Journal of Nuclear Materials 381 (2008) 124–128

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Application of a micromechanics model to the overall properties of heterogeneous graphite

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ARTICLE INFO

PACS:
02.60.Cb
62.20.Dc
81.05.Uw

ABSTRACT

This paper deals with the overall properties of polycrystalline graphite, a material mainly composed of voids and dense inhomogeneities embedded in a less dense matrix. First, we examine the overall average elastic properties and conductivities of such a material. Second, we evaluate the void shape effects on the overall Young's modulus. Finally, we compare the results obtained from the analytical model with experimental data from radiolytic oxidation of graphite.

1. Introduction

Because of its very good thermo–mechanical properties in a large range of temperatures, graphite is used for many industrial applications and particularly in the nuclear industry. Its manufacture involves complex methods of mixing and baking at high temperatures, resulting in a heterogeneous material made of coke filler particles, a coal–tar pitch binder matrix and pores of various sizes [1,2]. In the UK, advanced gas-cooled reactors (AGRs) use a dense and near isotropic type of graphite as a moderator and as a major structural component. During service, the microstructure of graphite is subjected to neutronic irradiation and radiolytic oxidation, leading to important microstructural changes. It is now well established that these changes are related to the bulk mechanical properties of the material [2,3]. These property changes and their relationships to the microstructure have been since long the subject of many studies and considerable effort has been made on the development of porosity due to radiolytic oxidation [4,5]. The aim of this paper is to use an analytical approach to understand and to evaluate the microstructural changes due to increasing pore volume fractions and their relationships with the overall mechanical properties of heterogeneous graphite.

Eshelby's paper [6] is a basic general theory of micromechanics of an inhomogeneity problem. Several extended theories have been proposed since Eshelby [7]. Among them, a mean field method has been used widely for its simplicity. In the context of the present study, a paper by Taya and Chou [8] is most significant, since the paper examined the overall elastic constants of a composite having two types of inhomogeneities. However, as shown later, the basic equations to derive the overall elastic constants in their study lack one critical point. Thus, this paper first presents our understanding of micromechanics of a material having two types of inhomogeneities. In the main development of analysis, we will assume that the shape of an inhomogeneity is ellipsoidal. Firstly, a spherical shape is assumed for two types of the inhomogeneities in the actual calculations, since the inhomogeneities we encounter in the type of graphite considered are nearly spherical or particulate. In addition, we will adopt the method using the extra strain due to the inhomogeneities in order to calculate the overall elastic constants. This strain method is different from that originally given by Eshelby, but can be more easily accommodated into the final expressions of the overall constants. However, we will show that the two methods are equivalent. Next, the overall thermo–mechanical properties of the same material are evaluated using the same principle and the influence of the stiffness of the particles and the shape of the pores on the elastic properties is shown. Finally, the numerical calculations of the overall Young's modulus for increasing pore volume fractions are compared with radiolytic experimental data from the literature.

2. Analysis

2.1. Elastic body consisting of three isotropic phases

Eshelby demonstrated that the disturbance of the stress field, created by the presence of one inhomogeneity in an infinitely extended and elastic matrix, can be reproduced by an equivalent inclusion. The equivalent inclusion must possess the same shape as the inhomogeneity it represents, and the same properties as the surrounding matrix in which it is embedded. The local 'stress-free' strain within the inclusion is here referred to eigen-

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0022-3115/$ - see front matter © 2008 Published by Elsevier B.V.
doi:10.1016/j.jnucmat.2008.07.015

strain and denoted ε^*. Eshelby also demonstrated that the eigenstrain within an ellipsoidal inclusion is uniform. The local disturbance of the strain field due to the presence of an inhomogeneity is reproduced in Eshelby's equivalent inclusion problem. The strain inside the inclusion ε is related to the eigenstrain by

$$\varepsilon = \mathbf{S}\varepsilon^* \tag{1}$$

where $\mathbf{S}$ is the Eshelby shape tensor, depending solely on the geometry of the inclusion.

