

Effects of Precursor Chemistry on the Microstructural Characteristics of Sol-Gel/Combustion Synthesized Y_2O_3 -MgO Nano-Composites

Chigozie K. Muoto, Eric H. Jordan, Maurice Gell and Mark Aindow

Institute of Materials Science, University of Connecticut, Storrs, CT 06269-3136

Single-phase nanostructured oxides have a strong tendency for grain coarsening during high temperature processing. One way to limit the grain growth in these ceramic materials is to introduce a second oxide phase, so that in the microstructure of the resultant composite material the grains of one phase pin the boundaries in the other, inhibiting boundary migration and grain coarsening. However, this is only effective when there is a homogenous distribution of the phases in the nano-composite oxide powder. The synthesis of such uniformly dispersed nano-composite oxides has been a major challenge and has attracted a great deal of research interest [1]. In our work we have been investigating the synthesis of such nano-composites by wet-chemistry routes [2]. We present here a study on the effect of solution precursor chemistries on the microstructures of MgO-50 vol% Y_2O_3 nano-composites.

Four different binary precursor mixtures were made from yttrium nitrate (Yn) or yttrium acetate (Ya), with magnesium nitrate (Mn) or magnesium acetate (Ma) using water as a solvent. The precursor solutions were dried and calcined at 500°C, cold-pressed and then sintered at 1350°C for 4 hours. The microstructures of the resultant composite oxides were investigated using a combination of: focused ion beam (FIB) sectioning, scanning and transmission electron microscopy (SEM and TEM). Precursor mixtures containing a nitrate and acetate gave much more homogeneous composites than those containing two nitrates or two acetates, as shown in figures 1 and 2. Backscattered electron (BSE) SEM images (e.g. fig. 1a) of the sintered samples revealed the sizes and distributions of the phases in the composite oxides. In such images the bright and dark regions correspond to the Y_2O_3 and MgO phases, respectively. Information about the grain sizes and crystal structure of the phases were revealed by TEM. The bright field (BF) images, selected area diffraction patterns (SADP) and microdiffraction patterns were all obtained from FIB-cut TEM sections through the sintered samples. Due to the non-uniform phase distribution, more grain growth occurred in the composites from the YnMn and YaMa mixtures than in those from the YaMn and YnMa mixtures.

The improved phase domain distribution observed in the samples produced from the acetate-nitrate mixtures is attributed to the increased rate of crystallization as a result of the excess heat evolved during calcining. Differential scanning calorimetry experiments show that precursor mixtures containing both acetate (reducer) and nitrate (oxidizer) salts undergo a highly exothermic combustion reaction whereas acetate- or nitrate-only precursor mixtures react endothermically making the decomposition and crystallization processes very sluggish and allowing for more extensive phase separation.

References

- [1] A. S. Edelstein and R. C. Cammarata, *Nanomaterials: Synthesis, Properties and Applications*, Taylor & Francis, New York, 1996.
- [2] e.g. M. Gell, E.H. Jordan, Y.H. Sohn, D. Goberman, L. Shaw and T.D. Xiao. *Surf. Coat. Technol.*, 146-147, 48-54 (2001).

