Microsymposium

MS71.O05

Size dependences of transition temperatures of nanoparticles by SAXS and XRD

G. Kellermann[1], A. Craievich[2]
[1]Federal University of Parana, Department of Physics, Curitiba, Brazil, [2]University of Sao Paulo, Institute of Physics, Sao Paulo, Brazil

SAXS technique alone and combined with XRD can be applied to determine the size dependences of crystal-to-liquid and liquid-to crystal transition temperatures of nanoparticles in dilute state. Since the volume of nanoparticles at the transition temperatures are expected to exhibit (weak) discontinuities, SAXS can be applied to determine the melting and freezing temperatures of nanoparticles as functions of their size. For this purpose, a set of samples, each of them with different nanoparticle size, is studied by in situ SAXS at varying temperatures. The variations in nanoparticle volume at the transitions lead to discontinuities in the 3D integral of SAXS intensity, ΔQ, in the slope of Guinier plots, ΔS, and in SAXS intensity at any q except at $q=0$, $\Delta I(q>0)$. Thus, the transition (melting and freezing) temperatures of a nearly monodisperse set of nanoparticles, can be derived from the discontinuities observed in the temperature dependence of V, S or $I(q>0)$. Since the transition temperatures are strongly dependent on the size of the nanoparticles, for samples containing nanoparticles with a wide size distribution the size dependences of the melting and freezing temperatures cannot be determined by applying the method outlined above. However, in the particular case of systems consisting of a dilute set of spherical nanoparticles with a broad radius distribution, N(R), the combined use of SAXS and XRD makes it possible to determine the radius dependences of the melting and freezing temperatures, MT(R) and FT(R), respectively. This is achieved by studying a single sample in situ, along a heating/cooling cycle, and simultaneously determining the temperature dependences of the SAXS intensity pattern and the area of XRD Bragg peaks. A few applications of these procedures will be described, namely the determination of the radius dependences MT(R) and FT(R) of (i) a nearly monodisperse set of Pb nanoparticles embedded in a lead-borate glass (Gorgeski et al, 2014) and (ii) a polydisperse set of Bi nanoparticles embedded in a sodium-borate glass [Kellermann and Craievich, 2002). Other relevant structural features of Bi nanoparticles embedded in borate glass could also be derived from the analysis of the same experimental results [Kellermann and Craievich, 2008].

[1] *Gorgeski, A., Kellermann, G. & Craievich, A.F. (2014). In preparation.,* ***[2]*** *Kellermann, G. & Craievich, A.F. (2002). Phys Rev B65, 134204.,* ***[3]*** *Kellermann, G. & Craievich, A.F. (2008). Phys Rev B78, 054106.*

Keywords: SAXS and XRD, Size dependence of transition temperatures