SCHOTT/Nicola O'Sullivan

The X-ray fluorescence (XRF) spectrometer can hold nearly 150 glass samples to characterize main and secondary constituents.

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Analyses Further **Progress**

SCHOTT relies on state-of-the-art analytical methods for quality assurance and the development of new products. This expertise is concentrated in the Analysis and Measurement Service (AMS) of Research & Development.

Classic wet-chemical analytical methods ensure the reliable characterization of the main, secondary and trace constituents in glass, glass ceramics and raw materials. As the solid test samples first have to be crushed, ground, screened and ultimately rendered soluable with acids or alkaline melts, testing processes are time-consuming and cost-intensive. If disturbances in the characterization occur because of further matrix elements or if the concentrations of the elements are too low for detection, additional separation and enrichment steps are necessary.

Modern solid-state analytical methods offer an alternative to classic wet-chemical processes. They allow a fast characterization of the composition of the glasses.

Glass standards are indispensable

A pioneering technology in the field of solid-state methods is the X-ray fluorescence analysis (XRF). This analytical method has been a routine procedure at SCHOTT for more than 30 years.

Standard materials in which the element concentrations are exactly measured are indispensable for the quantitative characteriaztion of the constituents. Since purchasable compositions are rarely comparable to SCHOTT special glasses, the company has developed specific glass standards in its own facilities that are carefully analyzed in Mainz using classic wet-chemical methods. "Thanks to the systematic recording of glass samples and their results, we have amassed an extensive database with whose help we can even characterize exotic glasses," explains Lothar Meckel, Head of Chemical Analysis.

The sample preparation is quite simple: specimens are cut out of the samples with a drill. The surface is then ground and pol-

X-ray fluorescence analysis

XRF functions as follows: highly energetic X-rays knock photoelectrons out of the inner shell of the atoms in the glass samples. These electrons leave behind unstable vacancies. Electrons from the outer shell, which have a higher energy level, fill these vacancies. Excess energy in the form of secondary X-ray photons are thus released. The emitted X-ray fluorescence radiation is decomposed by the diffraction on crystals. The wavelengths of this radiation are characteristic for the elements in the sample and for their atomic number. The intensity of the radiation is proportional to the concentration of the element in the sample.

ished. The results are available shortly after the sample arrives. The accuracy and precision of the measuring results are comparable to those obtained with wet chemistry. The measurable range of concentration for XRF extends to values of 50 mg/kg.

However, these methods have their limitations with lightweight elements, which are either impossible to characterize or cannot be characterized with the necessary precision. Glass powders and materials from which it is not always possible to obtain suitable test specimens are also problematic.

Fast and exact detection

Laser ablation-inductive coupled plasma mass spectroscopy (LA-ICP-MS) can be used as a supplement to characterize secondary constituents, traces and ultratraces. This method also requires carefully analyzed standard materials, which have been characterized beforehand with classic wet-chemical techniques. The decisive advantages of LA-ICP-MS are that it is highly exact (down to 50 µg/kg), fast and even the tiniest glass splinters can be analyzed without any major preparations.

With the combination of XFR and LA-ICP-MS, SCHOTT specialists are able to characterize all elements in the relevant ranges of concentration in glasses, glass ceramics and other materials. “These analytical methods play an important role in quality assurance and in the development of new products – in the interest of all customers,” stresses Lothar Meckel. ◂

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The classic wet-chemical analysis is indispensable: measuring samples with inductively coupled plasma optical emission spectroscopy (ICP-OES).

Laser ablation-inductive coupled plasma mass spectrometry

With this method, material is taken from a glass sample with the help of a laser. The test material is converted to argon plasma with inert gas. Positively charged ions of the elements are produced at temperatures of around 7,000 degrees Kelvin. These are then separated and identified using mass spectrometry.

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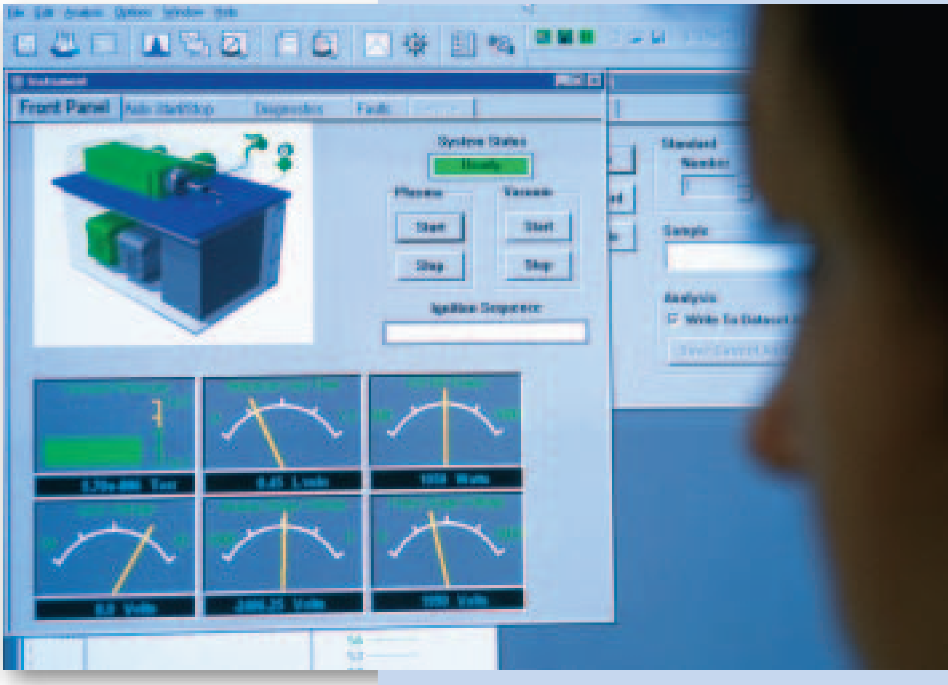

Laser ICP-MS is an analytical method that allows a fast characterization of secondary constituents, traces and ultratraces.