

***In situ* X-ray diffraction study of nanopowders Spark Plasma Sintering under very high pressure**

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SPS (Spark Plasma Sintering) is a pressure sintering technique that uses pulsed current heating. The very high heating and cooling rates allow a fast consolidation of the powders with limited grain growth. Nanostructuring allows modulating the mechanical, thermal or optical properties of materials. Therefore, SPS is widely used in the search for materials with remarkable properties. However, SPS has its limitations and one of the ways of improvement is the application of very high pressures during sintering. Increasing the pressure significantly lowers the densification temperature of the powders [1, 2]. SPS at very high pressures is therefore a promising process for the elaboration of bulk nanostructured materials, and, for the sintering of temperature unstable materials and materials that are difficult to sinter without the addition of a binder. Commercially available SPS devices are limited to 150 MPa. Modified devices can reach 1 GPa. But these pressures are insufficient to sinter some materials under their thermodynamic stability conditions (e.g. diamond, c-BN). We have pushed the limit to 10 GPa, by adapting a pulsed electric current heating to a high pressure synthesis equipment of PE type (Paris-Edinburgh press) [2, 3]. Our SPS-PE device is portable and suitable for *in situ* measurements on large facilities (synchrotron, neutron source) for the study of material transformation kinetics during the sintering process as a function of temperature, pressure and applied current characteristics. This type of real-time analysis (Fig. 1) is not possible on any other SPS device. We will present the results of sintering studies conducted on different oxide nanopowders and on nanodiamond.

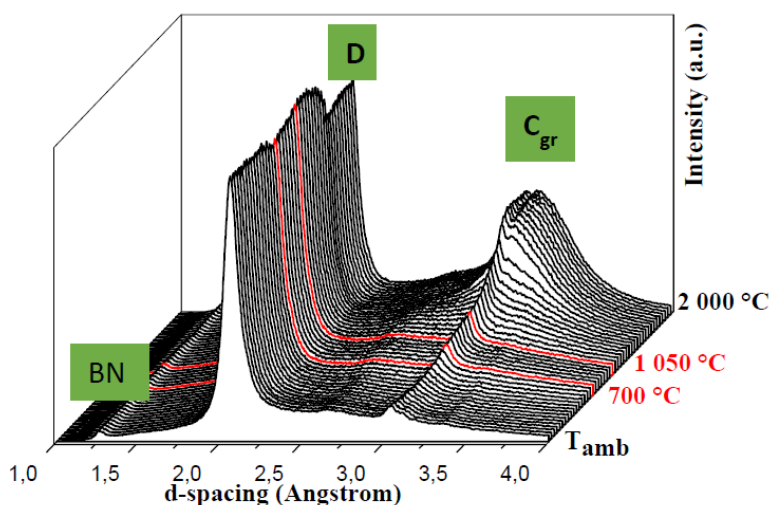


Figure 1. *In situ* X-ray diffraction monitoring of a diamond nanopowder (5 nm) sintering under 5 GPa (D, diamond; C_{gr}, graphite; BN, hexagonal boron nitride). The heating rate is 500°C/min. The acquisition rate of 10 diffractograms per second on the PSICHE beamline (synchrotron Soleil, France) allows to follow the grain growth in real time and to detect any graphitisation.

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