JOURNAL OF MATERIALS SCIENCE 21 (1986) 4262–4268

Some aspects of shock consolidation of polymeric, PVC–metallic and PVC–silica powder mixtures

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Industrial interest in reinforced plastics, plastic–metallic and plastic–ceramic aggregates has led to a preliminary examination of the effect that shock waves have on the properties of dynamically consolidated powders. Three PVC powders were compacted explosively either in pure double polymeric mixtures, or singly with 304 stainless steel, iron or SiO_2 powder. Pressures generated ranged from 3 to 9 GPa for polymeric mixtures and from 14 to 24 GPa for metallic and silica composites with PVC materials. The effect of the original particle size and density on the final properties of a compound is discussed and optimal conditions of densification and soundness are indicated, together with the potential industrial applications of these composites.

1. Introduction

The effects of the passage of a shock wave through an aggregate of a metallic or ceramic powder have been studied in the last twenty years in considerable detail because of their substantial technological potential. On the chemical side, synthesis of new materials, or desired phase changes, can be effected since preshocking of powders increases generally their reaction rate and shock-induced temperature. Exothermic reaction is often associated with the initiation of synthesis in these conditions, and activation (enhancing chemical reaction) follows. This is particularly noticeable in the case of increased catalytic activity of materials like carbon and rutile, dissolution of silicon nitride, hydration of neodymium oxide, and reactivity of zirconia [1, 2].

On the other hand, the considerable amount of plastic deformation, leading often to comminution of particles and the associated lattice distortion, combined with a general increase in the incidence and rate of propagation of microfaults, results in the storage of energy which can be utilized if sintering is required. In this case much lower pressures and temperatures are needed – by comparison with conventional treatment – and the consolidation of the powdered material is achieved on a very short time scale [3].

These properties of pre-shocked materials are of particular importance when sintering non-oxide ceramics of strong covalent properties, low diffusion rates and considerable intercrystal vacancies. Shock consolidation does also offer the possibility of producing non-sintered, high green density compacts in a variety of materials that will display high levels of flow stress, combined with a satisfactory degree of elasticity and a fair to good structural integrity. This is possible as a result of internal bonding of individual particles produced by high pressures, and purely local temperature effects which are associated with high-velocity intergranular impacts. It is this basic low-level temperature effect that makes the use of shock compaction particularly interesting and useful in the case of polymeric materials and their mixtures, because the degradation of physical and mechanical properties that often follows the application of conventional higher-temperature forming techniques (e.g. injection moulding) is absent. An extensive investigation of the morphology and properties of a selection of explosively compacted PVC powders indicated clearly that the low levels of adiabatic heat produced did not change the basic characteristics of the materials concerned, but created conditions in which improved strength and a high degree of densification could be attained [4–6].

Industrial demand for reinforced plastic materials on the one hand, and the ever growing interest in polymer–metallic (electronic industry) and polymer–ceramic (bearing design) integrally bonded mixtures on the other, have led to the initiation of the investigation into the basic properties and characteristics of explosively compacted powder mixtures. The possibility of increasing the strength of polymeric compounds by densification of two different polymer powders lies behind the compaction of such mixtures. These may possibly be used in preference to directionally pre-orientated (by cold forming) polymer components.

The present paper is concerned with reporting on the explosive compaction technique employed and the resulting effects of shock-consolidation on the mixtures of three PVC-type powdered materials, together with the preliminary results of investigations into the

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TABLE I Characteristics of "as supplied" materials

Material	Average particle size (μm)	Apparent density ($g cm^{-3}$)	Tap density ($g cm^{-3}$)	Theoretical density ($g cm^{-3}$)	Glass transition temperature (°C)	Decomposition temperature (°C)
P67/579	75	0.27	0.53	1.40	82	242
S57/116	160	0.56	0.66	1.40	82	237
S67/111	170	0.54	0.60	1.40	87	287

physical and mechanical properties of PVC–stainless steel, PVC–iron and PVC–silicon dioxide compacts.

2. Experimental materials and techniques

The ICI manufactured PVC powders had the initial properties given in Table I. The glass transition temperature was measured by means of a differential scanning calorimeter.

The initial average grain size of SiO_2 powder was $15\,\mu m$ and its theoretical density $2.32\,g\,cm^{-3}$. The starting sizes of the 304 stainless steel and iron powders were 130 and $140\,\mu m$, respectively. The volume fractions of the polymeric mixtures varied from 3 : 1 to 1 : 3, but those of polymer–metal and polymer–ceramic were limited to 1 : 1.

