

Properties and uses of consolidated cork dust

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Cork dust was consolidated without additives by heating under pressure. Four engineering properties of the compacted cork were measured: density, modulus, strength and fracture toughness. The thermal conductivity and the absorption of water by the compacted cork were also measured. The properties depend on the consolidation conditions, and (in a way which is not yet understood) on the origin of the cork dust itself, but not on particle size. The properties are compared with those of other common materials to identify possible uses.

1. Introduction

Cork is light, chemically inert and fire resistant. Its low density ($120\text{--}230\text{ kg m}^{-3}$) makes it a good thermal insulator, and its considerable loss coefficient (0.1–0.3) gives it a high coefficient of friction and the ability to damp vibrations and absorb sound. Its main uses are as stoppers for bottles (exploiting its compressibility and chemical stability) and as wall and floor coverings (because of its attractive appearance and its thermal and acoustic insulating properties), though there are many others [1, 2].

Cork dust is a by-product originated during the various phases of the industrial processing of cork. Its commercial value is insignificant and it is usually burned.

When heated at temperatures close to 200°C , chemical degradation of cork begins [3]. The ability of cork to bond to itself when heated, which is used to obtain the black cork agglomerates, has been known since 1892, but has not, until now, been used to consolidate the cork dust.

2. The consolidation of cork dust

Three grades of cork dust with average particle sizes of $100\ \mu\text{m}$, 1 and 3 mm were studied. The cells in cork can be described as corrugated hexagonal prisms [2, 4, 5], roughly $40\ \mu\text{m}$ high and $25\ \mu\text{m}$ across, so even the finest dust particles contain 20–50 cells.

The cork dust was consolidated in a Struers Prontopress, a small hydraulic press with heating element (Press 1), and in a specially constructed hydraulic press into which shaped moulds could be fitted (Press 2). This second press allowed independent control of temperature and pressure, and gave the most reproducible results. A weighed quantity of cork dust was put in the mould and pressed under conditions within the ranges listed in Table I. An example of the way in which pressure and heating are applied to the cork dust in the mould is shown in Fig. 1. The change in

weight after compaction is negligible. A release agent was sprayed on the mould surfaces before each run to prevent sticking. The best results were obtained at temperatures around 180°C and pressures of 32 MPa, when the consolidated cork dust has a very dark colour with a slightly “waxy” aspect.

3. The structure of consolidated cork dust

Samples of the consolidated cork dust specimens prepared using both presses were examined in the scanning electron microscope (SEM). The specimens were prepared for microscopy in three ways: by fracturing, by cutting using a fresh scalpel blade, and by polishing using wet silicon carbide paper followed by diamond paste. The microstructure could be seen most clearly on the polished surfaces, and there was no indication that polishing artefacts had been introduced. Specimens were sputter-coated with a thin layer of gold before being examined in the microscope to prevent electrical charging.

A typical polished surface is shown in Fig. 2. Some areas are smooth and are composed of tightly folded and compressed cell walls, while elsewhere the cork has retained vestiges of its cellular structure, and the material is not fully densified. One of these regions is

TABLE I Pressing conditions

	Prontopress (Press 1)	Custom-made press (Press 2)
Compressive force	0–4 tonnes	0–20 tonnes
Maximum pressure in specimens	$\approx 55\text{ MPa}$	$\approx 50\text{ MPa}$
Temperature	roughly 180°C	up to 250°C
Heating time	10–30 min	10–30 min
Mould shape	Discs	Discs and rectangular prisms

