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Inhomogeneous flow and the effective pressure concept in pressure sintering

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Many of the models proposed to describe pressure sintering of solids with a randomly distributed porosity have implicit in them the effect of porosity and it is incorrect to incorporate any extra pressure intensification factor. However, usually, pores are segregated into zones which leads to inhomogeneous deformation. A consequence of this inhomogeneity is to enhance densification rates by dislocation creep and particle sliding resulting in an anomalous pressure intensification factor. Some observations on microstructural development during hot isostatic pressing are also made.

1. Introduction

There have been a number of analytical treatments describing pressure sintering [1–4]. Sintering under these conditions has been visualized as being due to a number of creep mechanisms operating, activated by an externally applied pressure. In all these analyses, a problem arises as to what pressure one must use for formulating the densification equation. This is relevant as the applied pressure acts on a porous solid and not on a homogeneous material and could therefore be modified.

Coble [1] considered sintering of a powder compact under pressure and showed that this pressure manifests as stresses around pores activating various diffusional flow processes. He evaluated the "effective stress" present around pores on the basis of equations advocated by a number of workers, and suggested that the external pressure divided by the relative density could be a reasonable approximation. He went on to point out that as the densification equation contained a number of parameters which were known only approximately, it would not seriously matter as to which of the formulations for the effective stress was used in the equation.

Wilkinson and Ashby [2] have recently analysed the final stages of pressure sintering, modelling the porous powder compact in terms of a representative unit undergoing deformation. For the effective stress, they suggested the externally applied pressure itself and pointed out that any addition to this pressure term would be erroneous.

In all these models we assume the pores to be distributed randomly. This indeed is an idealization as pores in reality are always segregated. In a powder compact, pores are generally present only around powder particle boundaries and in a crept solid only in grain boundaries. These pores are not only segregated to start with but also persist in remaining near particle boundaries even during sintering. This segregation removes the macroscopic equivalence between idealized models and real examples rendering most of the models less accurate.

In this paper we shall re-examine the concept of "the effective pressure" and show how this concept is intrinsically incorporated in many of the models, making further modifications incorrect. We shall also define an effective pressure for solids containing segregated pores and evaluate the consequences of segregation on different flow processes operating to close the pores.

2. Pressure intensification and effective pressure

An easy way to visualize pressure intensification is through a force balance across a random cross-section in a porous solid. For a solid with random pores, every cross-sectional plane would contain an area fraction of voids designated as θ. This reduces the load-bearing area and effectively raises the stress level on any plane making the average stress level higher than in a pore-free solid. The pressure intensification factor can

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thus be written as

$$f = \frac{1}{1-\theta}. \tag{1}$$

This is not to imply that the effective pressure is isostatic and yet higher than the externally applied pressure; for, then the material would squeeze itself out! What is actually intensified are the stresses with pore areas excluded from the averaging procedure. Similarly, the stress invariants such as J_1 are raised at any point in the solid by $1/(1-\theta)$ suggesting that a pressure intensification factor $1/(1-\theta)$ ought to be included in the densification equations [5].

However, many of the models already have this factor already implicit in them. For example, let us consider a model for the final stages of sintering: a pore of radius a in a sphere of radius b such that

$$\theta = \frac{a^3}{b^3}. \tag{2}$$

The stress distribution at a point r from the centre in such a solid under an external pressure p is given by [6]

$$\sigma_1 = \sigma_2 = \frac{-pb^3(a^3+2r^3)}{2r^3(b^3-a^3)}$$

$$\sigma_3 = \frac{-pb^3(r^3-a^3)}{r^3(b^3-a^3)} \tag{3}$$

where σ_1, σ_2 and σ_3 are the principal stresses. The stress invariant J_1, which is the average of the principal stresses then becomes,

$$J_1 = \frac{\sigma_1+\sigma_2+\sigma_3}{3} = \frac{-pb^3}{b^3-a^3} = \frac{-p}{1-\theta}, \tag{4}$$

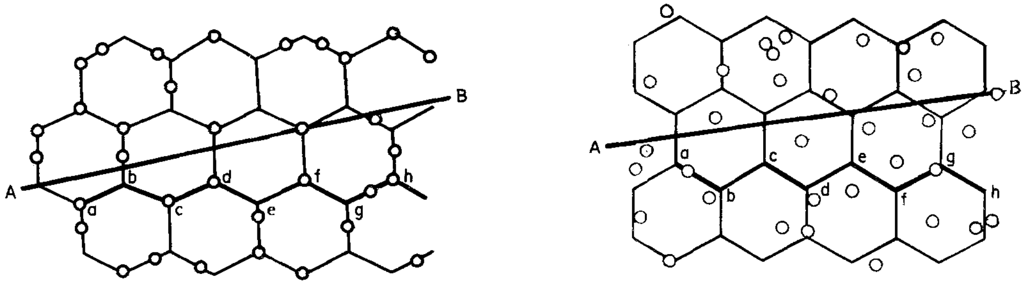
confirming that the existence of porosity has already been taken into account while formulating the model itself. Similar results can be obtained for other models as well, substantiating a recent suggestion that the introduction of a separate pressure intensification factor is erroneous.

