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Powder compaction with ultrasonic assistance

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The compaction behaviour of a ceramic powder can be improved by ultrasonic assistance, only when ultrasounds are used at pressures lower than a critical value, P_c. This critical pressure is connected with the limit of mobility of the powder grains under ultrasonic vibrations. Its value depends on the characteristics of the powder, as well as those of the ultrasounds: frequency, amplitude, time of application, etc.

1. Introduction

The properties of ceramic bodies are known to be highly dependent on their processes of fabrication, especially before firing. In this respect, ultrasonic vibrations have been used to assist the pressure during the powder compaction stage in order to increase the density [1–4], the Weibull modulus [5], or to decrease the force of compaction [2, 6, 7]. The best conditions for ultrasound application, however, have not been defined so far. The aim of this work was to study the intrinsic behaviour of a SiYAlON powder when compacted under ultrasonic assistance.

A Wolpert Machine, especially equipped, was used in which the compression set includes an upper piston on which the pressure is applied, a floating die containing the powder (1 g) and a lower pressing tool connected to the ultrasonic circuit. This circuit is composed of a generator (20 kHz), a transducer and boosters for selecting the vibration amplitude (up to 40 μm). The ultrasonic pulse is transmitted by a device licensed [8] by Legrand Company (Limoges, France), which prevents the transducer from contacting the pressing force. During compaction, the applied pressure can be recorded simultaneously with the movement of the upper piston or the thickness of the pellet from which its density is calculated, taking into account the diameter of the cylindrical die (15 mm).

2. Powder behaviour on compaction

2.1. Without ultrasound (US)

Samples of powdered SiYAlON (1 g) commercialized by "Céramiques et Composites" (Tarbes, France) were first compacted without ultrasonic assistance in a classical way.

Our results are shown in Fig. 1 where the heights, H (or thicknesses), of the compacts are plotted against compacting pressure, P (MPa). The results are well described, as shown in Fig. 2, by the following equation according to Balshin [9]

$$\log P = -C_1 V/V_0 + C_2 \qquad (1)$$

or

$$1/d = C_3 - C_4 \log P \qquad (2)$$

where C_1, C_2, C_3 and C_4 represent constants, V_0 and V the volume of the powdered sample before and after compaction, respectively, and d the density obtained under a pressure, P.

2.2. With ultrasonic assistance

When an ultrasonic pulse is now applied under a given load (frequency 20 kHz, amplitude = 20 μm, time = 1 sec) this leads to a surprising pressure drop equal to the compacting pressure which is thus brought back to zero, as shown in Fig. 3. Above a critical pressure, P_c, a reduction in the pressure drop is observed, followed by a slight decrease. Such a phenomenon appears to be highly dependent on the US vibration amplitude and the time of application, as can be seen in Fig. 3.

It is worth noting that a temperature rise in the sample (up to 70° C) occurs, which is roughly proportional to the magnitude of the pressure drop. This obviously results from the friction of the moving powder particles along the line of their rearrangement when applying ultrasound.

While pressure is now brought back to its initial compacting value, a reduction in the height of the compact which is equivalent to an increase in density can be noticed. The resulting density has been plotted against pressure in Fig. 2, where each spot, in coordinates ($1/d$, $\log P$) according to Equation 2, represents a powdered sample compacted at a given pressure in the case of an ultrasonic pulse of frequency 20 kHz, amplitude 20 μm, time 1 sec. This figure shows a break in the compaction behaviour of the powder on both sides of a critical pressure, $P_c = 33.7$ MPa. For pressures lower than P_c, powder compaction is improved when an ultrasonic vibration is applied. At higher pressures, on the contrary, ultrasound has really no effect on the green density.

These observations can be illustrated in Fig. 4 which represents the density of the compacted powder at any pressure in comparison with that obtained without ultrasonic assistance. It clearly appears that the extra density due to ultrasound increases with pressure up to a maximum of 6% at the critical pressure, P_c. But

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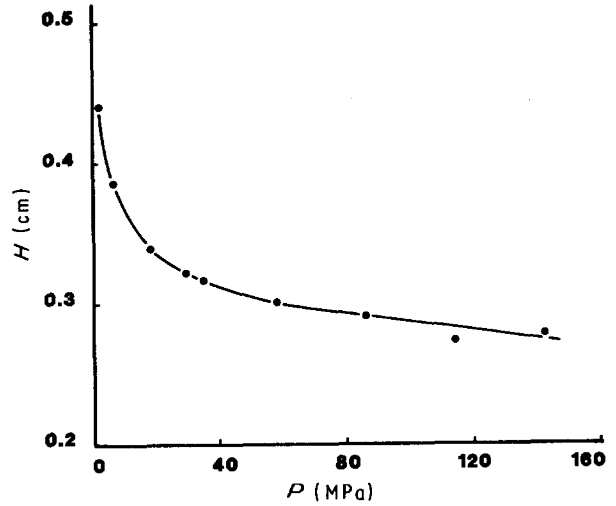

Figure 1 Thickness of the samples as a function of compaction pressure for 1 g SiYAlON.

