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Fast Firing of Electroceramics

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Fast firing of electroceramic material was investigated as a means of rapidly processing piezoelectric material. The green bodies were rapidly heated to their sintering temperature and sintered for a variety of processing times. Piezoelectric properties and microstructures were compared to material processed under the conventional processing conditions. The d_{33} coefficient, coupling factor and density compared well with conventional material whilst the grain size was considerably lower. Possible improvements due to the fine grain size are increased mechanical properties and lower dielectric loss.

Keywords: Fast-fire: piezoelectric: grain size

1. INTRODUCTION

Fast firing was introduced by Brook et al. as a means to obtain high density and fine grain sized ceramic materials[1,2,3]. The technique involves passing the ceramic material through a sintering temperature zone for a short period. It has been proposed that the densification and grain growth rate are both exponentially dependent on the respective activation energies. If the densification activation energy (ΔH_d) is greater than grain growth activation energy (ΔH_g), the densification proceeds faster than grain growth above a critical temperature. It has also been proposed that the high temperature

gradient within the ceramic during fast firing can increase the densification rate[4].

Compared to conventional furnace heating, the fast firing procedure reduces the processing time considerably, as demonstrated in Figure 1. The fast processing time has a number of possible advantages:

(i) the formation of fine grain sized material with possibilities for improvement in mechanical and dielectric properties
(ii) a reduction in furnace energy consumption and possibilities for continuous processing
(iii) reduction in lead oxide loss, which often occurs in lead based materials held at high sintering temperatures for long periods.

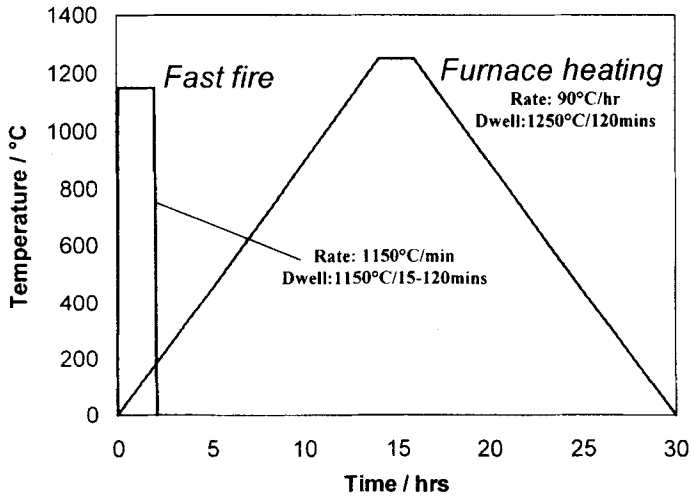

FIGURE 1 Demonstration of large reduction in processing time by using the fast firing process.

The aim of the paper is to investigate the effect of fast firing on a typical piezoelectric material and whether, under the correct conditions of temperature and time, it is possible to achieve high sintered density and fine grain size. The microstructure and piezoelectric properties of fast-fired electroceramics will be compared to conventional materials.

2. EXPERIMENTAL

The ceramic powder used for this study was PC4 from Morgan Matroc, a hard lead zirconate titanate (PZT) piezoelectric material. The powder was uniaxially pressed into green bodies of 30mm diameter and 2.4mm thickness at 35MPa to a green density of 55%. After a binder burnout procedure of heating to 450°C at 60°C hr^{-1} and a hold for 2hrs the material was sintered under conventional and fast fired methods. The sintering procedures were,

(i) conventionally processed material was heated at 90°C hr^{-1} to 1250°C and held for 2h and cooled at 90°C hr^{-1}

(ii) fast firing was conducted in a 5 zone furnace with the hot zone (1135°C) at the furnace centre. PZT green bodies were enclosed in an alumina crucible surrounded by loose PZT powder and pushed into the furnace hot zone in 1min and held at times of 0.25 to 2 hours to sinter. The ceramic was subsequently removed from the furnace in 1min.

