

# On the fabrication of ceramic windings

J. K. WRIGHT, R. M. THOMSON, J. R. G. EVANS

*Department of Materials Technology, Brunel University, Uxbridge, Middlesex, UK*

Components in the form of springs and windings were fabricated from technical ceramics by the extrusion of a polymer-ceramic suspension. Control of thermomechanical properties of the suspension allowed the ceramic to be extruded as a wire and to be plastically worked under gentle heating. The organic component of the blend was removed by pyrolysis before sintering the ceramic powder. This method may be particularly suited to the production of high-temperature ceramic springs, heating elements and ceramic superconductor windings for electrical power applications. Defects produced during removal of the polymer and the methods used to avoid them, shed light on the complex processes taking place in crowded suspensions during extraction of the organic vehicle.

## 1. Introduction

The rapid development of ceramic materials in electrical and mechanical devices has emphasized techniques for the fabrication of complex shapes from non-plastic solids. One of the ways of achieving this is to incorporate the ceramic powder in suspension in an organic vehicle and to employ one of the many processing routes open to the polymer technologist. In this way, extrusion [1], blow moulding [2] and, most importantly, injection moulding [3, 4] have been used to generate shape and form from assemblies of fine particles prior to sintering. The present work details techniques for the fabrication of ceramic windings which can be plastically formed at temperatures just above ambient. These techniques are valuable for the production of high temperature ceramics springs and have recently acquired a novel relevance to the manufacture of ceramic superconducting components.

Current work in this laboratory is directed towards understanding the properties of ceramic suspensions which favour such processing routes [5-7]. In particular, the removal of the organic component from the assembly of particles before firing is a priority. The most widely used method is a controlled pyrolysis in either air or a non-oxidizing atmosphere. The rapid generation of volatile degradation products at this stage may produce voids or cracks which cannot be removed by sintering.

Two models describe the process of binder removal from plastically formed ceramic suspensions. In the first, degradation of the binder proceeds uniformly throughout the body according to thermal degradation reaction kinetics. In the second, the reaction proceeds preferentially at the surface and the 'shrinking unreacted core model' of Szekely [8] applies. The latter is more applicable where a reactive gas is present, the former where the polymeric binder decomposes by thermal degradation in an unreactive gas, or where diffusion control limits reactive gas supply.

The choice of polymeric binders for plastic forming is influenced by many factors [3-7] including the

thermomechanical properties of the suspension. The blending of a main component in the form of a high polymer with a minor component in the form of an oil, wax or plasticizer is a common feature [3], and allows the mechanical properties to be modified for processing. In particular, it may allow the freedom to select the softening point of the suspension.

There is growing interest in the fabrication of ceramic springs [9-12] which have the advantage of surviving high service temperatures without metallurgical deterioration, oxidative damage or corrosion in severe environments. Helical springs have been prepared from ceramic materials by machining a helical groove in a cylindrical prefired compact [12]. A more attractive route, and one that lends itself to mass production methods, is the extrusion and subsequent plastic working of a ceramic suspension. In this context a water based system has been tried [9, 10] in which the suspensions is wound onto a rotating mandrel with suitable spacers. One of the difficulties was that drying shrinkage caused the winding to contract on the mandrel, making removal difficult or cracking the body. Elaborate precautions were therefore needed to overcome the effects of shrinkage stresses. In the present work this problem is overcome by controlling the softening point of the suspension while maintaining a high melt strength.

The ability to produce windings from ceramic components opens up new fabrication routes for superconducting ceramics [13] which would allow the production of windings for motors, electromagnets, generators and related equipment.

## 2. Experimental procedure

The materials used were as follows:

alumina	A16SG (Alcoa GB Ltd)
ceramic	YBa <sub>2</sub> Cu <sub>3</sub> O <sub>x</sub> (Cookson Group
superconductor	Research Centre, UK)
polystyrene	HF66 and HF555
	(BP Chemicals Ltd, UK)

TABLE I Composition of suspensions

Component	Density kg m <sup>-3</sup>	Composition no.							
		1	2	3	4	5	6	7	8
Superconducting ceramic powder	6300	-	-	-	-	-	-	-	88.78
Alumina	3980	85.14	85.11	85.40	85.50	84.25	86.28	86.07	-
Polystyrene	1060	6.75	9.02	8.85	7.91	8.94	9.50	6.15	6.12
Dibutyl phthalate	1047	6.75	4.51	-	-	-	-	-	-
Diethyl phthalate	981	-	-	4.43	4.39	-	3.17	-	3.40
Dimethyl phthalate	1190	-	-	-	-	3.83	-	-	-
Ethylene vinyl acetate	950	-	-	-	0.88	1.49	-	-	0.68
Polybutene	893	-	-	-	-	-	-	6.15	-
Stearic acid	941	1.35	1.35	1.33	1.32	1.49	1.06	1.64	1.02

di-n-octyl phthalate	Aldrich Chemical Co. Ltd
dibutyl phthalate	BDH Chemicals Ltd
dimethyl phthalate	
stearic acid	
ethylene vinyl acetate copolymer	
polybutene	H50 Amoco chemicals Ltd

