JOURNAL OF MATERIALS SCIENCE **34** (1999) 4811–4814

Analysis of ultrasonic velocity-porosity data in polycrystalline materials using rotation-iteration technique

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A modified regression analysis (rotation iteration technique) has been applied to establish the ultrasonic velocity-pore volume fraction relationship in some important polycrystalline materials (uranium dioxide, alumina and α-silicon carbide). The new values obtained are compared with the values reported for a single crystal and the results are discussed.

1. Introduction

Ultrasonic characterization of different polycrystalline materials using ultrasonic velocity has been widely reported in the literature. Variation of ultrasonic velocity as function of pore volume fraction has been studied in nuclear ceramics [1–5], structural ceramics [6–8], sintered powder metals [9], clay ceramics [10], sedimentary and metamorphic rocks [11] and superconducting ceramics [12]. The velocity-pore volume fraction data has been found to follow in general a linear relationship of the form

$$V_l = V_{lo}(1 - ap) \tag{1}$$

where V_l and V_{lo} are the longitudinal ultrasonic velocities in porous and non-porous material, p is the pore volume fraction and a is a constant. The data are fitted using the method of least squares. The method of least squares assumes that there is no error in the measurement of x coordinate values i.e., pore volume fraction p. There however definitely exists some error in the estimation of pore volume fraction of the experimental specimens. An improved regression analysis method which takes into account errors in both the axes has been recently proposed by Papadakis [13]. This approach fits the data points to a straight line corresponding to the major axis of the ellipse formed by the data points.

This paper explains the new regression technique (rotation-iteration) briefly and re-evaluates the ultrasonic velocity-pore volume fraction relationship in some important polycrystalline materials like uranium dioxide, alumina and α-silicon carbide and discusses the results.

2. Rotation-iteration technique

It is assumed that most of the data points lie within an elliptical outline described by the equation of the form in the x-y coordinate system.

$$Ax^2 + Bxy + Cy^2 + F = 0 \tag{2}$$

where A, B, C and F are constants.

Papadakis [13] has proposed that the best regression line through these points is the major axis of the ellipse which would take into account the variations in both x and y coordinates.

As seen from Fig. 1, the original coordinate system has to be rotated through an angle θ so that the major axis is parallel to the new x-axis (OX′). The angle of rotation θ is given by the equation

$$2\theta = \cot^{-1}\{(A - C)/B\} \tag{3}$$

The new intercept of the fitted equation on the y' axis is such that the sum of the squares of the deviation is minimum. The calculation of θ and the intercept is done by iteration as given in the following steps.

Step 1: Calculate the regression equation by the standard method of least squares in the x-y coordinate system, i.e.,

$$y = mx + c$$

where m is the slope and c is the intercept.

Step 2: Find the sum

$$S = \sum_{i=1}^{n} \{y_{i,\text{expt.}} - y_{i,\text{fitted}}\}^2 \tag{4}$$

where $y_{i,\text{expt.}}$ is the measured value, $y_{i,\text{fitted}}$ is the calculated value and n is the number of points.

Step 3: The x-y coordinates are rotated through an angle,

$$\theta = \tan^{-1} m.$$

Least square analysis is repeated in the x'-y' axes to find the new intercept c'. The steps 1–3 are repeated till the value of S becomes nearly a constant. When the change in S value and θ is minimum, the iteration is stopped. The final angle of rotation θ_f is given by the sum of individual θ values.

$\theta_f = \sum_i \theta_i$ and the final intercept C_f is value found in the last iteration.

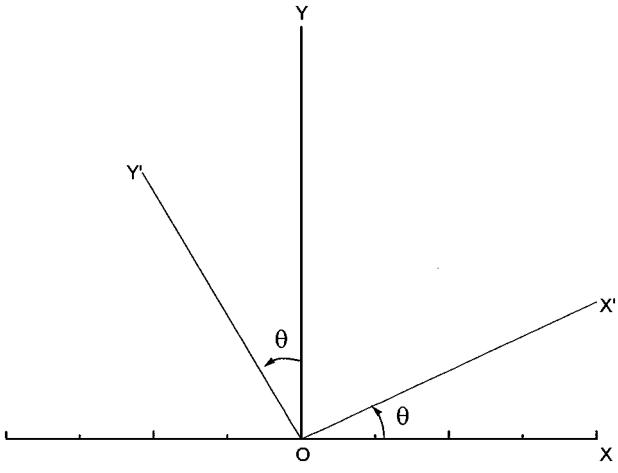


Figure 1 Rotation-iteration technique.

Software was developed for performing the analysis and was used in the analysis of velocity-porosity data of the polycrystalline materials.