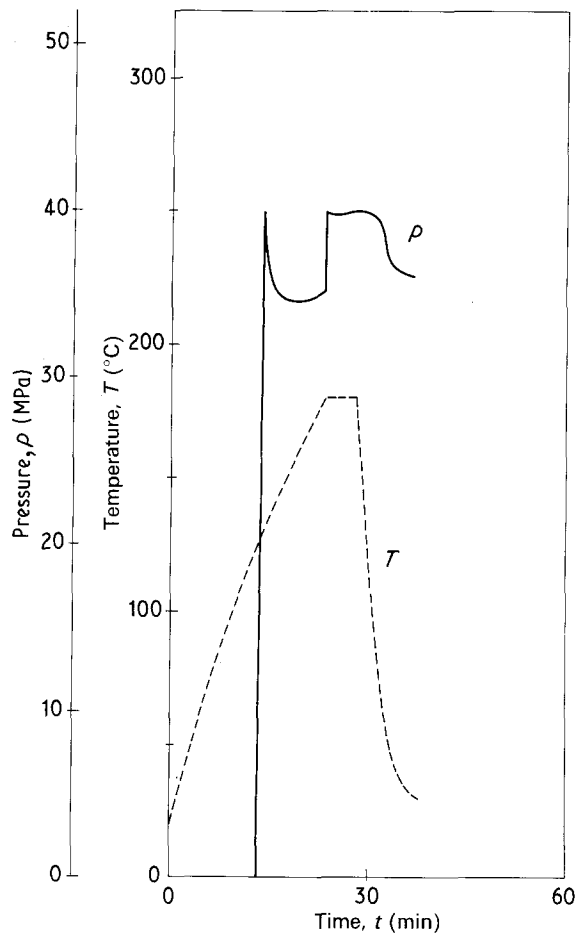


Figure 1 Changes of (—) pressure, p and (---) temperature, T , during the consolidation of cork dust.

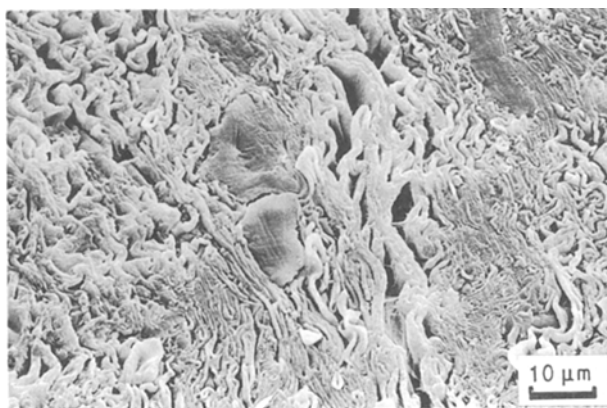


Figure 2 Polished section of consolidated cork dust showing some regions which are better compacted than others.

shown in greater detail in Fig. 3: the folded cell walls can be seen quite clearly.

All the specimens examined contained some cracks, but their nature and distribution varied. The samples made in Press 1 contained many large fissures in the plane of the specimens, normal to the pressing direction. They were mostly concentrated in the central plane of the disks, and there were more of them if shorter heating and compaction times and lower pressures were used. A possible explanation for this is that due to the low thermal conductivity of cork, for the shorter heating and compaction times, the cork



Figure 3 Detail of one of the less well compacted regions.

dust in the middle of the samples tended not to become hot enough to be compacted properly, but the fissures may also result from gases originated from the thermal decomposition of cork that escape during compaction.

The distribution of cracks in the specimens from Press 2 was rather different. A characteristic set (200 °C, 24 MPa) is illustrated in Fig. 4. These cracks are sharp and cut impartially across regions which are both fully densified and less well densified. Concentric cracks are often seen, and seem to be related to the flow patterns of the cork powder in the mould. They are probably shear cracks. The highest temperature and pressure used (200 °C, 48 MPa) resulted in many small cracks concentrated in the surface layers of the specimens, but there was often a central fissure as well.

Cork dust from different sources has been used. The microstructures looked similar, although the surface finishes were different, and some dust had more tendency to stick to the moulds than others. The colour of the compacted cork varies from dark brown to black depending on the moulding temperature.

4. The properties of consolidated cork dust

4.1. Density

The density of the consolidated cork dust samples was found by weighing them and measuring their dimensions (and thus volume) accurately. The measurement errors were less than 3%. The sample-to-sample variation was greater than this.