A direct, implosive compacting method was employed, using the ICI Trimonite No. 3 powdered explosive. The detonation velocity and the associated shock and pressure wave velocities depend on the thickness of the explosive charge, but, on average, would be of the order of $3000\,m\,sec^{-1}$.

The direct compacting system used consists of a cylindrical metal container filled (at tap density) with the powder to be compacted and of an outer plastic, tubular container that provides an annular space of required dimensions to accommodate the charge. Provision is made for the generation and propagation of a plane-wave detonation front along the cylinder.

Depending on the shape of the generated shock wave, the pressure may decrease towards the centre of a cylindrical specimen (hyperbolic wave), remain constant across it (conical wave), or increase in the centre (parabolic wave), and under-correct or overcompaction may result. Fig. 1 provides examples of this showing a soft centre (A), uniform hardness (B) and a central core crack (C). The success of the operation depends on the ability to generate a conical type of shock wave.

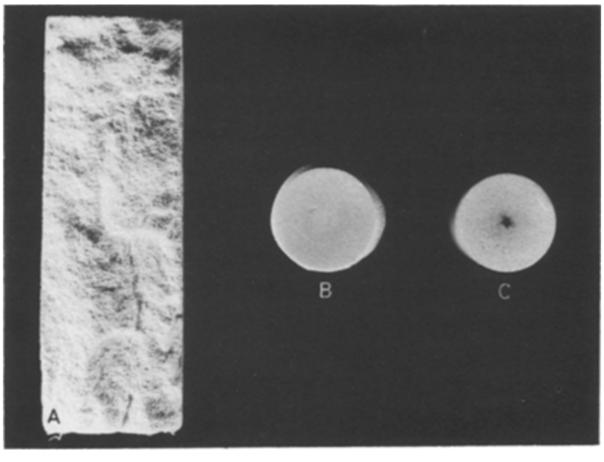

Figure 1 Under-correctly compacted and overcompacted S67/111–S57/116 polymer powder mixtures.

The morphological changes, including the examination of the nature of bonding, were established using SEM.

3. Properties of consolidated powders

3.1. PVC mixtures

Figs 2 to 4 below serve as examples of the morphology developed by shock consolidation with Figs 2 and 3 representing polished, and Fig. 4 fracture surfaces. The nature of bonding achieved is demonstrated by Figs 2, 8 and 9, and the effect that the volume fractions of the constituent materials have on the densification and compressive strength of the compacts is shown in Figs 5 to 7.

Three specific mixtures of S57/116–P67/579, S67/111–P67/579 and S67/111–S56/116 have been selected as being representative of this group of PVC materials.

3.1.1. Morphology

Fig. 2 indicates a considerable reduction in the particle size of both materials, by comparison with the original size (Table I), and the change in shape of both powders. In their original state, the particles of all three powders resembled quite closely the morphology of the uncompacted S57/116 (Fig. 2b), with P67/579 showing a number of small, randomly grouped nodules on the surfaces of its particles. Considerable integration of the latter material has taken place partly as the result of plastic deformation and partly in consequence of comminution of the individual particles. The S57/116 material appears to be less affected and maintains fairly uniform dispersion in the mixture. Interparticle bonding has been achieved and a voidless structure obtained. The individual grains of S57/116 have been reduced, on average, to about $100\,\mu m$ at a pressure of 6 GPa, and those of P67/579 to about $55\,\mu m$.

The effect of undercompaction, illustrated in Fig. 1, is also shown in Fig. 3 taken near the core of the specimen. Whereas clear interface bonding is evident in the upper and side areas, a series of large voids is present in the central portion. Plastic deformation of considerable proportions has altered the shapes of the individual grains in this (1 : 1) mixture, reducing their sizes. Sound compacts of this composite were produced when the compacting pressure was increased from about 4 to 5 GPa.

A fracture surface of the S67/111–S57/116 mixture (Fig. 4) reveals a voidless structure of well mixed and bonded powders. Geometrical similarity of the two materials makes it difficult to distinguish between

