Handling of radioactive materials prior to analysis by X-ray powder diffraction

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Materials under extreme conditions of radiation and temperature, as in nuclear facilities, need to be tested and analysed to understand the neutron-induced microstructural defects that might affect their mechanical properties at macroscale and thus affect the material performance. X-ray diffraction (XRD) is a widely use technique for structural characterization of materials in a bulk or powder form. Special care must be taken when manipulating radioactive material, especially in a powder form, since it can lead to unwanted radioactive contamination [1, 2]. Therefore, the handling and milling of radioactive materials (e.g. minerals-rocks, concrete) is carried inside of a hermetically sealed shielded glovebox under negative pressure [3]. Milling in ethanol of the bulk material was performed using an oscillating ball mill, producing a fine powder (after air-drying) with an average particle size of 4 microns, "wet" milling offers the advantage to produce a powder with an homogeneous size distribution and also to avoid the dispersion of the radioactive dust into the air. Radioactive samples for XRD analysis must fulfil two requirements: 1) small size to avoid excessive irradiation, and 2) a contention barrier between the sample and its surroundings to avoid radioactive contamination due to leaking of powder. To meet those requirements a drop-casting of material (approx. 15 mg) onto PEEK foil (6 μm) has been chosen as a suitable option. After air-drying of the sample, it is covered with a second layer of foil and sealed with fast-drying glue to avoid powder leaking. The thus prepared sample is now ready for XRD analysis in transmission mode [4]. The data collection is performed using a multipurpose diffractometer (Empyrean from Malvern-PANalytical) with a Co X-ray tube, the diffractometer posses a magazine and a robotic arm for automatic loading of samples, besides it can be operated remotely reducing the exposition to radiation of the operator. With the described procedure phase identification, quantification of amorphous content using the internal-standard method, and monitoring of changes in lattice parameters of the identified crystalline phase can be safely performed on radioactive samples.

One applicative example was the study of aggregates (majorly quartz, > 90 wt.%) under different levels of neutron fluences (up to 10^{20} n/cm^2). Where it was observed a progressive amorphization of quartz from 9 wt.% to 76 wt.%, at the same time volumetric expansion of the unit cell was observed (up to 11%), as both axes a and c increased with the neutron fluence. Crystal density (g/cm^3), calculated from the previously calculated lattice parameters, decreases (-10%) with the increase of neutron fluence irradiation.

In summary, the developed methodology represents an easy and affordable way to study the irradiated materials at laboratory scale.

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