Table I gives the compositions prepared during the course of this work. The mixing was performed on a Betol Laboratory twin-screw extruder model TS40-DV-L which produces continuous high shear mixing [14]. This device has been shown to be capable of dispersing agglomerated ceramic powders [15]. Table II gives the extrusion temperatures used and a screw speed of 60 r.p.m. was employed throughout. Composition 8, incorporating superconducting ceramic powder, was prepared on a twin-roll mill fitted with oil heating at 175°C.

Each composition was ashed to 650°C in air to determine the exact powder loading. Composition 8 was ashed, along with samples of ceramic powder, to 950°C in order to compensate for changes in the weight of the YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> powder caused by heating in air, and the results are given in Table III. The calculated volume loadings are also given assuming that the organic species are present in their original ratios.

Extrusion of wire was performed using a Davenport Capillary rheometer with a 19 mm diameter piston and a 2.5 mm diameter die. Temperatures in the range 125–190°C were used and the granulated feedstock was left for 10 min to equilibrate before extrusion. The extrudate was wound onto a rotating steel mandrel. The extrusion conditions are given in Table IV.

Plastic working of the extrudate was carried under gentle heating from a warm air blower in order to adjust

pitch and produce hooks or terminals and leads on the windings. After producing the desired shape, the components were supported on alumina powder and heated using the schedules described in Fig. 1. With one exception, these are modifications of a 5°C/hr ramp and were obtained using a Eurotherm 818P programmable temperature controller. After this treatment the fragile pre-fired alumina bodies were sintered in air at 1600°C for 1 h and the YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> windings in air at 930°C for 4 h. Thermogravimetric analysis was performed on finely powdered samples using a Stanton thermobalance either in air or in a nitrogen flow of 40 l h<sup>-1</sup>.

### 3. Results and discussion

#### 3.1. Development of formulations

The development of the formulations used in this work follows the principles previously described [3]. Alumina powder was used in most of the experiments on grounds of cost, uniformity and availability. The volume fraction of A16SG alumina suitable for injection moulding was 60 vol % [16]. The same proportion was initially selected for the extrusion of windings. A high volume fraction is desirable not only to reduce firing shrinkage [3] but also to reduce slumping during removal of the binder [16]. The volume loading was modified in later stages of the work for reasons described below.

No precise method currently exists for predicting the ideal volume fraction of ceramic powder from particle characteristics, and empirical tests were used for the superconducting ceramic powder. The pressed relative density of the powder was found to be 46% at 25 MPa nominal pressure, and the volume loading of powder which made a non-crumbling paste with linseed oil [17] was 45%. After extrusion trials a volume loading of 56% was selected. The pressed relative density gives an approximate, but generally slightly

TABLE II Compounding extruder temperatures

Composition no.	Temperature (°C)			
	Feed	Zone 2	Zone 3	Zone 4
1	160	170	185	200
2	160	170	185	190
3	160	170	180	190
4	160	170	180	190
5	150	160	160	160
6	150	180	200	200
7	160	190	200	210