The sintered ceramic material was subsequently ground to a sample size of 25mm diameter and 1mm thick for electrical characterisation. The density of the ceramic was calculated using the Archimedes' method and microstructural analysis of the polished and etched surface was performed using scanning electron microscope (SEM). The mean grain size was determined by a linear intercept method from a number of micrographs.

3. RESULTS

Figure 2 shows the variation in density of the fast fired sintered material as a function of dwell time at 1135°C. Densities of the order of 98% were obtained by holding the material at the sintering temperature for 1-2 hours, which compares well with the conventionally produced material. It should be noted that the high densities were obtained at sintering temperatures of 1135°C, lower than the conventional temperature of 1250°C.

The variation of piezoelectric coefficients (d_{33} and d_{31}) and coupling coefficient, k_p, as a function of fast firing time are shown in Figures 3 and 4 respectively. The d_{33} and d_{31} coefficient increase with fast firing dwell time and density and are similar to conventionally processed PC4.

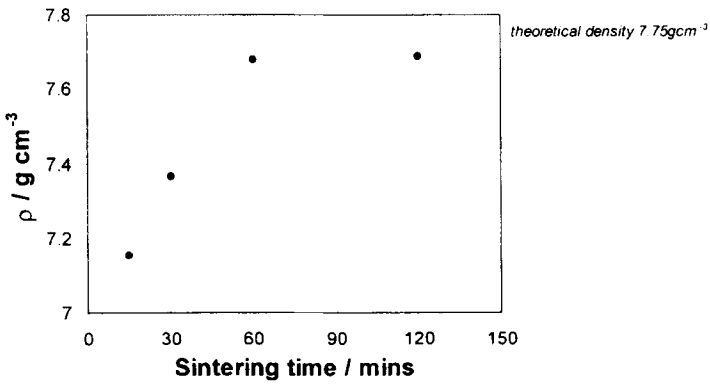

FIGURE 2 Density of fast fired piezoelectric as a function of time at 1135°C

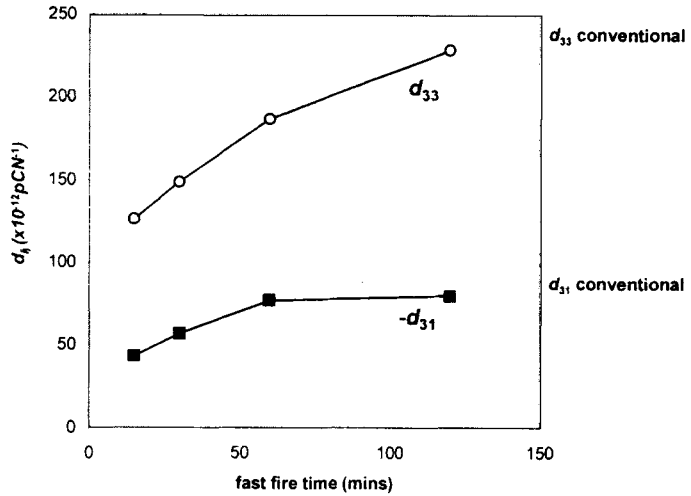

FIGURE 3 Variation of piezoelectric *d* coefficients with fast firing time

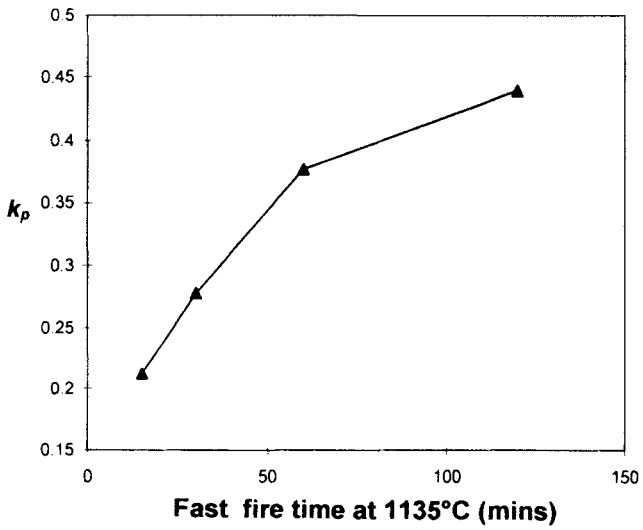

FIGURE 4 Variation of planar coupling coefficient with fast fire time. A typical value for conventional PC4 is 0.5.