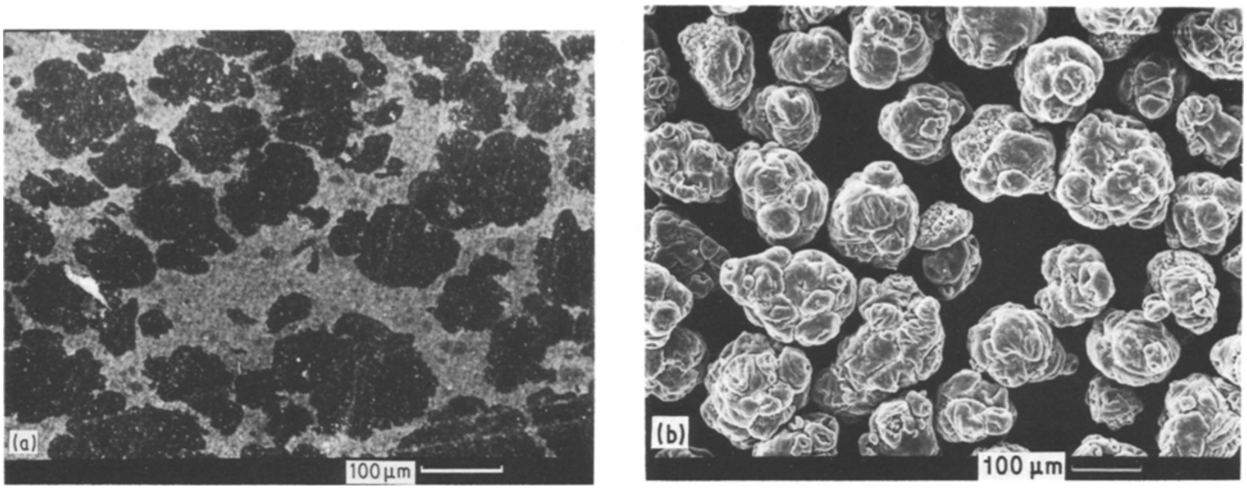

Figure 2 (a) Compacted mixture of S57/116 (dark) and P67/579 polymer powders (1 : 1) mixture; (b) morphology of the original S57/116 polymer powder.

them on a black and white photograph, but the actual SEM examination confirms this statement. Although no fragmentation is observed at a pressure of about 6.5 GPa, the plastic straining has reduced individual grains of both materials to about 120 μm.

3.1.2. Densification and strength

The degree of densification and the propensity to fracture are the two main parameters that decide the usefulness of the material for structural purposes. These basic characteristics are explored in Figs 5 to 7. It is clear from these figures that the post-compacting density is affected by the volume fraction of the constituent materials, as is the compressive stress to fracture. The nature of these relationships depends, however, on the initial properties of the agglomerate.

The mixtures of the P67/579 material with either of the other two S-type powders are characterized by a decrease in both the density and strength with the increasing volume fraction of P67/579 powder. The reason for this is likely to be the smaller particle size and lower strength of the latter (Table I and [5, 6]). The rates of energy absorption and of pressure attenuation increase directly with a decrease in the density of powder [7] and, consequently, a higher initial density of the agglomerate will result in higher green density and final strength. For a constant level of the energy delivered in these experiments, an increase in the proportion of the small-grained low tap density P67/579 material will affect adversely the degree of both the densification and interparticle bonding.

With all three powders having the same theoretical density of 1.40 g cm^{-3}, it is observed that the densities of the respective mixtures reduce from some 97 to 88% and 98.5 and 91.4% of theoretical for S57/116–P67/579 and S67/111–P67/579 compacts, respectively, within the ratio range of 3 : 1 to 1 : 3. The strengths of these compacts reduce correspondingly and equally from some 65 to 35 MPa. The similarity of these figures reflects, of course, the basic physical similarity of the two S-type powders. It is interesting to note that shock-compacted alumina and silicon carbide show the same pattern of behaviour [8, 9].

The properties of the compacts of the two S-type powders (Fig. 8), both of which possess very similar initial properties – with S67/111 showing lower tap density and slightly larger average particle size – are affected to a higher degree by their composition. The addition of S67/111 results in a decrease in the density from 98.5% for a 3 : 1 ratio of S57/116–S67/111 mixture to 89% for the ratio of 1 : 1, but a further increase in the volume fraction of this material restores the (3 : 1) properties at the reverse ratio of 1 : 3. Similarly,

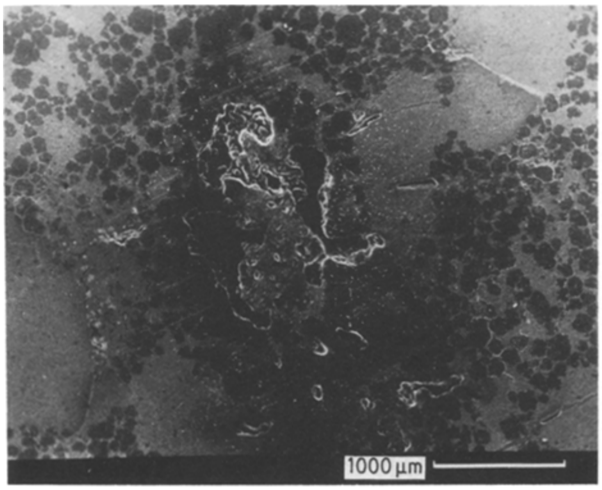

Figure 3 Compacted mixture of S67/111 (dark) and P67/579 polymer powders.

