The crystallization of nanosized crystals in amorphous matrix result of two interconnected phenomena. Starting from an amorphous material homogeneous at the molecular scale, incongruent crystallization occurs in fact after amorphous-amorphous phases separation processes. Such fluctuations can be evidenced through Small Angle X-ray Scattering (SAXS). Because crystallization of the phase separated areas is a stochastic phenomenon, related in particular to the nuclei size, at the global scale of the material crystallization and phases separation processes are very often observed simultaneous. As a consequence, the determination of their both kinetics require in principle coupled SAXS and Wide Angle X-ray Scattering (WAXS) in situ measurements. We synthesized through the sol-gel process amorphous silica-based material containing various amount of tin. Dense bulk samples are obtained without any high temperature and cooling process and the obtained materials are in a far from the thermodynamic equilibrium state. Tin oxide crystallization occurs during isothermal treatment realized at few hundred of degrees [1] through mechanisms that are far from the classical nucleation theory (CNT). The BM02 beamline at the ESRF is equipped with an experimental bench allowing simultaneous measurements of the WAXS and SAXS signals [2]. We will show how such set-up allows to determine at the second time-scale the kinetics of phases separation and SnO₂ crystallization processes.