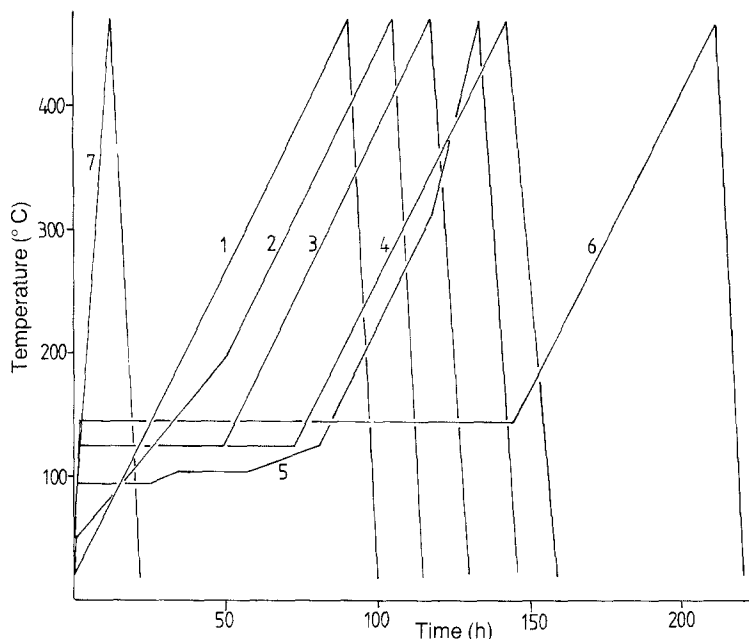


Figure 1 Heating schedules for removal of organic material from windings for compositions 1 to 7.

lower value than the ideal loading for processing of suspensions [15]. The critical powder volume concentration [18] measured by torque rheometry using a simple oil or carboxylic acid gives an approximate indication of maximum volume loading, but in the present work the small quantity of powder available necessitated a simplified and unfortunately more subjective test [17]. The fact that the pressed density and the oil absorption test indicated a lower volume loading than that obtained by twin-roll milling is indicative of an agglomerated powder.

The plastic-forming processes available for ceramic fabrication can in principle be applied to many different powders provided the guidelines for control of viscosity are followed [5]. Stearic acid was incorporated as a lubricant or processing aid because it has been shown to reduce the accumulation of iron in the suspension from abrasive wear of processing machinery [16]. It is likely that the stearic acid functions as a lubricant early in the mixing process before the particles have been encapsulated by the main binder component.

An injection moulding grade of polystyrene was selected as the main binder because it is non-crystalline and because its glass transition temperature, and hence the softening point of suspensions based upon it, can be controlled by diluent content. Previous work on injection moulding had revealed problems with unsteady flow using polystyrene with dibutyl phthalate as the organic component and 65 vol %

silicon powder [7]. With the suspensions used in the present work lower extrusion temperatures were employed and the quality of extrudate was not impaired by this problem.

Thus in composition 1 the weight ratio of polymer to diluent was 1 : 1. It was possible to prepare windings from this composition but they inevitably slumped at room temperature. This can be attributed to the lowering of the softening point by the diluent. Although it is unusual in polymer technology to incorporate plasticizers into 'polystyrene, the solubility parameter is in the range  $17.5-18.6 \times 10^3 (\text{J m}^{-3})^{1/2}$  for polystyrene [19] and  $19 \times 10^3 (\text{J m}^{-3})^{1/2}$  for dibutyl phthalate [20] suggesting mutual compatibility. The polymer-polymer interaction is low for polystyrene and therefore solution behaviour is expected rather than conventional plasticization. In fact polystyrene-diluent blends have been investigated previously for ceramic injection moulding [21].

A very useful feature of this system is that the softening point can be controlled by composition. Thus composition 2 was prepared in which the polystyrene-diluent ratio was 2 : 1, other factors being unchanged. This produced windings which could be

TABLE III Exact ceramic loadings and calculated volume percent

Composition no.	Wt %	Vol %
1	84.79	59.6
2	85.09	60.0
3	85.55	60.4
4	85.29	60.1
5	84.78	59.8
6	86.43	62.3
7	86.43	60.7
8	88.83	56.2

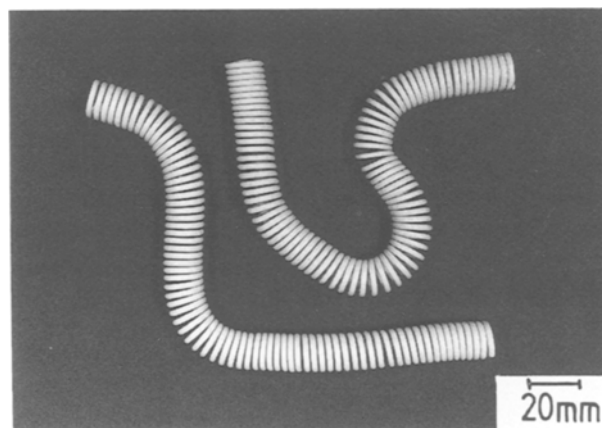


Figure 2 Flexible extruded windings of a 60 vol % alumina suspension prepared from composition 2.

