ICAR-CM2T
Ingénierie Matière

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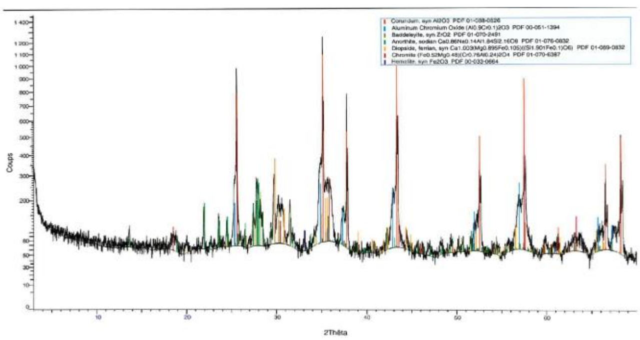
On the testing side: X-ray Diffractometry

X-ray diffractometry is a physico-chemical analysis technique based on the phenomenon of X-ray diffraction on crystalline material (ceramic, metal, etc.).

In the chemical characterisation of crystalline materials, if X-ray fluorescence spectrometry allows the determination of the elemental composition of the analysed sample (Fe, Al...), X-ray diffractometry allows to complete the analysis by determining the nature of the mineralogical phases present. The position of the diffraction lines is characteristic of the phases in the sample. A database lists the positions of the crystalline lines of all the mineralogical phases present: a mathematical treatment of the position of the lines observed on the diffractogram makes it possible to identify the crystalline phases contained in the sample.

Principle:

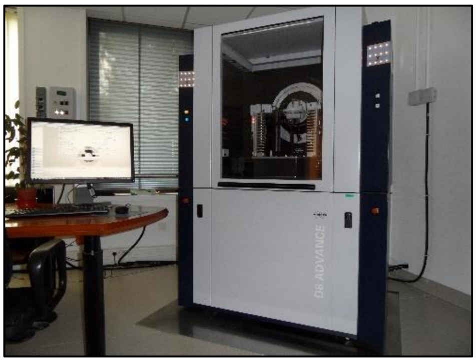
The X-ray beam produced by the tube is sent onto the sample (powder, solid or coating), rotating on itself, where it is deflected by the crystal lattice. The diffracted beam is characterised by the angle of incidence and reflection. The X-ray detector is responsible for collecting the diffracted signal over a wide angular range. The intensity of the peaks will be plotted as a function of the diffraction angle as shown below.



Example of a processed diffractogram

The position of these peaks is a true signature of the arrangement of atoms within a crystal (distance between atoms, between intra-crystal planes). The empirical relationship between the angles at which the peaks are observed and the distances between atomic planes is called Bragg's law.
X-ray diffraction can be used to distinguish between products with the same chemical composition but with different atomic arrangements.
In a mixture, it is possible to determine the nature of each of the crystalline phases present, provided that the signature of each of these phases is known beforehand.

To summarise, X-ray diffraction is based on the recording of a diffractogram and the analysis of the peaks of this diagram, which allows the characterisation of the crystalline phases present in the sample. ICAR-CM2T allows the characterisation and study of samples via its parent company: the Société Française de Céramique.



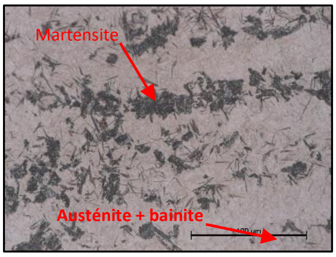
Example of a diffractometer

While the position of the peaks will give information on the identification of the crystalline phases present (qualitative analysis), their intensities, via calibration lines, will allow the quantities of each of the phases to be estimated, or will highlight a preferential orientation of the crystals concerned. The width of the peaks, particularly at mid-height, can provide information on the size and shape of the crystallites and also on the presence of internal stresses.

Examples of the use of this analysis:

- Dosing of quartz in a silica brick for a coking plant:
In this application, many bricks of very different sizes will be made of crystalline silica. When used in service at around 1100°C, it is essential that the starting materials consist of silica in the cristobalite form. If quartz is present in the initial brick, it will transform when heated into cristobalite with a very large increase in volume. This transformation is therefore not at all desired and it is important that the quartz content is as low as possible (<1%). X-ray diffraction will show the absence of quartz (brick conformity).

- Determination of austenite, bainite and martensite content in a metallurgical product before and after mechanical and thermal treatment.
(determination of CCT diagram).➢



- Presence of phase σ in a used refractory steel part and consequent embrittlement.

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-A similar analysis on used, corroded or modified materials can help to understand the phenomenon(s) that lead to the degradation of the refractory material or metal alloy in relation to corrosive agents. If the damage mechanism is highlighted, it will then be possible to make recommendations on the choice of a material that would be less loaded with phases that would tend to react subsequently. These levers make it possible to limit certain wear phenomena and increase the life span of materials operating in extreme conditions.

Corroded cement brick

Bibliography:

This selection of publications comes from Technical Survey made by the SFC (Société Française de Céramique) Documentation department. For more information on these documents of Scientific, Technical and Competitor Intelligence Survey: Monthly Survey reports, target specific Surveys, access to "CeramBase" Survey database, contact SFC to the address: **soc.fr.ceram@ceramique.fr**

SFC

- GHILOTTI D.

Bauxite shortage likely for refractories
Industrial Minerals, n°02, 02/21, pp. 49, 1 fig., ANG.
Consumers of refractory raw materials are likely to face shortages of bauxite due to continuing supply and logistical bottlenecks from China in the face of a low stock situation for destination markets.
Key words : REFRACTORY. MARKET. BAUXITE. TREND.

- VAßEN R., MACK D.E., TANDLER M, ET –AL.

Unique performance of thermal barrier coatings made of yttria-stabilized zirconia at extreme temperature (>1500°C)
Journal of the American Ceramic Society, Vol. 104, n°01, 01/2021, pp. 463-471, 6 fig., bibliography (28 réf.), ANG.
This paper shows that, under cycling conditions, it is not the time of use at elevated temperatures that leads to a reduction in the service life of thermal barrier coatings made of yttria-stabilised zirconia but the transient cooling rates. If the latter are reduced to 10K/s, the coating systems can operate in a plant with burners at temperatures above 1500°C without showing a reduction in service life. Evaluation of the peak energy release rates during rapid transient cooling in combination with the phase evolution during cooling helps to explain these findings.
Key words : COATING. THERMAL BARRIER. YTTRIA STABILIZED ZIRCONIA. HIGH TEMPERATURE. LIFETIME. COOLING.

TRAININGS TO COME (IN FRENCH ONLY)

- **From the 8th to 10th June 2021 in Moncel-les-Lunéville : (STM2) Failure analysis on forming tools : rootcause and solutions – (21h)**
- **From the 16th to 18th June 2021 in Moncel-les-Lunéville : (STR2) Implementation of refractory materials – (18h)**
- **From the 22nd to the 24th September 2021 in Moncel-les-Lunéville : (STR1) Refractory materials : Generalities – (18h)**

- **From the 22nd to the 24th September 2021 in Moncel-les-Lunéville : (STR1) Cast Iron Metallurgy and Applications – (21h)**
- **05Th October 2020 at Moncel-les-Lunéville : (ST4) Thermal calculation (7h)**
- **22 to 24 november 2020 at Moncel-les-Lunéville : (ST3.1) Durability of refractory materials –(18h)**
- **24 to 26 november 2020 at Moncel-les-Lunéville : (ST3.2) Used refractory recycling – (14h)**

And still the possibility to perform intra-company trainings throughout the year on metallic and refractory materials…

For more information…CONTACT US…

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