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DETERMINATION OF DYNAMIC PROPERTIES OF SINTERED COPPER POWDER FROM ULTRASONIC MEASUREMENTS

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The overall (effective) dynamic properties of sintered copper powder with air-saturated pores at moderate porosities are deduced from suitable ultrasonic measurements carried out at low porosities in the long-wavelength approximation. The effective dynamic properties of the two-phase composite at low and moderate porosities are analysed computationally, the properties of the pure matrix material being in the first step determined by extrapolation from suitable ultrasonic measurements carried out at low porosities, and by employing the Berryman's self-consistent single scattering theory. The presented results confirm the ultrasonic measurements to be extremely useful in estimating the influence of the volume concentration and shape of the inclusions on the overall dynamic properties of porous two-phase composites at porosities from a wide range of porosity.

KEYWORDS: ultrasonics, porous composite, inclusion shape

1. Introduction

One of the main objectives of material science of random composities is the formulation of stress-deformation relations that govern the mechanical response of a material under specific environmental conditions (loading). In the approximation of the linear elasticity theory, these relations can be written when the effective Lamé constant are known. Therefore, predicting the effective Lamé constants of macroscopically isotropic composites is of great engineering importance. The subsequent considerations are confined to this case and concerned with bulk samples of an isotropic two-phase solid. The inclusions are of the form of ellipsoidal pores with the same shape, and random size and orientation, the pores being air saturated or evacuated.

This paper grew out of the analysis, which the author performed to prepare the paper [1] for publication. In Ref. [1], for the sake of brevity the emphasis in the presentation of the results of analysis is laid on the propagation properties of sintered copper powder rather than on the overall dynamic properties. Due to the limitation in the desired size of this volume, it was decided that the concluding sections of the text concerned with the investigations of the dynamic properties should be presented as a supplementary article.

Because of the great engineering importance of the problem, there are numerous works devoted to the ultrasonic technique as a tool for the determination of the effective dynamic moduli of sintered metal powders. Therefore, it seems to be reasonable to point out the main reason which, in our opinion, justifies presenting the subsequent study as another paper concerned with the problem mentioned above.

According to the simple two-phase model, which is commonly used for the prediction of the elastic behaviour of a sintered metal powder, such a material is regarded as consisting of a solid matrix with the properties of a pure metal, in which inclusions are dispersed in the form of voids. Therefore, in such approach the matrix phase is treated in the linear acustic approximation as a perfectly elastic material with the real elastic moduli of the pure lossless metal. In this model, the matrix subdomains are regarded to be filled by a material which is free from both inelasticity (dissipation properties) and scattering centres. The apparent sensitivity of the velocity and attenuation of ultrasonic waves propagating through such a medium, to the changes in the inclusion shape is discussed an analysed for non-spherical inclusions, to some more or less limited extent, in such papers as [1–6].

The assumptions of the simple two-phase model are in contradiction to the really existing inelasticity in the matrix phases of the sintered metal powders, the inelasticity being mainly due to the lattice defects, impurities and some distributions of residual local stresses and imperfect adhesion between adjacent metal grains. These randomly occuring flaws contribute to highly variable scattering and dissipation properties of the matrix material and causes that the observed components of the Hooke's tensor are complex.

This paper presents an attempt of applying a more complicated model of two-phase media proposed by the author in the paper [1] to the determination of the effective dynamic moduli of sintered metal powders. In this model, the complex elastic moduli of the matrix phase of the composite under study are to be determined from ultrasonic measurement of the propagation velocity and attenuation of ultrasonic waves in two samples of the composite, the samples being characterized by different and small volume concentrations of the inclusions. In the present work, the influence of the pore shape on the effective dynamic moduli of the composite is examined computationally with the aid of an algorithm supplied by the self-consistent scattering approach of Berryman [4].

2. Formulation of the problem

As it was mentioned, the subsequent considerations are concerned with bulk samples of an isotropic two-phase (porous) solid of the form of sintered metal powder. The matrix material is composed of a large number of metal (copper) grains of random size, shape and orientation, the adjacent grains being joined toghether more or less closely (perfectly) by adhesion due to the sintering process under high hydrostatic press. The imperfection of the adhesion between adjacent grains and the other matrix material flaws as well as the size and orientation of the pores with the same ellipsoidal shape are also assumed to be random, the pores being air-saturated or evacuated. Under these assumptions, the matrix material may also be regarded as an isotropic solid material, although its elastic moduli differ from those of the polycrystalline metal, and are to be determined experimentally from ultrasonic measurets in a manner mentioned above.

