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MS-5-P-2839 Synthesis of rare earths oxides from waste minerals

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Rare earth elements like cerium and lanthanum are currently involved in a wide range of advanced technologies and in industrial chemistry. The initial rare earth (RE) mineral is extracted, mechanically and physically concentrated, giving a main concentrate and mineral wastes. These waste materials can be considered as “zero cost” initial sources of rare earth oxides. Thus, the development of a simple chemical process allowing synthesis of rare earth oxide phases from these waste minerals is part of sustainable and economic development. The waste is rich in monazite and allanite which contain different proportions of rare earth elements.

Different chemical processes were explored, in order to extract the interesting oxide phases from these complex mixed materials. Transmission Electron Microscopy coupled with Energy Dispersive Spectroscopy was performed at each step of the chemical process: it was the most efficient way to check the efficiency of the removal of chemical species, and the crystallisation and composition of the products. The first way explored was a solubilization of the minerals through an acid attack followed by a selective precipitation of Re (OH)x [1]. The precipitation of RE oxalates and their final annealing at 900 °C led to a new pyrochlore phase RE2Ce2O7 [2]. Another process started with an alkaline fusion, followed by a solubilization in nitric acid led to two distinct crystallographic phases, related to the initial compounds: monazite and allanite. Fig 1 shows two distinct morphologies: one can found spherical grains (Fig1: A grains) and platelets (Fig.1: B grain). The spherical grains crystallize in the cubic CeO2 structure (Fig.2). EDS analyses lead to the chemical formula of this oxide: Ce0.65La0.25Nd0.1O2. The diffraction pattern of the cubic shaped grain can be indexed in a double cell of a tetragonal structure along the [001] axis or in an ordered cubic double perovskite along the [001] axis (Fig.3). The EDS analyses show that this oxide phase contains mainly lanthanum and iron, with a La/Fe ratio around 2.

References

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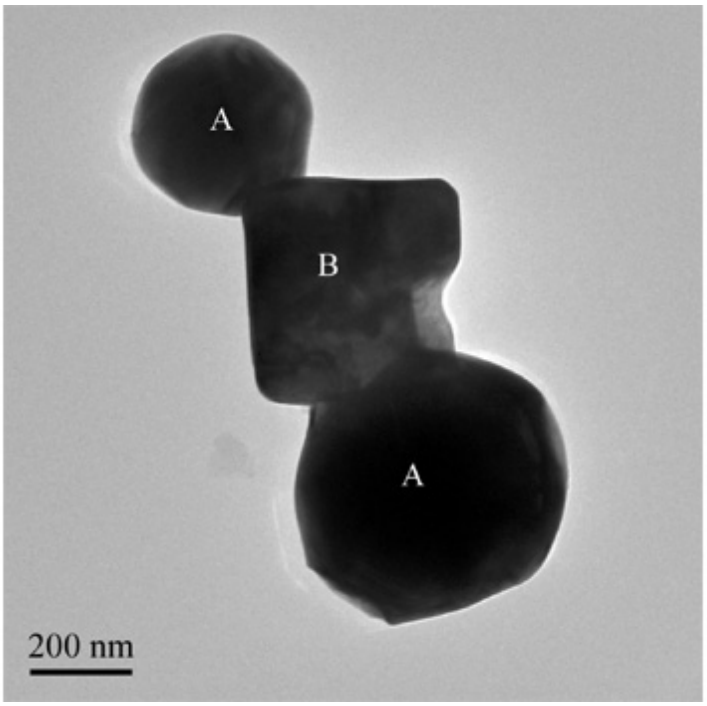

Fig. 1: Grains obtained after alkaline fusion. The grains labelled A are spherical and contain Ce,La,Nd,Th. The grain labelled B contains mainly La and Fe.

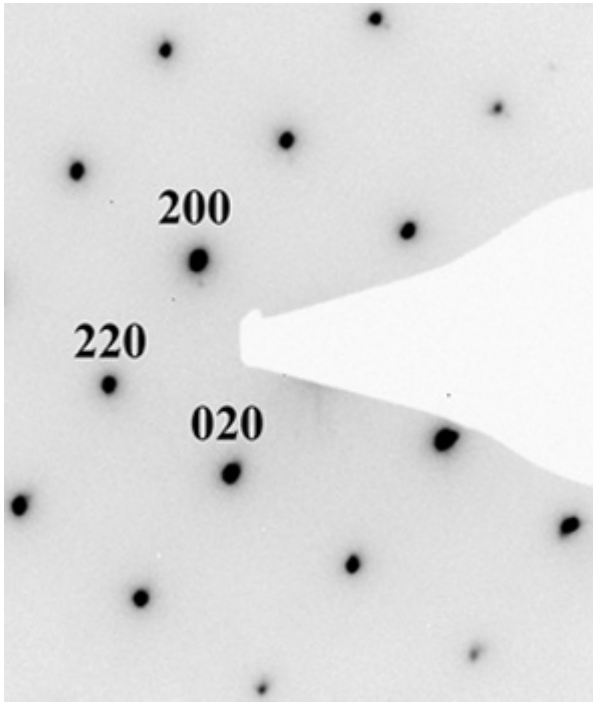

Fig. 2: diffraction pattern of a grain labelled A on figure 1. It is indexed in the CeO_2 fluorite structure along the [001] direction.

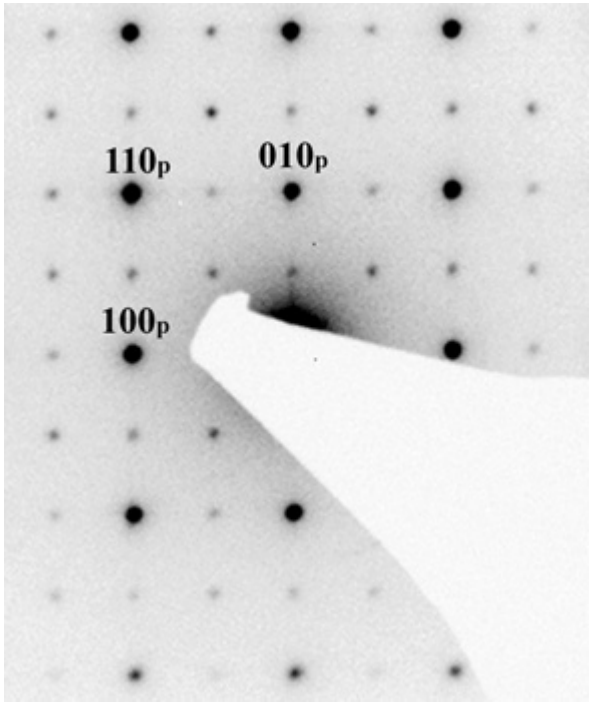

Fig. 3: diffraction pattern of a grain named B on figure 1, showing a doubling of the perovskite cell.