If we consider an elastic and infinite body, composed of an isotropic matrix (M), and of two different types of inhomogeneities, (Ω_1) and (Ω_2), which are perfectly embedded in it, we can express the equivalent eigenstrains of each Ω-phase by considering separately one representative inhomogeneity of each type. The volume fractions of the two domains Ω_1 and Ω_2 are respectively denoted f_1 and f_2, and f is the sum of these two quantities. The composite domain is subjected to an external force at infinity that generates a uniform stress $\boldsymbol{\sigma}^0$. The resulting external strain due to this force is denoted γ^0 when the body is elastically uniform. To consider the average stress and strain fields, we assume a large number of inhomogeneities is embedded in the matrix. These inhomogeneities do not necessarily have the same size, but they have the same shape and the same properties. The stress 'felt' by one inclusion of either one phase or another is equal to the average stress $\langle\boldsymbol{\sigma}\rangle_M$ within the matrix [9]. Each inhomogeneity is subjected to three strain contributions: the strain disturbance ε created by its own presence; to the external strain γ^0 due to the prescribed force; and to the strain disturbance $\mathbf{C}^{-1}\langle\boldsymbol{\sigma}\rangle$ due to the surrounding inhomogeneities. Considering (1), the total stress within one representative inhomogeneity and within the equivalent inclusion is related to the elastic strain through Hooke's law by

$$\boldsymbol{\sigma}^0 + \boldsymbol{\sigma} = \mathbf{C}^*(\gamma^0 + \mathbf{C}^{-1}\langle\boldsymbol{\sigma}\rangle_M + \mathbf{S}\varepsilon^*) = \mathbf{C}(\gamma^0 + \mathbf{C}^{-1}\langle\boldsymbol{\sigma}\rangle_M + \mathbf{S}\varepsilon^* - \varepsilon^*), \tag{2}$$

where $\mathbf{C}$ and $\mathbf{C}^*$ respectively represent the stiffness of the matrix and of one type of inhomogeneity. The unknown is the average stress within the matrix phase $\langle\boldsymbol{\sigma}\rangle_M$. A self-consistent scheme can be used to express the eigenstrain [7]. However, results must be considered with great care as it was shown by Budiansky [10] that this method could lead to unrealistic results in some particular cases. The residual stress $\boldsymbol{\sigma}^\infty$ within an inclusion is uniform [6] since the eigenstrain of the inclusion is also uniform and its expression is obtained from the one-inclusion problem by noting

$$\boldsymbol{\sigma}^\infty = \mathbf{C}(\mathbf{S}\varepsilon^* - \varepsilon^*). \tag{3}$$

In the case of a body composed of many inhomogeneities, the total stress within one equivalent inclusion is composed of the residual stress, which is due to its inherent eigenstrain, and the 'back-stress' generated by the presence of other inclusions [9]. Since the residual stress within an ellipsoidal inclusion is uniform, we can express the average stress within one inclusion of either one phase or another ($\Omega_1 \cap \Omega_2$) by

$$\langle\boldsymbol{\sigma}\rangle_{\Omega_1\cap\Omega_2} = \boldsymbol{\sigma}^\infty + \langle\boldsymbol{\sigma}\rangle_M. \tag{4}$$

Since the composite body is in stress equilibrium, the average stress within the matrix and within the inclusions are related by the average stress balance equation that is given by

$$f\langle\boldsymbol{\sigma}\rangle_{\Omega_1\cap\Omega_2} + (1-f)\langle\boldsymbol{\sigma}\rangle_M = 0 \tag{5}$$

The average stress balance equation and the expression of the residual stress within one inclusion (Eq. (2)) lead to the expression of the average stress in the matrix in terms of eigenstrain written as

$$\langle\boldsymbol{\sigma}\rangle_M = -f\mathbf{C}(\mathbf{S}\varepsilon^* - \varepsilon^*). \tag{6}$$

This is a fundamental equation to be used, which Taya and Chou [8] did not specifically give. Finally, the equivalency condition is given by

$$\mathbf{C}^*(\gamma^0 - f(\mathbf{S}\varepsilon^* - \varepsilon^*) + \mathbf{S}\varepsilon^*) = \mathbf{C}(\gamma^0 - f(\mathbf{S}\varepsilon^* - \varepsilon^*) + \mathbf{S}\varepsilon^* - \varepsilon^*). \tag{7}$$