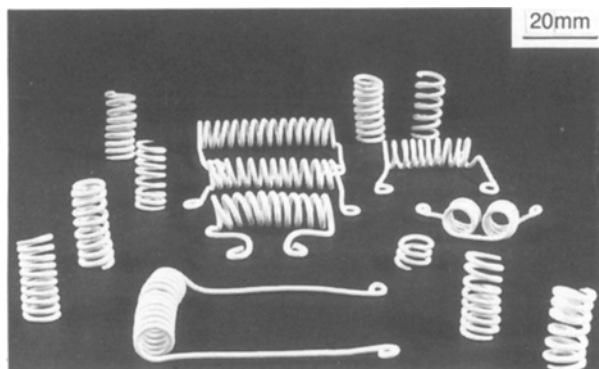


Figure 3 Sintered alumina helices showing the versatility of form and the problems of sagging during polymer extraction.

stored at room temperature and plastically worked under gentle heating to change their shape.

Further refinements of the formulations were necessitated by problems caused during removal of the binder by pyrolysis which are described in detail below. In particular, di-n-octyl phthalate (boiling point 384°C) and also di-methyl phthalate boiling point 284°C) were substituted for di-butyl phthalate (boiling point 340°C) in order to modify the volatility of the diluent. In composition 4 some of the polystyrene was replaced with an ethylene-vinyl acetate copolymer.

### 3.2. Preparation of windings

Material of composition 2 was wound into coils using the first set of extrusion conditions in Table IV. The flexible composite windings thus obtained could be handled without damage and stored at room temperature without slumping. They are shown in Fig. 2. An advantage of the low softening point was that it resulted in very low thermal stresses which would otherwise clamp the winding on the mandrel or cause cracking. The windings were then cut into shorter lengths and reworked by plastic forming under a warm-air blower in order to adjust the pitch and to produce leads and terminals. On cooling they retained their shape.

The windings were placed on a powder bed of alumina and heated using ramps 1 and 2 in Fig. 1. They were then transferred to a sintering furnace and fired at 1600°C to produce the range of components shown in Fig. 3 which shows the versatility of form that is possible with this process. The consequence of

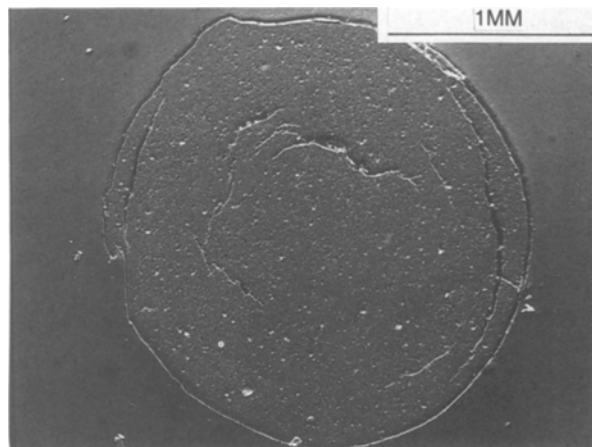


Figure 4 Polished section of a sintered ceramic wire prepared from composition 2 showing circumferential cracking.

slumping during reheating to remove the polymer are clearly evident in Fig. 3 and precautions are needed to prevent this deformation.

Unfortunately ceramic wire produced by this route presented circumferential sub-surface cracks shown in polished section in Fig. 4. This effect was present in the prefired state where it gave the appearance of a 'skin' in the fracture face (Fig. 5). No cracks could be detected in the extruded wire before polymer removal. A possible origin for these defects was thought to be the extrusion process. It is well known that radial separation of particles from matrix can occur in capillary flow [22] leading to inconsistent volume loadings of powder across the diameter of the extrudate. This could explain the cracking if consequent volume shrinkage on removal of the polymer was non-uniform.

In an attempt to eliminate these defects the full range of extrusion conditions shown in Table IV was explored. Springs could be prepared under most conditions with extrusion temperatures from 125–190°C. The die land length was reduced from 35 to 1 mm in order to reduce shear flow effects. The extrusion speed was also varied over a wide range, but at low extrudate velocities cooling in ambient air caused the extrudate to be too rigid to coil. Under each of these conditions the cracks were present after polymer removal using ramps 1 and 2 despite the excellent outward appearance of the components, and it was concluded that the defects were unlikely to have originated at the extrusion stage.