In the remainder of this paper, the standard abbreviations are used for the quantities involved in the description of the propagation of ultrasonic waves and the material parameters of the constituents of the heterogeneous material under study. Thus ω and t denote the angular frequency and time, respectively, ρ stands for the density, C_{ijkl} $i, j, k, l=1, 2, 3$ denote the components of a Hooke's tensor K and μ are the bulk and shear modulus, respectively, of an elastic isotropic solid. λ denotes the Lamé constant which is related to the moduli K and μ by the following formula:

$$\lambda = K - \frac{2}{3}\mu. \tag{2.1}$$

The effective material parameters of the isotropic two-phase solid as a whole and all the other quantities referred to this material are labelled by asterisks, i.e. ρ^* denotes the effective density, K^*, μ^* and κ^* denote the effective elastic moduli of the heterogeneous solid. Similarly, throughout the paper all the abbreviations with the sub- or superscripts m and i denote quantities referred to the isotropic material of the matrix and inclusion, respectively. Similarly as in [1], the ultrasonic waves propagating through the two-phase medium are assumed to be plane linearly polarized waves, which can be described by the following formulae:

$$u(r, t)^* = \mathbf{e}_q B^*_{pq} \exp(-i K^*_{pq} \mathbf{e}_p \mathbf{r}) \exp(i\omega t), \tag{2.2}$$

where

$$K^*_{pq} = (\omega / v^*_{pq}) - i\alpha^*_{pq}, \qquad p, q = 1, 2, 3, \tag{2.3}$$

B^*_{pq} stands for the amplitude of the wave, $\mathbf{e}_p$ and $\mathbf{e}_q$ are the unit vectors in the directions of the Ox_p and Ox_q reference axes of a Cartesian coordinate system fixed in the sample, respectively. Formulae (2.2), (2.3) describe an attenuated plane wave being polarized in the direction $\mathbf{e}_q$ and propagating in the direction $\mathbf{e}_p$ with the velocity v^*_{pq} and amplitude atenuation coefficient α^*_{pq}, henceforth referred to as attenuation coefficient.

Let us suppose that the velocity and attenuation coefficient are measured on ultrasonic pulses propagating through a single bulk sample of the two-phase composite under study, the pulses being generated by a transducer oscillating with the frequency ω normally or transversly to the coupling surface. On the strength of the definition of the bulk volume, the vlume of the sample is large enough to include a large number of inhomogeneities of each type occuring in the heterogeneous solid. Then it seems to be reasonable to suppose that the measured velocity and attenuation coefficient of the ultrasonic pulses are equal to the propagation velocity v^*_{pq} and attenuation coefficient α^*_{pq} of the respective ultrasonic wave appearing in formulae (2.3)

If

$$v^*_{pq} = 1/Z^{*(a)}, \quad \alpha^*_{pq} = -\omega Z^{*(b)}, \quad Z^* = [\rho^*/C(\omega)^*_{pqpq}]^{1/2} \tag{2.4}$$

then the expression given by formulae (2.2), (2.3) is a solution to the following equation of motion for the effective homogeneous (equivalent) medium:

$$C(\omega)^*_{ijkl}\, u^*(r, t)_{kjl} + \omega^2 \rho^* u^*(r, t)_i = 0. \tag{2.5}$$

Throughout the paper, the real and imaginary parts of complex quantities are denoted by the superscripts (a) and (b), respectively. If the effective response of the bulk sample of the composite is a plane attenuated wave given by formulae (2.2)–(2.4), then the equivalent homogeneous solid is characterized by the density ρ^* and components of the complex Hooke's tensor,

$$C(\omega)^*_{jklm} = C(\omega)^{*\,(a)}_{jklm} + iC(\omega)^{*\,(b)}_{jklm}, \tag{2.6}$$

which can be calculated from the macroscopic propagation parameters v^*_{pq} and α^*_{pq} of the wave, by making use of the following formulae:

$$C(\omega)^{*\,(a)}_{pqpq} = B(1-z^2), \quad C(\omega)^{*\,(b)}_{pqpq} = 2Bz,$$

where

$$B = (v^*_{pq})^2 \rho^* (1+z^2)^{-2}, \quad z = (\alpha^*_{pq}/\omega)\, v^*_{pq}. \tag{2.7}$$

