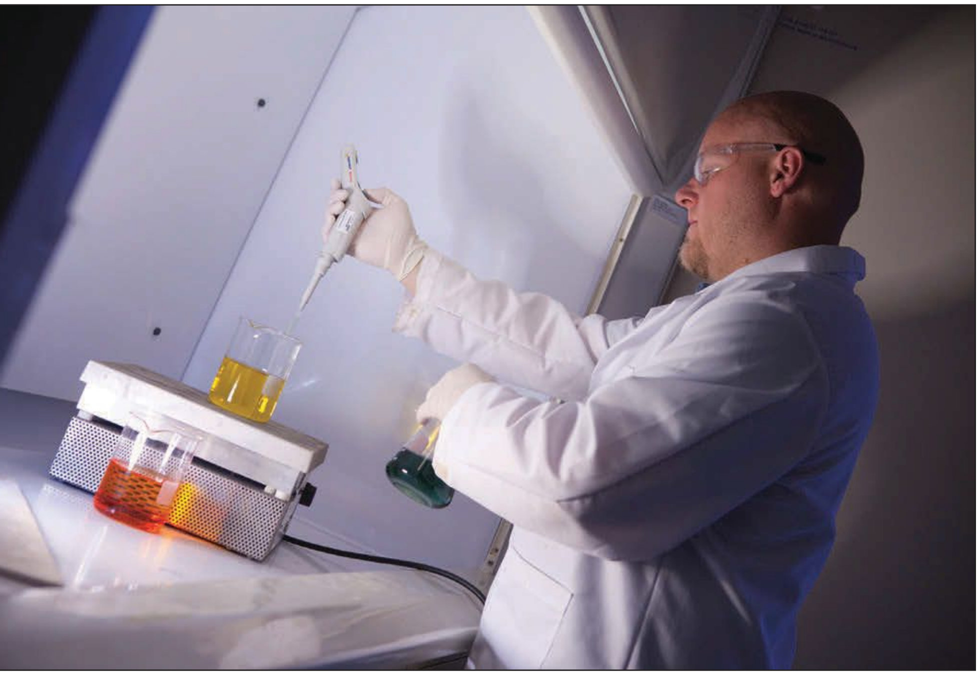
Overcoming Analytical Challenges for Ceramic Materials

An experienced testing lab can identify the right technique for ceramics analysis and provide results that help boost confidence in a material's quality.



Scientists use standard methods or develop new approaches for breaking down and preparing many common ceramic materials for analysis.

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Performance is critical for today's advanced applications of ceramics. Whether they are being used as electrical insulators or for thermal protection on a spacecraft, the failure of ceramic components can have serious, even life-threatening consequences.

One source of failures for ceramics is a mismatch between a material's actual composition and the expected specifications. A material's particle size, precise chemical composition, and the presence of any trace elements/compounds are all tied closely with performance. For this reason, both manufacturers of ceramic materials and those who use them need to know the exact make-up of their materials.

A trusted testing lab can provide tailored analysis that not only boosts confidence in a ceramic's quality, but can also be used to identify the source of a product failure or problem. Many analytical chemistry techniques (each with different benefits and drawbacks) are available for bulk and trace analyses of ceramic materials.

Elements commonly requested for trace analysis include sodium, magnesium, potassium, calcium, iron, zinc, selenium, silver, tin, antimony, lead and bismuth. Bulk analysis can determine the percent composition of an oxide such as alumina, silica, zirconia, and others (e.g., sodium oxide, magnesium oxide, potassium oxide, calcium oxide, titanium dioxide, chromium oxide, manganese oxide, iron oxide, zinc oxide, and hafnium (IV) oxide). In addition, a particle size analyzer can be used to measure particle size distribution for powdered ceramics.

Breaking Down Ceramics

The very characteristics that make ceramics ideal for harsh environments and demanding applications—resistance to corrosion and high temperatures—make it challenging to prepare ceramic materials for analysis. For standard metal alloys, preparation usually involves breaking down a sample by adding a combination of acids and then digesting the material via hot plate. However, most ceramic materials are highly resistant to acids, meaning that more aggressive methods are required to prepare ceramics for analysis.

Because of the challenges surrounding ceramic analysis, it is key to use a testing lab with scientists who are experienced in analyzing a variety of materials and that has invested in multiple analytical techniques and instruments. Scientists who conduct analyses every day can determine the best balance between sampling technique/preparation method and analytical technique to arrive at the desired level of detection for a given element/compound in a material.

At a testing lab, scientists use standard methods or develop new approaches for breaking down and preparing many common ceramic materials for analysis, such as silica, alumina, zirconia, titanium dioxide, zinc oxide, magnesium oxide, chrome oxide, and mixtures of two or more oxide bases. Analysis of new mixtures requires identifying potential analysis pitfalls and selecting the sampling technique and instrumentation that will produce the very best result.

