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Fracture toughness variability of foamed alumina cements

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Abstract

Preexisting micropores and microcracks within solid cement paste cause a variation in both the cell wall modulus of rupture and fracture toughness of foamed cements. Here, we determine for the variability of cell wall modulus of rupture by assuming that it follows a Weibull statistical distribution. Consequently, the variation of fracture toughness of foamed cements depends on the Weibull modulus of solid cement paste. A series of measurements on the Mode I fracture toughness of foamed alumina cements with various relative densities were conducted. Results indicate that the Weibull moduli of solid cement paste for foamed alumina cements are within the range of 7 to 10 except for the foamed alumina cement with a relative density of 0.167.

Keywords: Microcracking; Fracture toughness; Micromechanics

1. Introduction

For a number of years, foamed cements were utilized as lightweight cores in sandwich structures and thermal insulators because of their good creep resistance, high specific stiffness and strength, and low thermal conductivity. The unique properties of foamed cements are exploited in many engineering applications. The mechanical properties of foamed cements can be analyzed by employing a cell wall-bending model proposed by Gibson and Ashby [1]. Theoretical results from the cell wall bending model suggest that the strengths and fracture toughness of brittle foamed cements are affected by their cell geometry, relative density and cell wall modulus of rupture. Tonyan and Gibson [2] measured the elastic modulus and compressive strength of foamed portland cements and verified that the elastic properties of foamed portland cements are described well by the cell wall bending model. Brezny and Green [3,4] measured the fracture toughness and compressive strength of open-cell foamed ceramics and concluded that cell wall modulus of rupture controls their fracture toughness and strength.

Preexisting micropores and microcracks within solid cement paste of foamed cements primarily resulting from manufacturing might lead to a reduction of cell wall modulus of rupture. Also, the variation of cell wall modulus of rupture is influenced by the micropore and microcrack size distribution within solid cement paste. As a result of these defect variations, variable cell wall modulus of rupture for brittle foamed cements can be expected. In experiments, the cell wall modulus of rupture for open-cell foamed materials might be directly measured, giving a variation of cell wall modulus of rupture. Brezny et al. [5], using a technique of threading thin steel wire beneath a cell wall and connecting it to a tensile load cell, evaluated the cell wall modulus of rupture in open-cell foamed alumina and carbon material. The variation of cell wall modulus of rupture for open-cell foamed alumina was found to be significant and dependent on the volume of individual cell walls, leading to a cell size effect. On the contrary, the cell wall modulus of rupture for closed-cell foamed cements is difficult to analyze accurately and, to some extent, impossible to directly quantify due to their complex microstructure. Therefore, we propose to experimentally measure the variation of fracture toughness for foamed alumina cements with different relative densities. Based on experimental results, the variability of cell wall modulus of rupture in foamed alumina cements can be predicted and evaluated.

The variability of cell wall modulus of rupture can be accounted for by assuming that it follows a Weibull statistical distribution [6]. Huang and Gibson [7] investigated the fracture toughness of foamed ceramics using the Weibull

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statistical analysis. They showed that the effect of cell size on the fracture toughness of reticulated vitreous carbon foams is dependent upon the Weibull modulus of solid cell walls. Failure envelopes of brittle foamed materials under multiaxial loads were developed and presented by Huang and Chou [8]. It was found that the failure envelopes of brittle foamed materials are dependent on cell size, prescribed failure probability and the Weibull modulus of solid cell walls. In this paper, we aim at determining the Weibull moduli of solid cement paste and the variation of cell wall modulus of rupture within foamed alumina cements. The reliably measured fracture toughnesses of closed-cell foamed cements are more likely to accomplish. The variation of measured fracture toughnesses can be utilized to calculate the corresponding Weibull moduli of solid cement paste within foamed alumina cements with various relative densities. To do that, both the variations of cell wall modulus of rupture and fracture toughness for brittle foamed cements need to be analyzed first.