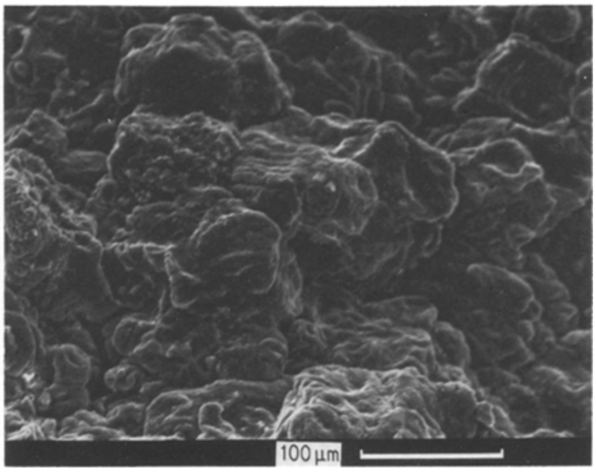

Figure 4 Compacted mixture of S67/111 and S57/116 polymer powders.

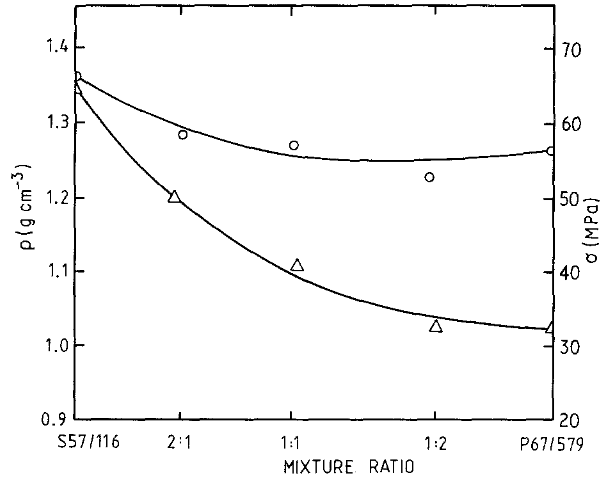

Figure 5 Variation of (△) compressive stress to fracture and (○) green density with the mixture ratio of compacted S57/116 and P67/579 PVC powders.

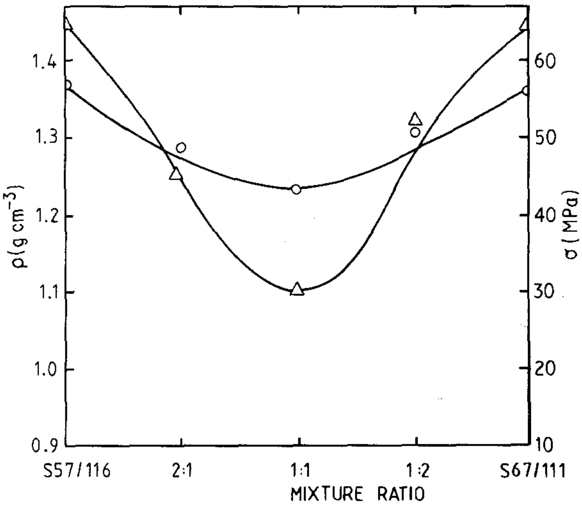

Figure 7 Variation of (△) compressive stress to fracture and (●) green density with the mixture ratio of the compacted S57/111 and S67/111 PVC powders.

the compressive stress to fracture reduces from 65 to 35 MPa and increases again to 65 MPa.

An explanation of this pattern of behaviour is sought again in the disparity in the original tap densities. The slight size advantage of the S67/111 material is offset by the lower initial density, but reasserts itself as the proportion of the powder is increased. These observations are confirmed by the results obtained when compacting the two materials singly [5, 6]. In this case, for a given consolidating pressure, the green density and the strength of S67/111 tend to be slightly lower than those of S57/116 powder.

Irrespective of the material(s) undergoing consolidation, the strength of the compact will depend on the nature and efficiency of interparticle bonding. Unlike the static or isostatic processes, shock compaction produces intimate bonds of sufficient strength to obviate, very often, any need for further sintering operations. Three basic mechanisms operate in these conditions, and all three can be identified at the same time in any given situation.

