"Slow operand" measurements by laboratory small-angle X-ray scattering

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Majority of recent small-angle X-ray scattering (SAXS) studies have been performed mainly in the large-facility, such as SPring-8 (JAPAN), APS (USA), and other synchrotron radiation facilities. Since high intensity of those source makes possible to realize time resolve measurements in a few seconds in non-distractive mode, "operand" measurements become popular and important to understand formation of nanostructures in many materials. In contrast, laboratory SAXS systems are usually regarded as the tool for static measurements. However, recent progress in source, optics (confocal mirror, low scattering slit) and detector makes us possible to measure nanostructure in a few minutes. Those systems can also be optimized for high energy source such as Mo solid or In-rich liquid metal targets. Combining these features, reaction continuing for a few days can be monitored non-destructively, which we call "*slow operand*" measurements. Two examples will give in this talk; First one is about low temperature aging (room temp., 65ºC, 120ºC) of Al-Zn-Mg-Cu alloys for 2 days. Second example is shape change of colloidal calcium phosphate (CCP) in real cheese for more than 5 days. In the former case, we have measured 1 mm thick aluminum sheet directly from solid solution treatment (SST) without any sample thinig using labo-SAXS with Mo source. We have also measured the sample with rolling following SST. Since all has been done in same room, the uncovered time before starting measurements are less than 5 minutes. Advantage in the second example is the physical distance between source and cheese factory. Since fresh curd (before salting) and cheese has been carried from real cheese factory in Rakuno Gakuen Univ. to labo. SAXS in Hokkaido Univ. with in 1 hour. Samples (curd or cheese) with 1.8 mm thick put into the glass cell and sealed. Shape of nanostructure of cheese corresponding to CCP changes from about sphere of 2.4 nm in diameter to disc like shape with 14 nm in diameter as shown in Fig.1.

Though there are many studies using SAXS [1, 2] including operand measurements in both case, such long time-span measurements have not been reported as far as we know. Nevertheless, there are several processes which are industrially important and occur slowly around room temperature. For those target, the slow operand technique with labo-SAXS must be very useful and important in addition to regular operand technique with large facilities.

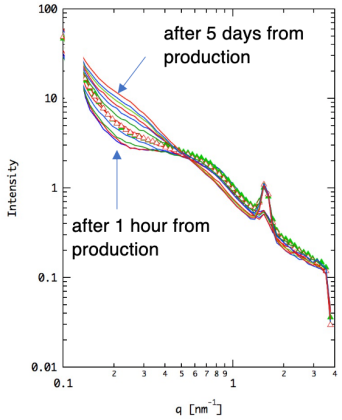

Figure 1. Time evolution of SAXS profiles of curd and cheese from 1 hour to 5 days after production .

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[2] ex. Ingham, B., Smialowska, A., Kirby, N. M., Wang, C. & Carr, A. J. (2018). *Soft Matter.* **14**, 3336.

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