Precision Lattice Parameter Measurement

Background

Lattice parameter measurements are used in many situations to characterize materials. For example, knowledge of the lattice parameters can provide information on the thermal properties of a material, an indirect method to determine the compositions in a solid solution, a measure of the strain state, or an analysis of the defect structure. Therefore, it is important to determine the lattice structure with the highest precision. Fortunately, X-ray diffraction can provide such information to an accuracy of several significant figures if care is taken during the experiment and subsequent analysis.

Background

If the crystal structure of a material is known, then the diffraction pattern can be indexed rather easily. However, in order to use this indexing to compute a precise value of the lattice parameter, careful analysis of the data is required. For example, in a cubic system, the "*d*" spacing corresponding to each diffraction line can be related to the lattice parameter *a* through

$$a^2 = d^2/(h^2 + k^2 + l^2)$$

where hkl are the Miller indices. If there were no systematic errors in the positions of the diffraction peaks, then there would be only random errors in

> ### Applications
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> |---|---|
> | Material Synthesis | Misfit Stress Analysis |
> | Analysis of Solid Solution | Alloy Formation |
> | Thermal Expansion | Defect Structures |
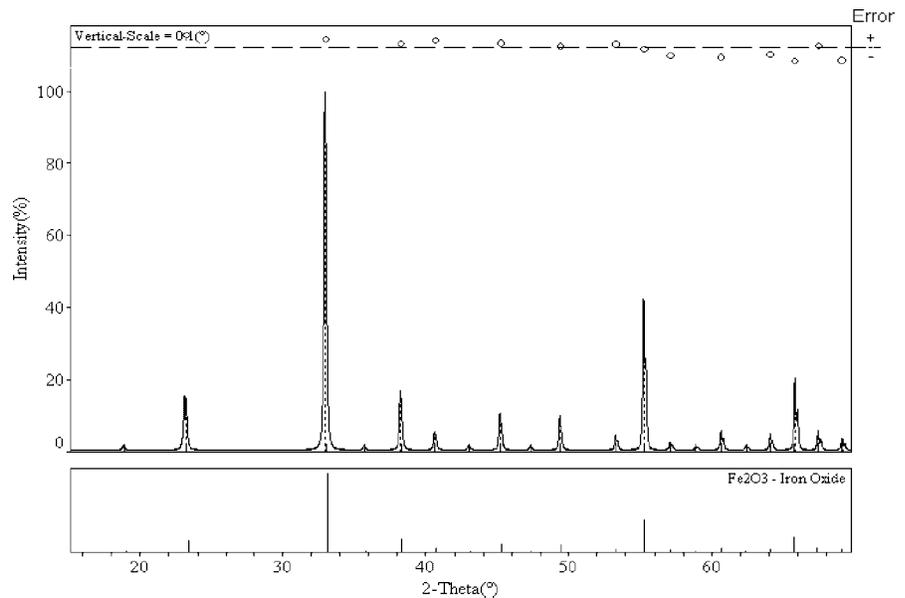> | Strain/Stress Determination | |

Figure 1 Presence of systematic errors in peak positions

the individual calculations of the lattice parameter. However, systematic errors occur for a number of reasons that require a sophisticated approach to compensate fully for such errors. An example is shown in Figure 1, where the diffraction pattern of a simple cubic material is shown. At the top of the figure is shown the error between the *d* spacing based upon the computed lattice parameter and the experimental peak position. Note that peaks at the low angles show a positive deviation while the higher angle peaks show a negative deviation.

Elimination of Systematic Errors

Systematic errors fall into three categories. the first is the lattice parameter itself, which if incorrect will result in an error pattern. Once the systematic errors due to the experimental setup have been removed, then the lattice parameters will be computed to provide a least squares fit.

The principal experimental errors are due to the zero-offset and sample displacement. The zero-offset occurs when the zero angle position of the diffractometer is off slightly. Such errors can generally be controlled during the alignment. Nonetheless, small zero offsets will be present. The second type

of error, sample displacement, is more important and is due to small vertical displacements of the sample off the rotation axis of the diffractometer. One way for this error to creep in is for dust particles to collect on the sample holder, causing a displacement of tens of microns. This small error is sufficient to shift the entire diffraction pattern by approximately 0.1 degrees, similar to that shown in Figure 1.

Computational models of the zero-offset and sample displacement errors have been derived and are commonly used to correct mathematically the raw data. Typically, a least squares refinement is run in which the lattice paramaters and one of the mechanical errors are refined simultaneously. More sophisticated refinements, such as the Rietveld method, will also take into consideration peak asymmetry, which is very important for low angle peaks.

Once the corrections for systematic errors are applied, it is possible to obtain lattice parameters routinely to an accuracy of 200 ppm. With more careful data acquisition and data refinement, accuracies can be pushed to the 20 ppm level.
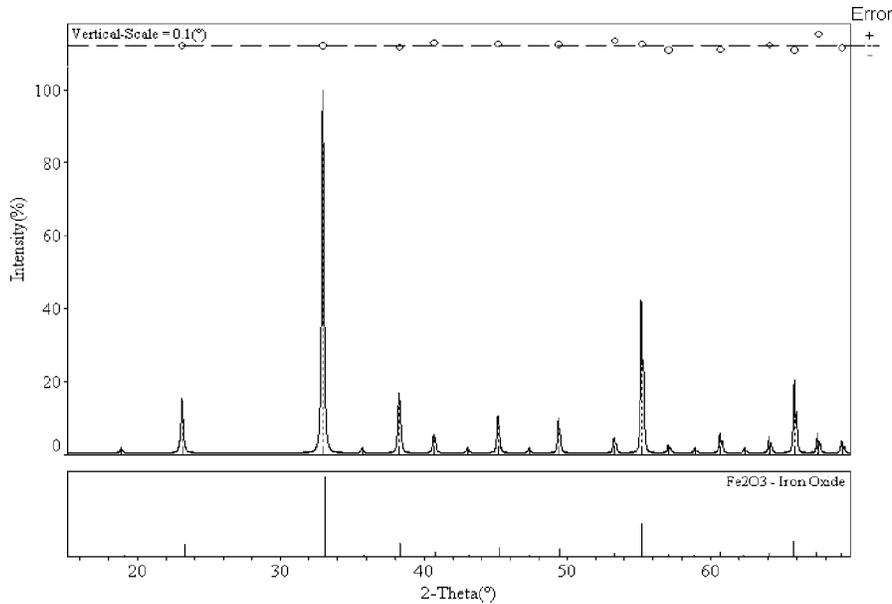

Figure 2 Corrections applied to diffraction pattern of Figure 1 to compensate for systematic errors.

Our Pledge

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- fiber analysis
- crystal orientation
- grazing incidence angle
- retained austenite analysis

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