Report : HE-3797

The coexistence of a high magnetic moment, with superconductivity in $A_xFe_{1-y}Se_2$ systems calls for systematic probes to check whether it is a microscopic coexistence or a nanoscopic phase separation and whether there is a structural divergence between the surface and the bulk. We have undertaken comprehensive single crystal X-ray diffraction studies to address these questions [1-3]. Our temperature dependent single crystal X-ray diffraction studies of superconducting $K_{0.8}Fe_{1.6}Se_2$ in transmission mode probing the bulk shows a phase separation in the system below 520 K where a first expanded phase with superlattice modulation coexists with a second collapsed phase with relative weight of around 85:15 [1]. To investigate the phase separation between the compressed and expanded phases at nanoscale, the $K_{0.8}Fe_{1.6}Se_2$ single crystal, is analyzed using the scanning nanofocus diffraction facility. Measurements were done with photons of energy 14 KeV, with a 300 nm focused beam-size on the sample. The sample is fixed in a moving stage for spatial scanning to permit the maps of regions of the sample. Within the same 300 × 300 nm^2 crystal surface-area, illuminated by the X-ray spot, we see the coexistence of compressed and relaxed lattice domains.

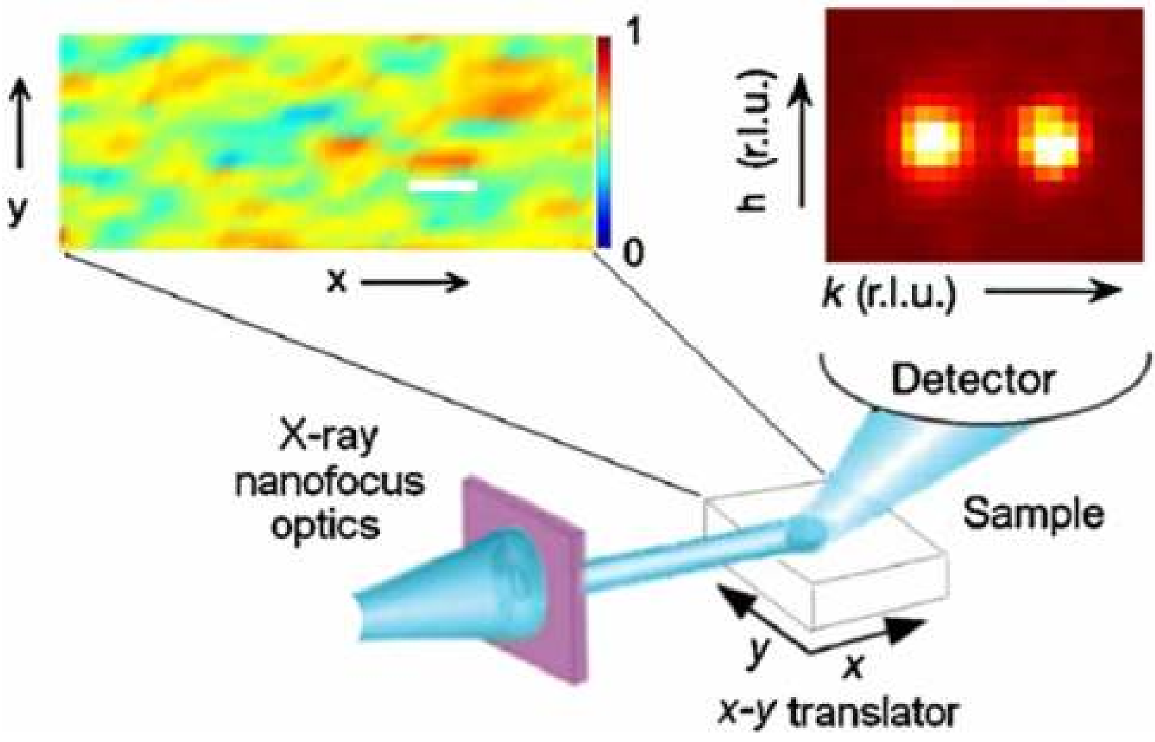

Figure 1. The X-ray nanofocus apparatus located at ID13 beamline providing stable 14 keV monochromatized X-ray beam of size approximately 300 × 300 nm2 by KB-mirror focusing optics. Upper-right panel shows a CCD recording the X-rays scattered by the sample. The room temperature coexisting two phases in K0.8Fe1.6Se2 are evidenced by the splitting of the main Brag peak reflections. The intensity of the compressed phase, I (c), and of the relaxed phase, I (r) is integrated over square subareas of the image recorded by the CCD detector in reciprocal-lattice (r.l.u) and then normalized to the intensity I0 of the tail of the main crystallographic reflection at each point (x, y) of the sample reached by the translator. The nanoscale phase separation is visualized in the color-maps showing the intensity distribution map [I (c) − I0] / [(I (r) − I0) + (I (c) − I0)]. The scale bar corresponds to 5 µm

Present observation of an intrinsic phase separation with the coexistence of a relaxed and compressed phase [2,3] suggest the importance of inhomogeneities in the $A_xFe_{1-y}Se_2$ systems. Indeed the phase diagram of involving Fe–Se show the possibility of coexisting phases for different ratios between Fe and Se [4], consistent with extreme sensitivity of the chemical composition to the superconducting properties of FeSe [5]. The coexisting chalcogen heights observed in the doped ternary chalcogenides [6] are found to be more pronounced in the $A_xFe_{1-y}Se_2$ systems [7].

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