

Nanostructure of the Iron Chalcogenide Superconductor $\text{Fe}_{1+y}\text{Te}_x\text{Se}_{1-x}$

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High- T_c superconductors have great potential in energy saving for real world applications, such as power transmission lines, electric motors, maglev trains, and etc. Discovery of the high- T_c iron-based superconductors, such as $\text{LaFeAsO}_{1-x}\text{F}_x$ [1], have attracted renewed interest in high- T_c superconductors. Among the iron-based superconductors synthesized so far, 11- type $\text{Fe}_{1+y}\text{Te}_x\text{Se}_{1-x}$ [2] has the simplest crystal structure, comprised of FeTe/Se layers only, which is ideal for investigating the underlying mechanism in superconductivity. Investigation on the structure of $\text{Fe}_{1+y}\text{Te}_x\text{Se}_{1-x}$ is essential to further understand the properties of this material. Scanning transmission electron microscopy(STEM) and electron energy loss spectroscopy(EELS) are applied to examine crystal and electronic structures of $\text{Fe}_{1+y}\text{Te}_x\text{Se}_{1-x}$ single crystals.

In STEM, incoherently scattered intensities, collected by the high angle annular dark field (HAADF) detector with a large inner cutoff angle, are proportional to atomic numbers. Fig. 1 shows HAADF images at low magnification of (a) $\text{Fe}_{1.07}\text{Te}_{0.75}\text{Se}_{0.25}$, (b) $\text{Fe}_{1.08}\text{Te}_{0.55}\text{Se}_{0.45}$, (c) $\text{FeTe}_{0.7}\text{Se}_{0.3}$ and (d) $\text{FeTe}_{0.55}\text{Se}_{0.45}$, along the c axis. The images show clear interconnected, fractal-like, contrast for all of the samples. The brighter regions contain higher Te concentration than the darker regions, since Te atoms are heavier than Se atoms. EELS can be recorded simultaneously in STEM mode. The characteristic energy of core loss edges in EELS, resulted from inner-shell ionization, provides direct chemical information. Quantitative analysis on Te content and energy-loss near-edge structure (ELNES) of the Fe- $L_{2,3}$ edge, especially the L_3/L_2 white-line intensity ratio, are studied by the STEM-EELS technique. Fig. 2(a) shows a HAADF image of $\text{FeTe}_{0.7}\text{Se}_{0.3}$ with a line demonstrating the path along which EELS spectra were recorded. A typical spectrum is shown in Fig. 2(b). The integrated Te- $M_{4,5}$ intensity along the scanning path after background subtraction is shown as the red curve in the Fig. 2(c). The change in the integrated intensity indicates that the fluctuation of Te concentration along the scan by 20% or more. The intensity ratio of the Fe L_3 and L_2 edge along the EELS scan was also calculated, shown as the black curve in the Fig. 2(c). The change in the L_3/L_2 intensity ratio shows a clear correlation with the Te content fluctuation. These results demonstrate direct evidences of phase separation in $\text{Fe}_{1+y}\text{Te}_x\text{Se}_{1-x}$ single crystals and changes in the d-electron states at the nanometer scale. Nanostructures of Oxygen annealed samples will be also discussed.

References

- [1] Y. Kamihara et al. *JACS* 130 (2008) 3296-3297
- [2] K. W. Yeh et al. *EPL* 84 (2008) 37002

