Delta-d – Comparator for the Si-28 lattice parameter

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PTB is developing measurement systems to determine the lattice parameter of Si-28. The goal is to verify the original value from the Avogadro project and provide a better assessment of the material by measuring a larger set of samples.

1 Introduction

In 2019 the kilogram was re-defined to trace back to natural constants instead of a physical prototype. Though the official definition is based on the Planck constant, the Avogadro constant (N_A), which is linked to the Planck constant via the molar Planck constant, was also updated. The redefinition relied on counting the atoms in a perfect silicon sphere. This required an accurate value of the lattice parameter of enriched Si-28. [1]

The lattice parameter was measured at INRIM. PTB aims to independently verify this value with two systems: *Abs-d* is a combined optical x-ray interferometer (COXI) capable of measuring the absolute (traceable) value directly. *Delta-d* is a relative measurement system to compare the lattice parameters of different samples, which will use a sample measured with abs-d as a reference to achieve traceability.

2 Atom counting and the lattice parameter

Silicon is a crystalline material with a diamond face-centred cubic structure, i.e. it has a highly regular structure consisting of repeated unit cells. The unit cell has length a (the lattice parameter), volume a^3 and contains eight atoms. Thus, it is possible to determine the number of atoms (N) inside a macroscopic crystal (here, an Si sphere) of known volume (V), and, given the atomic mass of Si (A_r), its mass.

$$N = 8\,V/a^3 \tag{1}$$

$$m = A_r\,N/N_A \tag{2}$$

This is a greatly simplified version of the calculation underlying the redefinition of N_A, neglecting impurities, crystal defects and many other correction factors considered by the Avogadro project. Initially, N_A was determined by re-arranging (2) and measuring the mass of a silicon sphere with the old mass definition. With N_A fixed, the mass of the silicon spheres produced for the Avogadro project can then be determined from their volume and they can be used as reference standards with very low uncertainty, traced back to the Planck constant via N_A.

The lattice parameter itself is very difficult to measure with low uncertainties. The available data has so far been limited to few measurements and samples. PTB is aiming to provide additional measurements of several of the Avogadro-Si-28 ingots with uncertainties of the order of 3×10^{-9}.

Rather than measuring a directly, the distance between the (220) lattice planes is measured. (220) is a convenient choice due to the strong Bragg reflex at an angle of 23.6° (with Cu Kα radiation).

$$a = \sqrt{8}\,d_{220} \tag{3}$$

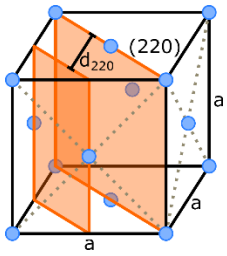

***Fig. 1** : Face-centred cubic unit cell with (220) planes. (Diamond FCC contains four additional atoms within the cell, which hinder the clear illustration of the lattice planes.)*

3 X-ray optics

Both systems use sets of thin (<0.5 mm), parallel lamellae structured into the silicon. A monochromatic x-ray beam impinges on the first *splitter* lamella at the Bragg angle and is diffracted by the lattice planes under investigation. The two beams hit the *mirror* lamella, which diffracts and refocuses the beams onto a third lamella, the *analyser*. The beams intersect inside the analyser material, giving rise to an interference pattern between the lattices of the splitter and analyser lamellae.

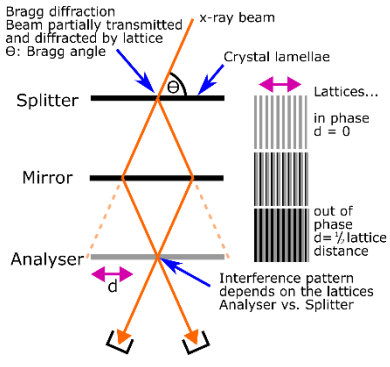

***Fig. 2** : Concept of a triple Laue x-ray interferometer, on which both abs-d and delta-d are based.*

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***Fig. 3** : The abs-d x-ray interferometer after machining. The relative motion of the two parts is tracked by an interferometer using mirrors polished into the material on the other side.*