Fig. 5 shows how the density varies with pressure and temperature, using samples from Press 2. The density rises to a plateau of about 1200 kg m^{-3} at a temperature, T , of 180 °C and a pressure, p , of 32 MPa. Increasing either T or p further did not increase the density.

The density of the cell-wall material of cork is not accurately known, but is likely to be near 1250 kg m^{-3} . This means that the cells in the cork dust particles have collapsed, during compaction, and that the relative density has increased from about 0.1 to about 0.95. Electron micrographs, shown earlier, confirm this.

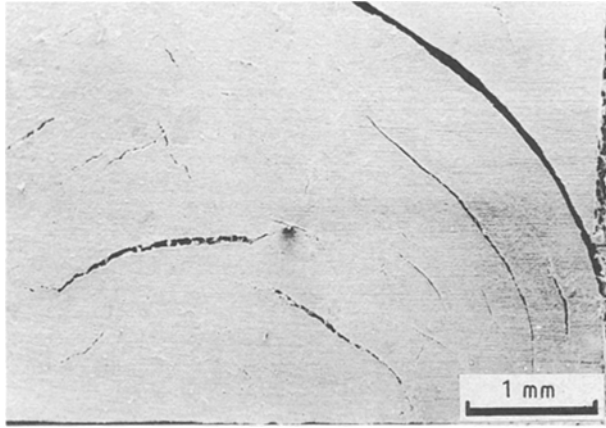


Figure 4 Cracking in a sample from Press 2.

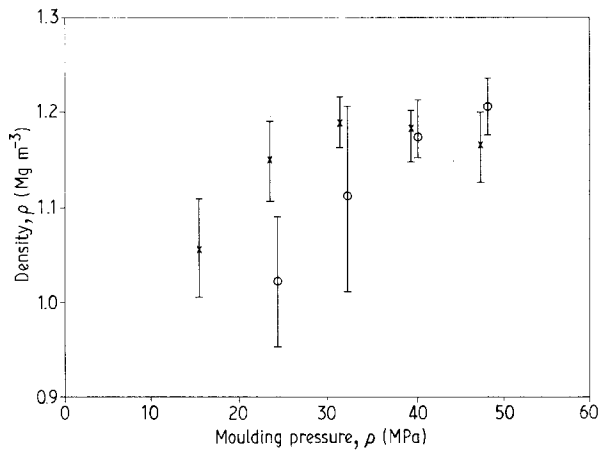


Figure 5 Density, ρ , as a function of consolidation pressure, p , and temperature, T , for a pressing time of 25 min (Press 2): moulding temperature: (○) 180 °C, (×) 200 °C.

4.2. Modulus, strength and fracture toughness

Small beams, about 40 mm × 9 mm × 5 mm (see Fig. 6a), were cut from the consolidated cork samples. They were tested in the three-point bending rig shown in Fig. 6b. A typical load, F ,–displacement, δ , curve, for an unnotched specimen, is shown in Fig. 7. Young's modulus, E , was calculated from

$$E = \frac{L^3}{4bt^3} \left(\frac{F}{\delta} \right)_0 \quad (1)$$

where L is the length of the beam between the outer loading points, b is the width and t the depth of the beam, and $(F/\delta)_0$ is the slope of the linear part of the loading curve. The yield strength, σ_y , was calculated from

$$\sigma_y = \frac{F_y L}{bt^2} \quad (2)$$

where F_y is the load at which the curve deviates from linearity. This was measured in the way shown in Fig. 7 for a 0.2% non-linear strain corresponding to $\delta = L^2 \epsilon / 4t \approx 0.08$ mm.