Formulae (2.7) are obtained by solving Eqs. (2.4) with respect to $C(\omega)^{*\,(a)}_{pqpq}$ and $C(\omega)^{*\,(b)}_{pqpq}$, $p, q=1, 2, 3$. Formulae (2.7) enable the effective complex moduli $C(\omega)^{*\,(a)}_{pqpq}$ and $C(\omega)^{*\,(b)}_{pqpq}$ to be determined from the measurements of the macroscopic parameters of the ultrasonic wave propagation, v^*_{pq} and α^*_{pq}, in the composite bulk sample under examination. Thus Eqs. (2.2)–(2.7) suggest an experimental method of performing the task of establishing the structure and frequency dependences of the propagation and effective material parameters of two-phase media with non-spherical inclusions. More straictly speaking, in accordance with (2.4), the dependence of the effective material parameters on the volume concentrations of the matrix and inclusion phases c_m and c_i, respectively, and on size, shape and orientations of the inclusions as well as on the frequency can be determined empirically from ultrasonic measurements, after preparing the respective bulk samples. This task is regarded as the main problem of this paper.

Equations (2.5) together with formulae (2.4) show how the overall macroscopic response of the composite material to dynamic loading of a transducer oscillating with the frequency ω, normally or transversely to the coupling surface, is determined by the effective material parameters. In view of that the dynamic effective material parameters are the essential parameters determining the utility of heterogeneous materials in engineering applications. For this reason, every method which enables us to establish the structure and frequency dependence of these parameters is of great importance.

In contrast to the simplicity of the above macroscopic relationships, which suggest the experimental assessment of the structure and frequency dependences of the propagation and material parameters of two-phase media, in theoretical attempts of finding these dependences, problems of great complexity are always involved. The dynamics of the multi-phase media with non-spherical inclusions is so complicated that, for a wide range of the volume concentrations c_i of the inclusions, we would be content with performing a computational analysis of the problem of the propagation of ultrasonic waves in such media. The computational investigations, some results of which are presented in Ref. [1] and in the next section of this paper, enable us to establish the desired dependences. Similarly as in paper [1], in performing such numerical analysis we make use of the self-consistent approach proposed by BERRYMAN [4] for analysing N-phase media, N being a natural number. According to the Berryman's approach, the self-consistent effective (equivalent) medium is determined by requiring the net scattered, long-wavelength displacement field to vanish on the average.

On employing the results of MAL and KNOPOFF [8] as well as of WU [9] in the way that was presented in Ref. [1], BERRYMAN [4] arrived at an algorithm for computational investigation of mechanical properties of N-phase media with ellipsoidal inclusions. Considering the two-phase media with ellipsoidal inclusions, the Berryman's concept yields the algorithm (2.8) given below which is employed in our computational analysis

$$K^* = (c_i K_i P^{*m}) / (c_i P^{*i} + c_m P^{*m}),$$

$$\mu^* = (c_i \mu_i Q^{*i} + c_i \mu_i Q^{*i}) / (c_i Q^{*i} + c_m Q^{*m}). \tag{2.8}$$

The quantities P^* and Q^* can be expressed in terms of WU's [9] tensor T in the following general form [4]:

$$P^* = \frac{1}{3} T^*_{ppqq}, \quad Q^* = \frac{1}{5}(T^*_{pqpq} - \frac{1}{3} T^*_{ppqq}), \tag{2.9}$$

where the formulae P, Q and T derived by WU [9] are also listed in the Appendix of [4]. These formulae will not be rewritten here. P^*, Q^* and T^* denote expressions obtained from the formulae for P, Q and T, after replacing the matrix material parameters by the respective parameters of the effective equivalent homogenous medium (material of type —*)

Among those data which are required by computational methods are the material parameters of the matrix phase. The material parameters of the matrix phase (sintered metal grains) are to be determined from ultrasonic measurements. To point out this concept let us notice that equation (2.5), together with formulae (2.2) – (2.4), define the overall effective response of a bulk sample made of the matrix material to the dynamic loading of the transducer, after replacing each of the asterisk by superscripts m. If the hypothesis of the possibility of finding the homogeneous equivalent medium is reasonable and the effective response of the bulk sample of the matrix material is a plane attenuated wave given by formulae (2.2)–(2.4), after replacing the superscripts * by m, the equivalent matrix material, henceforth called shortly matrix phase, is characterized by the density ρ^m and the components of the complex Hooke's tensor.

