ESRF	**Experiment title:** High temperature structural studies of the Ca-doped Bismuth Ferrites, $Bi_xCa_{1-x}FeO_3$	**Experiment number**: HE 2476
Beamline: BM01B	**Date of experiment**: from: 02 February 2007 to: 06 February 2007	**Date of report**: 15 February 2007
Shifts: 9	**Local contact(s)**: Mr. Wouter van Beek, Mr. Hermann Emerich	*Received at ESRF:*

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Report:

A series of temperature resolved high resolution X-ray diffraction scans of calcium-doped bismuth ferrites, $Bi_xCa_{1-x}FeO_3$, (x=1 and 0.8) were carried out in order to determine the lattice symmetry of this series of materials and any phase transitions.

The room temperature pattern of $BiFeO_3$ indicated that the structure is described by the rhombohedral space group *R3c* (# 161), as previously observed. $Bi_{0.8}Ca_{0.2}FeO_3$, however, showed some small splittings in several peaks, characteristic of a lower symmetry. It was found that the room temperature symmetry of this phase can be described by the monoclinic space group An (# 9).

Variable temperature powder diffraction patterns for $Bi_{0.8}Ca_{0.2}FeO_3$ were subsequently measured every 50°C, over the temperature range of 50~450°C, in the 2θ range of 1~35°. The sample was mounted in a 0.7mm quartz capillary. Nine diffraction patterns were collected for about four hours each. For diffraction patterns above 350°C, the sample undergoes an irreversible chemical phase separation into two perovskite phases (figure 1).

The monoclinic space group *An* is used for Rietveld refinements for all patterns of $Bi_{0.8}Ca_{0.2}FeO_3$. From the plot of scaled lattice parameters a_P, b_P and c_P, an *An* to *R3c* structural transition occuring between 100~150°C is observed (figure 2).

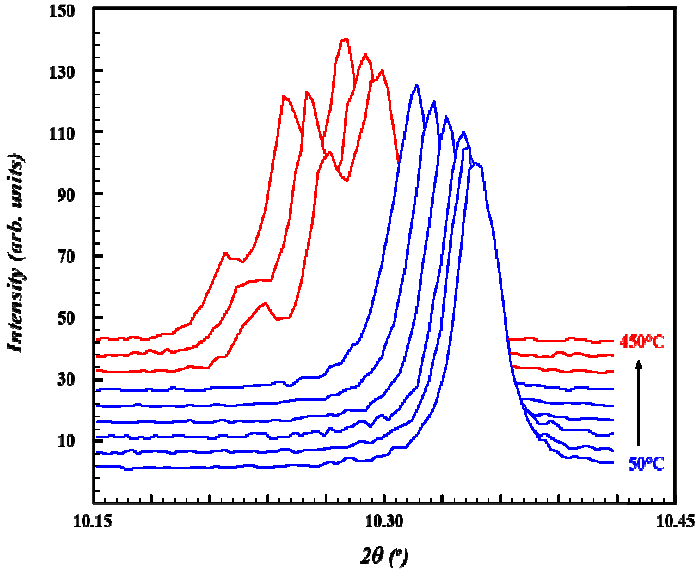

Figure 1. The evolution of the (1 1 0) peak of $Bi_{0.8}Ca_{0.2}FeO_3$ from 50 to 450°C. The patterns have been normalized.

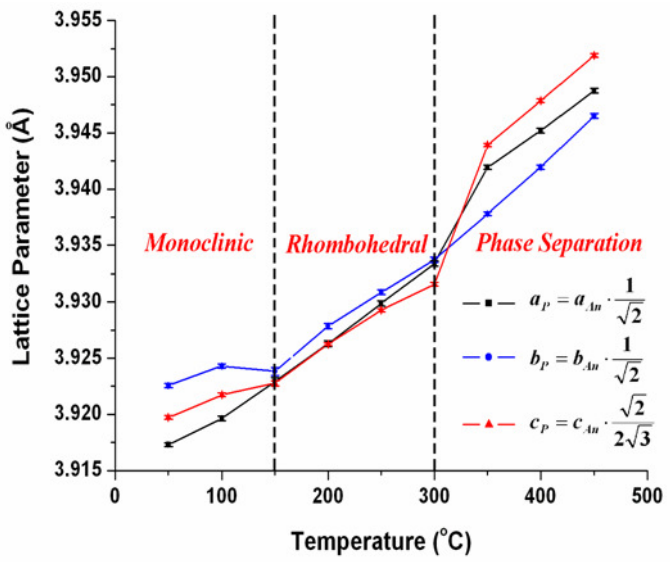

Figure 2. Scaled *An* lattice parameters of $Bi_{0.8}Ca_{0.2}FeO_3$. A transition to rhombohedral symmetry occurs between 100~150°C, and phase separation occurs above 300°C.