TABLE IV Extrusion conditions for the preparation of windings

Extrusion temperature (°C)	Piston speed (mm min <sup>-1</sup> )	Extrudate velocity (mm min <sup>-1</sup> )	Die land length (mm)
125	10	578	35
130	10	578	35
140	10	578	2
140	10	578	1
170	10	578	1
190	10	578	1
190	5	289	1
190	2.5	144	1
190	1.25	72	1

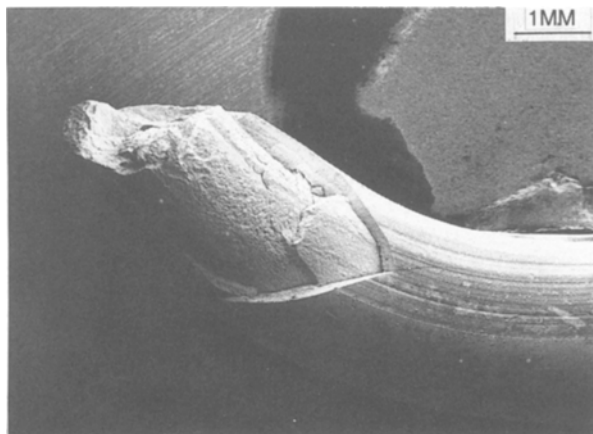


Figure 5 Fracture face of prefired ceramic wire prepared from composition 2 showing sub-surface circumferential cracks.

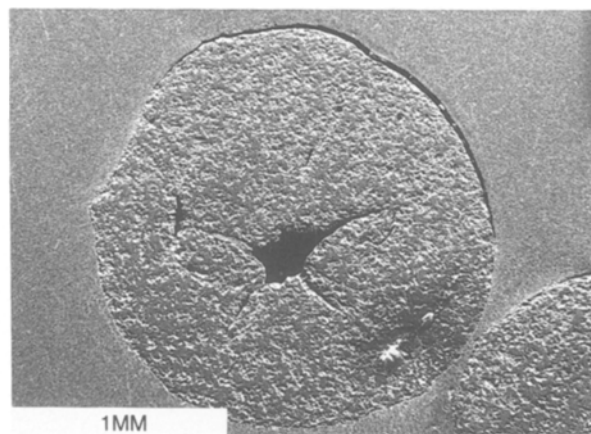


Figure 7 Polished section of sintered ceramic wire prepared from composition 4 showing internal hollow pipe.

### 3.3. Removal of the organic binder

The excellent thermomechanical properties of composition 2 were offset by the appearance of macroscopic defects which became apparent after removal of the organic vehicle. This was surprising in view of the small cross-section of the wire and the very slow heating rate of  $5^{\circ}\text{C h}^{-1}$ . Acting on the hypothesis that the defects were associated with the composition of the organic vehicle, two modifications were made to composition while keeping the volume fraction of alumina constant.

Diethyl phthalate, being a less volatile diluent was substituted for the dibutyl phthalate in composition 3. Ethylene vinyl acetate (EVA) copolymers have found a place in ceramic processing largely as a result of their role in hot melt adhesives [23]. The requirements of polarity and extensive solubility with other polymers are common to both applications. Therefore in composition 4 a small amount of EVA was substituted for polystyrene in an attempt to modify the polymer-ceramic interfacial interaction. In composition 5 an extremely volatile diluent, dimethyl phthalate, was substituted for the dioctyl phthalate in order to examine further the effect of diluent volatility on the incidence of defects.

Fig. 6 shows the thermogravimetric loss for composition 4 heated in static air, flowing nitrogen and

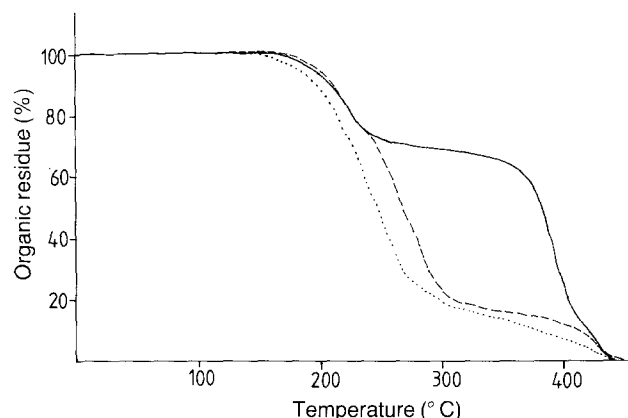


Figure 6 Thermogravimetric loss for composition 4 in powdered form in nitrogen, air and oxygen (heating rate  $1^{\circ}\text{C min}^{-1}$ ). (—)  $\text{N}_2$ ; ( $\cdots$ )  $\text{O}_2$ ; (---) air.

flowing oxygen. In nitrogen the curve reveals two stages, the first of which is the evaporation of the diluent. The second decomposition is that of the higher molecular-weight polymer components of the blend. The use of the inert atmosphere produced a more controllable rate of weight loss over a wider temperature range by comparison with the oxidizing atmosphere. Furthermore the maximum temperature to which the body must be heated to remove all the organic residue was not reduced by the use of oxidizing atmospheres. These two factors encouraged the use of inert atmospheres of nitrogen or argon in subsequent work.

