



Dyna

ISSN: 0012-7353

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Universidad Nacional de Colombia

Colombia

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Dyna, vol. 83, núm. 195, febrero, 2016, pp. 29-33

Universidad Nacional de Colombia

Medellín, Colombia

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## Crack growth in pyrographite under the conditions of radiation

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Received: November 27<sup>th</sup>, 2014. Received in revised form: April 12<sup>th</sup>, 2015. Accepted: December 10<sup>th</sup>, 2015.

### Abstract

The damage induced by radiation in the pyrographite is accompanied by significant plastic deformation. Microcracks arise in the edges of the radiated areas that develop radially in the direction of the unaffected matrix. The peculiarities in the formation of the tension state of the radiated area and adjacent unaffected areas are analyzed in order to explain the reasons behind the growth of cracks. The analysis is carried out on graphite disks with constant thicknesses that are exposed to radiation with high-energy electrons in an HVTEM-JEOL 1000. A differential equation for specific load conditions is obtained from the analysis of equilibrium conditions of a disc-shaped element.

**Keywords:** Pyrographite, crack, irradiation, HVTEM, stress state.

## Crecimiento de grietas en pirografito bajo condiciones de radiación

### Resumen

El daño inducido por radiación en el pirografito va acompañado por una significativa deformación plástica. En los bordes de la zona radiada surgen microgrietas que se desarrollan de manera radial en dirección de la matriz no afectada. Se analizan las particularidades en la formación del estado de tensión de la zona radiada, y en los campos adyacentes no afectados por la radiación para explicar las razones que originan el crecimiento de grietas. El análisis se lleva a cabo sobre discos de grafito con espesor constante, expuestos a radiación con electrones de altas energías dentro de un HVTEM-JEOL 1000. Del análisis de las condiciones de equilibrio de un elemento en forma de disco, se obtiene una ecuación diferencial para condiciones específicas de carga.

**Palabras clave:** Pirografito, grieta, irradiación, HVTEM, estado de esfuerzos.

### 1. Introduction

Exposure of pyrographite to radiation with high-energy electrons causes a type  $K \rightarrow A$  phase transition, accompanied by considerable plastic deformation. It was established that microcracks emerge at the edges of the irradiated zone, these microcracks then grow unceasingly in the radial direction within the irradiated matrix, Fig. 1.

To clarify the possible causes of the incubation and growth of cracks, it is necessary to examine the

peculiarities in the formation of a stress state in the irradiated zone as well as in the conjugated matrix zone that is not affected by the radiation. This cannot be caused by the change of the temperature field in the irradiated zone, since the heating magnitude in the HVTEM column by means of the electron beam does not exceed 40°C [1,2]. Besides, prolonged radiation experiments undoubtedly favor the balance of the temperature field gradients in both the sample thickness and the radial direction within the irradiated zone limits.

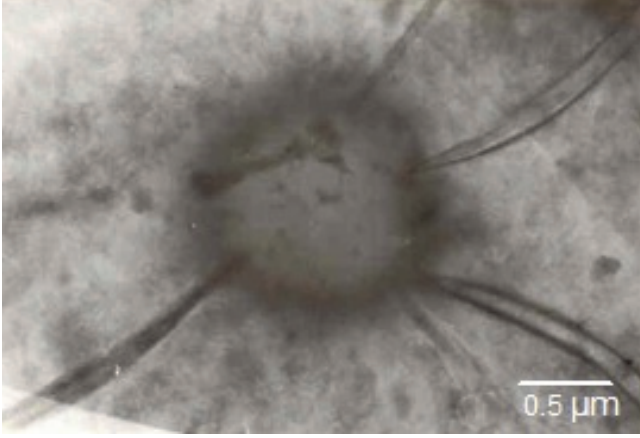


Figure 1. Cracks in pyrographite emerged as a result of irradiation with energy electrons: HVTEM-JEOL 1000.  
Source: The authors.