This method can be extended to the case where the matrix phase contains two different types of inhomogeneity, each type having different shapes and different properties. In the case of graphite, these can be considered the porosity and the filler particles. The 'back-stress' felt by one inclusion in either one phase or the other is expressed by

$$\langle\boldsymbol{\sigma}\rangle_M = -f_1\mathbf{C}(\mathbf{S}_1 - I)\varepsilon_1^* - f_2C(\mathbf{S}_2 - I)\varepsilon_2^*. \tag{8}$$

For one representative inhomogeneity of the phase Ω_1, having stiffness $\mathbf{C}_1^*$, the equivalency condition is written as

$$\begin{aligned}&\mathbf{C}_1^*(\gamma^0 - f_1(\mathbf{S}_1 - \mathbf{I})\varepsilon_1^* - f_2(\mathbf{S}_2 - \mathbf{I})\varepsilon_2^* + \mathbf{S}_1\varepsilon_1^*)\\ &\quad = \mathbf{C}(\gamma^0 - f_1(\mathbf{S}_1 - \mathbf{I})\varepsilon_1^* - f_2(\mathbf{S}_2 - \mathbf{I})\varepsilon_2^* + (\mathbf{S}_1 - \mathbf{I})\varepsilon_1^*)\end{aligned} \tag{9}$$

Similarly, for one representative inhomogeneity of the phase Ω_2, having stiffness $\mathbf{C}_2^*$, the equivalency condition is written as

$$\begin{aligned}&\mathbf{C}_2^*(\gamma^0 - f_1(\mathbf{S}_1 - \mathbf{I})\varepsilon_1^* - f_2(\mathbf{S}_2 - \mathbf{I})\varepsilon_2^* + \mathbf{S}_2\varepsilon_2^*)\\ &\quad = \mathbf{C}(\gamma^0 - f_1(\mathbf{S}_1 - \mathbf{I})\varepsilon_1^* - f_2(\mathbf{S}_2 - \mathbf{I})\varepsilon_2^* + (\mathbf{S}_2 - \mathbf{I})\varepsilon_2^*).\end{aligned} \tag{10}$$

Expressions for the eigenstrain in the equivalent inclusion of each phase are obtained by solving Eqs. (9) and (10), giving

$$\begin{cases}\varepsilon_1^* = (\mathbf{A}_1\mathbf{A}_4 - \mathbf{A}_2\mathbf{A}_3)^{-1}[\mathbf{A}_4(\mathbf{C} - \mathbf{C}_1^*) - \mathbf{A}_2(\mathbf{C} - \mathbf{C}_2^*)]\gamma^0\\ \varepsilon_2^* = (\mathbf{A}_1\mathbf{A}_4 - \mathbf{A}_2\mathbf{A}_3)^{-1}[\mathbf{A}_1(\mathbf{C} - \mathbf{C}_2^*) - \mathbf{A}_3(\mathbf{C} - \mathbf{C}_1^*)]\gamma^0\end{cases}, \tag{11}$$

where the matrix coefficients $\mathbf{A}_1, \mathbf{A}_2, \mathbf{A}_3$ and $\mathbf{A}_4$ are given by

$$\begin{cases}\mathbf{A}_1 = (1-f_1)(\mathbf{C}_1^* - \mathbf{C})(\mathbf{S}_1 - \mathbf{I}) + \mathbf{C}_1^*\\ \mathbf{A}_2 = f_2(\mathbf{C} - \mathbf{C}_1^*)(\mathbf{S}_2 - \mathbf{I})\\ \mathbf{A}_3 = f_1(\mathbf{C} - \mathbf{C}_2^*)(\mathbf{S}_1 - \mathbf{I})\\ \mathbf{A}_4 = (1-f_2)(\mathbf{C}_2^* - \mathbf{C})(\mathbf{S}_2 - \mathbf{I}) + \mathbf{C}_2^*\end{cases} \tag{12}$$

In the case in which the volume fraction of either one phase or the other is zero, the result is identical to the one-type of inhomogeneity problem.