Table V shows the incidence of defects in samples heated in an inert gas on the temperature ramps described in Fig. 1. All samples prepared from composition 2 presented circumferential cracks similar to those shown in Fig. 4. Compositions 3, which employed the less volatile dioctyl phthalate, produced 37% defect-free sections but this proportion of defects was unacceptably high. The partial substitution of EVA changed the appearance of the defects. Instead of circumferential cracks many sections revealed an internal semi-continuous pipe with occasional cracks emanating from the centre radially (Fig. 7).

Figures 4, 5 and 7 taken together with the thermogravimetric loss curve in Fig. 6 indicated the cause of the defects in these suspensions. It is well known that the viscosity of suspensions increases steeply with volume loading of powder in the high powder volume fraction regime [24]. The maximum volume loading for fluid flow is approximately 70% for this powder [25]. It is argued that evaporation of diluent from the surface of the wire during pyrolysis caused the effective volume fraction of powder at the surface to increase. Evaporation of all the diluent in composition 2 would provide a volume fraction of alumina of 0.68. Thus a rigid exoskeleton may be formed at an early stage. Organic components continued to emerge from the core of the wire and the resulting shrinkage produced stresses at a stage when the polystyrene was still present at particle junctions, forbidding interparticle movement. The circumferential cracks were thus a consequence of the core shrinking from the rigid outer layer.

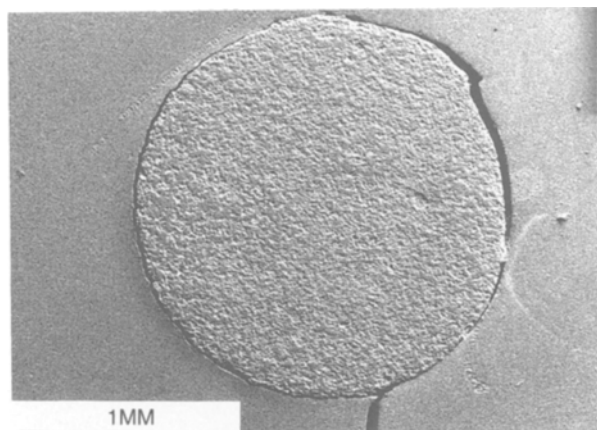


Figure 8 Polished section of sintered ceramic wire prepared from composition 4 after removing all the diluent at 140°C before removal of the polystyrene.

It is believed that the incorporation of EVA modified the decomposition behaviour and the mechanical properties of the suspension enough to prevent fracture of the brittle outer region of the composite. Nevertheless, hydrostatic tension was still present as the volatile phthalate evaporated from the wire and gave rise to a central semi-continuous void in the region of the wire where the composite retained fluid properties, together with some cracks emanating from this void.

Clearly the failure of ramps 1, 2 and 7 in Fig. 1 to avoid these stresses arises from the fact that the removal of diluent was too rapid in relation to its diffusion through the wire. Thus a steep radial concentration gradient was established. When composition 4 was heated according to ramp 6 which allowed a weight loss equivalent to the weight fraction of diluent to occur at 140°C before raising the temperature to 470°C, cracking was absent in 76% of the fracture faces of the sintered wire. Figure 8 shows a macro-defect-free section of this wire. The long heating schedule and the significant proportion of defects still make the process commercially unusable.

Figure 9 shows the thermogravimetric loss curves for compositions containing dimethyl, dibutyl and di-n-

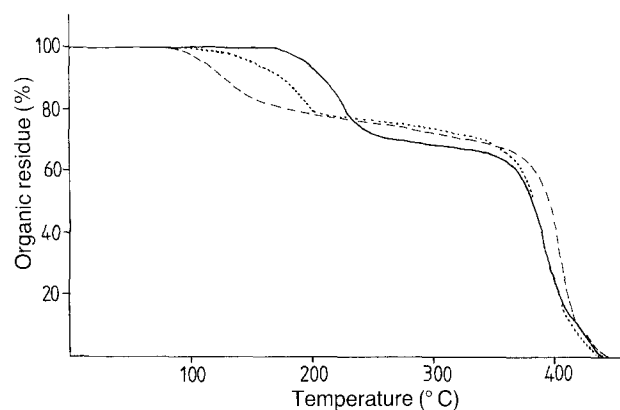


Figure 9 Thermogravimetric loss for compositions (····) 2, (—) 4 and (---) in powdered form in nitrogen (heating rate 1°C.min<sup>-1</sup>).