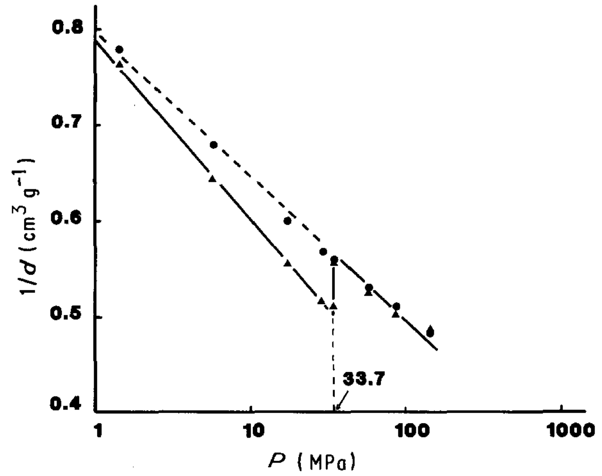

Figure 2 Compaction behaviour of a powdered SiYAlON (Δ) with and (+) without ultrasonic assistance. US was applied at each pressure under the same conditions: $f = 20\,\text{kHz}$, $A = 20\,\mu\text{m}$, $t = 1\,\text{sec}$.

for pressures above P_c, there is only a slight density increase and it is then no use applying ultrasound in such conditions. A previous knowledge of the intrinsic curve of ultrasonic assistance (Fig. 2), and of the critical pressure, P_c, is thus very important for all practical purposes.

3. Influence of the ultrasonic characteristics

As mentioned above, the critical pressure, P_c, is dependent on the characteristics of the ultrasound. Here we discuss in detail the influence of the ultrasonic vibration amplitude and time of application.

3.1. Amplitude

The influence of amplitude was tested by applying ultrasound at a given frequency (20 kHz) for the same time of application (1 sec). At each amplitude, we studied the evolution of the resulting density as a function of pressure where ultrasound is applied (cf. Fig. 2) in order to determine the critical value, P_c. These values have been plotted against the US amplitude in Fig. 5.

It can be seen that P_c increases linearly with amplitude up to $20\,\mu\text{m}$. Then a sudden decrease in P_c is observed before stabilization of the critical pressure around 10 MPa for amplitudes as high as $40\,\mu\text{m}$. As a consequence, it is better to use an ultrasonic vibration with an amplitude close to $20\,\mu\text{m}$ to enlarge the useful pressure field of the ultrasonic assistance.

Let us suppose, for example, that ultrasound is applied for 1 sec under a compacting pressure of 13.8 MPa. It can be expected from results given in Section 2.2 that the extra density due to ultrasound will be very small for values of $P_c < 13.8\,\text{MPa}$. Therefore, the ultrasonic amplitude must be contained between 8 and $25\,\mu\text{m}$, as deduced from Fig. 5.

These predictions are confirmed by the results of Fig. 6 where the relative density obtained after compaction has been plotted against US amplitudes. Indeed, ultrasound appears to be only efficient with amplitudes in the range 8 to $25\,\mu\text{m}$: for higher values, the green density is about the same as that obtained from powder compaction without ultrasonic assistance. Here, the best that could be expected would need a higher pressure of compaction near 30 MPa and, consequently, an ultrasonic amplitude very close to $20\,\mu\text{m}$ to keep the critical pressure over 30 MPa.

