Advanced characterization of materials. Relevance and challenges

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The most widely used experimental procedure for studying the structure of materials is X-ray diffraction. The analysis of single-crystal X-ray diffraction patterns reveals the geometry of unit cells and the coordinates of the atoms inside them. The problem is that atomic structures determined by applying this technique are *spatial* and *time* averages of many *local* and *instantaneous* structures, respectively. Thus results derived from analyses of single-crystal X-ray diffraction patterns do not describe neither local structures of point, linear, surface and volume defects, nor instantaneous configurations of oscillating or, more generally, moving atoms.

However, the properties of many materials depend more strongly on the local configurations of structural defects than on the features of their spatially averaged structure. Characterizations of static and dynamical defects such as thermal atomic oscillations, quantum dots, stacking faults, strains and others were made possible by using X-ray diffuse scattering techniques, which analyze the weak diffuse intensity between Bragg peaks. The technique of diffuse X-ray scattering at small angles (SAXS) was applied by first time to study substitutional Cu clusters in Cu-doped aluminum (GP zones). In Brazil there are several SAXS setups installed in laboratories located most of them in São Paulo State and two SAXS beam lines in operation at LNLS, connected to its 1.37 GeV UVX synchrotron source [1]. Diffuse X-ray scattering techniques yield useful information related to structural defects but they still only refer to space and time average structures.

The problem related to space and time averaging of structures determined by classical X-ray diffraction is expected to be solved by the use of recently developed new X-ray sources, namely X-ray free-electron lasers (XFEL) and fourth generation synchrotrons, which, under favorable conditions, allow the determination of *(i) nearly instantaneous structures without time averaging and (ii) local structures without spatial averaging*.

XFELs are now in operation in USA (LCLS) and Japan (SACLA) and under construction in Germany (European XFEL). These X-ray sources generate very short (tenths femtoseconds) and high power photon bunches, which totally destroy the sample but still produce useful diffraction patterns that may lead to the determination of nearly instantaneous structures. A pioneer serial crystallographic study of protein nanocrystals using an XFEL was conducted at LCLS [2] while the first single-shot structural study of metal nanocrystals using only one ten-femtosecond XFEL pulse has been recently performed at SACLA [3]. Another example of application of a modern synchrotron X-ray source to materials science – carried out at ESRF, France - is a time-resolved study of discontinuous crack propagation in silicon single crystals [4].

The first two fourth generation synchrotrons in the world are under construction, in Sweden (Max IV) and at LNLS, Campinas (Sirius) [5]. These sources are expected to produce X-ray beams with high lateral and longitudinal *coherence lengths*, thus allowing the determination of structures of crystalline and amorphous materials *without spatial averaging*. Max IV is currently being commissioned and will operate soon while Sirius is now under construction and will be open to users by 2019.

Novel applications of modern coherent X-ray sources require challenging developments of very stable optics systems, in situ preparation of nanoscopic samples [2], complex control systems, *big-data* analysis procedures and new advanced instruments such as fast detectors with high spatial resolution and dynamical range [6]. Progresses in all these relevant issues are being achieved.

The expected opening of Sirius to users will certainly bring new, exciting and challenging research opportunities to Brazilian and international materials science communities.

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