2. Weibull statistical analysis

Weibull [6] suggested that the failure probability of a brittle solid with a volume of V, subjected to a uniform tensile stress σ, could be described well by the following equation:

$$P_{\rm f} = 1 - \exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{1}$$

Here σ_0 is a scale parameter or normalizing stress, V_0 is a unit volume and m is the Weibull modulus of the brittle solid. Variable cell wall modulus of rupture in brittle foamed materials is accounted for by assuming that it follows a Weibull statistical distribution.

For brittle foamed materials with a critical macrocrack, the applied stresses are transmitted through the foamed materials as a set of discrete forces and moments acting at each cell wall. The macrocrack advances one cell size distance when the maximum tensile stress within the first unbroken cell wall ahead of macrocrack tip exceeds its cell wall modulus of rupture. Solid cell walls in brittle foamed materials loaded in bending produce a nonuniform stress field. The failure probability for individual solid cell wall within brittle foamed materials subjected to a nonuniform stress field is [9]:

$$P_{\rm f} = 1 - \exp\left[-\int_V\left(\frac{\sigma_{\rm s}}{\sigma_0}\right)^m\frac{{\rm d}V}{V_0}\right] \qquad \text{for } \sigma_{\rm s} > 0 \tag{2}$$

where $\sigma_{\rm s}$ is the induced normal stress acting at any point within individual solid cell walls. Note that only tensile stresses are taken into account in calculating the failure probability.

The induced tensile stress acting at any point in the bent solid cell wall can be expressed in terms of its cell wall modulus of rupture:

$$\sigma_{\rm s} = \sigma_{\rm fs} g \tag{3}$$

and the failure probability becomes:

$$P_{\rm f} = 1 - \exp\left[-\left(\frac{\sigma_{\rm fs}}{\sigma_0}\right)^m\int_V g^m\frac{{\rm d}V}{V_0}\right] \tag{4}$$

Here g depends on the loading configuration and cell geometry of the bent cell wall. In practice, Eq. (4) is difficult to carry out due to the complex microstructure in closed-cell foamed materials. Nevertheless, the failure probability can still be expressed as:

$$P_{\rm f} = 1 - \exp\left[-\left(\frac{\sigma_{\rm fs}}{\sigma_0}\right)^m\frac{V}{V_0}f(m)\right] \tag{5}$$

Here $f(m)$ is a function of the Weibull modulus of the solid cell walls, relying on the cell geometry of closed-cell foamed materials.

Mechanical properties of closed-cell foamed materials are the sum of three contributions: cell-edge bending, cell-face stretching and cell-fluid compression [1]. For closed-cell foamed cement materials, the contribution of cell-fluid compression can be neglected because the cell fluid is air instead of liquid. Based on the near-tip singular stress fields of a continuum model, Maiti et al. [10] were able to derive a theoretical expression for Mode I fracture toughness of brittle foamed materials. In their derivation, they assumed that cell wall bending is dominant and cell wall modulus of rupture is constant. When both the cell-edge bending and cell-face stretching are considered, the theoretical expression for Mode I fracture toughness of brittle, closed-cell foamed materials, $K^*_{\rm IC}$, becomes:

$$\frac{K^*_{\rm IC}}{\sigma_{\rm fs}\sqrt{\pi\ell}} = C_1\left(\phi\frac{\rho^*}{\rho_{\rm s}}\right)^{3/2} + C_1''(1-\phi)\frac{\rho^*}{\rho_{\rm s}} \tag{6}$$

where C_1 and C_1'' are microstructure coefficients, ϕ is the volume fraction of solid contained in the cell edges, $(1-\phi)$ is the remaining fraction of solid contained in the cell faces, ℓ is the cell size, ρ^* is the density of foamed materials and $\rho_{\rm s}$ is the density of solid cell walls. Huang and Liu [11] found that $C_1 = 0.65$, $C_1'' = 2.0$ and $\phi = 0.8$ for closed-cell foamed alumina cements; that is, 80% of solid alumina cement paste is contained in the cell edges.

