FREEZING TRANSITIONS IN MYOGLOBIN STUDIED BY POWDER DIFFRACTION

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Obtaining good quality single crystals remains one of the major barriers to the study of protein structure. Cryocooling reduces radiation damage and is widely employed for data collection with synchrotron radiation. However, cryocooling frequently leads to a reduction in crystal quality, motivating an in-situ powder diffraction study to understand the processes occurring during the freezing of a protein crystal.

Myoglobin (from horse skeletal muscle) has been studied using the high resolution powder diffraction beamline (BM16) at ESRF and we have observed a pair of first order structural phase transitions at 245K and 265K. The transitions occur above the freezing point of the mother liquor, and involve volume expansions of the order 2-3%. Fast cooling through the transitions traps the sample in the high temperature form, but introduces severe peak broadening, indicating a reduction in the crystal quality. Growth of the 250K phase over the course of several hours has been observed at constant temperature, as well as peakshape annealing effects during heating.

Although these transitions might be catastrophic for a single crystal experiment, they can be actively exploited in powder data, since the change in unit cell parameters partially alleviates the peak overlap problem. This leads to a significant increase in the amount of information that can be extracted from a powder diffraction experiment.

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STRUCTURE OF BINARY CaO-Al2O3 AND SrO-Al2O3 LIQUIDS BY COMBINED LEVITATION-NEUTRON DIFFRACTION

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The total structure factors, S(Q) of 50:50 mole % CaO:Al2O3 and 67:33 mole % SrO:Al2O3 composition liquids have been determined by neutron diffraction. Measurements were made on laser beam heated liquids at 2000-2100 K held in an aerodynamic levitator. Pure vanadium levitation nozzles were used. Reduction of the nozzle wall thickness decreased attenuation of scattered neutrons to give a twofold increase in signal intensity. The liquids comprise predominantly 4-coordinate aluminum with the group II metal in 6- and higher coordination with oxygen.

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