The passage of a shock wave through a porous agglomerate consists of a series of jumps from particle to particle, and thus creates localized but very specific effects. The impact at various velocities and angles between individual grains, initially separated by varying distances, results in the generation of hot-spots caused by the intergranular friction and is consequently responsible for local friction welding. At the same time, a localized increase in the temperature associated with the detonation of the charge can often produce surface melting of the particles and will result in fusion welding. Jetting – the well known mechanism of explosive welding – can also be present when the right angular and speed conditions of impact are present. The combination of the effects of some, or of all, of these mechanisms will be responsible for the degree of densification, bonding and, ultimately, of the strength of the compact.

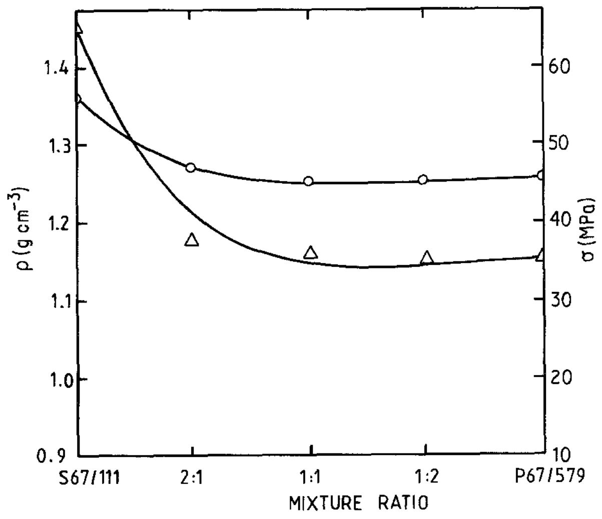

Figure 6 Variation of (△) compressive stress to fracture and (●) green density with the mixture ratio of the compacted S67/111 and P67/579 PVC powders.

The three types of bonding are shown in Figs 8, 9 and 3. Fig. 8 shows localized friction welding, Fig. 9 the large areas of fusion welding, and Fig. 3 the characteristic "explosive welding wave" on the interfaces of three grains (lower left-hand corner).

3.2. PVC–metal mixtures

A limited number of experiments carried out so far permits only a brief outline of the basic properties of these mixtures, but is sufficient to indicate their

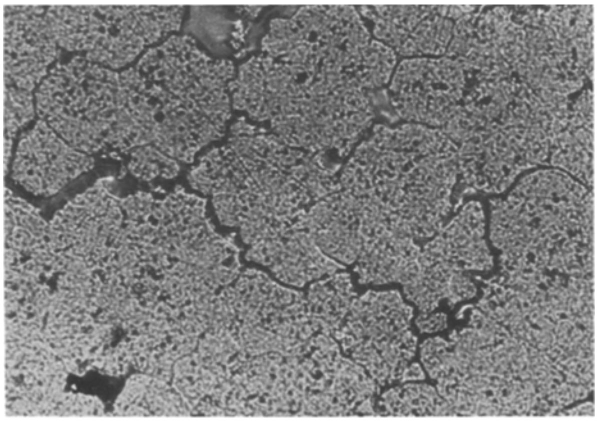

Figure 8 Bonding of individual grains effected by local friction welding × 270.

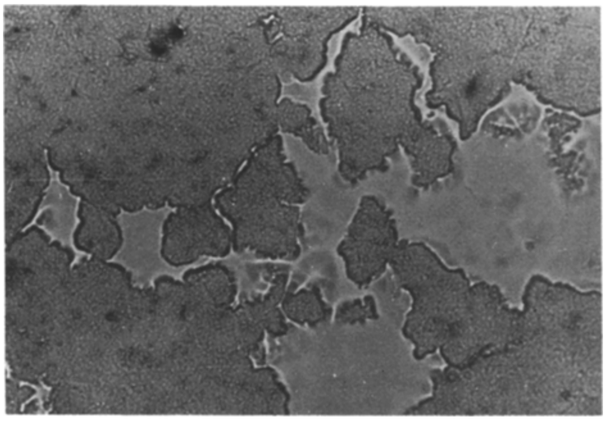

Figure 9 Fusion bonding of compacted polymer particles.

technological potential in the development of new materials for the electronics industry.

The P67/579 material was compacted in mixtures with 304 stainless steel or iron powder. The compaction was carried out at the optimal pressure (for P67/579 powder) of about 6.5 GPa. The morphologies of the consolidated compacts are given in Figs 10 and 11 for the steel and Fig. 12 for the iron mixtures.

