Control of PbO Loss During Sintering of PZT: Laboratory vs Industry

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The relatively low melting point of lead oxide (approx. 900 °C) has always presented an issue in the processing of lead zirconium titanate (PZT) piezoelectric ceramics. The loss of PbO at high sintering temperatures (up to 1300 °C) can cause undesirable changes in stoichiometry, phase composition and electrical properties of the final ceramic product. In high-volume production, the PbO loss per piece is low and, with a small excess of lead oxide in the initial powder composition, it is usually sufficient to keep sintered samples in enclosed crucibles. Small-scale lab processing requires better atmosphere control, usually implemented by surrounding the sample in a lead oxide-containing powder bed. Such control is required in order to prepare samples for detailed composition- microstructure- property studies.

In this work, a typical industrial sintering program with slow heating rate and long dwell time was used to sinter hard PZT samples (NCE40 supplied by Noliac) at 1260 °C in a laboratory furnace. It was found that conventionally used powder beds such as PZT or $PbZrO_3$ mixed in different ratios with ZrO_2 were either difficult to separate from the crucible/samples or not able to sufficiently prevent the weight loss of the samples. Excessive PbO loss was indicated by the presence of ZrO_2 secondary phase in sintered samples using the scanning electron microscope. Weight loss of individual samples, and their resulting electrical properties, varied depending on the composition and particle size of the powder bed. An alternative powder bed consisting of ZrO_2 sand reacted with PbO was found to sufficiently reduce the PbO loss in the samples (no secondary phase detected) while being easily separated from both the samples and crucible after sintering, and maintaining good piezoelectric properties in the sintered samples.

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