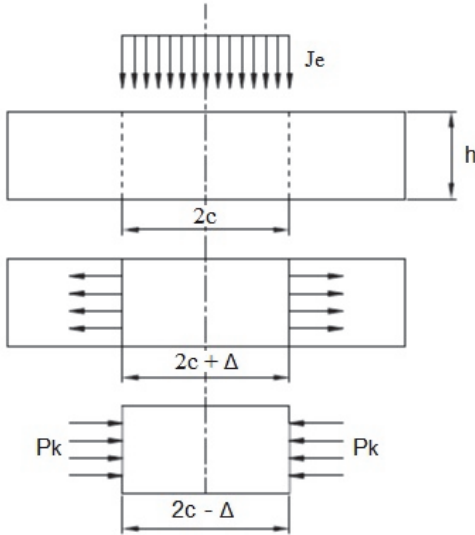


Figure 2. Radiation scheme pyrographite samples: Experiments conducted within the HVTEM, JEOL 1000.  
Source: The authors.

## 2. Theoretical approach

The radiation scheme of pyrographite samples in the form of flat disks performed within the HVTEM is shown in Fig. 2.

As a result of a prolonged high energy electron bombing, the amorphization is developed in the pyrographites, which leads to a decrease in the material specific volume in the irradiated zone [3]. Considering that the irradiated zone is surrounded by a matrix that is not affected by radiation, a forced interaction consequently arises between it and the loosened of the irradiated zone's material [4].

From the equilibrium conditions of a circular sheet element of constant thickness analysis, it is possible to obtain a differential equation for a determined loading scheme. Following the plate theory methods [5,6], we obtain the equilibrium equation for the plate and solve it for the initial and boundary conditions that are derived from the supposed scheme

of interaction between an irradiated zone wrapped by a sample matrix. The differential equation is obtained as follows:

$$\sigma_r + \frac{d\sigma_r}{dr} - \sigma_\tau = 0$$

or

$$\frac{\alpha(\sigma_r \cdot r)}{dr} - \sigma_\tau = 0 \quad (1)$$

where  $\sigma_r$  is the radial stress, and  $\sigma_\tau$  is the tangential stress.

The link between the components of the stress tensor  $\sigma_r$ ,  $\sigma_\tau$ ,  $\sigma_z$  and the deformations  $\varepsilon_r$ ,  $\varepsilon_\tau$  can be expressed in terms of the Generalized Hook's Law:

$$\varepsilon_r = \frac{1}{E} [\sigma_r - \mu(\sigma_\tau + \sigma_z)]$$

$$\varepsilon_\tau = \frac{1}{E} [\sigma_\tau - \mu(\sigma_r + \sigma_z)] \quad (2)$$

where E is the Young modulus,  $\mu$  is the Poisson modulus.

Supposing that in the conditions of our problem  $\sigma_z = 0$ , it is possible to obtain the expressions for  $\sigma_r$  and  $\sigma_\tau$  from the eq. (2) as follows:

$$\sigma_r = \frac{E}{1 - \mu^2} \cdot (\varepsilon_r + \mu\varepsilon_\tau)$$

$$\sigma_\tau = \frac{E}{1 - \mu^2} \cdot (\varepsilon_\tau + \mu\varepsilon_r) \quad (3)$$

It is not difficult to prove that

$$\varepsilon_r = \frac{du}{dr}$$

but

$$\varepsilon_\tau = \frac{u}{r} \quad (4)$$

where  $\varepsilon_r$  is the radial deformation,  $\varepsilon_\tau$  is the tangential deformation,  $u$  is the radial displacement.