The above analysis is valid only when the models are statistical macroscopic equivalents of a porous body. The equivalence is readily established when the pores are randomly distributed. However, when they are segregated, as in a powder compact where they are found only in particle boundaries, or in a crept sample where they are present in grain boundaries, this equivalence is destroyed. In the following sections we shall examine the consequences of pore segregation and show how it could lead to an anomalous pressure intensification.

3. Pore segregation and pressure intensification

Localization of pores in certain regions results in increased porosity compared to the average value in these regions with a concomitant reduction in other regions. Fig. 1a shows a distribution of pores segregated to particle boundaries, and Fig. 1b a randomly distributed porosity. This representation of the randomly distributed porosity was obtained by plotting 300 random points on a computer. Over this plot, we have superimposed the grain structure to locate randomly distributed pores against grain-boundary segregated ones. These figures show that porosity on particle boundary planes (a . . . h) is significantly higher than on any random plane (AB) when pores are segregated. When the pores are randomly distributed as in Fig. 1b, there is no such difference.

Inhomogeneous distribution of pores results



a) a-h SCAN ALONG PARTICLE BOUNDARIES
A-B A RANDOM LINE SCAN

b) a-h SCAN ALONG PARTICLE BOUNDARIES
A-B A RANDOM LINE SCAN

Figure 1 (a) Porosity segregating to particle boundaries leads to higher effective porosity levels in regions around the boundaries a–h and depleted levels at other regions A–B. (b) For randomly distributed porosity, particle boundaries have the same porosity as elsewhere.

in a shear stress localization leading to localization of strain to the regions around planes having a higher porosity. Localization of stresses can also be qualitatively seen from an analysis of stress distribution in a sheet with closely spaced circular holes subject to biaxial stress. Bailey and Fidler [7] used a point-matching technique for this analysis although a more accurate route would be through finite element techniques [8]. The result of the point-matching technique shows that σ_θ, the tangential stress, reaches a maximum, and σ_r, the radial stress a minimum on pore segregated planes. The flow rate which depends on $|\sigma_r - \sigma_\theta|$ is thus higher for pore-segregated regions and lower for planes far away from them. The resultant flow rate is thus inhomogeneous and symmetric models are, therefore, not accurate enough to describe the overall densification. In the following section, we shall describe an approximate method for evaluating the overall densification by separating these regions into two types: one deforming more, and the other less.

4. Inhomogeneous flow and its effect on overall flow rate

A rough approximation to visualize the inhomogeneous flow would be to divide the total volume of a porous solid into two zones: a boundary zone (with a higher porisity) where the flow rate is higher, and an inner zone with less porosity where the flow rate is subdued. Let the volume of the boundary zone be x and its porosity $k\theta$, where k is greater than one and increases with the severity of segregation. The remaining volume $(1-x)$ would then have a porosity $\theta(1-kx)/(1-x)$. The total densification rate can then be represented as the averaged sum of two densification rates, although a more accurate procedure would be to divide the volume into small elements and integrate the flow rates. We shall evaluate the flow rate for power law creep using Wilkinson and Ashby's [2] equation which in our notation becomes

$$\dot{\theta} = \frac{B\theta(1-\theta)}{(1-\theta^{1/n})^n}(p/n)^n \qquad (5)$$

where $\dot{\theta}$ is the closure rate, B a constant and n the stress exponent for creep. The overall densification rate in our analysis thus becomes

$$\dot{\theta}_s = B(p/n)^n\left(\frac{xk\theta(1-k\theta)}{(1-(k\theta)^{1/n})^n} + \frac{\theta(1-kx)(1-x-\theta+k\theta x)}{(1-x)\left\{1-\left[\theta\left(\frac{1-kx}{1-x}\right)^{1/n}\right]^n\right\}}\right) \qquad (6)$$

where $\dot{\theta}_s$ is the rate of densification when segegated pores are present. The difference between $\dot{\theta}_s$ and $\dot{\theta}$ can be described as due to an apparent pressure intensification factor, f_s, which intensifies the flow rate due to the segregation of pores.

f_s then can be written as

$$\left(\frac{\dot{\theta}_s}{\dot{\theta}}\right)^{1/n}. \qquad (7)$$

f_s depends on the intensity of segregation k, the volume of segregated zone x, and more significantly on the stress exponent n.