The cell wall modulus of rupture can be expressed in terms of the fracture toughness of foamed materials from Eq. (6) and then are substituted into Eq. (5), giving:

$$P_{\rm f} = 1 - \exp\left[-\left(\frac{K^*_{\rm IC}}{K^*_0}\right)^m\frac{V}{V_0}f(m)\right] \tag{7}$$

The scale parameter K_0^* is:

$$K_0^* = C_1 \sigma_0 \sqrt{\pi \ell} \left(\phi \frac{\rho^*}{\rho_s} \right)^{3/2} + C_1'' \sigma_0 \sqrt{\pi \ell} (1 - \phi) \frac{\rho^*}{\rho_s} \tag{8}$$

Rewriting Eq. (7) as:

$$K_{IC}^* = K_0^* \left[\frac{1}{f(m)V} \frac{V_0}{V} \ln \left(\frac{1}{1 - P_f} \right) \right]^{1/m} \tag{9}$$

At the same time, the mean fracture toughness of brittle closed-cell foamed materials can be calculated from Eq. (7):

$$\begin{aligned} \overline{K_{IC}^*} &= \int_0^\infty (1 - P_f) dK_{IC}^* \\ &= \int_0^\infty \exp \left[- \left(\frac{K_{IC}^*}{K_0^*} \right)^m \frac{V}{V_0} f(m) \right] dK_{IC}^* \\ &= K_0^* \left(\frac{1}{f(m)} \frac{V_0}{V} \right)^{1/m} \Gamma \left(1 + \frac{1}{m} \right) \end{aligned} \tag{10}$$

where $\Gamma(1+1/m)$ is the gamma function. Therefore, the ratio of the fracture toughness for a prescribed failure probability and the mean fracture toughness can be found from Eqs. (9) and (10), regardless of the cell geometry of solid cell walls:

$$\frac{K_{IC}^*}{\overline{K_{IC}^*}} = \frac{\left[\log \left(\frac{1}{1 - P_f} \right) \right]^{1/m}}{\Gamma \left(1 + \frac{1}{m} \right)} \tag{11}$$

From Eq. (11), it is clear that the fracture toughness of brittle closed-cell foamed materials is not a constant, but rather depends on the Weibull modulus of solid cell walls and the prescribed failure probability. The Weibull modulus of solid cell walls can be determined from the variations of measured fracture toughness of brittle closed-cell foamed materials in experiments.

3. Experimental method

Foamed cement specimens are made from alumina cement (Asano, Japan), water, superplasticizer and preformed air bubbles. The chemical compounds of the alumina cement we used include 5% SiO_2, 53% Al_2O_3, 0.8% Fe_2O_3, 38.2% CaO and 2.3% TiO_2. Alumina cement slurry with a water/cement ratio of 0.6 and with a superplasticizer/cement ratio of 0.5% was made first. Different amounts of preformed air bubbles were mixed mechanically with alumina cement slurry, giving a series of specimens with varying relative densities. Five different design relative densities of foamed alumina cements were considered here: 0.15, 0.2, 0.3, 0.35 and 0.45; the density of solid alumina cement paste is 1780 kg/m^3. The wet foamed alumina cements were cast into steel molds after complete mixing. One day later, the foamed alumina cement specimens were removed from steel molds and cured in water at room temperature for 7 days. Some foamed alumina cement specimens with visible cracks and flaws were observed and thus discarded. Foamed alumina cement specimens were then trimmed on both ends before mechanical testing.

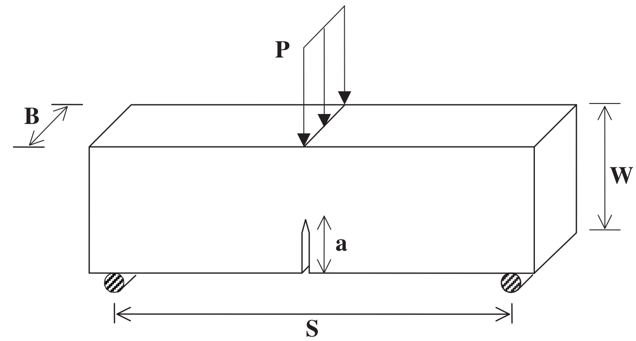

Fig. 1. The dimensions and loading configuration for notched beam specimens.