2.2. Equivalency between the energy and the direct strain balance approach

The stress and strain disturbances by an inhomogeneity accompany changes in elastic energy and potential energy of the system on application of a prescribed loading. Originally, Eshelby developed this method to calculate these energy changes. When a disturbance is reproduced by an equivalent inclusion with equivalent eigenstrain ε^*, the increase in elastic energy is given as

$$\Delta E = \frac{1}{2}\int_V \sigma_{ij}^0\varepsilon_{ij}^* \mathrm{d}V, \tag{13}$$

where V includes all the domains of inhomogeneities. This gives the increase in elastic energy of

$$\Delta E = \frac{f}{2}\sigma_{ij}^0\varepsilon_{ij}^*, \tag{14}$$

per unit volume of the body. Thus, the total elastic energy per unit volume is

$$\bar{E} = E^0 + \Delta E = \frac{1}{2}\sigma_{ij}^0\gamma_{ij}^0 + \frac{f}{2}\sigma_{ij}^0\varepsilon_{ij}^*, \tag{15}$$

where

$$E^0 = \frac{1}{2}\sigma_{ij}^0\gamma_{ij}^0 \tag{16}$$

is the elastic energy per unit volume before the disturbance. $\overline{E}$ is defined as

$$\overline{E} = \frac{1}{2}\sigma^0_{ij}\overline{\gamma}_{ij}. \tag{17}$$

Summarizing, we have

$$\frac{1}{2}\sigma^0_{ij}\overline{\gamma}_{ij} = \frac{1}{2}\sigma^0_{ij}\gamma^0_{ij} + \frac{f}{2}\sigma^0_{ij}\varepsilon^*_{ij}. \tag{18}$$

Next, using $\overline{\gamma}_{ij} = \overline{C}^{-1}_{ijkl}\sigma^0_{kl}$, $\gamma^0_{ij} = C^{-1}_{ijkl}\sigma^0_{kl}$ and rewriting ε^*_{ij} in terms of σ^0_{kl} by using the equivalency condition, we can rewrite this equation as a quadratic form of σ^0_{ij}. The proportional factor of each term contains $\overline{\mathbf{C}}$, $\mathbf{C}$, $\mathbf{C}^*$ and $\mathbf{S}$. Comparing the factor of each term, we can obtain the components of $\overline{\mathbf{C}}$, which is the stiffness of the material containing the two populations of inhomogeneities.

2.3. Application to the overall thermo–mechanical properties

2.3.1. Overall stiffness

The overall stiffness $\overline{\mathbf{C}}$ of a composite material which is constituted of three different elastic phases can be obtained using either an energy approach or a direct strain approach. These two approaches are identical as one is derived from the other and results are thus equivalent, as shown later. In this work, the expression of the overall stiffness was obtained from a direct strain approach. Since each phase is assumed elastic, Hooke's law can be applied to the overall composite material. The strain balance equation is thus written as

$$\overline{\mathbf{C}}^{-1}\boldsymbol{\sigma}_0 = \mathbf{C}^{-1}\boldsymbol{\sigma}_0 + \mathbf{f_1}\varepsilon^*_1 + \mathbf{f_2}\varepsilon^*_2 \tag{19}$$

Replacing the eigenstrains ε^*_1 and ε^*_2 by their expressions given by (11), the overall stiffness $\overline{\mathbf{C}}$ is obtained with

$$\begin{aligned}\overline{\mathbf{C}}^{-1} = \mathbf{C}^{-1} &+ \mathbf{f_1}(\mathbf{A_1A_4} - \mathbf{A_2A_3})^{-1}[\mathbf{A_4}(\mathbf{C} - \mathbf{C}^*_1) - \mathbf{A_2}(\mathbf{C} - \mathbf{C}^*_2)]\mathbf{C}^{-1} \\ &+ \mathbf{f_2}(\mathbf{A_1A_4} - \mathbf{A_2A_3})^{-1}[\mathbf{A_1}(\mathbf{C} - \mathbf{C}^*_2) - \mathbf{A_3}(\mathbf{C} - \mathbf{C}^*_1)]\mathbf{C}^{-1}\end{aligned} \tag{20}$$

2.3.2. Overall thermal conductivity

The local heat flux of a conductive material at any point is obtained by Fourier's equation written as

$$\mathbf{q} = -\mathbf{K}_i\nabla T, \tag{21}$$

where $\mathbf{K}_i$ is the thermal conductivity of the ith constitutive phase, and ∇T is the gradient of temperature within the region considered.