octyl phthalate esters. The temperature at which the initial weight loss occurs reflects the volatility of the phthalates. It should be noted that each composition incorporated slightly different weight fractions of diluent. These curves are for finely divided material where diluent diffusion effects are not considered to be rate controlling and they therefore represent the behaviour of the outer region of the wire. Samples of composition 5 containing dimethyl phthalate were heated on ramps 1 to 5 in Fig. 1, and 98% of sections examined showed circumferential cracks. It is likely that the low temperature loss of diluent shown in Fig. 8 is unfavourable because the weight loss from the outer skin occurs at a very early stage. This hypothesis is compatible with the observations that dioctyl phthalate as a diluent (composition 3) produced 36% defect-free sections (Table V).

Further evidence for the importance of non-uniform removal of binder from these suspensions is presented by the observation that all attempts to use oxygen-containing atmospheres for pyrolysis of the organic phase resulted in severe cracking. Figure 6 shows that weight loss of polystyrene occurred at a lower temperature in oxidizing atmospheres, and it is also likely to be more pronounced at the surface of the wire because of the rate-limiting effect of oxygen diffusion. Furthermore, it is possible that oxidative damage to the phthalate ester enhanced volatilization.

TABLE V Defects observed by optical microscopy of polished sections of sintered ceramic wire

Composition no.	Ramp no.	Number of sections examined			
		Cracks	Voids	Total	Percentage without macrodefects
2	1	32	0	32	0
	2	11	0	11	0
3	1	65	0	103	37
	2	13	0	18	28
4	1	0	7	7	0
	2	13	8*	22	5
	7	0	12	12	0
	6	3	5	34	76
	5	14	0	14	0
5	2	24	0	24	0
	3	14	0	16	13
	4	43	0	43	0
	5	25	0	25	0
	1	7	0	85	93
7	1	2	0	128	98
8	6	3	2	21	76

\*Some samples show both voids and cracks.

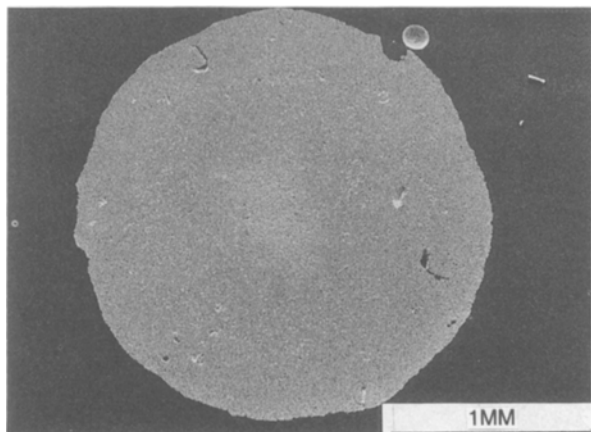


Figure 10 Polished section of sintered wire of  $\text{YBa}_2\text{Cu}_3\text{O}_x$ .

If the preferential loss of diluent from the surface of the ceramic wire is the main explanation for this type of defect, it follows that factors which impede volatilization should reduce the incidence of defects. Windings of composition 3 were heated on ramp 1 in nitrogen but submerged in an alumina powder bed in a tall cylindrical alumina crucible. Of 86 sections examined, 17 presented cracks. This scrap rate of 20% is significantly lower than the 63% observed with the wire exposed to the flowing gas stream. This experiment confirms the source of the problem but does not present a satisfactory solution.

The hypothesis employed to explain the persistent appearance of defects in ceramic windings prepared by extrusion involves a competition between the loss of diluent and the asymptotic approach to infinite viscosity as the effective volume loading of ceramic powder at the surface increases. It follows that a ceramic suspension which started at a higher volume fraction of powder would undergo less shrinkage as the binder was removed from particle junctions. The effective volume loading at which shrinkage ceases due to particle contact can be considered to be similar to, or not slightly lower than, the volume loading at which viscosity becomes effectively infinite [25]. Thus composition 6 was prepared using dioctyl phthalate as a diluent with 62 vol % A16SG alumina. This composition did not extrude evenly because of the high volume loading of powder. Table V shows that 93% of sections examined were free from macroscopic defects.

