

Pressing Ceramic Sample Discs

Sample discs of ceramic materials can be used for X-ray fluorescence (XRF) analysis, infrared spectroscopy and optical emissions spectroscopy.

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Ceramic sample preparation is a technique that involves the formation of a solid pellet of material for analysis through methods that require flat, round samples known as sample discs. The sample discs are either produced by fusion of the sample with salt or through pressing in a press and die. Molds control the size of the sample discs.

Sample discs can be used for X-ray fluorescence (XRF) analysis, infrared spectroscopy and optical emissions spectroscopy. XRF discs do not require a binder or holder if the sample coheres under pressure. The prime requirement for an XRF binder is that it does not contribute impurities.

Pressing results in a sample disc that holds together and has a flat, compositionally uniform surface. The disc is then placed in the sample holder of the XRF spectrometer. Running a series of standards or known samples helps to confirm that the chosen procedure



Standard 12-ton benchtop press.

results in accurate, reproducible data.

In contrast to loose powder, a sample disc has the advantage that the element concentration detected by the X-ray is higher because the material is more compact. In addition, a smooth surface is preferable to a rough one from an optical point of view.

Pressing Details

Pressing a sample disc is the most common procedure for many applications. Generally, a pressed disc should fulfill the following quality criteria:

- Homogeneous
 - Absolutely solid to avoid loose particles
 - Stable and storable
- Benchtop hydraulic presses are commonly used to prepare ceramic sample discs. Laboratories around the world use these presses for materials research, quality and performance testing of physical properties, lab testing, laminating, and many other applications. More specifically, benchtop hydraulic presses are used to

process ceramics, composites, construction materials, cosmetics, pharmaceuticals, powder metals, printed circuit boards, rubber, silicone and other elastomers, soil, thermoplastic resins, and thermosets.

Compacting sample materials into discs is achieved through the use of a test cylinder outfit (TCO). The TCO comprises a cylinder, plunger, base plug and an ejector. Before the sample process begins, it is important that a proper plate kit assembly is acquired. A KBr buffer plate and 6 x 6-in. single hardened steel platen with centering grooves is recommended for use with pellet dies and TCOs. A hardened plate kit that mounts to the top bolster and a 90-mm diameter is generally best. A 6 x 6-in. hardened steel plate with centering grooves should then be mounted to a moving bolster on top of the ram. Centering the die and TCO are important for operator safety.

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After the proper press structure is in place, more information is required in order for the TCO to turn a ceramic material into a sample disc. Important factors to consider include the finished compacted dimensions of the desired disc (diameter x height), the material compaction ratio or loose fill height required in the cylinder, mate-

rial particle size and hardness, and the specific compacting pressure required on the material. The chemical reaction in coordination with corrosion issues must also be taken into consideration.

It is important to keep in mind that fine material particles migrating up between the plunger and the cylinder can cause stress concentration in the cylinder wall. It is critical to thoroughly clean the TCO after each pressing.

A hydraulic press is used to compact the material in the TCO. The press used in the sampling process can be manually actuated (hand pump) or automated (electric motor and pump with push button close). As discussed previously, the press should be equipped with a hardened plate kit to ensure mechanical integrity and proper die alignment. **C**

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