In situ time resolved study of fast chemical reactions using X-ray powder data. H.K.R. Dunn, G.B.M. Vaughan, Å. Kvick, E.S.R.F, 6 rue Jules Horowitz, BP 220, 38043 Grenoble cedex 9, FRANCE.

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Spinel ferrites, such as MnFe₂O₄, are used in fields such as electronics. However, the synthesis of these compounds is often complex, requiring several time and energy consuming steps. Self-propagating Hightemperature Synthesis (S.H.S.) is a rapid, single step, energy-efficient technique which may be used to produce compounds such as spinel ferrites. The time scale of such reactions is the order of seconds. As the purity and microstructure of the final products is a function of the reaction path, we have undertaken a study to determine the reaction mechanisms of such reactions. Ferrites were synthesized from simple metal oxides, metals, and perchlorates, packed into pellets (1g, Ø 13mm). S.H.S. reactions were then initiated in situ using a mechanical ignition system. Time resolved 2-D powder diffraction data were collected on an ESRF Frelon CDD detector (1024x1024 pixels with a time resolution of 60ms) using an energy of 50 keV. The diffraction patterns were obtained in transmission mode, in order to collect bulk information on the reaction mechanism. A total number of 260 frames were recorded as the reaction front passed the probing X-ray beam, giving information on the preheated, reaction and cooling regions. After treatment and integration of the images, the data were analyzed by Rietveld refinement in G.S.A.S. Insight into the reaction mechanisms were obtained from the microstructure and quantitative phase fractions. Using the insights thus gained, the reactions were further tuned to obtain the desired results.