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Quantitative Analysis of Multi-Phase Steels of Ferrite and Austenite

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Quantification of phases in a multi-phase system in steels is often crucial in determining the properties. Magnetic balance [1], image analysis [2] and X-ray powder diffraction [3] are the most common used methods for the quantitative analysis of phases in steels, each of which has its advantages and limitations. However, because of the microstructure, such as preferable orientation, residual stress and so on, large deviations could be observed among the results measured using these methods. The present study was carried out using samples of mixture of ferrite and austenite powder, which are texture-free and residual stress-free. Samples with 2, 5, 10, 20, 50 vol % of ferrite (pure Fe) powder bided by a lubricant known as Mid Wax with austenite powder (stainless steel 316 L). Measurement by image analysis uses scanning electron microscopy and image was enhanced by electron back-scattering diffraction (EBSD). The direct method was used in X-ray powder diffraction. The results form the both magnetic balance and X-ray powder diffraction showed a good accuracy with absolute error limit of ±4 vol % [4]. This also indicates that low detect limit for these two methods would be of 4 vol %. For magnetic balance microstructure plays no rule in determine the volume fraction therefore, very useful in case of highly deformed or preferable orientated samples. However, in case of mixture of non-magnetic phases this method would not be applicable. While X-ray powder diffraction could measure multi-phases structure without any restriction and with the help of whole pattern treatment by the direct method even correction of the preferable orientation on the intergraded intensities could carried out. The result from the image analysis shows a significant deviation. This may be the results of surface modification during sample preparation, such as polishing and etching; the difference in the volume fraction in the bulk and at the surface.

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Use of quantitative X-ray diffraction for academic and industrial applications

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X-ray diffraction is a fast method to collect mineralogical information. This information can subsequently be processed in different ways to obtain quantitative results. For some industrial applications, mineral quantification is an essential part of quality control of final products and it is used for characterizing raw materials necessary for production. Also for academic purposes, the quantitative mineralogical composition of natural rocks, sediments and building materials is useful in for instance provenance studies, where the source of materials as a whole or the source of its individual components is to be retraced. Such a study is currently carried out by our group in the North Sea area where a clay mineralogical quantification is - used to retrace the source area of the mud sediments. The spatial distribution and nature of natural deposits is also an example of a useful application. Our research group is currently carrying out an investigation of the Cayo formation in Ecuador where the zeolite deposits show a variable mineralogy throughout the stratigraphic profile. Depending on the type and amount of zeolites compared to the type and amount of other minerals, a different origin, genetic history and application can be proposed. For the quantification, a profile summation program (RockJock ®) and the Rietveld approach (Topas Academic ®; TA) are - used.

For both RockJock and TA, it is not a requisite to mix the sample with a known amount of internal standard, although its use is advised and even indispensable if amorphous material is present. During sample preparation a small amount of sample is spiked with 10% of ZnO and ground in methanol with a McCrone micronising mill ® to < 10 μm. Random mounts are prepared by side-loading and hkl-reflections are measured.

In RockJock, quantification of the unknown sample is based on the summation of patterns of pure minerals measured under identical instrumental settings and entered in the program. If the Rietveld refinement is used for quantification, there is no need to measure the constituent minerals separately because calculations are based on crystallographic information. This implies that for the Rietveld refinement, the crystal structure must be known, whereas this is not required for RockJock. It is however a marked advantage that this crystallographic information can be adapted / refined to take into account peak shifts or changes in intensity. These may arise from solid solution which is frequently observed in for example cement minerals (also present in hydraulic limes). For such particular cases, the Rietveld method should be preferred. The positive effect on the refinement of a diffraction pattern of a hydraulic lime by changing the cell parameters is obvious.

Clay minerals are very difficult to quantify with the present Rietveld method because of their inherent defect and variable structure and their tendancy for preferred orientation. In RockJock this difficulty is faced by basing their quantification on the intensity of the 060-reflection. This is shown by examples of mixtures that have been prepared from pure clay phases and quantitatively analyzed by both methods. So, for clay-bearing samples the use of Rockjock is the method of choice. In practical use, the Rietveld method requires more experience and a more profound mineralogical and crystallographic knowledge in comparison with the RockJock program.