Notched beam specimens of $40 \times 50 \times 210$ mm for foamed alumina cements with different relative densities were produced and loaded under three-point bending. A central saw-cut notch with a depth of 20 mm was made for each notched beam specimen. The dimensions and loading configuration are illustrated in Fig. 1. Also, square beam specimens of $20 \times 20 \times 210$ mm for measuring the modulus of rupture of solid alumina cement paste were made and tested. During mechanical testing, the crosshead of an Instron machine moved at a constant speed of 0.5 mm/min. The corresponding load and deflection for each specimen were recorded on a computer up to the point at which fracture occurred. Then, the Mode I fracture toughness of foamed alumina cements and the modulus of rupture of solid cement paste were calculated from the applied loads at failure.

4. Results and discussion

The actual density of each foamed alumina cement specimen was measured before mechanical testing. The measured relative densities of foamed alumina cements are 0.167, 0.196, 0.297, 0.374 and 0.463. Air bubbles within foamed alumina cements are deformed under a loading of heavier alumina cement paste as the design relative density is increased, causing adjoining cells to collapse and coalesce before a complete hardening. As expected, the measured relative density is slightly higher than the design relative density. A typical microstructure of foamed alumina cements is shown in Fig. 2. Huang and Liu [11] have characterized

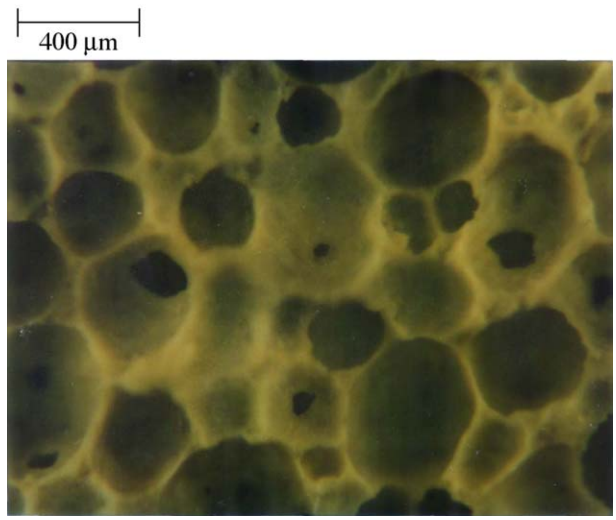

Fig. 2. A typical microstructure of foamed alumina cements.

the cell lengths along three principal directions for foamed alumina cements with different relative densities. They reported that the microstructure of foamed alumina cements is axisymmetric due to the effect of gravitational loading of solid cement paste. But, the aspect ratio of cell microstructure is close to 1. The mean cell lengths along the gravitational direction for foamed alumina cements we studied here are listed in Table 1. To the first-order approximation, the mechanical properties of the foamed alumina cements are isotropic.

The ratio of precut crack depth to cell length for notched beam specimens is greater than 10 to eliminate the short crack effect on fracture toughness of foamed materials as suggested by Huang and Gibson [7]. The Mode I fracture toughness of notched foamed alumina cement specimens loaded in three-point bending can be calculated from the applied concentrated force at failure, P, using the following equation [12]:

$$K_{\mathrm{IC}}^{*}=\frac{PS}{BW^{3/2}}f\left(\frac{a}{W}\right) \tag{12}$$

where S, B, W and a are the span, width and depth of notched specimens and the notch depth, respectively. For the notched beam specimens, $S=160$ mm, $B=50$ mm, $W=40$ mm and $a=20$ mm. The function $f(a/W)$ is given by [12]:

$$f\left(\frac{a}{W}\right)=\frac{3\left(\frac{a}{W}\right)^{0.5}\left\{1.99-\frac{a}{W}\left(1-\frac{a}{W}\right)\left[2.15-3.93\left(\frac{a}{W}\right)+2.7\left(\frac{a}{W}\right)^{2}\right]\right\}}{2\left(1+2\frac{a}{W}\right)\left(1-\frac{a}{W}\right)^{1.5}} \tag{13}$$