Figure 5 & 6 show secondary electron micrographs of polished and etched surfaces of the conventionally processed material and fast fired material. For the conventional material the grain size is ~4μm whilst for the fast fired ceramic the grain size is ~2μm. The finer grain size is associated with the lower sintering temperature and faster processing time.

Hsueh et al. have shown that fine grain size yields a lower dielectric constant and lower dielectric loss[4]. In terms of mechanical properties, a fine grain size can lead to improved strength and toughness.

CONCLUSIONS

Fast firing has been demonstrated as a means of rapidly sintering electroceramic materials. The density and the piezoelectric properties measured are similar to conventionally sintered materials. The advantages of the fast firing process are energy savings due to the faster processing times, the possibility of reducing lead oxide loss and the fine grain size of the material. Further work will examine any mechanical property enhancements (strength, hardness etc.) or electrical property enhancements (in particular dielectric loss) achieved due to the fine grain size. A detailed study of the densification mechanism by dilatometry is also required to determine activation energies for densification.

Acknowledgements

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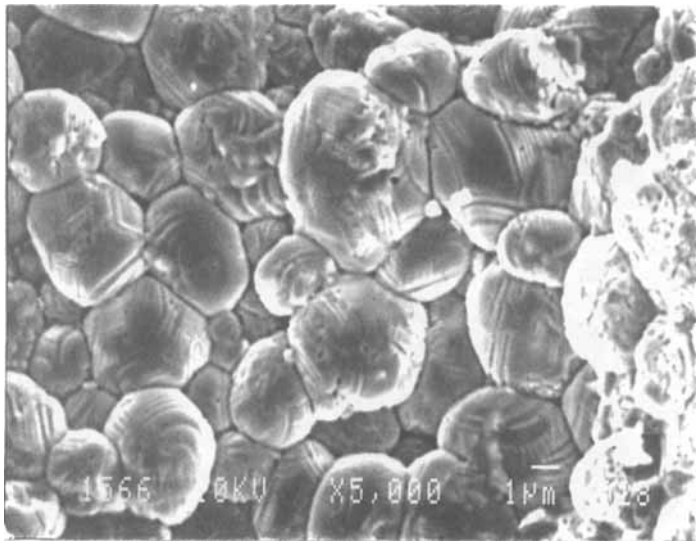

FIGURE 5. Secondary electron image of a polished and etched surface of conventionally sintered PC4 at 1250°C for 2h.

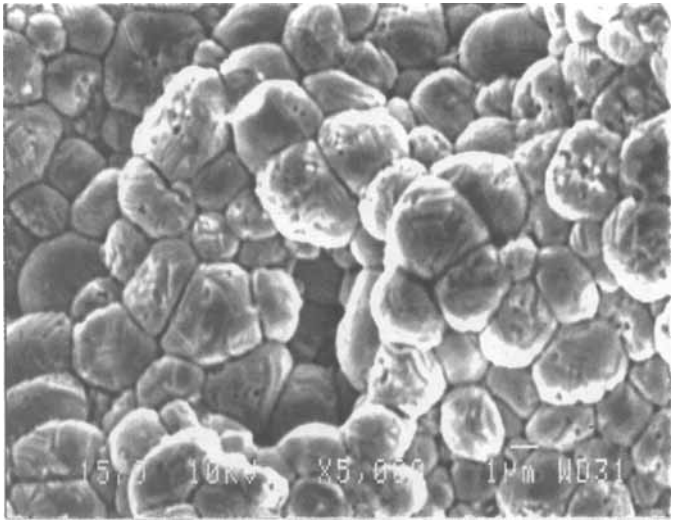

FIGURE 6. Secondary electron image of a polished and etched surface of fast fired PC4 at 1150°C for 2h. A reduction in grain size is observed.

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