We can numerically evaluate f_s for various flow processes assuming values for k and x. To estimate k, we can divide the volume into two zones, one containing pores and the other pore-free. If the volume of the pore-segregated region is x, then the effective porosity there is enhanced by $1/x$. If we assume x to be the volume of the mantle surrounding the particles (which contain the pores in their boundaries), then

$$k = \frac{1}{x} = \frac{1}{1-(1-t/d)^3}$$

where t is the thickness of the mantle, and d is the particle diameter. Assuming t/d to vary from 0.1 to 0.5, k is evaluated to lie between 4.3 and 1. The actual values of k would depend on porosity, pore shape as well as the particle size and shape. For spherical powders and spherical voids, k is unity when the number of pores per particle is less than one.

Some interesting results emerge when the functional dependence of f_s, k and θ are plotted (Figs. 2 and 3). These show that for $n = 1$, there is no pressure intensification, while for higher n values pressure intensification exists. This in physical terms, would mean that diffusional creep processes which have unit stress exponent are unaffected by segregation. In this case the increased flow rates from pore-segregated zones is exactly compensated by the subdued rates from pore-depleted regions. For dislocation creep ($n \sim 4$) or for particle sliding by boundary deformation ($n \sim 2$), the pore-closure rate is enhanced by segregation and this increases with increasing k and θ. For flow rates with n less than unity, as in the case of inertial flow, pore segregation actually decreases the flow rate.

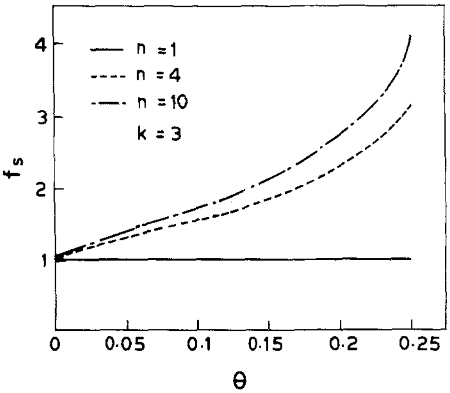


Figure 2 Variation of pressure intensification factor f_S with porosity θ for segregated pores.

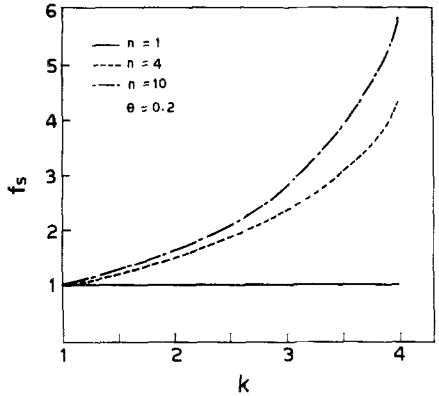


Figure 3 Variation of pressure intensification factor with segregation index *k*.

There are not many experimental results available in the literature to compare the predictions of the above analysis. A rather indirect verification can, however, be made from a recent observation be Beere [9]. He found the pressure intensification during hot-pressing of Cu containing 500 Å Al_2O_3 particles to be high, ranging between 1 and 16 for porosites 0.1 to 0.5. These results according to Beere were significantly higher than that for alumina [10, 11] where the pressure intensification ranged from 1.2 to 2. We believe this difference is due to power-law creep being more active in metallic systems with dispersed particles than in a single-phase ceramic. The fine-grained aluminium oxide, for instance, is not expected to get into the power-law creep region at the temperatures and pressures used, and it is more likely that the diffusional creep was the predominant operating mechanism giving rise to a low pressure intensification factor.

It is of interest to speculate on the applicability of the above analysis for evaluating optimum process parameters during hot isostatic pressing. This is somewhat difficult, as all the flow mechanisms operate more or less together during hot-pressing. In spite of this limitation we believe that this analysis is useful for prescribing optimum operational conditions for hot isostatic pressing. The concept of pressure intensification could be useful in assessing whether recrystallization near the particle boundaries or particle sliding could be promoted during hot-pressing. By promoting recrystallization or even sliding, it is possible to reduce the effect of prior particle boundaries which exist as thin continuous films around the particles in the microstructure. Boundary migration or sliding could possibly disperse or break the deleterious necklace-like structure of the second phase around the particles. As these boundaries are known to be responsible for poor, low, cycle-fatigue strength of sintered products it would be useful to evaluate the conditions necessary for operating dislocation-creep or boundary-sliding mechanisms during hot isostatic pressing.

5. Conclusions

(1) Suggestions made in earlier studies to include a distinct pressure intensification in most of the models for pressure sintering appears to be incorrect since they already have these factors implicit in them.

(2) All the pressure sintering models are for randomly distributed pores, and when pores are segregated as they usually are, these models have to be modified to take into account the segregation.

(3) Segregation of pores does not alter the diffusional flow processes but significantly enhances dislocation creep and grain-boundary sliding.

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