DOI: 10.1017/S1431927603440816
Microsc Microanal 9(Suppl 2), 2003
Copyright 2003 Microscopy Society of America

Characterization of Porous 95/5 PZT Using Quantitative Image Analysis

D.F. Susan, A.C. Kilgo, C.S. Watson, and R.H. Moore

Sandia National Laboratories, Albuquerque, NM 87185

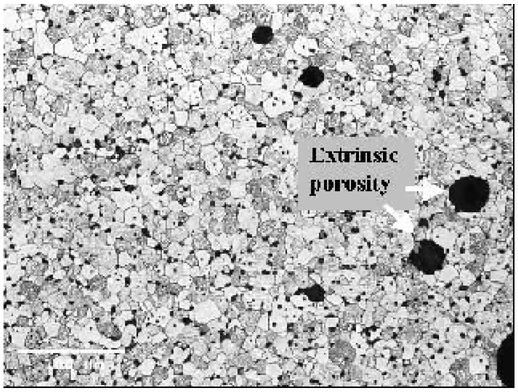
Porous 95/5 lead zirconate titanate (PZT), a ferroelectric material used in power supply applications, has a complex microstructure containing the following features (Figure 1): PZT grains, ferroelectric domains within the PZT grains, "intrinsic" (sintering) porosity, second phase particles such as zirconia (ZrO_2), "extrinsic" porosity, and other defects. To produce extrinsic pores, polymer spheres (PMMA or polystyrene) are added to the green body and burned out during sintering to reduce the density. The amount and morphology of pores and other constituents have important effects on mechanical and electrical properties. In this study, quantitative image analysis (QIA) techniques were used to characterize the structure of PZT materials. The parameters analyzed included PZT grain size, volume percent of intrinsic and extrinsic porosity, and pore size distribution. The QIA results show good agreement with independent density and particle size measurements. Effects of processing on microstructure were investigated with QIA and possible effects are discussed.

Figure 2 shows grain size histograms from five PZT samples final sintered at 1350°C. The differences in grain size between samples A-C and samples D and E were found to be statistically significant. Further analysis showed that larger grain size in samples D and E corresponds to higher lead content. Figure 3 displays results for measurements of porosity content. PMMA pore-former was added at two different loading levels of 0.9 wt.% and 1.38 wt.%, corresponding to theoretical volume percent levels of ~5.8% and ~8.8%, respectively. QIA was successful at differentiating between these two extrinsic porosity levels. When polystyrene is added (with lower density than PMMA) on a wt. % basis, QIA is able to distinguish the corresponding higher volume percent. Density can be estimated based on the combined amounts of intrinsic and extrinsic porosity and fairly good agreement was found between density values calculated from QIA data and results obtained by bulk density measurements.

Figure 4 exhibits extrinsic pore size distributions obtained by QIA for PZT with five different pore-former classes. Since the pores are spherical, the Schwartz-Saltykov correction was performed to correct the raw data for 2-D sectioning effects [1-4]. Comparisons to independent particle size analysis of the starting PMMA powders indicate pore shrinkage during sintering. Current work is underway to determine if the amount of pore shrinkage is consistent with theoretical predictions. The QIA results of this study, considering the inhomogeneity of the microstructure and the limited sampling of polished cross-sections, show the usefulness of this technique for understanding processing-microstructure relationships in complex PZT materials.

References
[1] S.A. Saltykov, Stereometric Metallography, 2nd Ed., Metallurgizdat, Moscow, 1958, pg. 446.
[2] E.E. Underwood, Chpt. 6 - Quantitative Microscopy by DeHoff and Rhines, McGraw-Hill, 1968.
[3] J.C. Russ, Computer-Assisted Microscopy, pp. 233-239, Plenum Press, 1990.
[4] J. Takahashi and H. Suito, *Met. Mat. Trans. A*, 34A, pp. 171-181, Jan. 2003.
[5] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract No. DE-AC04-94AL85000.

https://doi.org/10.1017/S1431927603440816 Published online by Cambridge University Press