By analogy with the elasticity problem, the overall thermal conductivity $\overline{\mathbf{K}}$ is determined by

$$\begin{aligned}\overline{\mathbf{K}}^{-1} = \mathbf{K}^{-1} &+ \mathbf{f_1}(\mathbf{A}'_1\mathbf{A}'_4 - \mathbf{A}'_2\mathbf{A}'_3)^{-1}[\mathbf{A}'_4(\mathbf{K} - \mathbf{K}^*_1) - \mathbf{A}'_2(\mathbf{K} - \mathbf{K}^*_2)]\mathbf{K}^{-1} \\ &+ \mathbf{f_2}(\mathbf{A}'_1\mathbf{A}'_4 - \mathbf{A}'_2\mathbf{A}'_3)^{-1}[\mathbf{A}'_1(\mathbf{K} - \mathbf{K}^*_2) - \mathbf{A}'_3(\mathbf{K} - \mathbf{K}^*_1)]\mathbf{K}^{-1}\end{aligned} \tag{22}$$

where $\mathbf{K}$ is the thermal conductivity of the matrix and $\mathbf{A}'_1$, $\mathbf{A}'_2\mathbf{A}'_3$ and $\mathbf{A}'_4$ are given by

$$\begin{cases}\mathbf{A}'_1 = (1-f_1)(\mathbf{K}^*_1 - \mathbf{K})(\mathbf{S}_1 - \mathbf{I}) + \mathbf{K}^*_1 \\ \mathbf{A}'_2 = f_2(\mathbf{K} - \mathbf{K}^*_1)(\mathbf{S}_2 - \mathbf{I}) \\ \mathbf{A}'_3 = f_1(\mathbf{K} - \mathbf{K}^*_2)(\mathbf{S}_1 - \mathbf{I}) \\ \mathbf{A}'_4 = (1-f_2)\,(\mathbf{K}^*_2 - \mathbf{K})(\mathbf{S}_2 - \mathbf{I}) + \mathbf{K}^*_2\end{cases} \tag{23}$$

The overall thermal conductivity $\overline{\mathbf{K}}$ is given by Eq. (22), which has the same form as Eq. (20) that gives the expression of the overall elastic matrix $\overline{\mathbf{C}}$. Using the elasticity-heat transfer analogy, the study of the Young's modulus can then be similarly repeated for the study of the thermal conductivity.

3. Results and discussion

3.1. Influence of voids and filler particles

Considering a three-phase composite material which is composed of an elastic matrix, voids and elastic filler particles, the overall Young's modulus of this material can be calculated for different void volume fractions and for filler particles having different elastic properties by using the analytical approach developed above. The Young's modulus of the matrix phase (cf. filler particles) is denoted E (cf. E_1). Filler particles having the following elastic properties were considered: $E_1/E = 10$, $E_1/E = 5$, $E_1/E = 1$, and $E_1/E = 1/10$. Fig. 1 shows the overall Young's modulus normalised against by the one of the matrix for void volume fractions between $0\ \mathrm{cm^3/cm^3}$ and $0.4\ \mathrm{cm^3/cm^3}$.

In Fig. 1, it can be seen that the overall Young's modulus decreases faster with increasing void volume fractions with stiff filler particles. This approach gives similar results as the one proposed by Taya and Chou [8]. However, our approach is more accurately developed since the average stress in the matrix phase is expressed in terms of residual stresses within one inclusion, and the expression of the overall elastic stiffness is given with a clearer and easily computable form.