The fracture toughness, K_{IC} , was calculated from the failure load, F_f , of centre-notched samples (notch

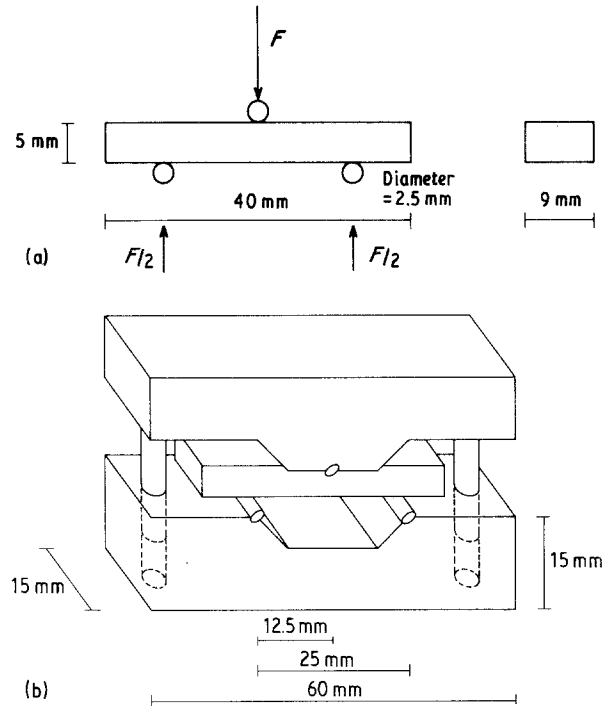


Figure 6 (a) Dimensions of test sample and testing forces. (b) The three-point bend jig.

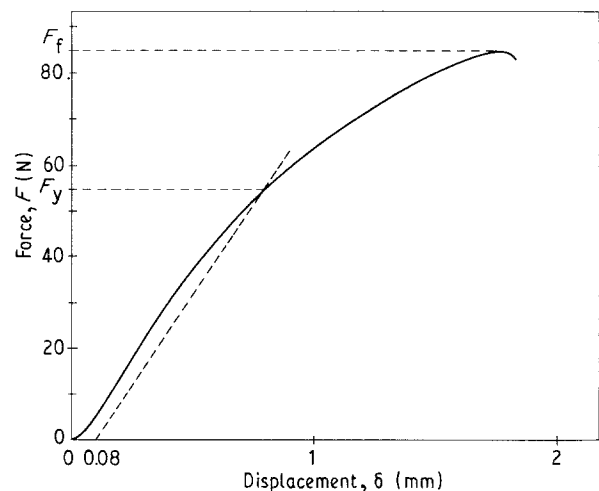


Figure 7 Typical force, F , – displacement, δ , curve in bending. The yielded load is determined in the way shown, for a non-linear deflection of 0.08 mm.

depth $a \approx 0.3t$) using

$$K_{IC} = \frac{3 F_f L}{2 bt^2} (\pi a)^{1/2} \quad (3)$$

The measurement precision for E was $\pm 3\%$, for σ_y was $\pm 7\%$ and for K_{IC} was $\pm 25\%$.

The results are plotted against density in Fig. 8. There is a considerable scatter, particularly in the results from Press 1. Thick samples were less well consolidated, and had poorer properties than thin ones, probably because heat diffusion through the cork powder is very slow. There is no indication that the particle size of the cork dust has any important effect on properties.

Samples prepared in Press 2 had a more uniform appearance, and a narrower range of properties, than those from Press 1.

