Contribution ID: **108** Type: **Oral presentation**

PCA analysis of in situ X-ray powder diffraction and imaging data: new approaches named differential scanning diffraction and imaging

Wednesday, 14 September 2022 11:45 (15 minutes)

Eutectic mixtures have wide industrial importance in many fields including alloys, refrigeration industry, electronics, and chemicals purification. Typically, eutectic mixtures are characterized by differential scanning calorimetry (DSC), able to identify the transition temperatures, possible hysteresis and investigate the energetic features of transformations. Despite its fundamental role, DSC is not able to give compositional, structural and morphological information. Such features are accessible exploiting diffraction and imaging techniques, not easily applicable to liquid/solid phase transitions. A novel approach, able to shed light on such complex liquid/solid transformations is proposed, by coupling principal component analysis (PCA) to simultaneous *in situ* XRPD and imaging, with an *ad hoc* modified sample holder exploiting a Linkam environmental chamber within a D8 Bruker diffractometer, equipped with an USB microscope. In this way, the experiment is simultaneously probed by XRPD and imaging by a cold/hot stage microscopy approach. PCA can blindly analyse *in situ* data from both solid and liquid phases, with PCA scores being the reaction coordinate of both melting and crystallization steps. Analyzing the in situ XRPD and imaging data by PCA, the phase transition can thus be analysed similarly to traditional (DSC) but probing structural (XRPD) and morphology (microscopy) effects instead of energy ones. Melting and crystallization points can be identified, together with hysteresis between downwards and upwards temperature ramps by both XRPD and imaging. PCA analysis of *in situ* data allowed the development of new analysis methods, named "Differential scanning diffraction" (DSD) and "Differential scanning Imaging – DSI). DSD also allows the identification of each solid phase during the phase transformation and the quantification by both PCA and traditional Rietveld methods. DSD/DSI is thus a powerful complementary tool to DSC for phase transition characterization. Figure 1 reports DSD and DSI data for a KCl/NaBr/water eutectic mixture. The phase transition is well identified with imaging and diffraction able to identify a solid amorphous phase, named "mush ice". It is evident by looking at the different melting temperatures (red curve in Figure 1) "seen" by diffraction (DSD) at -30℃ and imaging at -34 ℃ (DSI).

Primary author: MILANESIO, Marco (Università del Piemonte Orientale)

Co-authors: LOPRESTI, Mattia (Università del Piemonte Orientale); Dr PALIN, Luca (Dipartimento di Scienze ed Innovazione Tecnologica, Università degli studi del Piemonte Orientale "Amedeo Avogadro"); MANGOLINI, Beatrice (Cold Car S.p.A.,); Dr CONTEROSITO, Eleonora (Università del Piemonte Orientale)

Presenter: MILANESIO, Marco (Università del Piemonte Orientale)

Session Classification: MS

Track Classification: Bright Radiation Sources and Novel Software Applications