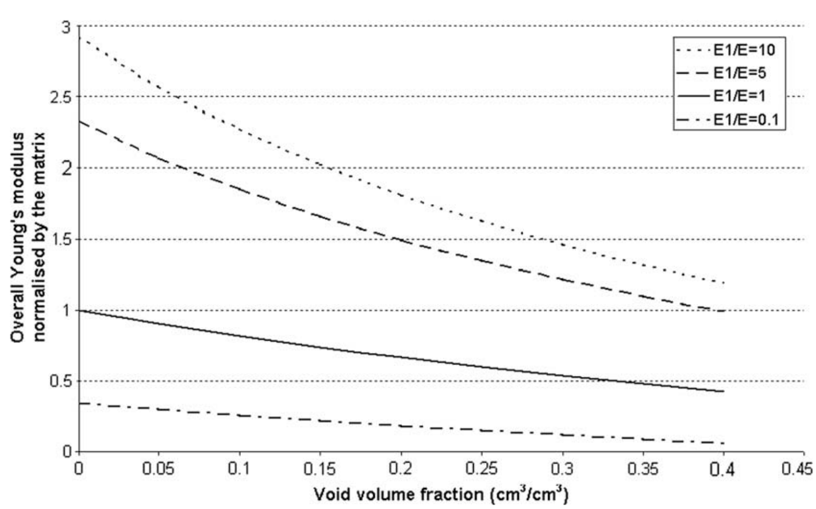

Fig. 1. Evolution of the overall Young's modulus against the void volume fraction for different particle stiffnesses.

3.2. Influence of the mean void shape

In this study, the influence of the shape of the voids is analysed. Filler particles are assumed spherical, and have a Young's modulus $E_1 = 2\,E$. The volume fraction of filler particles in polycrystalline graphite was estimated to $f_1 = 0.6\ \mathrm{cm}^3/\mathrm{cm}^3$ using X-ray tomography images. Three different kinds of void aspect ratios can be considered:

- Longitudinal voids, elongated in the direction of the prescribed stress ($a/c < 1$).
- Spheres ($a/c = 1$).
- Transverse voids, elongated in the transverse direction to the prescribed stress ($a/c > 1$).

For increasing void volume fractions, the effect of different void shape on the overall Young's modulus normalised against the overall Young's modulus at zero void volume fraction is shown in Fig. 2.

In Fig. 2, the void aspect ratio is defined by the ratio between the transverse radius a and the longitudinal radius c. The overall Young's modulus of a material containing longitudinal voids decreases linearly with increasing void volume fractions, with such voids having little effect on the average stress within the matrix. Spheroidal voids have a larger effect on the overall Young's modulus, but the largest effect is due to those elongated in the transverse direction to the applied stress. In this case, increasing void volume fractions reduce the overall Young's modulus rapidly at low volume fractions and slower at high void volume fractions.

3.3. Comparison with graphite behaviour

Since polycrystalline graphite is mainly composed of ~20% of voids and coke filler particles which are embedded in a binder matrix [2], the overall Young's modulus of graphite containing increasing void volume fractions f_2 can be calculated using the approach developed above. The input parameters to define in the analytical model are:

- The volume fractions and aspect ratio of the voids and of the filler particles.
- The elastic properties of the filler particles and the matrix.

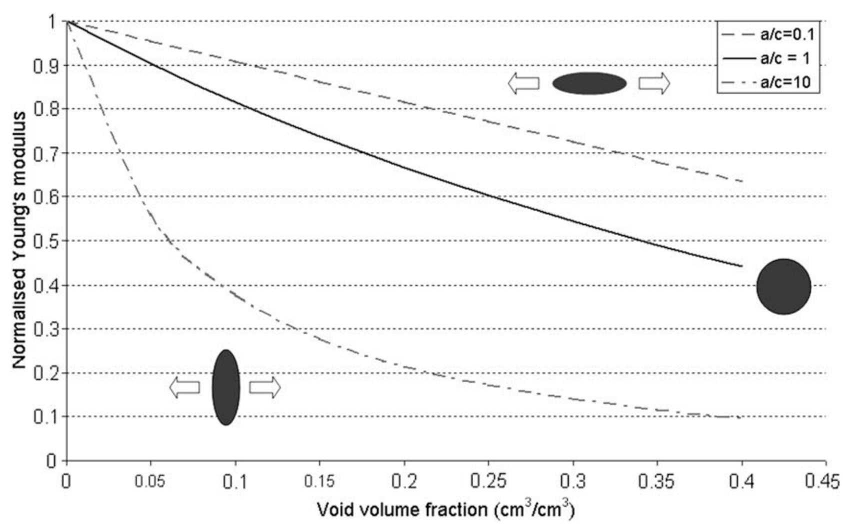

Fig. 2. Effects of the shape of voids on the overall Young's modulus normalised by the overall Young's modulus at zero void volume fraction.