The fracture toughness of each notched beam specimen is found once the maximum applied force is determined experimentally. Consequently, the variation of measured fracture toughness for foamed alumina cements is obtained.

To determine the Weibull modulus of solid cement paste, Eq. (7) can be rearranged as:

$$\ell n\left[\ell n\left(\frac{1}{1-P_{\mathrm{f}}}\right)\right]=m[\ell n(K_{\mathrm{IC}}^{*})]+\ell n\left(\frac{V}{V_0}\right)+\ell n\left[\frac{f(m)}{(K_0^{*})^{m}}\right] \tag{14}$$

The measured fracture toughnesses are ordered according to their magnitude and are assigned a corresponding failure probability. The measured fracture toughness and the assigned failure probability are plotted in a figure for foamed alumina cements with any particular relative density. The slope of the plot of $\ell n[\ell n(1\text{-}P_{\mathrm{f}})^{-1}]$ against $\ell n(K_{\mathrm{IC}}^{*})$ gives the Weibull modulus of solid cement paste. Such a plot for foamed alumina cements with a relative density of 0.196 is shown in Fig. 3. The slopes of the lines, giving the Weibull moduli of foamed alumina cements with the relative densities of 0.167, 0.196, 0.297, 0.374 and 0.463, are calculated and listed in Table 1. For a material with a

Table 1
The measured relative density, cell size, fracture toughness, Weibull modulus and cell-wall modulus of rupture for foamed alumina cements with various relative densities

$\overline{\rho^*/\rho_s}$ (–)	$\bar{\ell}$ $(10^{-6}$ m)	$\overline{K_{\mathrm{IC}}^{*}}$ $(KN-m^{-3/2})$	m (–)	$\overline{\sigma_{\mathrm{fs}}}$ (MPa)
0.167	267.2	14.04	2.09	4.90
0.196	249.7	21.15	8.13	6.36
0.297	240.0	37.66	7.38	7.07
0.374	235.1	52.42	7.73	7.53
0.463	228.3	68.04	10.20	7.66
1.000	–	–	7.37	6.76

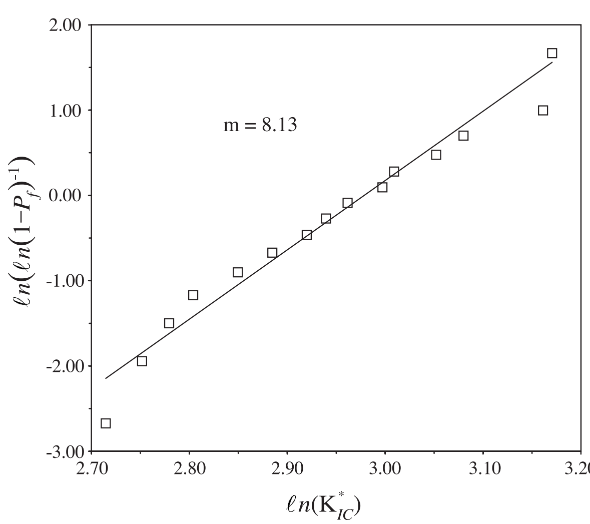

Fig. 3. The measured fracture toughness plotted against its corresponding failure probability for foamed alumina cements with a relative density of 0.196. The slope of the plot gives the Weibull modulus, m.

constant flaw population, m should be constant. It is seen that the results are not consistent with this: for foamed alumina cements with relative densities of 0.463, 0.374, 0.297 and 0.196, their Weibull moduli are roughly 8 while for those with a relative density of 0.167, the Weibull modulus is reduced to 2.

