homogeneous only when quantities ΔH and/or Δeff tends to zero. Since these conditions are difficult, even impossible to be fulfilled, we rather try to find the range of Δeff values for which we can tolerate an error on ΔH .

VI. RESULTS AND DISCUSSIONS

The visible values of thermal diffusivity as regards tested samples are presented in Table (I) and are deduced from an arithmetic mean made according to five measures read on the corresponding thermogrammes according to the Degiovanni's method [3]. We compare afterwards the calculated and experimental values of thermal diffusivity

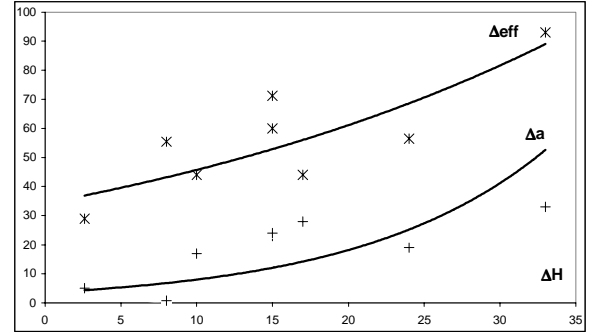


FIG 5 : Comparisons between the difference variations in effusivity and in diffusivity according to the homogenization condition.

Figure 5 represents variations in the relative difference of the respective thermal effusivity between both environments that make the dispersed material (column 9) and of the relative error between experimental and calculated values of thermal diffusivity (column 7) respectively. And this is depending on homogenization (column 8)

We notice that :

- for a relative difference about a_{eq} of the order of 5%, we can tolerate a relative difference on thermal effusivity of both environments reaching almost 30 % (figure 5). In these conditions, and with a good approximation, the dispersed material can be considered as homogeneous

- The relative error between experimental and calculated values is all the more important than the relative difference on a_{eq} .

VII. CONCLUSION

Theoretically speaking, the proposed model has been founded on the extension of the quadripole notion. Through this model, we have been able to model in transitional rate of flow the thermal transfer through a cylinder, only one face of which is subject to a thermal brief impulse. This transfer can be symbolically represented by a matrix system that links the temperature and the flux of the two faces. By identifying the equations given by the model and those of the supposedly equivalent environment, we have been able to deduce the sample thermal features according to thermo-physical and geometric parameters of the different constituents. It is especially us who create the homogenization conditions of such a sample.

As for our experiments, we have designed a simple experimental measurement assembly of thermal diffusivity, founded on the boxes model. The sample heating is made through a non intrusive excitation (Laser or Halogen lamps) and results have been operated from a thermogramme.

As a first step, we have measured the thermal features of used homogeneous samples to develop our dispersed samples. This enables us to corroborate our model.

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Nomenclature:

- Z** : cylinder glazed with ZrO₂ (Dioxide de Zirconium) stabilized in weight by 5% of CaO (Lime); $a = 5.3 \cdot 10^{-7} \text{ m}^2/\text{s}$ et $\kappa = 1.2 \text{ W/mk}$
- B** : Timber cylinder; $a = 3.5 \cdot 10^{-7} \text{ m}^2/\text{s}$ et $\kappa = 0.28 \text{ W/mk}$.
- C** : concrete cement cylinder; $a = 3.5 \cdot 10^{-7} \text{ m}^2/\text{s}$ et $\kappa = 0.55 \text{ W/mk}$.
- P** : Plaster cylinder; $a = 7.3 \cdot 10^{-7} \text{ m}^2/\text{s}$ et $\kappa = 0.55 \text{ W/mk}$.
- G** : Commercial graphite cylinder; $a = 600 \cdot 10^{-7} \text{ m}^2/\text{s}$ et $\kappa = 76.7 \text{ W/mk}$.
- e₁** (m) : Thickness of environment 1(matrix).
- e₂** (m) : Thickness of environment 2 (second phase).
- a_{eq1}** ($10^{-7} \text{ m}^2/\text{s}$): thermal diffusivity calculated with **a_{eq1}** (equation 9)
- a_{eq2}** ($10^{-7} \text{ m}^2/\text{s}$): thermal diffusivity calculated with **a_{eq2}**. (equation 10)
- a_{eq}** ($10^{-7} \text{ m}^2/\text{s}$): Average value of thermal diffusivity calculated with **a_{eq1}** and **a_{eq2}**.
- a_{exp}** : Visible thermal diffusivity according to the Degiovanni's studying method.
- Δa (exp/analy)** : the relative error between visible value **a_{exp}** and **a_{eq}**.
- ΔH** : Relative difference between diffusivity value **a_{eq1}** and **a_{eq2}** : homogenization condition of a dispersed material.
- Δeff** : the relative difference between the effusivity values of environment (1) and environment (2):
- P/C. P/Z. P/B. and P/Gr are respectively samples of dispersed materials whose matrix is made from Plaster in the first phase and from Cement, ZrC, Wood and Graphite in the second phase.
- C/P. C/Z. C/B. and C/Gr are respectively samples of dispersed materials, whose matrix is made from Cement in the first phase. In the second phase, the said matrix is respectively made from Plaster, ZrC, Wood and of Graphite