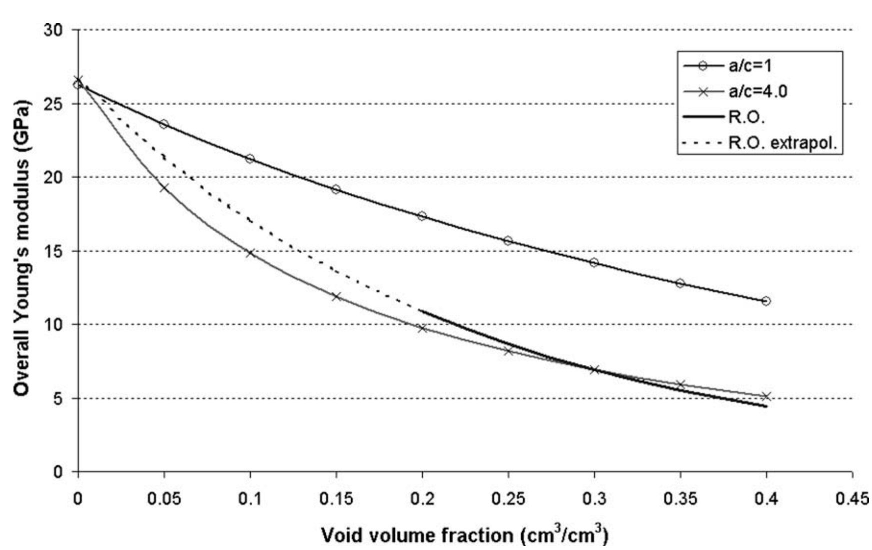

Fig. 3. Analytical results compared with radiolytic oxidation experiments [12].

In quasi-isotropic graphite, coke filler particles are nearly spherical, their volume fraction was estimated to $f_1 = 0.6\ \text{cm}^3/\text{cm}^3$ using X-ray tomography images, and assumed constant. The elastic properties of the matrix were previously estimated from nano-indentation [11], i.e. $E = 15$ GPa. The Young's modulus of the coke filler particles was calculated so that the initial point at zero-void volume fraction coincides with the extrapolating curve plotted from radiolytic oxidation data from the literature [12]. The Young's modulus of the coke filler particles was estimated to $E_1 = 41$ GPa. Fig. 3 compares the results obtained using the three-phase analytical model considering spherical and transverse voids, and those obtained from radiolytic experiments [12].

In Fig. 3, the dashed line represents the experimental data extrapolated to zero-void volume fraction. As seen in Section 3.2., the decrease of the overall Young's modulus is more rapid at low void volume fractions when the mean void aspect ratio $a/c > 1$. In this case, a good agreement between the numerical results and the experimental curve was obtained for $a/c = 4.0$.

4. Conclusion

The present paper showed the application of Eshelby's theory and the mean-field method to the prediction of the overall thermo–mechanical properties of a three-phase composite material. It was also demonstrated that the energy approach gives equivalent results to a direct strain approach. The effect of the elastic properties of the filler particles and increasing void volume fraction on the overall Young's modulus were analysed using this approach. Finally, the analytical calculations of the overall Young's modulus with increasing void volume fractions of isotropic polycrystalline graphite were compared to experimental data from radiolytic oxidation in the literature. A good agreement was found for a mean void aspect ratio $a/c = 4.0$. In a future paper, the present authors will also extend this theory to the prediction of the overall tensile strength of a composite material, the presence of n families of inhomogeneities, and to the case of a material containing non-ellipsoidal inhomogeneities.

Acknowledgements

The financial support of British Energy Generation Ltd. is gratefully acknowledged. The views expressed in this publication are those of the authors and do not necessarily represent the views of the sponsor.

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