To determine the matrix phase elastic moduli K^m and μ^m, we employ a sequence of values of K^* and μ^* (deduced from measurement of the values of v^*_{pq} and α^*_{pq}) for two distinct porosities, say c_{i1} and c_{i2}, both the porosities belonging to the range of low porosity. In this porosity range, it is to be expected that the effective elastic moduli of the sintered metal powder become linear functions of porosity as the latter approaches sufficiently small values c_{i1} and c_{i2}, and, consequently a linear extrapolation of these quantities beyond the limits $c_{i1} \leqslant c_2 \leqslant c_{i2}$ is possible. Carrying out such an extrapolation for the limiting case $c_i = 0$, we obtain an estimation of the values of the matrix elastic moduli, K^m and μ^m. It can be done by using the following formulae given by author in Ref. [1]:

$$K^m = K^*_1 - c_{i1}(K^*_1 - K^*_2)/(c_{i1} - c_{i2})$$

$$\mu^m = \mu^*_1 - c_{i1}(\mu^*_1 - \mu^*_2)/(c_{i1} - c_{i2}), \tag{2.10}$$

where the symbol F^*_ε, $\varepsilon = 1, 2$, denotes the value of the effective quantity F^* (F^* stands for K^* and μ^*) at the porosity $c_{i\varepsilon}$

3. Numerical results

Numerical calculations were performed for frequency $f = \omega/(2\pi) = 4Mc/\text{sec}$. The following values were taken as the material parameters of the composite (sintered copper powder) under analysis [1]: $\rho^m = 8.92$ g/cm³, $\rho^i = 0.001347$ g/cm³, $K^{(a)}_m = 1.41268*10^{11}$ Pa, $K^{(b)}_m = 1.2159*10^9$ Pa, $\mu^{(a)}_m = 4.878*10^{10}$ Pa, $\mu^{(b)}_m = 4.78857*10^7$ Pa, $K^{(a)}_i = 1.595*10^5$ Pa, $K^{(b)}_i = 0$, $\mu^{(a)}_i = 0$, $\mu^{(b)}_i = \omega\eta/\rho^i$, where η denotes the dynamic viscosity of air ($\eta_i = 1.8*10^{-4}$ Poise). The values of $K^{(a)}_m$, $K^{(b)}_m$, $\mu^{(a)}_m$ and $\mu^{(b)}_m$ were calculated from the linear extrapolation of formulae (2.10) and (2.7), by using the following results of our own experiments: $c_{i1} = 0.0068$, $c_{i2} = 0.0176$, $c_{L1} = 4758$ m/sec, $c_{T1} = 2325$ m/sec, $\alpha_{L1} = 19.52$ m^{-1}, $\alpha_{T1} = 7.97$ m^{-1}, $c_{L2} = 4677$ m/sec, $c_{T2} = 2304$ m/sec, $\alpha_{L2} = 24.78$ m^{-1}, $\alpha_{T2} = 12.25$ m^{-1}. Some results of the numerical calculations are presented in Figs. 1–8. These results visualize how, at the loading frequency $f = 4$ Mc/sec, the dynamic elastic moduli (Lamé constants) K^* and μ^* of the sintered copper powder depend on the volume concentration of the ellipsoidal inclusions, their shape and size. The calculations were carried out for both prolate ($a > b = c$) and oblate ($a = b > c$), air-saturated spheroids under the assumption that the shape of each pore in the bulk sample under examination is to be characterized by the same value of the shape

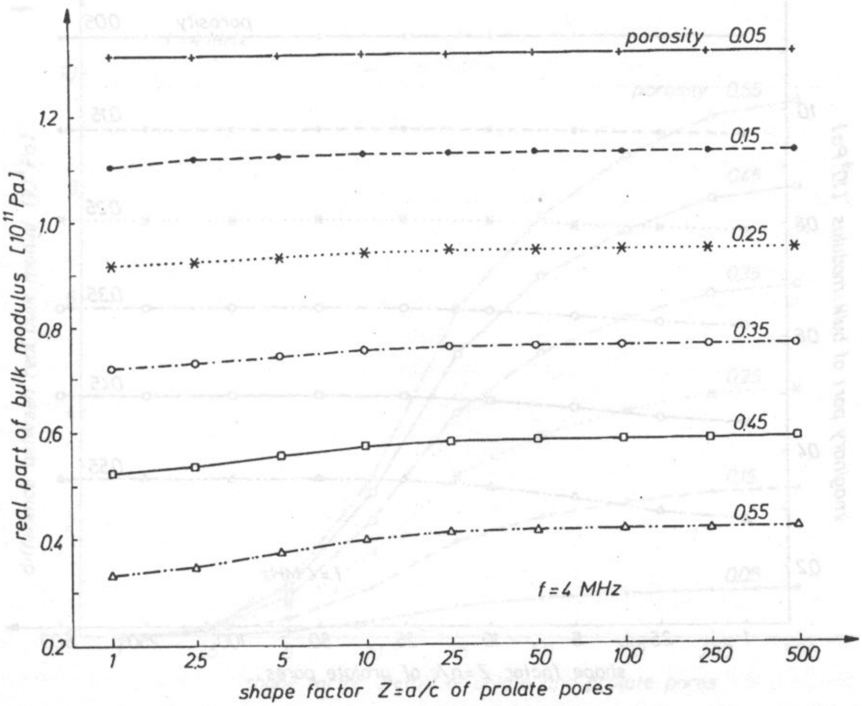

Fig. 1. Real part of bulk modulus of sintered copper powder as a function of the porosity and shape factor $Z=a/c$ of the air-saturated prolate pores.