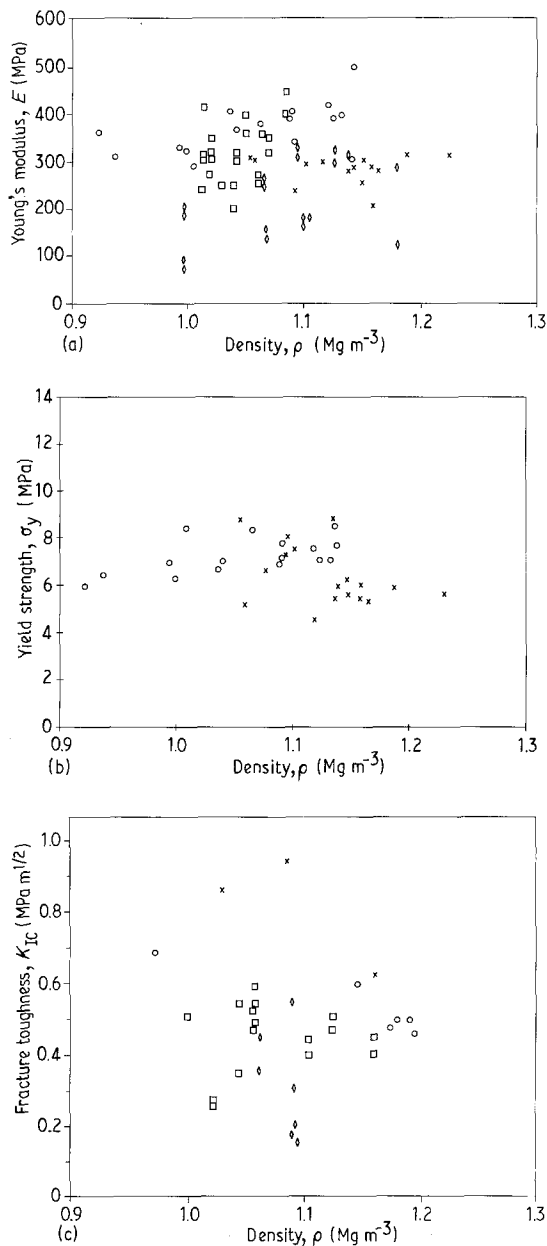


Figure 8 Properties of consolidated cork dust plotted as a function of density. (a) Young's modulus, E . (b) Yield stress, σ_y . (c) Fracture toughness, K_{IC} . Press 1: (\square) Thin specimens, (\diamond) thick specimens; Press 2: moulded at (\circ) 180 °C, (\times) 200 °C.

4.3. Compression behaviour

Cube specimens of consolidated cork powder (edge length of ≈ 14 mm) obtained at a pressure of ~ 45 MPa were tested in compression parallel and perpendicular to the direction of compaction. The strain rate used was $\approx 1.2 \times 10^{-3} \text{ s}^{-1}$. The stress-strain curves obtained are shown in Fig. 9. There is some anisotropy, the modulus in the direction of compaction (535 ± 55 MPa) being slightly smaller than that in transverse directions (765 ± 25 MPa). Both are larger than the moduli measured in the bending tests.

The compression curves are quite different from those for natural cork (see insert in Fig. 9). The consolidated cork powder has collapsed cells and its stiffness is comparable to that of cork at very large strains, as shown by the dashed line in the insert of Fig. 9 which