A similar procedure is applied to analyze the modulus of rupture for solid alumina cement beam with a volume of V. The corresponding modulus of rupture of a solid alumina cement beam for a prescribed failure probability is found to be:

$$\sigma_{\mathrm{fs}} = \sigma_0 \left[2(m+1)^2 \left(\frac{V_0}{V} \right) \ell \mathrm{n} \left(\frac{1}{1-P_{\mathrm{f}}} \right) \right]^{1/m} \quad (15)$$

Again, the measured modulus of rupture and the assigned failure probability are plotted in Fig. 4. The slope of the plot of $\ell n(\ell n(1\text{-}P_{\mathrm{f}})^{-1})$ against $\ell n(\sigma_{\mathrm{fs}})$ gives the Weibull modulus of solid alumina cement beam, $m=7.37$. Also, the mean modulus of rupture of solid alumina cement beams under three point bending is found to be 6.76 MPa.

From experimental results, it is noted that the Weibull modulus of solid cement paste can be regarded as a constant except for the foamed alumina cements with a relative density of 0.167. The reason for that might be due to the coalescence of preformed air bubbles into larger cells for lower relative density foamed alumina cements, introducing some flaws and cracks within solid cement paste. Meanwhile, the difficulty in complete mixing of cement paste and preformed air bubbles as the relative density is decreased could be another factor. It is expected that a smaller Weibull modulus of solid cement paste will cause a reduction in strengths and fracture toughness of foamed alumina cements.

Assuming that the cell wall modulus of rupture in foamed alumina cements is constant and equal to that of solid alumina cement beam, the mean fracture toughness of each relative density foamed alumina cement can be calculated from Eq. (6). The calculated mean fracture toughnesses for foamed alumina cements we studied are shown in Fig. 5; $C_1=0.65$, $C_1''=2.0$, $\phi=0.8$, $\sigma_{\mathrm{fs}}=6.76$ MPa and the mean cell size listed in Table 1 are used. We had looked at the microstructure of the foamed alumina cements as a function of relative density. The degree of openness tends to increase at low relative densities and most likely is not constant over the entire relative density range that was evaluated. From Fig. 5 it is seen that the measured fracture toughness is higher than the calculated fracture toughness for higher relative density foamed alumina cements; presumably, the volume of solid cell walls is much smaller than that of solid alumina cement beam, giving a size effect on the cell wall modulus of rupture. On the contrary, the measured fracture toughness is lower than the calculated fracture toughness for lower relative density foamed alumina cements. Since the calculated fracture toughness depends nonlinearly on relative density, it is seen that there is a change in the slope of the theoretical line for Eq. 6 in Fig. 5.

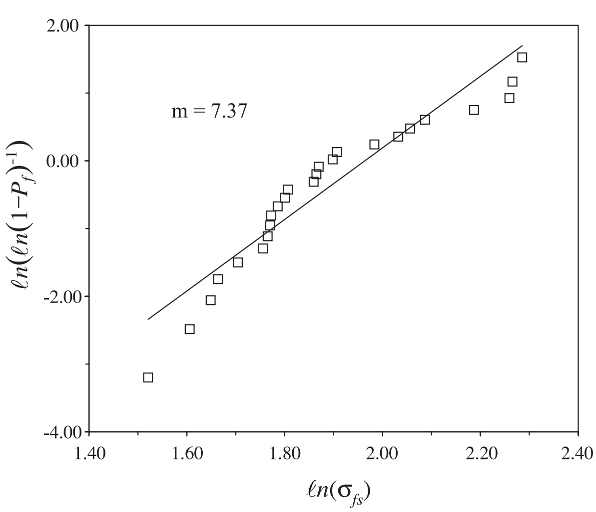

Fig. 4. The measured modulus of rupture plotted against its corresponding failure probability for solid alumina cement paste ($\rho^*/\rho_S=1.0$).

