Piezoelectric Ceramics

AN212

The particle size of piezoelectric ceramics plays a crucial role in calcination and sintering temperature, which affects the processing time, and, subsequently, the performance of the final pressed component. It is for this reason that particle size analysis is used at different stages of the manufacturing process; beginning with the raw materials through the final stage of the creation of a spray-dried powder. The LA-960 is ideally suited for tracking this synthesis.

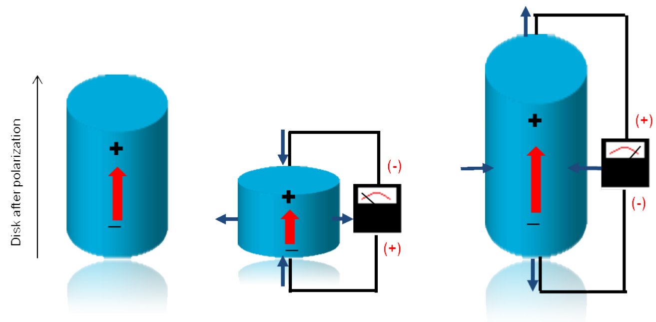

Figure 1. Cyclic expansion and contraction of piezoelectric material

Introduction

In 1880, French physicists Jacques and Pierre Curie* discovered that mechanical stress generates electrical energy in naturally occurring crystals such as quartz and tourmaline. They called this phenomenon the "piezoelectric effect." Conversely, when an electric field is imposed, the piezoelectric material deforms. The Curie brothers called this the "inverse piezoelectric effect." If an alternating charge is applied, cyclical expansion and contraction then follows (Figure 1). The magnitude of this dimensional change is proportional to the magnitude of the electrical energy. This unique ability makes piezoelectric material useful for various applications such as Dry Powder Inhalers (DPI), SONAR, ultrasonography, Tire Pressure Monitoring Systems (TPMS), barbecue igniters, and many more. This unique ability continues to prompt scientists to explore various synthesis routes and fabrication processing techniques for piezoelectric ceramic powders such as:

- Lead Zirconate Titanate – $Pb(Zr_{1-x}Ti_x)O_3$
- Barium Titanate – $BaTiO_3$
- Sodium Bismuth Titanate – $Na_{0.5}Bi_{0.5}TiO_3$ (NBT)
- Bismuth ferrite – $BiFeO_3$

Of these, the most studied and widely used material for this fabrication is lead zirconate titanate (PZT).

PZT Processing

PZT is often fabricated by a solid-state reaction among PbO, TiO_2 and ZrO_2. And by appropriate adjustment of the zirconate-titanate ratio, PZT properties can be optimized for different applications. In other words, the efficiency of electrical to mechanical energy conversion and vice versa is dependent of the ratio of zirconate to titanate. The shape of the final pressed PZT (Figure 2) also affects the net energy conversion as each physical dimension determines its own electrical resonances. For instance, a disc will exhibit radial and thickness resonance, where as a rod will have length, width, and thickness resonances.

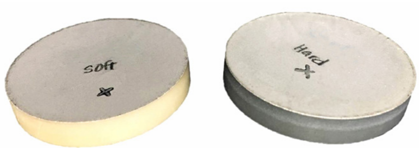

Figure 2. Final pressed soft and hard PZT discs

The Importance of Particle Size

While PZT processing through a solid-state reaction is relatively simple and inexpensive, it typically leads to particle aggregation and poor powder homogenity. Particle aggregation in turn, leads to high calcination and sintering temperature and time, which raises the overall cost of the PZT production. The presence of large particles also lowers the compaction strength of the pressed component and ultimately leads to lower performance of the final piezoelectric product. To mitigate this issue, particle size is monitored and controlled throughout PZT synthesis (Figure 3, below) with emphasis on analysis at the raw material inspection, mixing, milling, and spray-drying PZT powder steps.