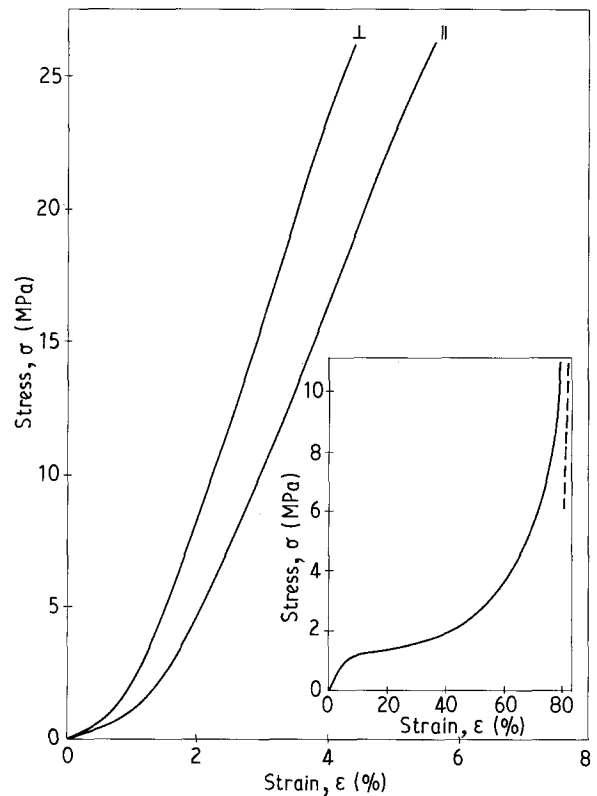


Figure 9 Compression curves of consolidated cork samples: parallel (\parallel) and perpendicular (\perp) to the direction of compaction. The insert shows the compression curve for natural cork.

has a slope equal to the average Young's modulus of the consolidated cork powder in compression.

4.4. Water absorption

The relative mass change, $\Delta M/M_0$, of consolidated cork powder samples immersed in water at room temperature is plotted as a function of time in Fig. 10 (full curve CC). ΔM is the mass increase and M_0 is the initial mass of the sample. The curve is an average curve for four samples prepared in Press 2 at 180 °C, 24 MPa. The scatter was $\approx 10\%$ of the average value. To facilitate comparison, the water absorption curve for a natural cork of good quality is also shown (full curve NC). The absorption curves were obtained from specimens of comparable dimensions (≈ 20 mm \times 10 mm \times 5 mm). At saturation, the relative mass change, $\Delta M/M_0$, of the consolidated cork powder is about 12%. A comparison of values of ΔM (to eliminate the effect of M_0) for a given time (dashed curves), shows that the compacted cork powder absorbs less water than natural cork, probably because of a change in the chemistry of the cork itself caused by heating.

4.5. Thermal conductivity

Lee's disc method was used for determining the thermal conductivity of the consolidated cork powder using plate samples ≈ 1 mm thick. The average value obtained is about $0.04 \text{ W m}^{-1} \text{ K}^{-1}$, while the value for natural cork is about $0.045 \text{ W m}^{-1} \text{ K}^{-1}$.

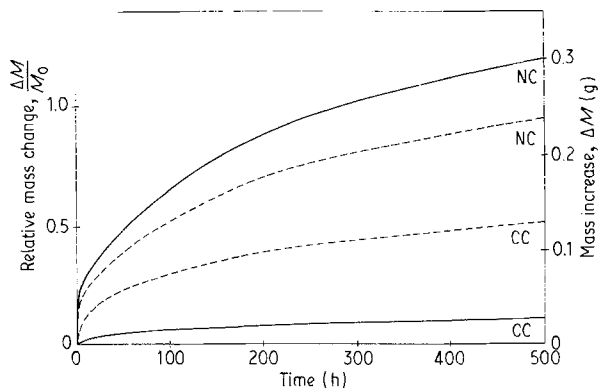


Figure 10 Water absorption curves for compacted cork (CC) and for natural cork (NC): (—) $\Delta M/M_0$; (---): ΔM .

5. Discussion and applications

Cork is a cellular solid. Its low modulus and low thermal conductivity are a direct result of its low relative density (about 0.1) and geometry of the solid cell walls [2, 6].

Consolidated cork powder has a relative density in the range 0.9–0.98. The density of the cork cell walls can, in principle, be calculated from its chemical composition [7], but in practice this varies, and is not accurately known. The collapse-stress, σ^* , of the cells, i.e. the compressive stress at which they buckle is known [2]: it is roughly 1MPa (cf. compression curve for cork in Fig. 9). If the gas within the cells is initially at atmospheric pressure (0.1 MPa), and the cork dust is consolidated at a temperature T , and a pressure, p , then, if the gas in the cells is compressed by the pressure, the resulting relative density can be calculated from the ideal gas law. In calculating the compression of the gas, we must allow for the presence of a volume fraction, D_0 , of (incompressible) solid. The initial relative density of cork is of course equal to D_0 . A volume, V_0 , of cork contains a volume $(1 - D_0)V_0$ of gas. A pressure, p , at a temperature, T , compresses this to a volume, V_{gas} , given by