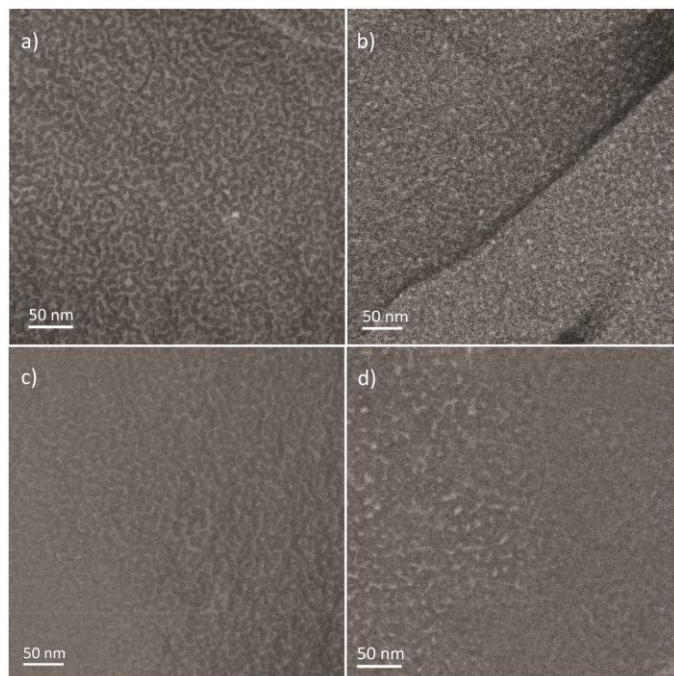


Figure 1. HAADF images at low magnification sample (a) $\text{Fe}_{1.07}\text{Te}_{0.75}\text{Se}_{0.25}$, (b) $\text{Fe}_{1.08}\text{Te}_{0.55}\text{Se}_{0.45}$, (c) $\text{FeTe}_{0.7}\text{Se}_{0.3}$ and (d) $\text{FeTe}_{0.55}\text{Se}_{0.45}$.

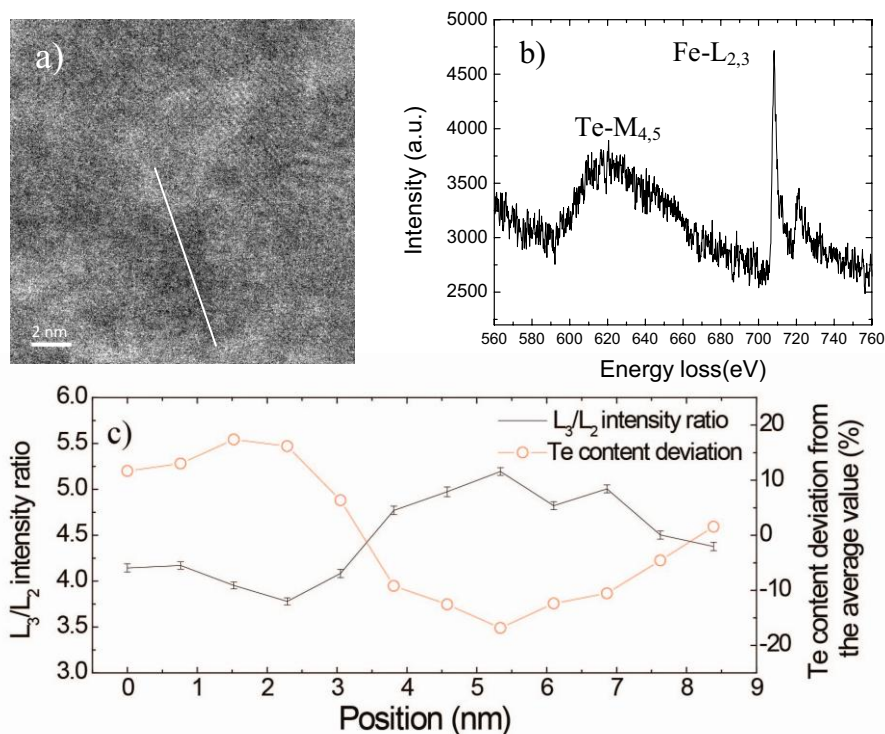


Figure 2. (a) A HAADF image of $\text{FeTe}_{0.7}\text{Se}_{0.3}$ with a line demonstrating the path along which EELS spectra were recorded. (b) a typical energy loss spectrum. (c) The red and black curves show variation of $\text{Te-M}_{4,5}$ intensity and L_3/L_2 white-line intensity ratio along the EELS scan, respectively.