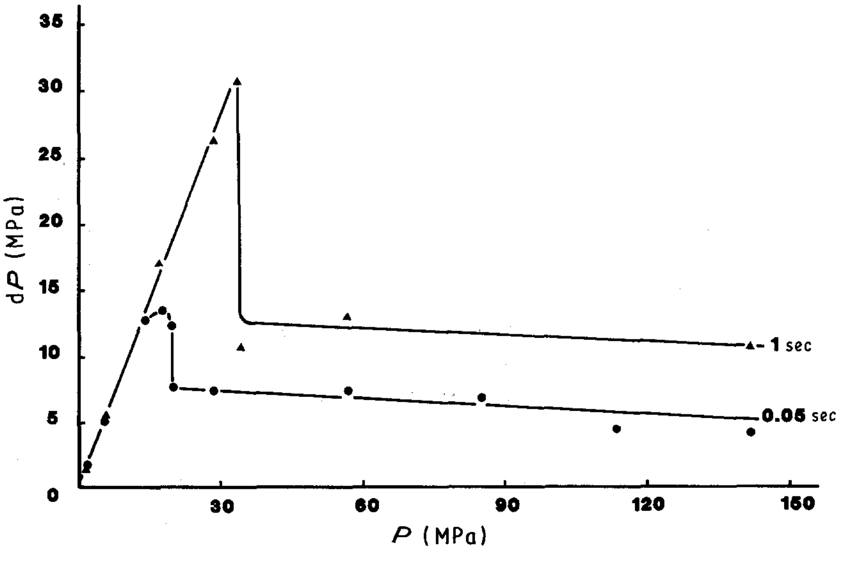

Figure 3 Pressure drop due to US application at various compacting pressures. (x) $f = 20\,\text{kHz}$, $A = 20\,\mu\text{m}$, $t = 1\,\text{sec}$. (Δ) $f = 20\,\text{kHz}$, $A = 20\,\mu\text{m}$, $t = 0.05\,\text{sec}$.

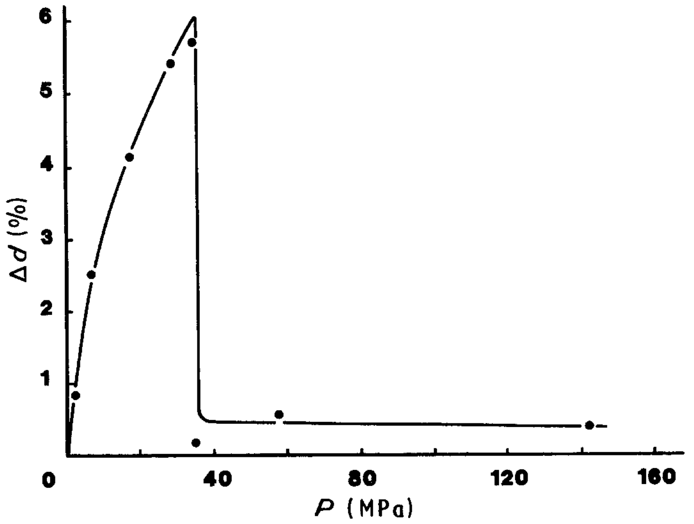

Figure 4 Extra density (%) due to ultrasonic assistance ($f = 20\,\text{kHz}$, $A = 20\,\mu\text{m}$, $t = 1\,\text{sec}$) as a function of compaction pressure.

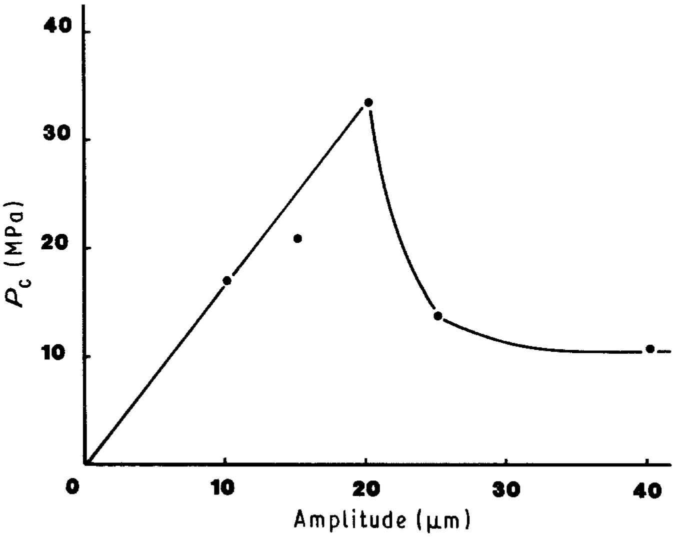

Figure 5 Relationship between the critical pressure, P_c, and the amplitude of the ultrasonic vibration, with $f = 20\,\text{kHz}$ and $t = 1\,\text{sec}$.

3.2. Time of US application

The influence of time was considered keeping constant both values of US frequency (20 kHz) and amplitude (20 μm), the critical pressure, P_c, being determined for each application time by drawing the intrinsic graph ($1/d$, $\log P$) as before.

Our results, shown in Fig. 7, indicate that P_c increases with time, the influence of which being particularly sensitive for application times below 1 sec. These data obey an exponential law which can be written as follows

$$P_c = a \log t + b$$

with $a = 11.1$ and $b = 33.5\,\text{MPa}$.

Now, let us consider the relative density obtained under 27.6 MPa as a function of time of application of ultrasound (Fig. 8). Here again, the efficiency of the ultrasonic assistance is highly dependent on the critical pressure value. For application times for which $P_c < 27.6\,\text{MPa}$, i.e. $t < 0.3\,\text{sec}$ (cf. Fig. 7), the density after compaction remains approximately constant and equal to that obtained from compaction in the absence of ultrasonic assistance. On the other hand, an increase in density is observed for longer pulses for which P_c exceeds the compacting pressure (27.6 MPa). For reasons of stabilization of density with time, however, it is no use applying ultrasound for longer than 1 sec.