In composition 7, the volatile phthalate diluent was replaced with a low-molecular-weight polybutene which is fluid at room temperature. This blend had a number of disadvantages compared with the poly-

styrene-phthalate blends. It did not mix easily and the stearic acid content had to be increased. It has a higher softening point than composition 3, making the preparation of windings less easy. Windings prepared from it were more fragile, probably because of incomplete compatibility of the polymer blend. However, by using a low-molecular-weight polymer to replace the volatile phthalates, the problem of defects was solved. Indeed the two cracks observed in 128 sections examined may have been associated with contamination in the blend and not with the polymer removal process. Clearly, careful tailoring of the composition is needed to balance the influence of additives on softening point, mechanical strength and volatility, but the viability of the process is established.

### 3.4. $\text{YBa}_2\text{Cu}_3\text{O}_x$ wire

Figure 10 shows the polished section of a  $\text{YBa}_2\text{Cu}_3\text{O}_x$  ceramic wire after removal of the organic binder on ramp 6, and sintering at  $930^\circ\text{C}$  for 4 h, indicating that this fabrication process is effective with such powders.

Some of the defects which appeared in these components were related to problems of degassing the suspension before extrusion (Fig. 10), but 24% of sections examined showed defects related to removal of the organic vehicle. Further work to establish the optimum volume loading is thus desirable for this powder.

No attempt was made to test the electrical properties of the wire, but the impurity contents of the organic materials used were assessed by energy dispersive X-ray analysis of the ash obtained from the organic species alone, and are shown in Table VI. The impurity residue introduced from the organic vehicle would be approximately 40 p.p.m. based on the ceramic, and further iron impurity may result from processing machinery. The deleterious effect of the impurities on electrical properties may mean that high-purity organic materials need to be synthesized for the processing of ceramic superconductors by this route. Sintering at  $930^\circ\text{C}$  for 4 h produced a linear shrinkage of 10 to 12% in the  $\text{YBa}_2\text{Cu}_3\text{O}_x$  ceramic. A sintered winding is shown in Fig. 11, which also shows alumina springs of both square and round sections, indicating that the slumping problem can be overcome by proper setters. This shows the excellent surface finish that can be achieved by the process.

## 4. Conclusions

1. Ceramic windings can be prepared by extrusion of a polymer-ceramic suspension and removal of the organic phase before sintering.
2. An amorphous thermoplastic polymer was

TABLE VI Impurity elements detected in the ash of the organic components by energy dispersive X-ray analysis: the impurities are listed in order of decreasing abundance

Organic component	Residue (wt %)	Impurity elements
Polystyrene	<0.01	Zn, Ca,
Stearic acid	<0.01	Ca, Al,
Ethylene vinyl acetate	<0.01	Si, Ca, Al
Dioctyl phthalate	No visible ash	-

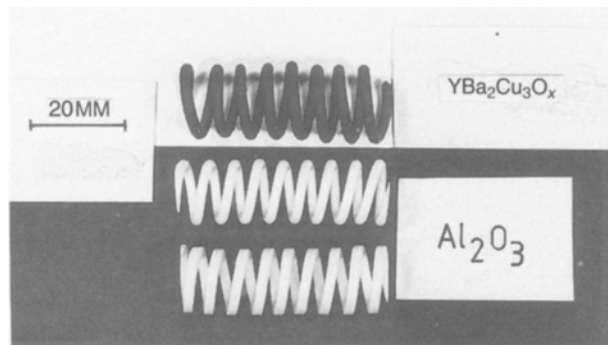


Figure 11 Sintered coil of alumina showing excellent surface finish. (a) Round section; (b) square section. Sintered coil of  $\text{YBa}_2\text{Cu}_3\text{O}_x$  formed from composition 5.

blended with a diluent so that the softening point of the suspension was just above ambient. The wire could then be plastically worked under gentle heating to produce complex windings with hooks or terminals.

3. Removal of the diluent by slow heating occurred preferentially at the surface of the wire resulting in a higher effective volume fraction of powder in this region. This was thought to result in the development of a rigid exoskeleton. As the inner ceramic suspension shrank away from the outer layer, characteristic defects were produced. These took the form of circumferential cracks or internal pipe-like voids.

4. Thermogravimetry was used to characterize the loss of the diluent from the outer region of the wire and the less volatile diluent produced the lower proportion of defects.

5. These defects could be avoided if the initial ceramic volume fraction was increased to a level that still permitted extrusion but reduced the volumetric shrinkage resulting from removal of organic vehicle from particle junctions.

6. Alumina and  $\text{YBa}_2\text{Cu}_3\text{O}_x$  ceramic windings were prepared by this method. Provided the ceramic volume loading is correctly chosen, the method, should be applicable to ceramic and metal powders in general.

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