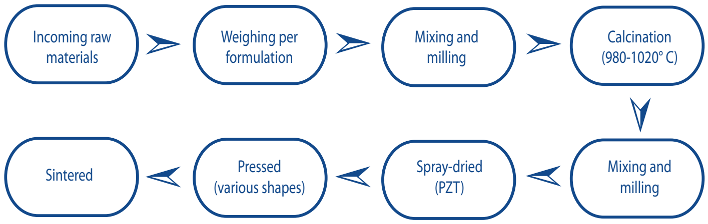

Figure 3. PZT processing steps

Measurement Examples

The Partica LA-960 Laser Diffraction Particle Size Analyzer, with a dynamic size range from 10 nm to 5 mm, was used in this study. Mie theory was selected to offer better accuracy when converting angular scattering data collected from the laser diffraction system into particle size distribution. Unlike the Fraunhofer approximation, the applicability of Mie theory is not limited to small angle forward scattering or particles larger than 50 µm. For this reason, choosing appropriate refractive indices for the particulate and the medium is important [1].

Two example raw materials [2] and two PZT powders were measured in 0.1 wt % aqueous sodium pyrophosphate solution (Darvan C is another commonly used surfactant).

Lead accounts for approximately 60 weight percent of PZT [3]. Since lead oxide is a hazardous material with a rather high vapor pressure at 980-1020°C (calcination temperations), lead may volatilize and change the product composition. It is, therefore, desired that the starting powder is less than 10 µm diameter with a medium size of 1-2 µm. A real refrative index of 2.20 and imaginary value of 1i was used here after investing the influence of the optical modal with the Method Expert wizard in HORIBA's LA-960 software. In this case, lead II oxide will need to be milled before processing.

An overlay of the particle size distirbution of soft and hard PZT (refractive index 2.32-1i) is displayed here with the soft PZT exhibiting a bi-modal size distribution. The final PZT is typically milled and spray-dried to less than 10 µm with a median size of 1-3 µm. The goal is to achieve single modal narrow distribution since a broad particle size distribution often indicates low powder homogeneity and purity. That brings the validity of the measured soft PZT bi-modal distribution into question. To confirm these results, the PSA-300 static imaging analysis system was used [1] (Figure 6). While optical microscopy was not able identify particle sizes lower than 0.5 µm, the image captured distinctively showed the existence of two populations.

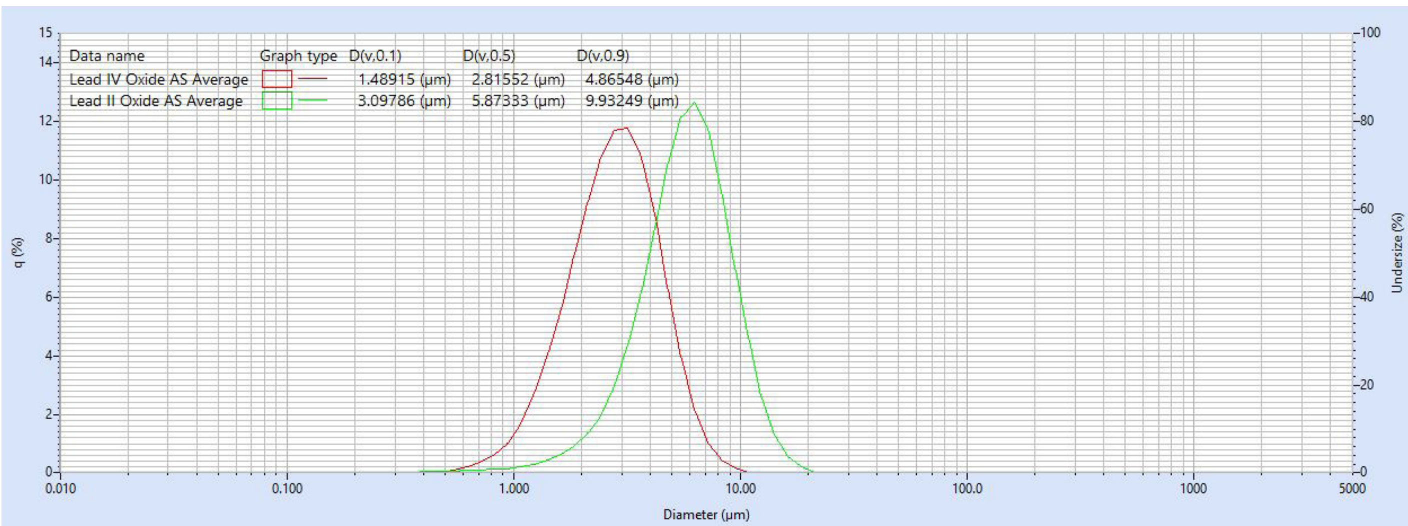

Figure 4. An overlay of passing and non-passing lead oxides are shown here using lead IV and lead II oxides as examples, respectively.