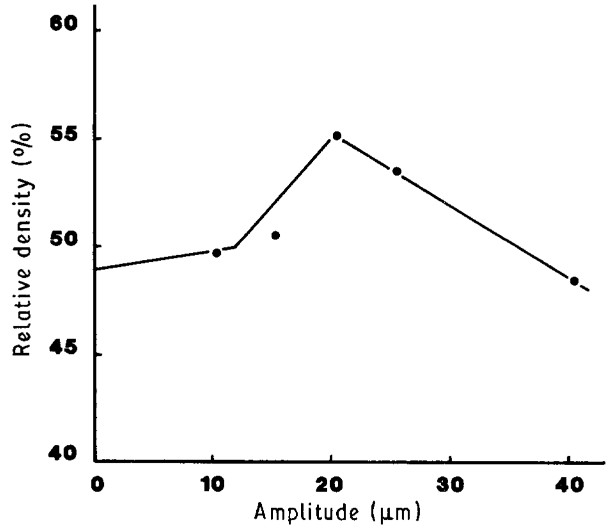

Figure 6 Relative green density after compaction (13.8 MPa) with ultrasonic assistance ($f = 20\,\text{kHz}$, $t = 1\,\text{sec}$) as a function of the US amplitude.

4. Conclusion

The best conditions for ultrasound application for improving the compaction behaviour of a powder have been achieved in this work by investigating the influence of the compacting pressure, as well as that of parameters such as amplitude of the ultrasonic pulse and time of application.

The main result to be kept in mind is the existence

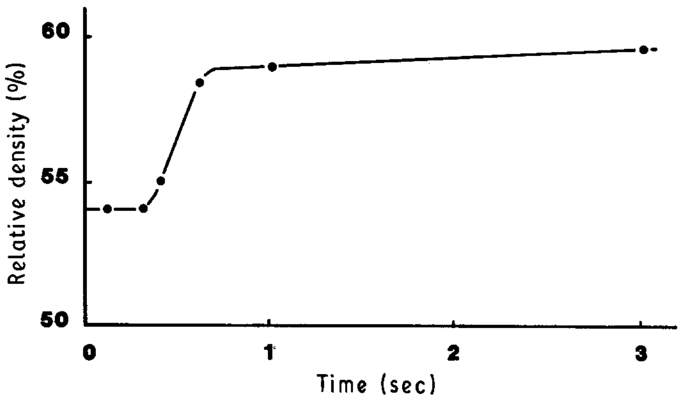

Figure 7 Influence of time of the US pulse ($f = 20\,\text{kHz}$, $A = 20\,\mu\text{m}$) on the critical compacting pressure.

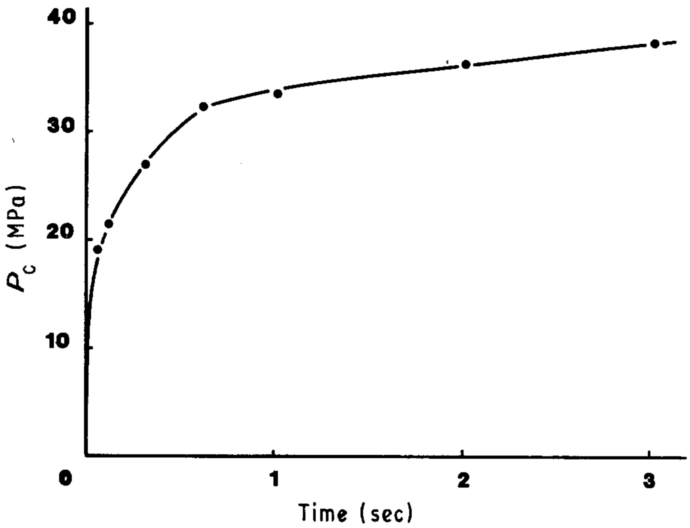

Figure 8 Time dependence of the relative density after compaction (27.6 MPa) with ultrasonic assistance ($f = 20\,\text{kHz}$, $A = 20\,\mu\text{m}$).

of a critical compacting pressure, P_c, below which ultrasound can only be applied efficiently. For compacting pressures above P_c, ultrasonic assistance has no effect on green densities.

This critical pressure, which is related to the limit of mobility of powder particles due to ultrasound, is highly dependent on the US frequency, the vibration amplitude and the time of application. As a consequence, it is essential to draw the intrinsic curves in order to have a previous knowledge of P_c and its relationship with the ultrasonic parameters, before making use of the method in each case and expecting suitable performances.

Taking advantage of these results, we are now endeavouring to determine the effect of ultrasonic assistance on the microstructure of the compacts when ultrasound is used under the best conditions. This will be the topic of a further paper.

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