3.2.1. PVC P67/579–304 stainless steel

A post-compacted fracture surface of a 1 : 1 mixture (Fig. 10) shows that the particles of both polymer (light areas) and steel are practically halved, having been both plastically deformed and fragmented. The structure is not entirely voidless, but the degree of porosity is small. The originally spheroidal polymer particles are flattened and more flaky as a result of the deformation experienced, and are consequently better suited to densification.

A larger magnification of a polished part of the surface (Fig. 11) indicates good intermeshing of the individual particles of the two materials with possible interface bonding, although the nature of the latter cannot be assessed at this stage.

3.2.2. P67/579–iron powder

A nominally one-to-one, post-compacted polished surface is shown in Fig. 12. A reduction in particle size has been achieved – again at a pressure of about 6.5 GPa – but to a lesser degree than in the case of the steel mixture. The interparticle boundaries are less clearly defined as a result of the polishing and the mixing of the two constituents is less efficient, with large areas of light polymer and relatively small areas of the iron powder showing. Again, intermeshing between the two materials and bonding of the individual grains occurred leading to a voidless and reasonably homogeneous structure.

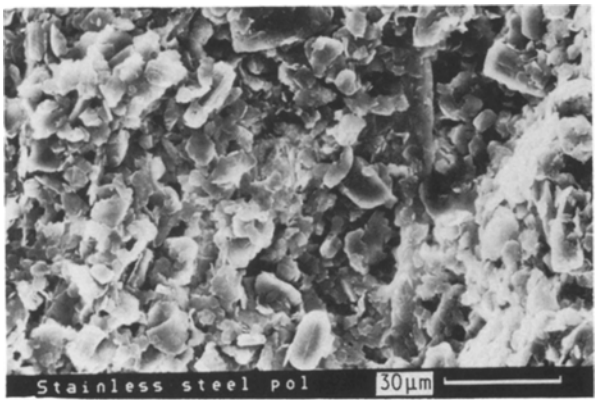

Figure 10 Morphology of the compacted 304 stainless steel and polymer P67/579 (1 : 1 mixture) × 160.

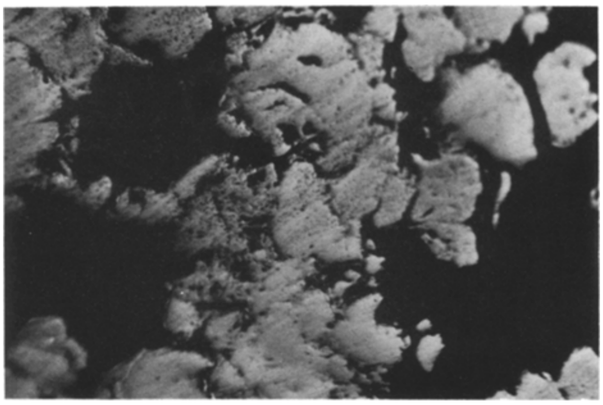

Figure 11 Interparticle boundaries in a compacted 304 stainless steel and P67/579 (1 : 1) mixture.

Both iron–polymer and steel–polymer compacts can be machined and drilled (as shown in Fig. 13) but defective structures having large polymeric inclusions and the resulting incipient cracking will result if the pre-consolidation mixing is not carried out carefully. Fig. 13 affords an example of this problem.

3.3. Compaction of silicon dioxide and P67/579 powders

Experimentation with mixtures of silicon dioxide and PVC-type polymers arose out of an industrial interest in the frictional properties of these agglomerates combined with their good wear resistance.

Incorporation of such composites into bearing design is a logical step, but requires more detailed knowledge of both the likely behaviour of SiO_2 and that of its effect on a polymer powder. Accordingly, experiments with both types of compact were conducted.

3.3.1. Compaction of SiO_2

The relevant information is given in Figs 14 and 15,

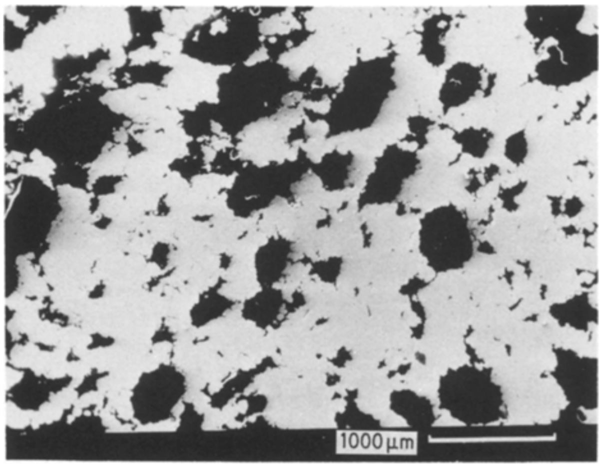

Figure 12 Morphology of compacted iron and P67/579 (1 : 1) powder mixture.