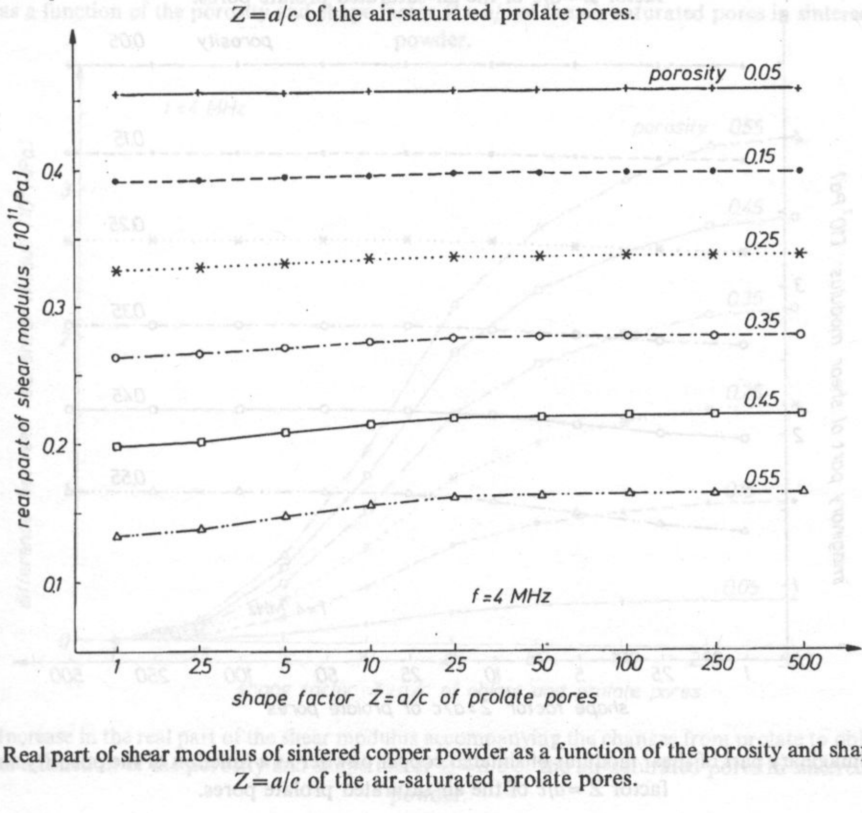

Fig. 2. Real part of shear modulus of sintered copper powder as a function of the porosity and shape factor $Z=a/c$ of the air-saturated prolate pores.

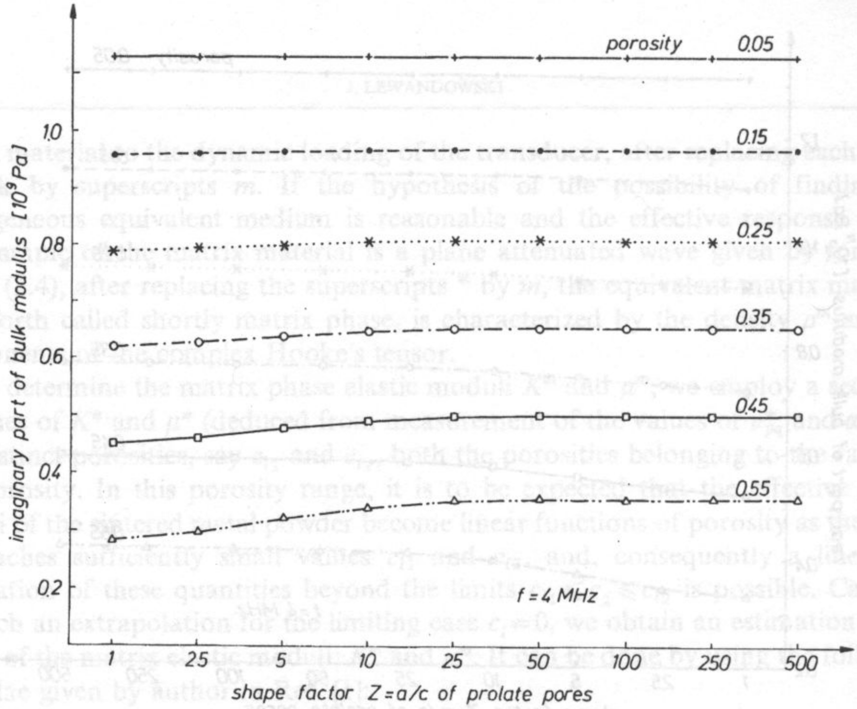

Fig. 3. Imaginary part of bulk modulus of sintered copper powder as a function of the porosity and shape factor $Z=a/c$ of the air-saturated prolate pores.

