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IMPROVEMENT OF THE WEIBULL MODULUS OF CERAMICS PRESSED WITH A VIBRATORY ASSISTANCE

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Résumé - Les défauts de compaction introduits lors du pressage des poudres interviennent fréquemment comme microfissures initiant la rupture fragile des céramiques. Une assistance vibratoire à la compaction réduit la dispersion de taille et limite la sévérité des défauts, ce qui entraîne une augmentation du module de Weibull. L'étude du pressage de différentes poudres céramiques montre que c'est sur le réarrangement initial des agglomérats que se manifeste l'effet des vibrations.

Abstract - The defects of compaction induced during the pressing of powders frequently act as microcracks which initiate the brittle fracture of ceramics. A vibratory assistance to the compaction reduces the scattering of size and limits the severity of flaws, which leads to an increase of the Weibull modulus. The study of the compaction of different ceramic powders shows that the vibrations mainly influence the initial rearrangement of agglomerates.

I - INTRODUCTION

The basic equation of linear fracture mechanics relates the strength of a piece ($\sigma_f$) to the toughness of the material ($K_C$) and the "equivalent" size of the critical "microcrack" ($a_c$):

$$\sigma_f \sim \frac{K_C}{Y a_c^{1/2}}$$

(where $Y$ is a dimensionless constant which depends on the geometry of the loading and on the microcrack configuration).

Many efforts have been made to increase the toughness of ceramics, in developing new materials: silicon nitride, silicon carbide, partially stabilized zirconia etc. However, such efforts would be useless if they were not accompanied by a correlative improvement of the processes to limit, or even to decrease, the $a_c$ value.

For ceramics processed in the usual way (powders → forming → thermal treatments and sintering) there is a general agreement on the importance of the forming stages on the occurrence of microcracks. For instance gross voids can be introduced by large powder agglomerates, and circumferential cracks can be induced by differential sintering in inhomogeneously compacted bodies /1/. A bad control of $a_c$ does not allow one to control the scattering of strengths in a batch of identical pieces, i.e. it leads to a low Weibull modulus: this is the main problem for the development of engineering ceramics, because most potential users are more afraid of such a scattering than of the value of the mean strength in itself. This study is thus devoted to a technique - the vibratory assistance to the pressing - which noticeably improves the Weibull modulus of sintered ceramics.
II - EXPERIMENTAL

Fig. 1 shows the schematic of the experimental equipment. The pressing of powders is performed at R.T. (20°C) in a floating cylindrical die, where the inferior punch is mechanically coupled with one or another of two devices, able to generate acoustic waves in the ultrasonic (20 kHz) or in the sonic (50 Hz) range. The amplitudes of the vibrations of the punch are 50 μm at low frequencies, and can vary from 10 to 40 μm at high frequencies.

The samples are disc-shaped, 30 mm in diameter and 4 mm in thickness in the aspressed state, 25 mm in diameter and 3 mm in thickness (depending on shrinkage) in the sintered state. Various ceramic powders are studied (alumina, silicon carbide, SiAlONS, tungsten carbide); however, all are in the micron or the submicron range (e.g. A 16 SG Alcoa or RC 172 DBM Reynolds alumina, with a mean particle size of 0.6 μm). Various organic binders are used, and all the powders are agglomerated into free-flowing granules (e.g. by spray-drying, or by screening through sieves) before pressing.

The sintering can be performed in a laboratory furnace (electric heating, low thermal inertia) or in industrial furnaces. The latter case is chosen for SiAlONS.

II - RESULTS AND DISCUSSION

Neither the high frequency nor the low frequency vibrations increase the green densities of compacts by more than ± 0.5 to 1 % (green densities depending on the nature of the powders and on the maximum pressing pressure, but always remaining between 56 and 60 %). This shows the vibratory assistance does not noticeably improve the pressing efficiency. However, it can improve the mechanical properties of sintered samples, by reducing the microcrack severity.

The samples are broken by using a biaxial flexure technique, and the strength data are analyzed in accordance with the usual two-parameter Weibull probability function:

\[ F = 1 - \exp \left( - \left( \frac{\sigma_f}{\sigma_o} \right)^m \right) \]

where "m" is the Weibull modulus. The LnLn (1/F) vs Ln σ_f diagrams are drawn by using at least 30 samples, to obtain sufficient accuracy.