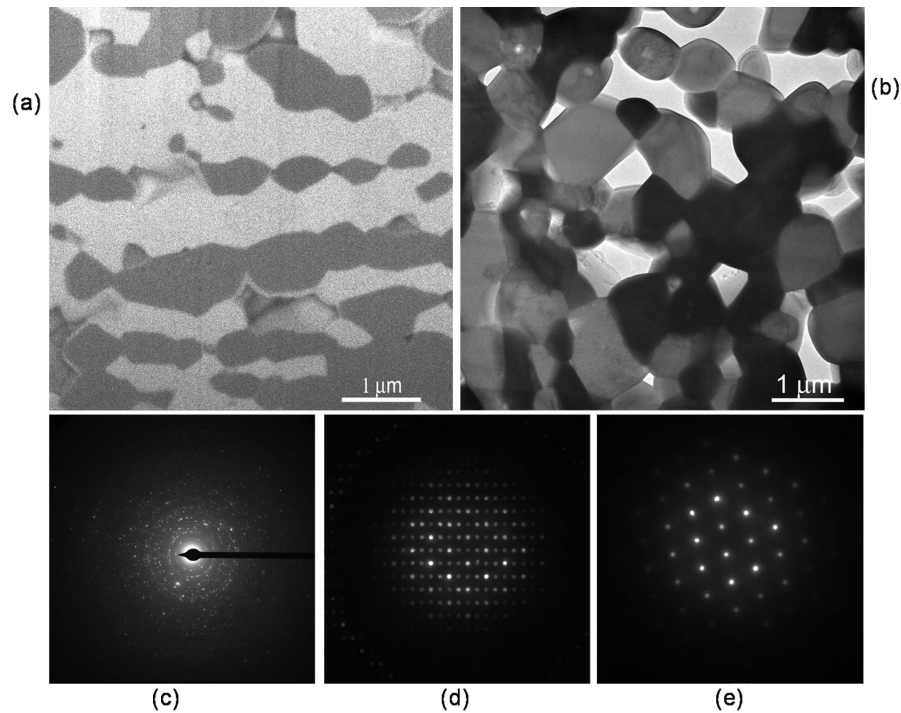


Fig. 1. Microstructural data from the $\text{Y}_2\text{O}_3\text{-MgO}$ composite made using the YnMn precursor mixture. (a) BSE SEM image of a FIB-milled section; (b) BF TEM image from a FIB-cut thin section; (c)-(e) diffraction data from the section in (b); (c) SADP; (d)&(e) microdiffraction patterns from $[011]$ Y_2O_3 and $[110]$ MgO zone axes, respectively.

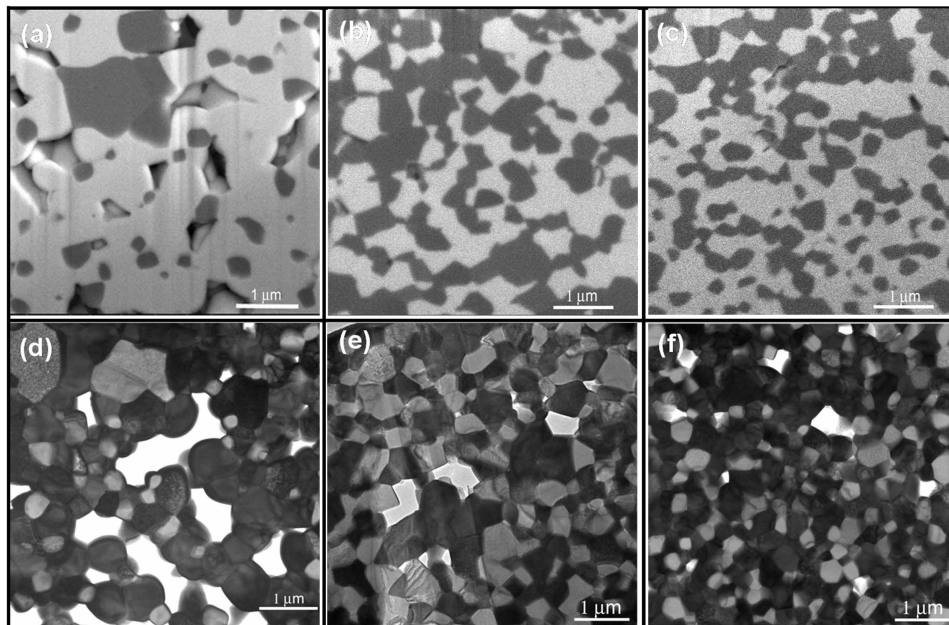


Fig. 2. Microstructural data from the $\text{Y}_2\text{O}_3\text{-MgO}$ composites made using the other three precursor mixtures. (a), (b), (c) BSE SEM images of FIB-milled sections and (d), (e), (f) BF TEM images from FIB-cut thin sections for composites made from the YaMa, YaMn and YnMa precursor mixtures, respectively.