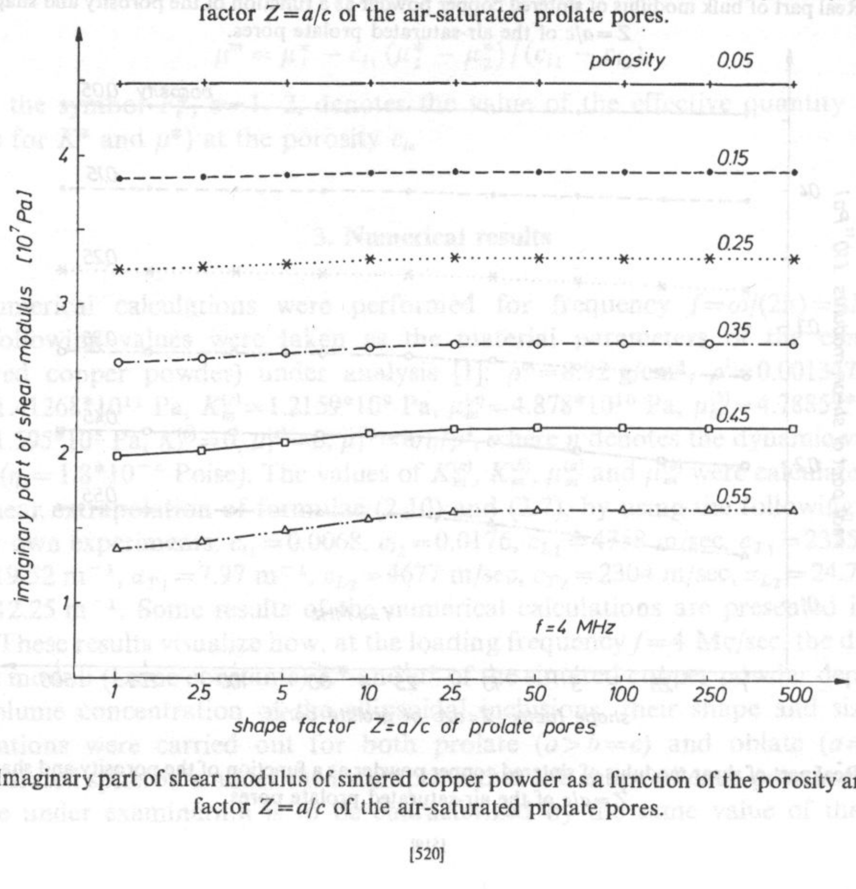

Fig. 4. Imaginary part of shear modulus of sintered copper powder as a function of the porosity and shape factor $Z=a/c$ of the air-saturated prolate pores.