Fig. 2 and 3 show the results for alumina and SiAlON materials, pressed without and with ultrasonic assistance (US). The US is clearly beneficial, because it leads to an increase of "m" from 6.5 and 6 (without US) to 15 and 14 (with US). The highest strength values are not sensibly increased, contrary to the lowest ones, which agrees with the previous statement that the US assistance results in a decrease of the "equivalent" size of the largest flaws.

Fig. 4 shows the compaction response of alumina powders without and with US. Without US, the \( \frac{\rho}{\rho_o} \) vs Ln P diagram exhibits its usual features: two linear domains, with a slope variation which defines the yield point. The low pressure domain is usually attributed to the particle sliding and the agglomerate rearrangement, whereas the yield point is the beginning of the particle deformation and the agglomerate fracture, such a yield point being a qualitative index of the fracture stress of agglomerates. With US, the density under low pressures (< 0.1 MPa) is strongly increased (by ~ 20 %), which means the US assistance is "equivalent" to an increase of pressure of about one order of magnitude. Besides, the yield point is not so well defined as it is without US. However, in the high pressure field (> 20 MPa), the density is only slightly improved by the US (0.5 to 1 %, as previously noticed). Hence, the ultrasonic assistance appears to improve the particle sliding and/or the agglomerate rearrangement, and to lead to a more homogeneous state with a better packing, which is shown by the higher density. Beyond the yield point the increasing pressure results in a progressive elastic deformation of particles and a breaking of the softest agglomerates, but it does not seem to modify the main features of the...
packing. That means even a high pressure cannot heal some "vault effects" around loosely packed hard aggregates. Thus the largest microcracks appear to be induced by the initial stage of the pressing. This is confirmed by tests where the US is applied in different ranges of pressure. For a low pressure US (point A in Fig. 4, and curve A in Fig. 5) the Weibull modulus is \( \approx 16 \), whereas for a high pressure US (point B in Fig. 4 and curve B in Fig. 5) it is \( \approx 10 \) only. All powders lead to similar results, i.e. the US has a beneficial influence on the "m" value (with a typical increase from \( = 8-10 \) to \( = 12-16 \)) only if applied at pressures below the yield point. The highest values of "m" here found (\( \approx 16 \)) may seem rather low in comparison with certain values given in the literature. However, we indeed found \( m < 16 \) in most of the tests we performed on some commercially available ceramics, advertised to have a high Weibull modulus. Finally, it must be underlined that the specimens were broken in their as-sintered state, without any surface machining, and that a polishing should lead to higher values for both \( \sigma_f \) and "m".

As far as the amplitudes of vibrations are concerned, the lowest amplitude (\( \approx 10 \mu m \)) always leads to better results than the highest one (\( \approx 40 \mu m \)). Working on the low frequency (50 Hz) device, at a vibrational amplitude of \( \approx 50 \mu m \), leads to similar results as the 40 \( \mu m / 20 \text{ kHz} \) equipment, which does not make it possible to judge a possible influence of the frequency.

### III - CONCLUSION

This study points out the importance of the low pressure stage in the pressing of ceramic powders and shows that a bad initial rearrangement of agglomerates leads to flaws - probably gross voids as the one shown in Fig. 6, which corresponds to the fracture surface of an alumina sample pressed without the US - which cannot be eliminated by the subsequent high pressure stage. It also demonstrates that a noticeable improvement can be obtained with the help of ultrasonic assistance.

### IV - ACKNOWLEDGEMENTS

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### V - REFERENCES


Fig. 1  Schematic of the devices for vibration assistance

Fig. 2  Weibull's diagram for A16SG Alcoa alumina, pressed (1) without US, (2) with US

Fig. 3  Weibull's diagram for Ceraver SiAlON, pressed (1) without US, (2) with US

Fig. 4  Effect of US on compaction of RC172IIBM Reynolds alumina powders: (1) without US, (2) with US
Fig. 5 Weibull's diagram for RC 172 DBM pressed with US: (1) US applied below the yield point (A, in Fig. 4); (2) US applied beyond the yield point (B, in Fig. 4).

Fig. 6 Fracture initiating void in alumina pressed without US.