ESRF	**Experiment title:** **Exploratory time-resolved XRD studies of combustion synthesis reactions in applied magnetic fields**	**Experiment number**: MI-517
Beamline: ID11	**Date of experiment**: from: 23/11/00 to: 25/11/00	**Date of report**: 2/3/01
Shifts: 6	**Local contact(s)**: Å. Kvick	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Q. A. Pankhurst*, University College London, U.K.
Prof. I. P. Parkin, University College London, U.K.
Miss L. Affleck*, University College London, U.K.
Miss H. Spiers*, University College London, U.K.
Mr. D. Morrison, University College London, U.K.
Dr. M. V. Kuznetsov*, ISMANAM, Moscow, Russia

Report:

An exploratory set of high-resolution time-resolved X-ray diffraction experiments were undertaken on the Materials Science beamline, ID11, at ESRF. X-ray diffraction patterns were recorded at a rate of one every 110 ms, comprising 50 ms exposure time and 60 ms readout time from a 1024×1024 pixel FRELON CCD camera. Excellent counting statistics were obtained, as illustrated in Figure 1.

Three different SHS reactions were tested :

1. $BaO_2 + 6\ Fe + 3\ Fe_2O_3 + 4\ O_2 \rightarrow BaFe_{12}O_{19}$,
2. $0.5\ Li_2O_2 + 2\ Fe + 1.5\ Fe_2O_3 + 1.25\ O_2 \rightarrow LiFe_5O_8$,
3. $MgO + ZnO + Fe_2O_3 + 2\ Fe + 0.75\ NaClO_4 \rightarrow MgZnFe_4O_8 + 0.75\ NaCl$.

Over the course of the experiment a total of 34 reactions were set up and run, using a variety of different experimental arrangements: powder reactions; pellet reactions at consolidation pressures ranging from 0.5 to 2 tonnes; reactions in zero field; reactions in a transverse field of 200 mT; reactions in a transverse field of 1.1 T; reactions in air; reactions in a flow of oxygen gas of 2, 4 or 8 litres per minute; reaction ignition using nichrome wire; reaction ignition using tungsten wire; and reaction ignition using the reagents themselves.

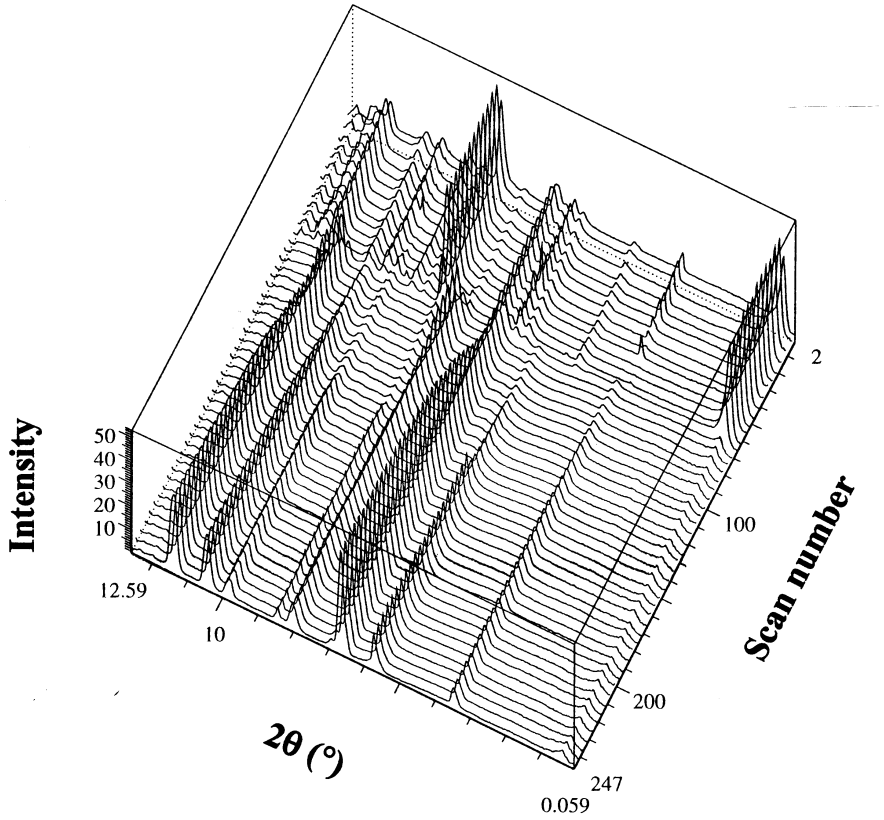

Figure 1. A plot of every fifth scan recorded from a combustion synthesis reaction of MgO, ZnO, Fe, Fe_2O_3 and $NaClO_4$ to form the magnesium ferrite product $Mg_{0.5}Zn_{0.5}Fe_2O_4$. The combustion wavefront reached the X-ray beam at approximately scan number 60. Note that significant changes are evident in the X-ray pattern both before and after this point.

Difficulties were encountered in attaining reliable ignition of the pelletised green mixtures, and consequently most of the reactions were performed on powders. This in turn led to some problems, especially in the 1.1 T applied field runs, where substantial product restructuring and movement during and after combustion led in most cases to a loss of signal quality as the combustion wave traversed the mixture.

Inspection of the data revealed many features reflecting the nature of the combustion process. These ranged from relatively slow changes, such as peak shifts due to lattice contraction of the product phases on cooling, through to relatively fast transients, which are most likely evidence of momentary macroscopic restructuring in the powder as the combustion wavefront passes.

One outcome of this preliminary experiment is that the method is clearly capable of delivering high-resolution data of significant interest regarding the materials chemistry of these solid-state combustion reactions. It is also clear that continued efforts are required to perfect the ignition of pelletised mixtures in order to avoid the complications that can arise due to powder movement during the reactions.