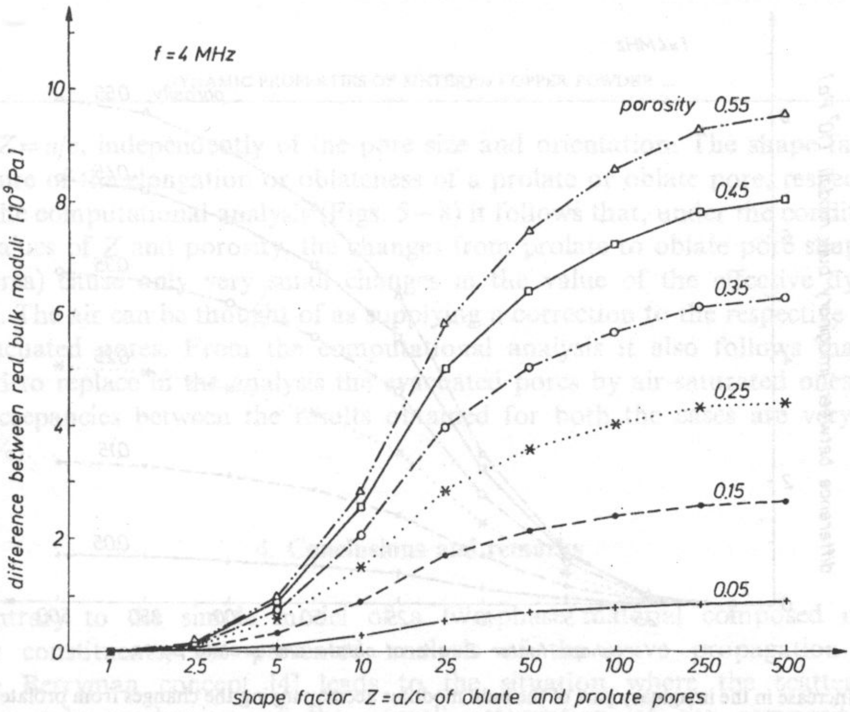

Fig. 5. Increase in the real part of the bulk modulus accompanying the changes from prolate to oblate pore shapes as a function of the porosity and shape factor $Z = a/c$ of the air-saturated pores in sintered copper powder.

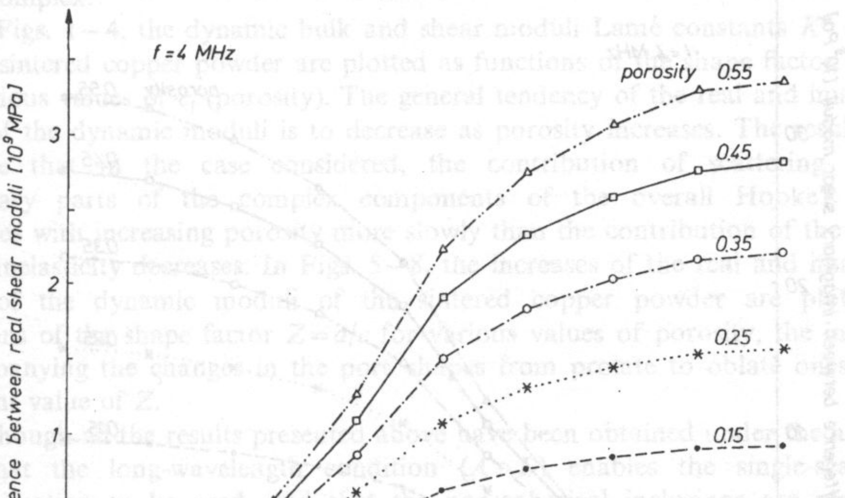

Fig. 6. Increase in the real part of the shear modulus accompanying the changes from prolate to oblate pore shapes as a function of the porosity and shape factor $Z = a/c$ of the air-saturated pores in sintered copper powder.

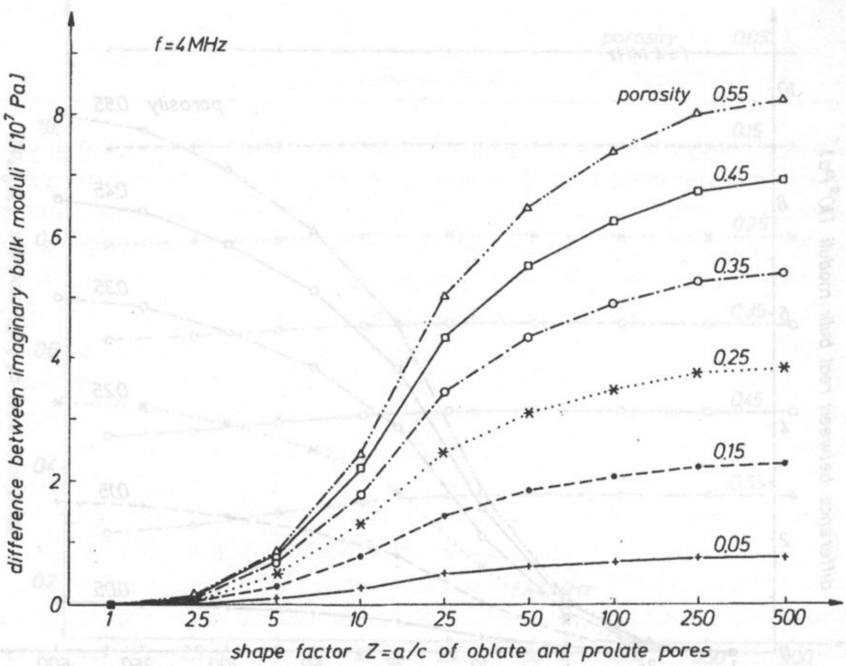

Fig. 7. Increase in the imaginary part of the bulk modulus accompanying the changes from prolate to oblate pore shapes as a function of the porosity and shape factor $Z=a/c$ of the air-saturated pores in sintered copper powder.