3. Analysis of data

Ultrasonic velocity-porosity data of polycrystalline materials of different types (nuclear, structural) are analysed using the rotation-iteration technique. Panakkal [3] analyzed the ultrasonic velocity data of sintered uranium dioxide in the range of pore volume fraction of 0–0.28 and fitted in the data into a linear equation (Equation 1), given by

$$V_l\,(\text{km/s}) = 5.416(1 - 1.35p) \qquad (5)$$

by the standard method of least squares. By applying rotation-iteration technique, the data gives a relationship

$$V_l = 5.457(1 - 1.436p) \qquad (6)$$

The velocity of non-porous UO_2 has increased to 5.457 km/s which is the reported single crystal value [14]. The rotation-iteration technique thus gives a better extrapolated value considering the fact that the data have been taken from measurements made by different authors over a wide range of porosity. Fig. 2a presents the experimental data and the old (—) and new (---) fitted lines.

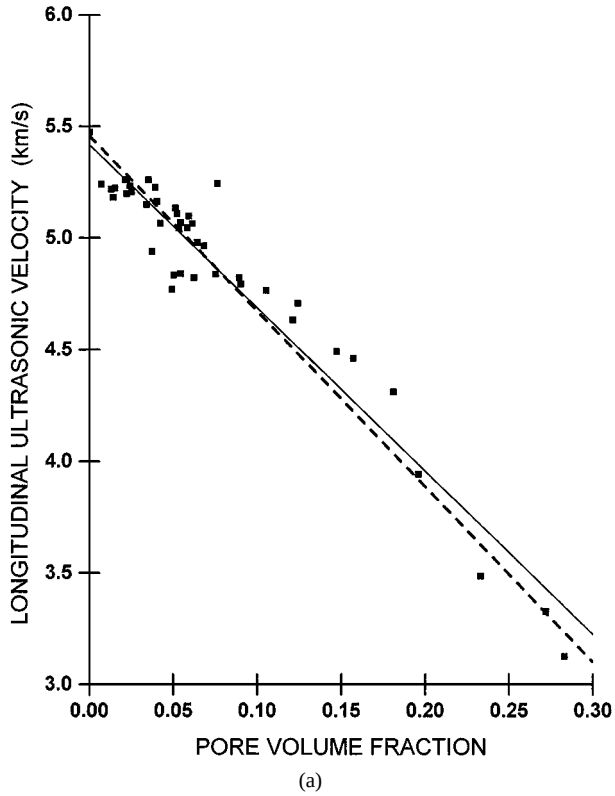
An attempt was also made to fit the data covering the pore volume fraction range of 0–0.28 into the Wylie equation [11] proposed for sedimentary and metamorphic rocks.

$$1/V_l = 1/V_{lo} + kp \qquad (7)$$

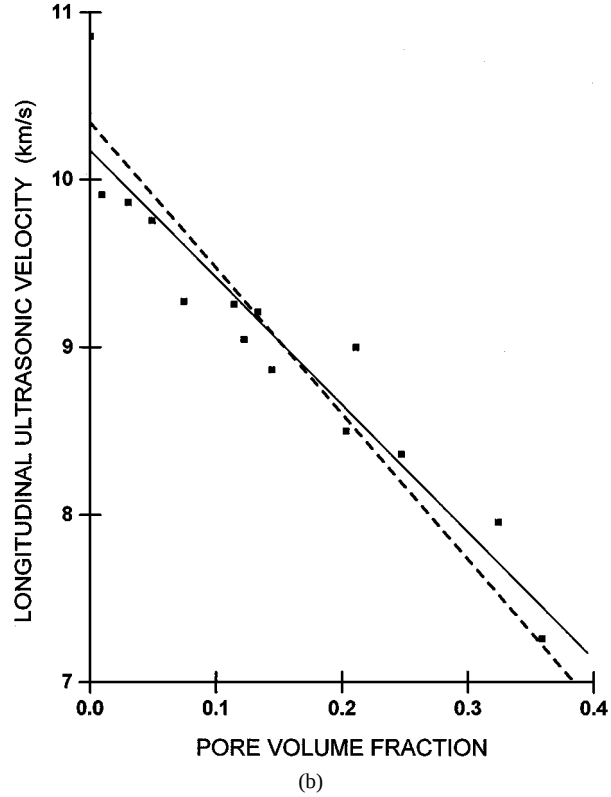
where k is a constant.

The non-porous value obtained was 5.615 km/s which was higher than the reported single crystal value and hence not used for the analysis of the data of UO_2.