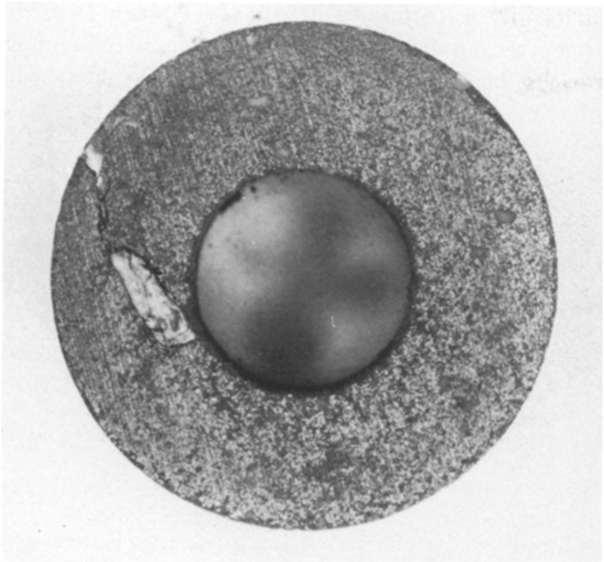

Figure 13 A specimen machined and drilled out of a cylindrical compact of stainless steel and P67/579 mixture.

with the morphology of the original powder shown in Fig. 14a.

A compact of the powder obtained at a pressure of about 22 GPa (Fig. 14b) shows severe cracking associated with both the release wave and, to a lesser extent, fragmentation of the material. The effect of the tensile release wave can be counterbalanced by the material and wall thickness of the metallic container, but the effect of the excessive pressure is counterproductive in terms of densification and strength. These points were clearly demonstrated in the course of explosive compactions of alumina [10, 11] and of single polymers [6].

Fig. 15 indicates that – as is the case with the alumina and polymers – three distinct zones exist when a process parameter is plotted against a material property. In Zone 1, with the increasing pressure, but low detonation velocity of the charge (the velocity of detonation depends on the thickness of the layer of explosive), consolidation of the powder is due simply to a rearrangement of the particles and the resulting lattice distortion is comparable with that produced by isostatic compaction. In the case considered here, Zone 1 extends to a pressure of about 16 GPa. In the following Zone 2, both plastic deformation and fragmentation of the particles take place with the consequent increase in and, ultimately, maximization of the density. This is achieved at a pressure of some 20 GPa. Further increase in the velocity of detonation and, therefore, of the pressure is undesirable because of the destructive effects of the shock and release waves, and also because of the degradation of material properties. The distortion energy generated is comparable with that encountered in the cold-working of metals. These effects are clearly seen in Fig. 15, where pressures in excess of 20 GPa reduce the density to the level already attained at a pressure of 14 GPa. The associated cracking was already indicated in Fig. 14b.

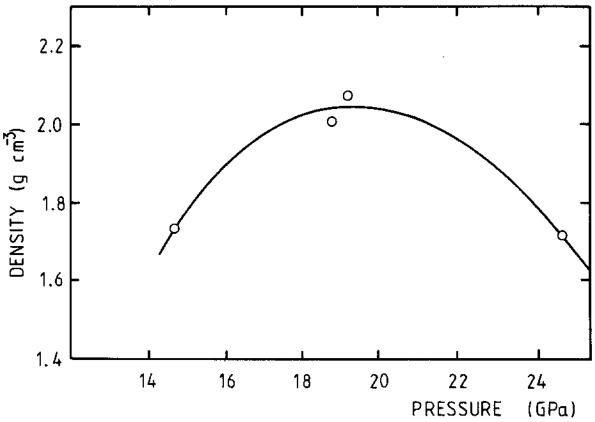

Figure 15 The relationship between the green density and compacting pressure in silicon dioxide compacts.

With the theoretical density of uncompacted SiO_2 being 2.32 g cm^{-3}, the maximum densification attained amounts to some 89%.

3.3.2. Mixtures of SiO_2 and polymers P67/579 and S57/116

One-to-one mixtures of silicon dioxide with each of the two PVC powders were compacted.