After substituting eq. (4) in the eq. (3) we obtain:

$$\sigma_r = \frac{E}{1 - \mu^2} \cdot \left( \frac{du}{dr} + \mu \frac{u}{r} \right)$$

$$\sigma_\tau = \frac{E}{1 - \mu^2} \cdot \left( \frac{u}{r} + \mu \frac{du}{dr} \right) \quad (5)$$

After substituting eq. (5) in the equilibrium differential equation (1), the latter can be expressed as:

$$\frac{d^2u}{dr^2} + \frac{1}{r} \frac{du}{dr} - \frac{u}{r^2} = 0$$

or

$$\frac{d}{dr} \left[ \frac{du}{dr} + \frac{u}{r} \right] = 0 \quad (6)$$

Finally, it can be presented in the following form:

$$\frac{d}{dr} \left[ \frac{1}{r} \cdot \frac{d(ur)}{dr} \right] = 0 \quad (7)$$

Observations of the zone irradiated by HVTEM show an absolute absence of extinction contours, even in the very moment when microcracks start to form. This circumstance reveals the absence of bending during the interaction of forces in the irradiated zone and the surrounding matrix, i.e., the latter has compression characteristics at the edges of the zone. The integration of eq. (6) provides a general solution for radial displacements in the zone affected by radiation:

$$u = c_1 r + \frac{c_2}{r} \quad (8)$$

where  $c_1$  and  $c_2$  are integration constants, the values of which come from the contour conditions.

In our problem, these can be expressed as  $r = c$ ;  $\sigma_r = -p_k$ ;  $r = \infty$ ;  $\sigma_r = 0$  for the matrix outside the zone of radiation, and  $r = c$ ;  $\sigma_r = p_k$ ;  $r = 0$ ;  $u = 0$  for the irradiated zone.

To determine the integration constants in solution (8), we inserted them in eq. (5) and consequently obtain:

$$\begin{aligned} \sigma_r &= \frac{E}{1 - \mu^2} \left[ c_1(1 + \mu) - c_2(1 - \mu) \frac{1}{r^2} \right], \\ \sigma_\tau &= \frac{E}{1 - \mu^2} \left[ c_1(1 + \mu) + c_2(1 - \mu) \frac{1}{r^2} \right]. \end{aligned} \quad (9)$$

Considering the contour conditions, eq. (9) for the plate placed outside the irradiated zone obtained the following form:

$$\frac{E}{1 - \mu^2} \left[ c_1(1 + \mu) - \frac{c_2}{c^2}(1 - \mu) \right] = p_k \quad (10)$$

from which the expression for the integration constants  $c_1$  and  $c_2$  can be presented as

$$\begin{aligned} c_1 &= 0, \\ c_2 &= \frac{p_k c^2 (1 + \mu)}{E} \end{aligned} \quad (11)$$

Substituting expression (10) in eq. (8) and (9), we obtain

$$u = \frac{p_k c^2 (1 + \mu)}{Er} \quad (12)$$

$$\sigma_r = -\frac{p_k c^2}{(1 - \mu)r^2} \quad (13)$$

$$\sigma_\tau = \frac{p_k c^2}{(1 + \mu)r^2} \quad (14)$$

Similarly, for the irradiated zone, the substitution of the corresponding boundary conditions leads to the following expressions:

$$\begin{aligned} \frac{E}{1 - \mu^2} \left[ c_1(1 + \mu) - \frac{c_2}{c^2}(1 - \mu) \right] &= -p_k, \\ u(r = 0) &= c_1 r + c_2 \frac{1}{r} = 0 \end{aligned} \quad (15)$$

From which we deduce:

$$\begin{aligned} c_2 &= 0, \\ c_1 &= \frac{p_k(1 - \mu)}{E} \end{aligned} \quad (16)$$

After substituting the values of  $c_1$  and  $c_2$  in eq. (8) and (9) we obtain:

$$u = -\frac{p_k(1 - \mu)^2}{E} \quad (17)$$

$$\sigma_r = p_k \quad \text{and} \quad \sigma_\tau = p_k \quad (18)$$

### 3. Discussion of the results

The material in the irradiated area has been under the effect of a biaxial stress state in compression, the magnitude of which can be determined by the degree of amorphization process development. The surrounding matrix is also in a state of biaxial stress. However, the compression forces decrease in the radial direction that is inversely proportional to the square of the radius measured from the center spot of radiation, but the stress forces, which are equal by modulus to compression forces, act in the tangential direction. A physical approach to the problem is to determine the magnitudes of the contact pressures  $p_k$ , which act in the limits of the irradiated area. To find the value of this pressure, we use the solution proposed by Lamé [5], which describes the emergence of a contact pressure when a cylindrical piece joins a sheet having a central perforation with a certain tightening  $\Delta c$ :

$$u_2 - u_1 = \Delta c \quad (19)$$

where  $u_2$  is the sheet displacement in the borders of the irradiated zone described by eq. (12),  $u_1$  is the zone of displacement in the boundary described by eq. (17),  $\Delta c$  is the tightening intensity.