Trace Analysis with DC Arc-AES

Direct current arc (DC Arc) atomic emission spectrometry (AES) has long been the primary method for trace analysis of elements in ceramic products. It offers quick trace detection in the range of 0.1-5.0 ppm (parts per million.) One benefit of this method is that it analyzes the material directly in its native form, making it less likely that contamination might be introduced into the sample using other preparation methods.

For DC Arc analysis, the ceramic material must be pulverized and blended to form a powder. Because this technique uses a very small sample size (only 10 mg of material), it is important that the sample be very representative of the overall material. Careful preparation is required to obtain the proper particle size and homogeneity so as to not skew the analysis results.

For materials that are particularly challenging to break down or when contamination is a primary concern, DC Arc may be the best—or in some cases, the only—way to obtain results. While DC Arc instruments continue to be replaced with other techniques that offer potentially higher accuracy and repeatability of results, the DC Arc still produces phenomenal results for almost any material.

ICP-AES for Bulk Analysis and ICP-MS for Trace Analysis

Inductively coupled plasma (ICP) approaches are newer options that are useful for trace and bulk analysis of those ceramic materials that can be converted into a solution for analysis. Inductively coupled plasma atomic emission spectrometry (ICP-AES) is effective for higher/bulk compositions from around 0.05-50 wt%, while inductively coupled plasma mass spectrometry (ICP-MS), although not applicable to all materials, can provide trace level analysis down to near 0.001 wt% and lower. These techniques can be used together when both bulk and trace analyses are desired.

One advantage of ICP-MS for trace analysis is that it uses a much higher sample mass (usually about 0.5 g of sample), which will be more representative of the original material than the 10 mg used for DC Arc analysis. However, unlike DC Arc, ICP-MS requires the ceramic to be dissolved into a solution without introducing contamination or elemental interferences with the instrument. For some materials, this isn't possible.

ICP sample preparation can involve using a fusion technique. For this approach, a powder (or flux) is mixed with the material and heated to around 1,000°C, which melts the flux and breaks down the sample. The resulting material forms a crystal that can be extracted and converted into a solution for analysis. While this type of sample preparation is useful for hard-to-digest ceramic samples, the addition of flux for fusions may result in slightly elevated levels of detection for some elements.

A variety of analytical chemistry techniques is available for bulk and trace analyses of ceramic materials.

New sampling/sample preparation techniques have been developed using a dedicated microwave system that provides a high-temperature, high-pressure closed system. The microwave is used to heat ceramics to extremely high temperatures for an extended period of time, which facilitates the breaking of the strong chemical bonds present in ceramics so that the sample can be converted into a solution. This method avoids some of the impurities that can be introduced when using a fusion preparation.

Rapid Analysis with XRF

Although most ceramic materials can be analyzed with DC Arc or ICP methods, X-ray fluorescence (XRF) offers another option. For ceramics, this approach provides faster, more cost-effective bulk and trace analysis of concentration ranges from 1 ppm and higher.

The very characteristics that make ceramics ideal for harsh environments and demanding applications make it challenging to prepare ceramic materials for analysis.

For XRF trace analysis, ceramic materials are prepared as pressed pellets by crushing or grinding the material to particle sizes less than 50 microns. Typically, 12 g of the resulting sample is then mixed with 3 g of a cellulose or wax binder. Pressing the material and binder under high pressure forms a stable pellet used for the analysis.

For XRF bulk analysis of compositions of about 0.05 wt% and more, ceramic materials are prepared as glass beads. This process involves crushing the material to a particle size of less than 250 microns and then mixing 1 g of the powder with 10 g of a flux such as lithium metaborate or lithium tetraborate. A fusion/fluxer instrument is then used to create a glass bead from the material and flux, and the bead is then analyzed using XRF.

The XRF technique features relatively simple sample preparation and shorter measurement times, yet it requires matrix-matched standards and reference materials for the most accurate results. While some instrument manufacturers offer additional software that provides users with a general calibration that may be applied to a variety of materials, XRF is most accurate when used to analyze commonly encountered materials or “project-specific” materials that are well calibrated, rather than a range of materials.

Careful Consideration

Whether conducting bulk or trace analysis, ceramic materials come with a variety of challenges that require careful consideration to determine the best sampling and analysis technique for a given material. New ceramic blends continue to be developed to meet the needs of next-generation applications such as additive manufacturing and the expanding hybrid and electric automobile market, and these new blends will each come with their own set of analysis challenges. Using a testing lab that is experienced in many sample preparation methods and that has invested in the very latest analyses techniques can ensure accurate, timely, and useful analyses results for even the most challenging ceramic materials. CI

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