$$V_{\text{gas}} = \frac{p_0}{(p - \sigma^*)} \frac{T}{T_0} (1 - D_0) V_0 \quad (4)$$

where p_0 and T_0 are the initial pressure and temperature. Taking $p_0 = 0.1$ MPa, $p = 30$ MPa, $T_0 = 300$ K and $T = 473$ K, equation 4 gives

$$\frac{V_{\text{gas}}}{V_0} = 5.4 \times 10^{-3} (1 - D_0) \quad (5)$$

The volume of the cork after compression is $D_0 V_0 + V_{\text{gas}}$, and its relative density is

$$D = D_0 \left/ \left(D_0 + \frac{V_{\text{gas}}}{V_0} \right) \right. \quad (6)$$

Taking the initial relative density as $D_0 = 0.1$ gives $D = 0.95$.

There are, however, difficulties with this simple argument. The first is that the cells, after consolidation, would contain gas under a pressure of almost 30 MPa, and this should cause re-expansion of cork cells. But could the cork cells survive such an internal

pressure? At the edge of a cork dust particle, as it is compressed, a pressure difference of roughly this magnitude must appear across the cell walls, giving a membrane stress in the wall itself which is larger, by the ratio of the wall thickness to cell size, that is, by the factor $1/D_0$, or about 10. The large pressure gradient could drive the gas, by diffusion, out of the cells; or the membrane stress could rupture them. Experiments were carried out to resolve this question. If a high gas pressure were contained in the cells after compaction, then reheating at 1 atm would cause massive re-expansion of the consolidated cork powder. Samples were reheated at 200 °C for 1 h. There was almost no expansion. We conclude that the gas has escaped from the cells by diffusion.

The ability of the cork dust particles to bond to each other is remarkable. Its origin must be related to the chemistry of cork and to the effect of temperature on the cork components. Thermal degradation of cork starts at temperatures close to 200 °C [3]. The bonding effect may be due to tannins, which are an important cork constituent, but it is not impossible that the hemi-celluloses and waxes also play a role. Preliminary experiments showed that cork dust bonds to pinewood almost as strongly as the cork bonds to itself, and that a mixture of cork dust with ceramic powders can be consolidated under the conditions used for the cork dust alone.

The modulus of the consolidated cork dust is larger, by a factor of about 10, than that of cork itself. But, despite the high relative density, it is still far lower (by another factor of at least 10) than that of the cell walls [2]. The micrographs of the lamellar structure shown in Section 3 suggest that the cell walls, though folded and collapsed, are not necessarily bonded. On loading, the walls can slide or fold further, giving a low stiffness.

Compaction folds the cell walls producing a layered structure, the layers lying normal to the direction of compaction (Fig. 3). Further loading in this direction bends the cell walls, whereas loading in the transverse direction applies axial stresses to them. This, we think, is the reason why the modulus in the compaction direction is lower than that in the direction normal to it.

The strength, too, though larger (by a factor of about 5) than that of cork, is still well below that of the cell walls themselves [2]. Failure is by delamination, giving a fibrous fracture, adding further to the evidence that the cell walls do not bond strongly to each other. The fracture toughness data suggest the same thing.

Cork itself has the attractive properties of low density, high resilience, high thermal resistance, chemical stability, low permeability and fire resistance. Consolidated cork powder retains the last four properties, with improvement in the impermeability to water. But the consolidation process destroys the first two properties. Consolidated cork powder can be painted, machined, cut with a saw and does not fracture when penetrated by a 5 mm diameter nail.

Consolidated cork dust is not so much in competition with natural cork but with woods and polymers

because its density is similar to these materials. Nevertheless, consolidated cork dust does not have as good mechanical properties as the materials it competes with, but because it is so cheap in comparison to these materials, some uses may be found that are financially viable.

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Received 17 July

and accepted 16 December 1991