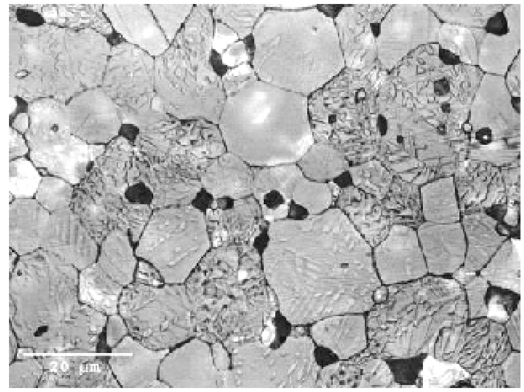
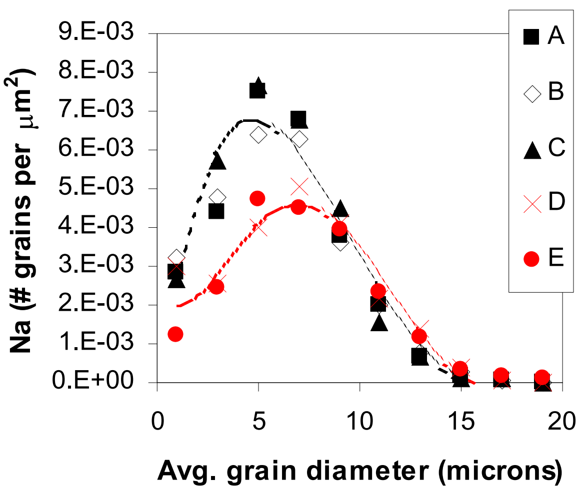


FIG. 1a. Etched microstructure of PZT with extrinsic pores (original magnification 100x).
FIG. 1b. Higher magnification image of grain structure and intrinsic porosity. Sample was more deeply etched to show domain structure within the grains (original magnification 1000 times).



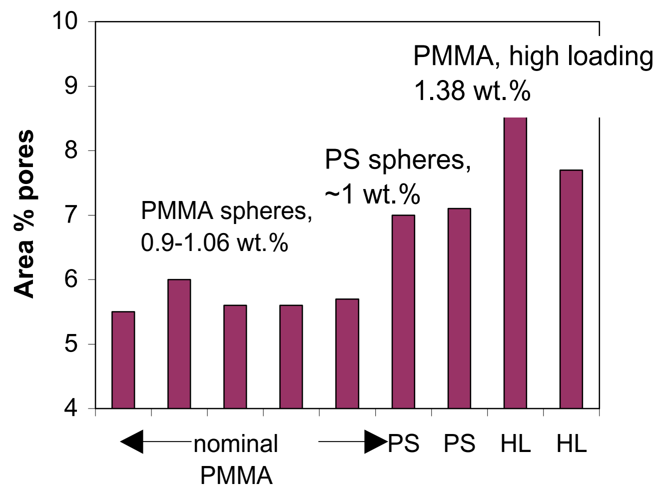


FIG. 2. Grain size histograms for PZT samples sintered at 1350°C.
FIG. 3. Volume percent of extrinsic porosity in PZT with different processing conditions.

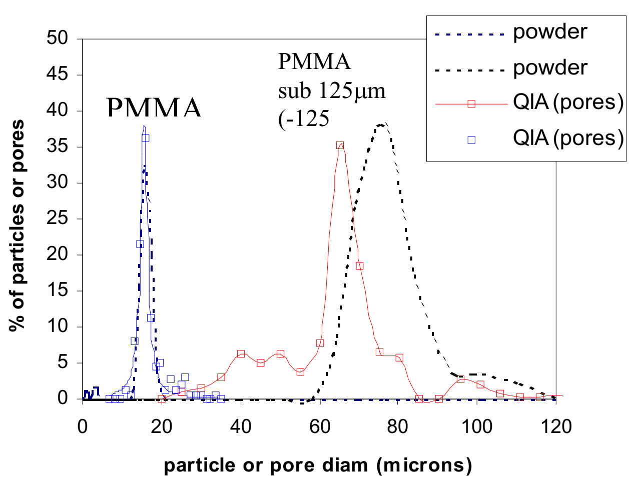


FIG. 4. Pore size distributions obtained by QIA and original pore-former particle distributions obtained by laser diffraction particle size analyzer.

https://doi.org/10.1017/S1431927603440816 Published online by Cambridge University Press