From nanomaterials to the bulk, one instrument to characterize them all

In materials science, characterizing the materials goes along with synthesizing them. One of the best and fastest characterization techniques is X-ray diffraction. This technique has mostly been restricted to well crystallized, homogeneous powders or bulk materials. However, research projects at the Institut NEEL are now focusing on more complex materials, such as nanomaterials, ultrathin films and layers, or striped alloys. These challenging cases, as well as increased demand from our researchers, have called for a dramatic evolution of our instrumentation. In that perspective, acquisition of a state-of-the-art X-ray diffractometer, together with development of new sample holders adapted for very small samples, was becoming essential.

Powder X-ray diffraction, i.e. measuring the diffraction of X-rays for a powder of randomly oriented crystallites, is a powerful but simple method for materials characterization. The diffraction pattern for a given material is usually unique (like DNA for humans) so we can identify known materials with the help of databases. And, for new materials we can “solve” the material’s structure, that is deduce the periodic arrangement of its atoms. However, this technique is usually restricted to well crystallized materials, meaning materials where the atoms are arranged periodically over at least microns range. Furthermore at least a few hundred milligrams of ground powder have been necessary.

But, we are now confronted with “nanomaterials” exhibiting very small crystallite sizes (<50 nm), as well as materials produced by high-pressure synthesis or spray-drying techniques that yield only microgram quantities. We have thin film materials (down to a few nm thickness) deposited on substrates. But we also have bulk samples (textured intermetallic alloys) that are difficult to grind into fine powders without altering the material. For these present-day research topics, “classical” X-ray diffractometers are obsolete: The signal acquired by the X-ray detector is too noisy, too broad, too weak. And longer acquisition times will not resolve the problem, especially as more and more researchers are requesting access.

In response to these new needs, the Institut NEEL has acquired a state-of-the-art X-ray equipment, the Bruker company’s “D8 Endeavor-Eco” diffractometer (Fig. 1). One of this unit’s main features is a fast, “linear”, X-ray detector, a linear array of silicon-pixels pixels extending across the circular powder-diffraction pattern. It can acquire the diffraction data 30 times faster than a classical, stepping, single scintillation detector. And, this new detector can discriminate as a function of the X-ray energy, so it can filter out parasite radiation and/or scattering from the sample, thus reducing background noise.

Furthermore, the instrument has an automatic robotic arm (Fig. 2) that fetches and returns the samples, bringing much faster sample turnaround, especially for unattended functioning overnight. This equipment has reduced the waiting times for access to X-ray characterization from weeks to hours, allowing researchers to optimize their synthesis strategy on a dramatically shorter time scale.

A major improvement has been the development in our laboratory of new sample supports (Fig. 2). Analyzing small amounts of powders was challenging as the contribution from the sample support could dominate the resulting diffractogram. In addition to bulky materials, we can now handle very small amounts of materials using a “zero-background” sample support. This is a “miscut” single crystal silicon wafer, whose crystal axes are orientated such that none of its diffraction spots fall on the detector array. Thereby, weak contributions such as from nanoparticles, can now be measured precisely.

This instrument and the specific developments of these new sample-supports provide our researchers with a versatile, high turnaround instrument fulfilling most of their needs in X-ray characterization of materials. One year after its commissioning, more than 2000 measurements have been performed on the D8 diffractometer by more than 70 research staff and students for characterizing materials ranging from nanopowders to bulk materials.

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