High Temperature X-ray Diffraction

It is often difficult or even impossible to understand processes occurring at high temperatures. Conventional experimental approaches, quenching the sample from run temperature and investigating the sample at room temperatures often lead to inconsistent and hardly interpretable results and above that are extremely time consuming. Therefore, an X-ray diffraction system was built up (Fig.1) which allows recording X-ray patterns at temperatures up to temperatures of 1900ºC under different atmospheric conditions (vacuum, inert gases, air). We can study processes which are relevant for different industrial applications, such as crystallisation of slag or oxidation of metals during hot rolling.

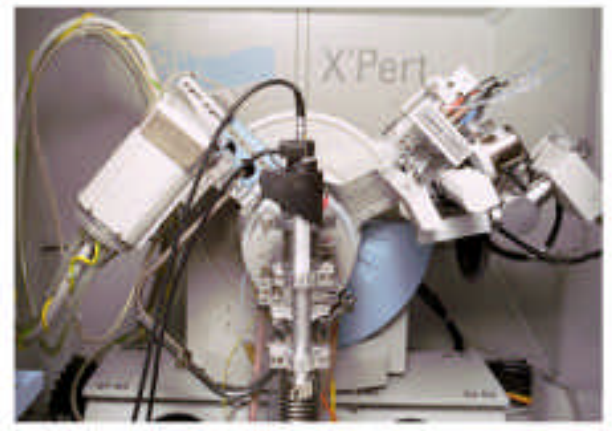
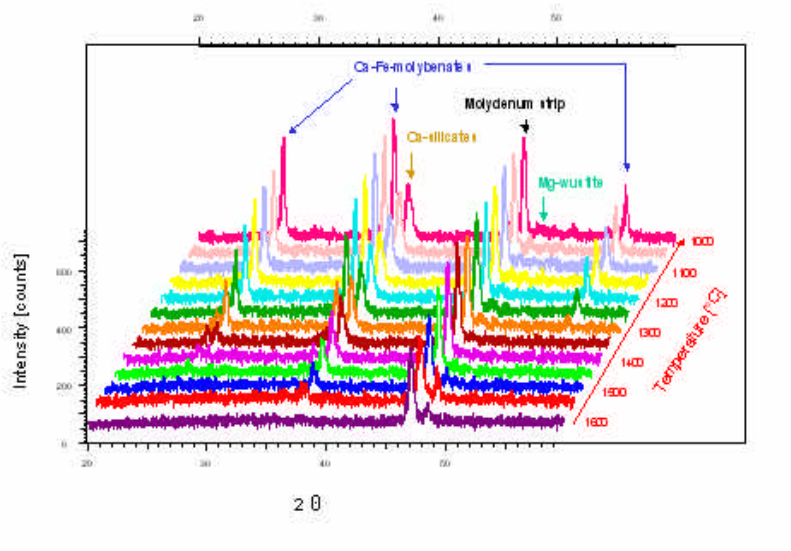


Fig 1: X'Pert diffractometer with High-Temperature set-up.

Oxidation During Hot-rolling

X-ray diffraction may be used to analyse the oxidation process during the cooling step in the hot rolling of steel. On the way to the coil the strip material is cooled with enormous amounts of water in the plant. X-ray diffraction is carried out in the laboratory. To simulate the cooling step, a strip (1 x 10 cm) of the chosen steel quality is heated up under an inert atmosphere to the finishing temperature e.g. 888ºC. In the experiment, humid air is blown into the XRD HT-chamber while rapidly cooling the strip to the coiling temperature. Within the coil, the steel strip is not longer exposed to the atmosphere and the cooling rate is relatively low. A slow cooling of the strip under nitrogen atmosphere simulates this.

Different cooling paths and atmospheric conditions can be simulated for different points of a coil (inside, outside). This can be done on the same time-scale as in the plant and the results can be monitored in-situ.



Crystallisation of Molten Slag

The physical properties and the usability of slag are controlled by its mineralogical composition. Controlling factors of this compositions are both the bulk chemistry and, the cooling history. Therefore, it is extremely important to know, which phases crystallize at which temperature. Of course, in an experiment slag can be heated up to a certain temperature, subsequently quenched and finally, the cold slag can be studied. However, some phases are not quenchable and other ones change their composition during quenching. A solution is to X-ray the molten slag at the temperature of interest. The crystallisation sequence can be visualized and allows the validation of thermo dynamical calculations. Based on this knowledge an optimal cooling path for slag can be derived.

Summary

High-Temperature X-ray diffraction (HT-XRD) is an important analytical tool to study chemical reactions, phase transformations and structural variations at elevated temperatures. Results are directly available within a small amount of time. The equipment at CRC allows the simulation of processes close to industrial applications.