In this case the tightening is a result of the swelling of the irradiated zone due to amorphization development stimulated by radiation. We determined its size from the conditions of substance mass conservation in the irradiated zone during the radiation process applied to it:

$$4\pi c^2 \cdot h \gamma_k = 4\pi(c + \Delta c)^2 \cdot (h + \Delta h) \gamma_{rz} \quad (20)$$

where  $\gamma_k$  is the initial crystalline pyrographite density,  $\gamma_{rz}$  is the present value of the material density in the irradiated zone,  $h$  is the sample thickness, and  $c$  is the irradiated zone radius.

After certain transformations, eq. (20) can be represented in the following form:

$$\gamma_k = \left(1 + \frac{\Delta c}{c}\right)^2 \cdot \left(1 + \frac{\Delta h}{h}\right) \cdot \gamma_{rz} \quad (21)$$

Recognizing that during the pyrographite amorphization process the deformation develops isotropically, i.e.,  $\varepsilon_x = \varepsilon_y = \varepsilon_z$ , and  $\gamma_{rz}$  is the density of totally amorphized pyrographite  $\gamma_a$ , and the crystalline pyrographite is expressed as  $\gamma_k$ , then according to the mixture law:

$$\gamma_{rz} = \gamma_a \alpha + \gamma_k (1 - \alpha) \quad (22)$$

where  $\alpha$  is the amorphization degree.

Eq. (21) can be expressed in the following form:

$$\gamma_k = \left(1 + 3 \frac{\Delta c}{c}\right) \cdot [\gamma_a \alpha + \gamma_k (1 - \alpha)] \quad (23)$$

Solving eq. (23) with respect to  $c$  and substituting eq. (22) in it we obtain

$$\Delta c = \frac{c}{3} \left[ \frac{\alpha(\gamma_k - \gamma_a)}{\gamma_a \alpha + \gamma_k (1 - \alpha)} \right] \quad (24)$$

Substituting eq. (24) in eq. (19), we have that for  $r = c$

$$\begin{aligned} \frac{p_k c \cdot (1 + \mu_k)}{E_k} + \frac{p_k c (1 - \mu_k)}{E_a} \\ = \frac{c}{3} \left[ \frac{\alpha(\gamma_k - \gamma_a)}{\gamma_a \alpha + \gamma_k (1 - \alpha)} \right] \end{aligned} \quad (25)$$

where eq. (26) is for the contact pressure:

$$p_k = \frac{\frac{\alpha(\gamma_k - \gamma_a)}{\gamma_a \alpha + \gamma_k (1 - \alpha)}}{3 \left[ \frac{1 + \mu_k}{E_k} - \frac{1 - \mu_a}{E_a} \right]} \quad (26)$$

where  $\mu_k$  and  $\mu_a$  are Poisson coefficients for the crystalline and amorphous pyrographite, respectively; and  $E_k, E_a$  are Young moduli for the crystalline and amorphous pyrographite, respectively.

