Elemental Analysis of Nanosized Glass Powders for Industrial Applications



Application Note

The RT100-B LIBS system overcomes the analytical challenges of nanosized industrial glass particles.

Summary

Nanomaterials possess unique physical, chemical and biological properties. When reduced to the nanometer scale, these particles can exhibit properties not expressed in the bulk material. These new properties can add value to products such as consumer goods, but they can also cause unwanted changes.

In this application note, we describe the use of Laser Induced Breakdown Spectroscopy (LIBS) for the rapid elemental analysis of industrial-grade nanosized glass powders, using Applied Spectra's RT100 LIBS series instrument.

The RT100-B identified differences in the intensity of critical elements between pre- and post-treated powders. These changes explained the altered physical appearance of the powders which occurred during processing.

Introduction

The use of nanoparticles in industrial applica-

tions is a rapidly growing, multi-billion dollar industry. These highly useful materials can add valuable properties to many consumer products, from personal care items, such as cosmetics, shampoos, deodorants and sunscreens, to high-performance lubricants and glass windshields. Engineered nanomaterials can exhibit unique physical, chemical, and biological properties due to their size.

For example, silver nanoparticles in deodorants and plastic food storage containers have antimicrobial properties. Nanoparticles of titanium dioxide can make sunscreens transparent when applied to the skin, and a coating of nanoparticles on glass can repel water and dirt from windshields and skyscraper windows. These characteristics are exhibited by nanoparticles, but not bulk materials.

Industrial-grade glass and glass nanoparticles must meet specific performance specifications, which are influenced by elemental content and purity levels. During glass manufacturing, physical and chemical changes can occur

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in the material, thus altering important material properties such as clarity, strength and purity. This is especially true for nanosized glasses. When reduced to the nanometer scale, particles can become highly reactive due to the large relative increase in surface area that accompanies miniaturization. These materials can undergo chemical reactions to form undesirable compounds when in contact with contaminants.

Glasses are notoriously difficult substances to analyze. They are not readily digestible by acids; they are heat resistant and insoluble, which makes the determination of their elemental composition difficult.

The RT100 series LIBS system was used to overcome these analytical challenges. Using this system, the elemental composition of processed glass powders and unprocessed glass powders was easily identified. These elemental differences were used to determine that the chemical composition of the different samples of nanoglass powders could be linked to the treatment of materials during processing.

Operating parameters

- Sample: Glass nanopowders
- Laser spot size: 5 micron
- Laser Wavelength: 266 nm
- Laser power:
 - 3 mJ
 - 1.5 Hz
 - 10 laser pulses per spectrum.

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Figure 1. Applied Spectra's RT100 Series LIBS instrument

Process

Nanosized glass powders were analyzed using the RT100-B LIBS instrument (Figure 1). Six different samples were analyzed for this study. Samples labeled as "R" were raw glass powders prior to material processing steps (pre-processing). The samples labeled as "L" were taken after material processing steps (post-processing). Each of the "R" and "L" samples was heated to three process temperatures of 200 °C, 250 °C or 350 °C. The samples were pressed into disc pellets under 9 tons of pressure (Figure 2).

These discs were placed in the sampling chamber and five locations on each sample were analyzed. Each spectrum was a collection of 10 laser pulses which improved the signal-to-noise ratio (Figure 5).

Results

Si, B, Ba, and K were the primary elements of interest in this LIBS study (Figure 3). In, Sr, Al, Y and Ga were also observed. The primary elements were used to distinguish between the post-processed and unprocessed nanosized powder glass materials heated to different temperatures.

The post-processed glass powders (L group) that were heated at 250 °C, exhibited a barium (Ba) signal intensity (@ 455.4 nm) that was significantly different from the samples heated at 200 °C or 350 °C (Figure 4). The unprocessed glass powders (R group) did not show any significant difference in the barium signal intensity. As seen from Figure 3, barium ionic line intensity at 455.4 nm was the maximum of all emission lines and Ba (II) at 455.4 nm and was used to normalize other emission lines.

A significant change in the L/R ratio was observed for elements such as Si. The normalized line intensity of other main constituent elements such as K and B showed no significant differences between L and R group at three different processing temperatures, indicated

Figure 2. Pellets of nanosized glass powders, prepared by pressing under 9 tons of pressure: (a) post-processed nanoglass (L); (b) pre-processed nanoglass (R).



(b)



by the normalized intensity ratio of close to 1. This suggests that some elements are more affected by processing conditions than other elements.

Conclusions

LIBS spectra collected using the RT100-B can determine important differences between processed and unprocessed nanosized glass powders in different industrial applications. Changes that occur during processing can be readily detected with the RT100-B. Raw materials can also be analyzed for contaminants with the RT100-B LIBS system.

Thus, manufacturers can save both time and money by preventing contaminated or undesirable raw materials from entering the manufacturing process. If unusual characteristics appear, LIBS can be used to pinpoint when contaminants entered the manufacturing stream and processing procedures can be assessed with this system. These differences can verify changes in glass quality that will make the difference between glass that meets strict specifications and glass which fails to meet specific standards

Figure 3. Representative LIBS spectra of nanosized glass powers.



Figure 4. LIBS emission line intensity of Ba at 455.4 nm in arbitrary units (A.U.):

(a) pre-processed (R group) nanosized glass powder at three different temperatures, $% \left({R_{\rm g}} \right) = 0$

(b) post-processed (L group) nanosized glass powder at three different temperatures, and

(c) Ratio of Ba LIBS intensity between L to R samples.









Figure 5. Photomicrograph and spectrum of glass nanopowder disc using Applied Spectra's Axiom software package.



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