

New usage for old reactor

The latest measurement instrument of the TU Delft measures the crystal structures of many different materials and is unique within the Netherlands. The so-called Pearl neutron powder diffractometer was opened on 24 September at the RID reactor institute. "It is difficult to overestimate the diversity of applications."

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Lambert van Eijk places a sample in the centre of the diffractometer (right).

Seventy guests active in the field of international neutron research witnessed the first use of the instrument through a video link to the reactor hall. They were shown a concrete container that connects the instrument with the reactor core. They also saw Dr. Lambert van Eijck, who pointed out the semi-circle with the neutron detectors behind it to the camera man. RID director Prof. Bert Wolterbeek then opened the shutter to bombard the sample with neutrons from the reactor core. A graph immediately came up on the screen. Following a slow build-up of radiation, a series of peaks emerged. The audience applauded this convincing demonstration. To experts, the peaks indicate distances between atoms in the crystal grids of the sample. Instead of monocrystals, the sample contains several cubic centimetres of fine crystalline powder. Thanks to a smart method known as the 'Rietveld refinement' researchers can translate the peaks from the diffractometer into distances between atoms in the crystal structure linking them together.

Wider use of the nuclear reactor

The Pearl project (not an acronym) was launched in 2009, when Prof. Katia Pappas received an invitation from Berlin to come to TU Delft to stimulate the wider use of the nuclear reactor in Delft. A neutron diffractometer seemed an ideal application for this purpose, because it enables researchers to use neutron radiation from the reactor to identify the crystal structures of many types of materials. Moreover, this type of instrument did not yet exist in the Netherlands. A year later, a budget of eight hundred thousand Euros had been secured, enabling them to recruit Van Eijck to design and build the instrument. Van Eijck worked closely with the Australian physicist Dr. Leo Cussen to create an instrument that could match others in Europe in terms of sensitivity and resolution, despite the small size of the reactor. In 2012, the final design was completed and construction got under way.

At the focal point of the device is a sample as large as a phalanx bone filled with fine crystalline powder. Neutrons are dispersed through the powder over a wide angle of nearly 180 degrees in a pattern of peaks and troughs forming a fingerprint of the crystal structure. In 1966, the eminent crystallographer Dr. Hugo Rietveld (who was present at the opening and was honoured during the ceremony) developed a method at ECN that he used to establish a link between the crystal structure and the diffraction pattern. This method, known as the 'Rietveld refinement' and used around the world, assumes a certain structure and calculates the diffraction of that structure. If that pattern eventually matches the



The sample is surrounded by a semi-circle containing 1,408 detectors.

measurement - following a number of iterations - the crystal structure will be determined. The sample is surrounded by a semi-circle (11-160 degrees) containing 1408 detectors behind a tight aluminium wall. Every detector is composed of a 2-millimetre wide strip of lithium and zinc sulphide with an optic fibre. When the lithium is struck by a neutron, it will break up into an alpha particle (two protons and

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two neutrons) and a tritium ion (one proton with two neutrons). These particles produce a light flash in the zinc sulphide that is transmitted by the optic fibre to a photodetector, which converts it into an electric pulse. There is one problem, however: the same also happens with incident gamma radiation. Thanks to colleagues from the Rutherford Appleton Laboratory near Oxford, the detector can now differentiate between gamma and neutron flashes, only allowing the latter to pass through. >>

The value of crystal structure knowledge is difficult to overestimate. Without this knowledge, the development of new materials for water storage, fuel cells, magnetic cooling and lithium batteries would be impossible. And this also applies to the development of pharmaceutical materials, coatings for turbine blades and catalysts.

Various researchers at the opening symposium talked about how they improve their materials step by step using the diffractometer. They are given a profile of the material, deduce the crystal structure based on this profile and make modifications to improve performance. The modified material is placed back in the instrument to check if the chemical changes had a positive

effect. This is how they improve their materials step by step. The nearest facilities for neutron diffraction are located in Oxford, Paris and Grenoble. Now, researchers can also come to Delft for this purpose. Despite the relatively low capacity of the reactor (2 megawatts), Pearl can easily match the other centres, according to Van Eijck. He adds that he is still working on the reduction of the background radiation.

The day was concluded with a dinner in the company of the technical directors of various major companies interested in the possibilities of neutron diffraction. <<

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