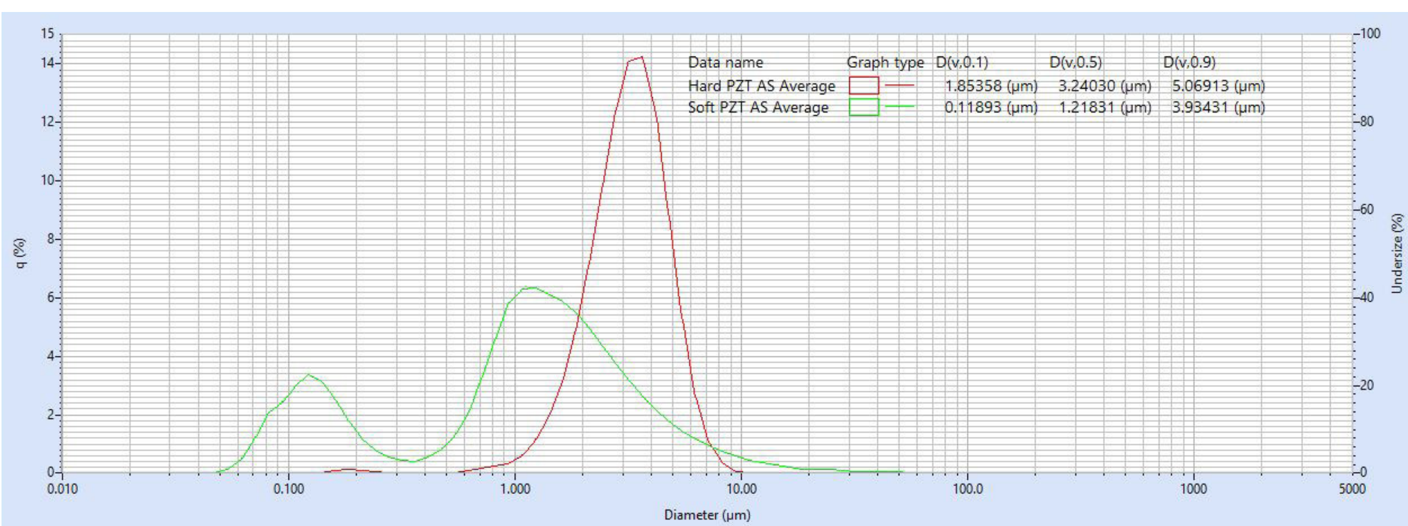

Figure 5. An overlay of soft and hard PZT with hard PZT displaying a single modal distribution, typically preferred for further pressing of final PZT component.

Figure 6. Static image analysis of the soft PZT sample.

Conclusions

This application note shows how the laser diffraction technique can be effectively used as an inspection tool for the synthesis of piezoelectric ceramics. While PZT is used as an example in this paper, the manufacturing of many other lead-free piezoelectric components are also highly dependent on particle size. The LA-960 can assist in the development and quality control of the process by rapidly determining the particle size to guide the manufacturing process, thereby, reducing production costs.

** Pierre Curie's wife, Marie Curie discovered radioactivity. She made history by being the first woman to win Nobel Prize. Together, the Curie family took home five Nobel Prizes. That made history too.*

References

1. ISO 13320 (2009) Particle size analysis - Laser diffraction methods.
2. Sigma Aldrich lead oxides.
3. Elena Aksel and Jacob L. Jones "Advances in Lead-Free Piezoelectric Materials for Sensors and Actuators" Sensors 2010, pp 1-2.