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DIFFRACTION THERMOMETRY AND DIFFERENTIAL THERMAL ANALYSIS

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Rapid diffraction patterns (0.9s) were collected during the formation of a Si substituted TiC phase by a self-propagating high-temperature synthesis (SHS) reaction on the D20 diffractometer at the Institut Laue-Langevin, Grenoble, France. The observations were used to propose a powder diffraction method for conducting differential thermal analysis (DTA) as part of in-situ phase transition studies. The thermal expansion coefficients for TiC were used to establish the sample temperature from the Ti(C,Si) lattice parameter during cooling. A lattice parameter anomaly during the precipitation and growth of Ti_3SiC_2 from the Ti(C,Si) phase was interpreted as the release of latent heat. An interpolating background function was subtracted and the exothermic peak extracted. The transition enthalpy was estimated using two techniques: 1. The area of the peak was determined numerically, 2. An Avrami Equation was fitted to Quantitative Phase Analysis results for Ti_3SiC_2 precipitation. The coefficients so derived were used to fit the temperature excursion directly using the heat of formation as the only variable parameter. These gave estimated transition enthalpies of –489 kJ/mol and –456 kJ/mol, respectively. Such a large enthalpy is consistent with intermediate phases, $Ti_5Si_3C_x$ = –579 kJ/mol and TiC = –185 kJ/mol, known to be associated with Ti_3SiC_2 formation at lower reaction rates. Conditions for optimising this method for rapid in-situ studies of phase transitions have been established.

Keywords: DIFFERENTIAL THERMAL ANALYSIS, PHASE TRANSITIONS, IN-SITU DIFFRACTION

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COMPARATIVE SANS AND PCS INVESTIGATIONS OF THE INFLUENCE OF TREHALOSE AND GLYCEROL ON THE TEMPERATURE DEPENDENT CONFORMATIONAL PROPERTIES OF A HYDROSOLUBLE MODEL SYSTEM: POLY(ETHYLENE OXIDE)

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The mechanism of protein stabilization by glassy solvents is not entirely clear, and the stabilizer effective for a given protein is often discovered empirically. Binary, Poly(Ethylene Oxide)/D_2O, and ternary, Poly(Ethylene Oxide)/$C_{12}H_{22}O_{11}$/D_2O and Poly(Ethylene Oxide) /$C_2H_7O_3$/D_2O, mixtures have been investigated using SANS and PCS spectroscopy. The results indicate that at low temperatures glycerol ($C_2H_7O_3$) appears to impart to better stability than glassy trehalose ($C_{12}H_{22}O_{11}$). Conversely, trehalose affects the swelling properties of the polymer, stabilizing its conformation, at higher temperatures better than glycerol.

Keywords: PEO NEUTRON SCATTERING LIGHT SCATTERING

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AN INTEGRATED APPROACH TO HANDLING NON-MEROHEDRAL TWINS

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Non-merohedral twins present several challenges even when the individual components can be indexed separately. Handling overlapped reflections and applying an absorption correction are especially problematic.

The combination of CrystalClear (Rigaku/MSC) - with its TwinSolve module - and CrystalStructure, which utilizes the robust and easy-to-use twin refinement utilities provided by CRYSTALS, allows users to readily handle merohedral and non-merohedral twins alike.

TwinSolve handles the indexing, cell refinement, reflection integration, absorption correction and file output. Data can be read from images collected on a variety of CCD or image plate detectors. Cell parameters for twin components are extracted and refined by TwinSolve. All components having the same cell may be refined together so that only one set of cell dimensions is produced. Integration for each component is performed and absorption corrections are applied using numerical (shape-based) information and/or Cartesian spherical harmonics (up to eighth order). Reflections can be saved in SHELX HKLF5 format (all components in a single file) or to a file containing only non-overlapped reflections for a single component.

Twin laws are passed to CrystalStructure via a CIF. During data evaluation, CrystalStructure flags the twin component(s) for each reflection. Since the refinement of a twinned structure is typically more difficult than for a single crystal, the battery of constraints and restraints available in CrystalStructure can provide additional information to help stabilize the least-squares process.

Keywords: NONMEROHEDRAL TWINNING SOFTWARE

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DATA QUALITY AND THE MODERN CCD DETECTOR

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Modern area detectors are capable of collecting high quality datasets. The quality of these datasets is partially a function of collecting redundant data and the sensitivity of the detector. The speed of data collection also plays a role as complete datasets are routinely collected on a single crystal. Further improvements in data quality can be achieved by collecting data at low temperature. This improvement is readily seen by comparing data collected on the same crystal at room temperature and low temperature (i.e. 97K). Macromolecular crystallographers rely on low temperatures to extend the life of a crystal in the x-ray beam. While this is not necessary for most small molecules there are benefits to be derived from collecting low temperature data. For well-behaved small molecules the difference peaks after the final cycles of refinement are often located in the middle of a covalent bond. An example will also be shown in which this residual density was found in a hydrogen bond. This example shows that low temperature data collection can aid in the study of both electron density and displacement of that density in the formation of a hydrogen bond.

Keywords: LOW TEMPERATURE ELECTRON DENSITY HYDROGEN BOND