As there is no data on the physical properties of pyrographite in the amorphous state, eq. (26) can only be used to estimate the tendency in the change of the localized stress state to the degree of amorphization development. From this it is observed that in the initial state, when  $\alpha = 0$ , the tightening  $\Delta c = 0$ . The maximum value of  $\Delta c = \frac{c}{3} \left( \frac{\gamma_k - \gamma_a}{\gamma_a} \right)$  is reached when pyrographite is amorphized totally in the irradiated zone. At this moment the contact pressure reaches the value of

$$p_k = \frac{\frac{\gamma_k - \gamma_a}{\gamma_a}}{3 \left[ \frac{1 + \mu_k}{E_k} + \frac{1 - \mu_a}{E_a} \right]} \quad (27)$$

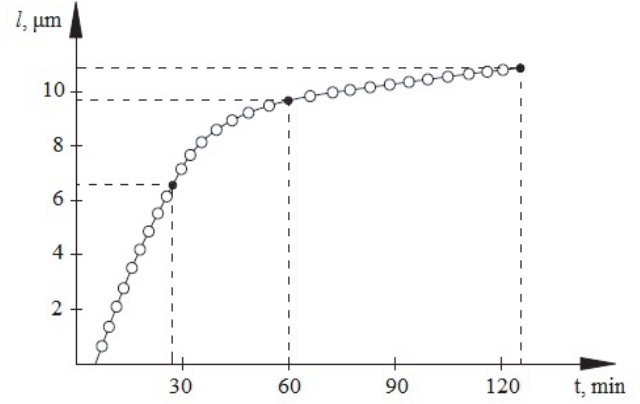


Figure 3. Kinetics of the development of a crack under irradiation conditions with high-energy electrons; experiment within the HVTEM-JEOL 1000; sample material-pyrographite  
Source: The authors.

When the character of stress distribution in the matrix zone not exposed to radiation described by formula 11 is analyzed, the reasons for the emergence of cracks at the border of the irradiated area and their propagation in the radial direction become clear.

For  $r = c$ , the stress forces in the tangential direction reach their maximum value. The resistance under these forces is much weaker in pyrographite compared with the effects of compression forces [7]. The fact that the cracks propagate along the crystallographic directions  $[010]$ ,  $[110]$ ,  $[100]$ ,  $[010]$ ,  $[110]$ ,  $[100]$  indicates that the fracture anisotropy resulting from the crystallographic structure of pyrographite expresses itself, and the characteristics of the deformation and stress state in an anisotropic approximation can be considered to be approximations. In Fig. 3, the kinetics of crack development in a matrix of pyrographite unaffected by the radiation is observed. It can be seen that the curve consists of two sections:

- I. A section in which the crack grows at a rate of  $1.10^{-5}$  cm/sec, and
- II. A section in which the crack develops at a rate of  $1.1 \times 10^{-7}$  cm/sec.

#### 4. Conclusions

1. We established that when a stream of high-energy electrons impacts pyrographite, amorphization is stimulated in it, which can be described as a transformation of the crystalline phase into the amorphous state. We studied the influence of the radiation parameters on the amorphization kinetics.

2. We proposed a phenomenological model of amorphization by radiation, which considers a possibility of a direct phase transformation of the type  $K \rightarrow A$ , as well as the inverse transformation  $A \rightarrow K$ . Based on this, we obtained a formula that describes the kinetics of amorphization by radiation in the following form:

$$\alpha(t) = \frac{1 - e^{-\frac{K_1}{2}\left(1 + \frac{K_1}{K_2}\right)t^2}}{1 + \frac{K_2}{K_1}} \quad (28)$$

3. We detected the effect of pyrographite amorphization under the influence of a high-energy electron stream inside an HVTEM. We proposed a phenomenological model that describes the kinetics of amorphization in pyrographite due to radiation using reference points put in the material surface. This was performed by means of the implantation of ionized copper atoms.

We studied the deformation and stress state of pyrographite, both in the zone irradiated by electrons as well as in the zones adjacent to the matrix unaffected by radiation.

We proposed a method to determine the energy threshold of radiation damage in pyrographite using data from the kinetics analysis of its amorphization under conditions of radiation by high-energy electrons performed inside the HVTEM column under various stress acceleration values.

### Acknowledgment

The authors express their gratitude to all the staff at the Laboratory 20, Baikov Institute of Metallurgy, Russian Academy of Sciences, for their support. We also thank the Secretary of Research of the National Polytechnic Institute of Mexico, SIP, for their support for Project 20131380, 20141031.

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