

# TA010

## INORGANIC CHEMISTRY

### EVIDENCE FOR THE MICROWAVE EFFECT DURING THE HYBRID SINTERING OF ZNO

**J. Binner, J. Wang and B. Vaidhyanathan**

Institute of Polymer Technology and Materials Engineering,  
Loughborough University, Loughborough, United Kingdom

Many investigators have reported unexpected effects resulting from the use of microwave radiation as an alternative energy source during the processing of materials. This has included apparent evidence for accelerated kinetics for a range of processes in ceramic, polymeric and organic systems [1-10]; enhanced sintering of ceramic powder compacts, including lower sintering temperatures [1,2]; and reduced activation energies [1-3,11]. Whilst it is now probably true to say that it is generally, though not unanimously, accepted that a 'microwave effect' exists, uncertainty still occurs because of:

- The inability to vary the energy source without simultaneously affecting a wide range of other variables.
- Uncertainties associated with temperature measurement. Pyrometry is often used with microwave heating whilst thermocouples are used in the conventional experiments. When a single technique is used, it is usually a shielded thermocouple - although the presence of the metallic shielding is known to distort the local microwave field. Finally, the surface temperature is usually measured. With conventional heating this will be the hottest part of the specimen, whilst with microwave heating it will be the coolest. This makes a direct comparison of results problematic at best.

The existence of these uncertainties has the direct effect of making clear analysis of the potential economic gains of using microwave processing rather problematic. As an example consider the simple case of an apparent lowering of sintering temperature by 100°C, a not unusual claim. Whether this reduction is genuine or merely an artefact of the difficulty of measuring temperature in a microwave environment will substantially affect the calculations of energy required for the sintering process. This will in turn affect the economics of the process. Although authoritative claims are often made by those doing the research, companies are understandably nervous of adopting a technology which they feel cannot be precisely and independently quantified.

In the present work, a microwave/conventional hybrid sintering furnace has been used to investigate the effect of the fraction of microwave power on the densification of ZnO for identical time/temperature profiles. Microwave-immune, optical fibre thermometers (OFT) have been used to record the temperature at the centre and edge of the samples to provide an indication of any temperature gradients present. The results appear to show that densification can be significantly enhanced by the presence of a microwave field, particularly at high power levels in the intermediate densification regimes and with finer precursor powders. More than 20% extra densification was achieved when physically small, submicron ZnO samples were sintered at 680°C for 1 hour using 1000 W of microwave power in a hybrid regime compared to an identical time-temperature profile achieved with pure conventional heating. Whilst significant temperature gradients were observed across large sample sizes, it is believed that they were less than 15°C for the smaller size samples used. Therefore it is unlikely they are the origin of the effect although further work is currently underway to completely eliminate this possibility. Work is also ongoing with respect to studying grain growth during the hybrid annealing of ceramics and the diffusion of O<sup>18</sup> in a variety of different ceramic materials. All the results

to date suggest that if a microwave effect exists, it is most likely to be associated with grain boundary (or interface) diffusion, rather than bulk diffusion.

## REFERENCES

1. Janney MA & Kimrey HD, Mat Res Soc Proc **189** 215-227 (1991).
2. Janney MA, Kimrey HD, Schmidt MA & Kiggans JO, J Am Cer Soc **74** [7] 1675-1681 (1991).
3. Lewis DA, Mat Res Soc Proc **269** 21-31 (1992).
4. Giguere RJ, *idem*, 387-396.
5. Katz JD, Blake RD & Kenkre VM, Ceram Trans **21** 95-105 (1991).
6. Willert-Porada M, Krummel T, Rohde B & Moormann D, Mat Res Soc Proc **269** 199-204 (1992).
7. Boch P, Lequeux N & Piluso P, *idem*, 211-216.
8. Fathi Z, Ahmed I, Simmons JH, Clark DE & Lodding AR, Ceram Trans **21** 623-629 (1991).
9. Binner JGP, Hassine NA & Cross TE, J Mat Sci **30** 5389-5393 (1995).
10. Booske JH, Cooper RF, Dobson I & McCaughan L, Ceram Trans **21** 185-192 (1991).
11. Janney MA & Kimrey HD, in Ceramic Powder Science II, Am. Ceram. Soc., 919-924 (1988).
12. Janney MA, Calhoun CL & Kimrey HD, Ceram Trans **21** 311-318 (1991)