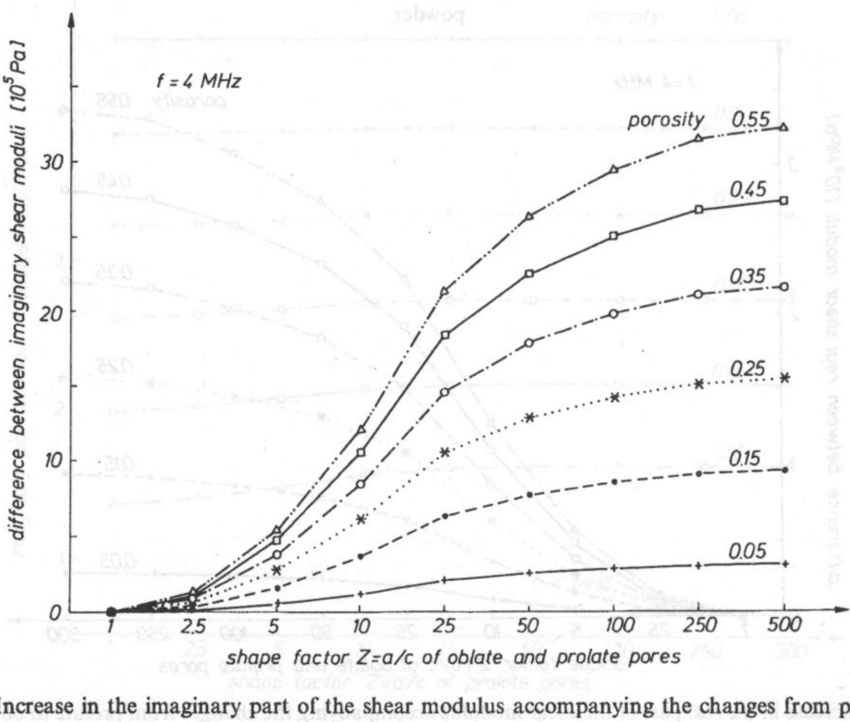

Fig. 8. Increase in the imaginary part of the shear modulus accompanying the changes from prolate to oblate, pore shapes as a function of the porosity and shape factor $Z=a/c$ of the air saturated pores in sintered copper powder.

factor $Z=a/c$, independently of the pore size and orientation. The shape factor is a measure of the elongation or oblateness of a prolate or oblate pore, respectively. From the computational analysis (Figs. 5–8) it follows that, under the condition of fixed values of Z and porosity, the changes from prolate to oblate pore shapes (or vice versa) cause only very small changes in the value of the effective dynamic moduli. The air can be thought of as supplying a correction to the respective results for evacuated pores. From the computational analysis it also follows that it is justified to replace in the analysis the evacuated pores by air-saturated ones, since the discrepancies between the results obtained for both the cases are very small ($<1.5\%$).

4. Conclusions and remarks

Contrary to the simple model of a two-phase material composed of two lossless constituents, the presented analysis of the wave propagation based on the Berryman concept [4] leads to the situation where the scattering is not the only mechanism of the overall attenuation of the composite, but in lasticity of the materials of the phases themselves contribute also to this effect. It occurs since the elastic moduli of the both phases are assumed to be complex.

In Figs. 1–4, the dynamic bulk and shear moduli Lamé constants K^* and μ^* of the sintered copper powder are plotted as functions of the shape factor $Z=a/c$ for various values of c_i (porosity). The general tendency of the real and imaginary parts of the dynamic moduli is to decrease as porosity increases. The results also indicate that in the case considered, the contribution of scattering to the imaginary parts of the complex components of the overall Hooke's tensor increases with increasing porosity more slowly than the contribution of the matrix phase inelasticity decreases. In Figs. 5–8, the increases of the real and imaginary parts of the dynamic moduli of the sintered copper powder are plotted as functions of the shape factor $Z=a/c$ for various values of porosity, the increases accompanying the changes in the pore shapes from prolate to oblate ones under constant value of Z.

Although all the results presented above have been obtained under the assumption that the long-wavelength condition ($\Lambda \gg D$) enables the single-scattering approximation to be used, and that the non-spherical inclusions are randomly oriented in a bulk sample of the composite, it can be stated that the presented results confirm the conclusion of paper [1] that the ultrasound is extremely useful in estimating the elastic and inelastic properties of the heterogeneous matrix phase as well as in determining the influence of the volume concentration and shape of the inclusions on the overall dynamic properties of the multi-phase composite under study.

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