A similar evaluation was made in alumina and α-silicon carbide samples (Fig. 2b and c). Table I presents the new constants of the fitted equations and extrapolated values of longitudinal ultrasonic velocity V_{lo} for non-porous material. The last column lists the reported non-porous value based on single crystal measurement. The reported values on Aluminium oxide samples (6, 15) were used to do the modified regression analysis and the value increased from 10.173 to

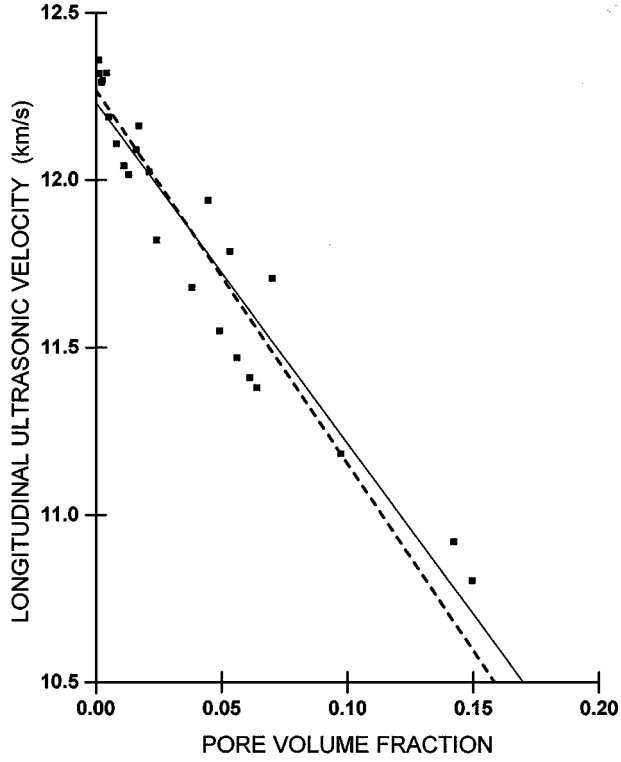


(a)



(b)

Figure 2 Ultrasonic velocity-porosity relationship using rotation-iteration technique; ("—" standard linear regression, "---" rotation-iteration technique). (a) UO_2, (b) Al_2O_3 and (c) α-SiC. (*Continued*).



(c)

Figure 2 (*Continued*).

TABLE I Longitudinal ultrasonic velocity V_l as a function of pore volume fraction p, $V_l = V_{lo}(1 - ap)$ using standard linear regression (LR) and rotation-iteration techniques (RIT)

Material		V_{lo} (km/s)	Constant a	V_{lo} (km/s) for single crystal	No. of iteration
UO_2	LR	5.416 [3]	1.35	5.457 [14]	6
	RIT	5.457	1.44		
Al_2O_3	LR	10.173 [6, 15]	0.75	10.858 [16]	5
	RIT	10.339	0.84		
α-SiC	LR	12.232 [1, 8, 16, 17]	0.83	11.684 [1]	6
	RIT	12.269	0.91	(12.257)[a]	

[a]Average value of non-porous polycrystalline material.

10.339 km/s. The value of longitudinal ultrasonic velocity of non-porous alumina based on single crystal measurement reported was 10.858 km/s [16]. The rotation-iteration technique gives a value closer to the single crystal value. If the analysis is done with values obtained with the more accurate measurement techniques available to-day, a better agreement is expected. In the case of α-silicon carbide, the value obtained by the rotation-iteration technique increased to 12.269 km/s. A study of published data on silicon carbide shows that there is a wide variation in the extrapolated values of the longitudinal ultrasonic velocity for non-porous α-silicon carbide, i.e., 12.125 to 12.382 km/s depending on the technique of fabrication [1, 8, 17, 18]. It may be noted that the extrapolated value reported from polycrystalline specimens has always been more than the reported single crystal value, viz., 11.684 km/s [1]. The difference in the method of fabrication and purity of sample may be the possible reasons for the discrepancy. However, the rotation-iteration technique gives a value closer to the average of extrapolated values of V_{lo} (12.257 km/s) reported by different authors.

4. Conclusion

The use of the rotation-iteration technique for evaluation of the ultrasonic velocity-porosity relationship is demonstrated taking examples of nuclear and structural ceramics. The non-porous values of longitudinal ultrasonic velocity obtained show, in general, closer agreement with single crystal values. The discrepancy observed in α-SiC is probably due to the different fabrication techniques used. Further, the values measured on polycrystalline α-SiC of higher density are larger than the single crystal values. The value obtained by the rotation-iteration technique is in close agreement with the average of the polycrystalline samples. The new ultrasonic velocity-pore volume fraction relationship obtained using the rotation-iteration technique gives a more realistic and meaningful relationship.

Acknowledgements

The authors would like to express their sincere thanks to Mr. H. S. Kamath, Head, Advanced Fuel Fabrication Facility for his appreciation and help during the work. They would also like to thank Mr. D. S. C. Purushotham, Associate Director, Nuclear Fuels Group and Mr. M. S. Ramakumar, Director, Nuclear Fuel, Automation and Manufacturing Group of Bhabha Atomic Research Centre for their keen interest in this work.

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Received 28 October 1997
and accepted 1 April 1999