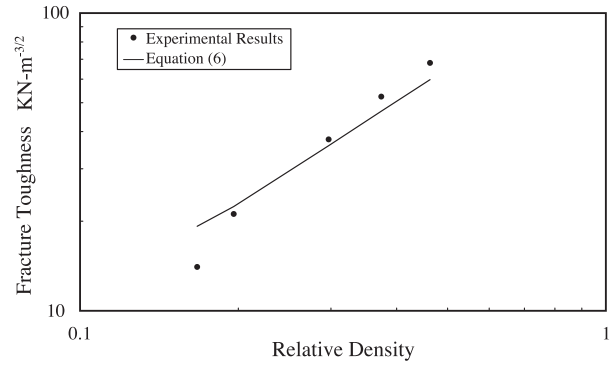

Fig. 5. The fracture toughness of foamed alumina cements with different relative densities.

Once the mean cell length, relative density and fracture toughness of foamed alumina cements are known, their corresponding mean cell wall modulus of rupture can be determined from Eq. (6):

$$\overline{\sigma_{\mathrm{fs}}} = \frac{\overline{K_{\mathrm{IC}}^*}}{\sqrt{\pi \bar{\ell}} \left[C_1 \left(\sigma \frac{\overline{\rho^*}}{\rho} \right)^{3/2} + C_1'' (1-\phi) \frac{\overline{\rho^*}}{\rho_{\mathrm{s}}} \right]} \quad (16)$$

The mean cell wall modulus of rupture is obtained from the measured mean fracture toughness of foamed alumina cements through Eq. (16) when $C_1=0.65$, $C_1''=2.0$ and $\phi=0.8$ are used. The variability of cell wall moduli of rupture in foamed alumina cements with different relative densities is thus plotted in Fig. 6. From Fig. 6, it is noted that the cell wall modulus of rupture increases gradually up to a peak value and then drops significantly as the relative

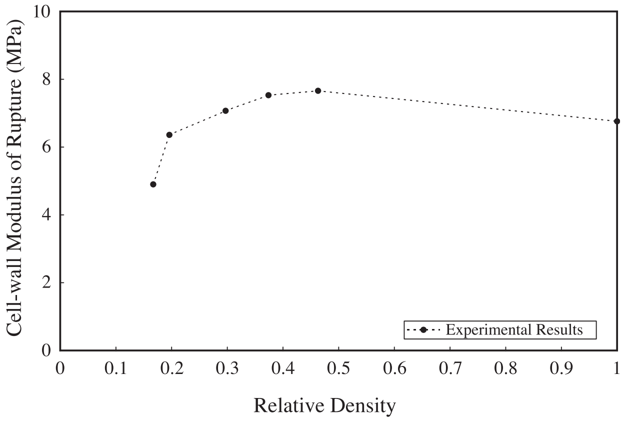

Fig. 6. The variation of cell-wall modulus of rupture for foamed alumina cements with different relative densities.

density of foamed alumina cements is decreased. The relative density with which foamed alumina cements have a peak value of cell wall modulus of rupture is roughly 0.25.

5. Conclusions

The variability of cell wall modulus of rupture is taken into account by assuming it follows a Weibull statistic distribution. It is found that the fracture toughness of brittle closed-cell foamed alumina cements depends on the Weibull modulus of solid cement paste and the prescribed failure probability. The Weibull modulus of solid cement paste can be determined from the variation of measured fracture toughnesses for foamed alumina cements with various relative densities. The Weibull moduli of solid cement paste in foamed alumina cements are found to be within the range of 7 to 10. However, the Weibull modulus becomes much smaller as the relative density is less than 0.2, leading to a significant decrease in both the cell wall modulus of rupture and the fracture toughness of foamed alumina cements. Meanwhile, experimental results indicate that the cell wall modulus of rupture increases gradually up to a peak value and then drops significantly as the relative density of foamed alumina cements is decreased.

Acknowledgements

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