Ceramic and P67/579 mixture (Fig. 16) forms an interesting aggregate in which the original disparity in particle size is less apparent, with the polymer having

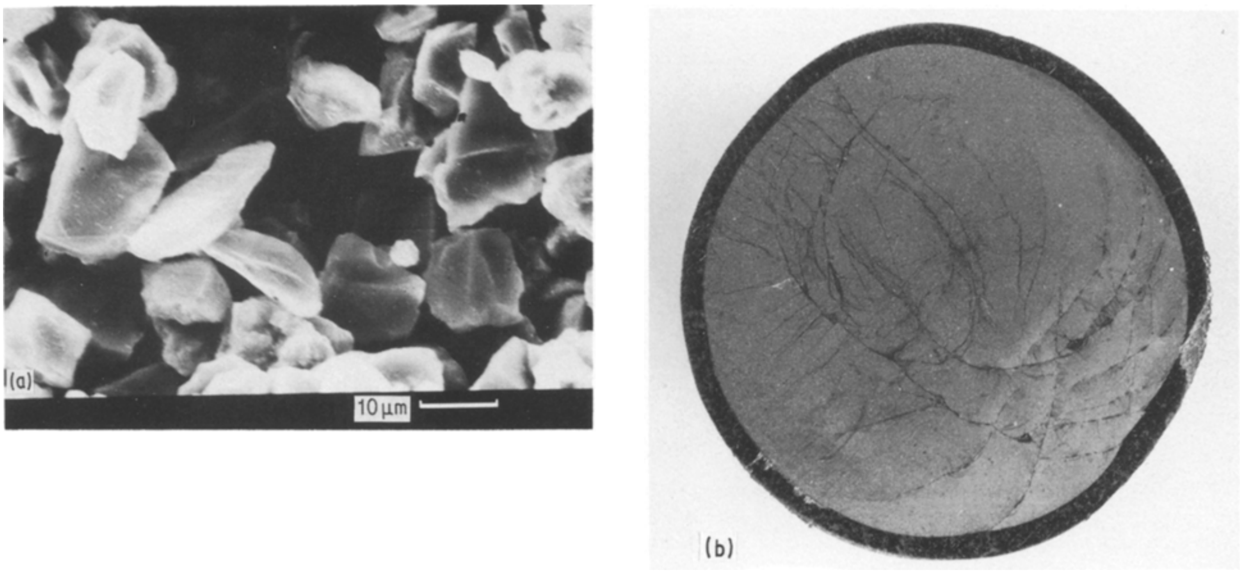

Figure 14 (a) Uncompacted SiO_2 powder; (b) compacted sample of SiO_2 powder showing cracks produced by the release wave.

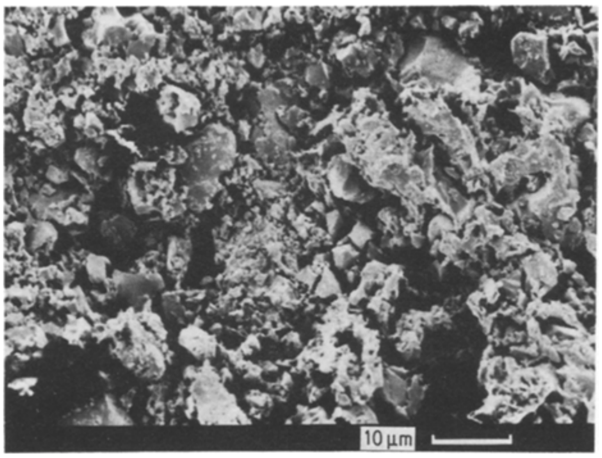

Figure 16 Morphology of the compacted (1 : 1) mixture of SiO_2 and P67/579 powder at a low compacting pressure.

undergone comminution and the silica having been both deformed and fragmented. Fig. 16 corresponds to the beginning of Zone 2 and shows the presence, at this stage, of porosity. Cohesive structures showing interparticle bonding have been obtained when operating nearer the optimal pressures.

Fracture surfaces of SiO_2 and S57/116 (not shown here) also indicate a good degrees of adhesion and of fragmentation of the silica.

In terms of green density, both compacts attain the same maximum level of $1.8\,g\,cm^{-3}$ or 89% of theoretical. Machining of the specimens to bush shapes in the "green" state does not involve any difficulties, but the wear and frictional properties are still under investigation.

4. Conclusions

Shock consolidation of mixtures of either polymeric or polymer–metal/ceramic powders affords means of producing new composite materials of well-defined and well-bonded structures which are of potential industrial interest.

This preliminary investigation of problems involved in the manufacture of such compacts has produced sufficiently encouraging results to warrant further in-depth examination of the properties of polymeric powder mixtures, with particular stress on silica-type additives.

Acknowledgement

The authors wish to thank the Polymer Directorate of the SERC for their financial support.

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Received 17 January
and accepted 14 March 1986