In abs-d all lamellae are made from a single piece of Si-28 with the same lattice parameter. The *analyser crystal* is separated from the *forecrystal* during the manufacturing process. Depending on the relative offset between the interfering lattices, the lattice planes will overlap to interfere either constructively or destructively. The phase change is observed with the x-ray interferometer as the analyser is moved. An optical interferometer measures the distance travelled, correlating phase shift and distance to measure the lattice parameter in metres (d_{220}≈192 pm). The abs-d lamella pattern is more complex as it is in fact two x-ray interferometers; the second is necessary to track and correct the angle between the crystal parts to ensure good signal contrast. [2]

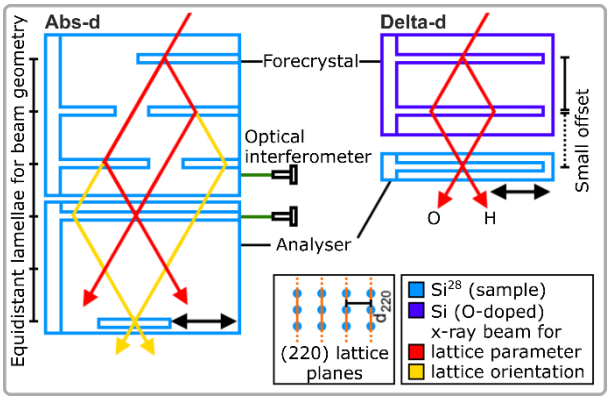

***Fig. 4** : Schematic representation of the two systems.*

Delta-d uses the simpler three-lamella arrangement. However, the forecrystal is made from oxygen-doped or natural silicon; only the analyser is made from Si-28. This alters the lattice parameter of the forecrystal slightly, so that instead of constructive/destructive interference (i.e. an *infinite* Moiré) a *finite* Moiré interference pattern arises. As the lattice periods are very similar, the Moiré pattern still has a very long period (µm to mm range) directly observable with an x-ray camera.

$$T_{Moirè} = \frac{T_{fore}\ T_{analyser}}{|T_{fore} - T_{analyser}|} \qquad (4)$$

Moiré patterns change direction depending on the relative alignment of the two underlying lattices, therefore angular tracking can be done directly with the same x-ray interferometer. However, delta-d lacks a system for the traceable measurement of the lattice parameter in metres. Instead, a measurement compares Moiré patterns observed with different analyser crystals. This provides information about the *relative change* in lattice parameter from one analyser to the next, as the ratio of the Moiré periods corresponds to the ratio of the lattice periods. The unknown lattice parameter of the forecrystal cancels out when comparing two Moirés acquired with the same forecrystal. Absolute values can be determined by using the abs-d analyser as a reference, since its d_{220} value is already known.

$$\frac{d_{220,2}}{d_{220,1}} = \frac{T_{M,2}}{T_{M,1}} \qquad (5)$$

4 Advantages and disadvantages of delta-d

The delta-d setup has several advantages over a complete, absolute x-ray interferometer.

Smaller samples: A delta-d “sample” consists only of a comparatively small analyser lamella. This is easier to produce and consumes much less Si-28 than an x-ray interferometer, which must be built in full (including the forecrystal) for each new sample.

Much smaller offcuts from an Si-ingot can be used to produce a viable sample. Thus, larger, more representative sample sets can be selected to better characterise the lattice parameter in different locations within an ingot, or to compare different ingots.

Flexible sample positioning: x-ray interferometers are very rigid systems. Delta-d can, in principle, position the analyser freely using a hexapod. This allows rasterised measurement of the entire lamella surface, instead of just one small region. Thus, much more data and a more robust lattice parameter can be obtained from each analyser.

No vacuum: Abs-d operates in a vacuum chamber for the benefit of its optical interferometer. Delta-d can operate at atmospheric pressure. This greatly simplifies the design as well as experimental work.

Indirect measurement: Delta-d requires a reference to determine absolute values. Without the analyser from abs-d it can only provide relative measurements, which would limit the usefulness of the data. The comparison is a two-step measurement with an inherently higher uncertainty. Furthermore, the overall uncertainty will also increase with abs-d as an extra step along the traceability chain.

References

[1] N. Kuramoto, in *Analytical Sciences,* **37**:177-188 (2021)

[2] E. Massa et al., in *Metrologia,* **48**:44-49 (2011)

[3] B. Andreas et al., in *Meas. Sci. Technol.,* **31**, 2020