

## Tracking the Structural and Chemical Evolution of Nanostructured Materials by *In-Situ* Experiments

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Bulk nanostructured materials show extremely high strength compared to bulk materials, and have received unprecedented attention in material research also due to its potential applications in industry. Severe plastic deformation is an emerging and quite efficient route to generate nanocrystalline materials out of normally immiscible systems. However, the main issue on nanostructured materials is its thermal stability [1]. In this contribution, the thermal stability of nanostructured materials will be explored at a very high-spatial resolution using modern spherical transmission electron microscopy (TEM) via simultaneous *in-situ* imaging and spectroscopy analysis. CuCr nanostructured alloy was utilized for *in-situ* monitoring the structural evolution and chemical composition changes simultaneously.

A coarse-grained Cu-Cr composite material (43wt % Cr, 57wt % Cu, and corresponding to 48 at % Cr and 52 at% Cu, respectively) was deformed by high pressure torsion (HPT) at room temperature. The initial microstructure of the composite consists of a Cu matrix with Cr particles (volume fraction of about 50%, mean diameter of about 50  $\mu\text{m}$ ) produced by PLANSEE (Reutte, Austria). Disks with a diameter of  $\sim 8$  mm and a thickness ( $t$ ) of  $\sim 0.8$  mm were HPT deformed for different numbers of turns  $n$  under a constant pressure of 6.25 GPa with a rotation speed of 0.2 rotations/minute. HPT disks which were deformed for 25 turns (equal strains = 400) were used for *in-situ* annealing in TEM at 25°C, 212°C and 414°C after preparing TEM specimens from the edge of the disk, where the highest strain has occurred.

For a comparison, normally immiscible Cu-Fe alloy system was mechanically alloyed directly from blended powders and vacuum arc-melted bulk respectively which contain different levels of content of oxygen impurity, with a series of compositions (100- $x$ ) at.%Cu -  $x$  at.%Fe ( $x = 0, 5, 15, 25, 35$ ), by means of high pressure torsion.

A JEOL 2100F field emission microscope (200kV) equipped with an image-side  $C_s$ -corrector which possesses a 1.2 Å resolutions at 200 kV was used. The aberration coefficients were set to be sufficient small. The HRTEM images were taken under slightly over-focus.

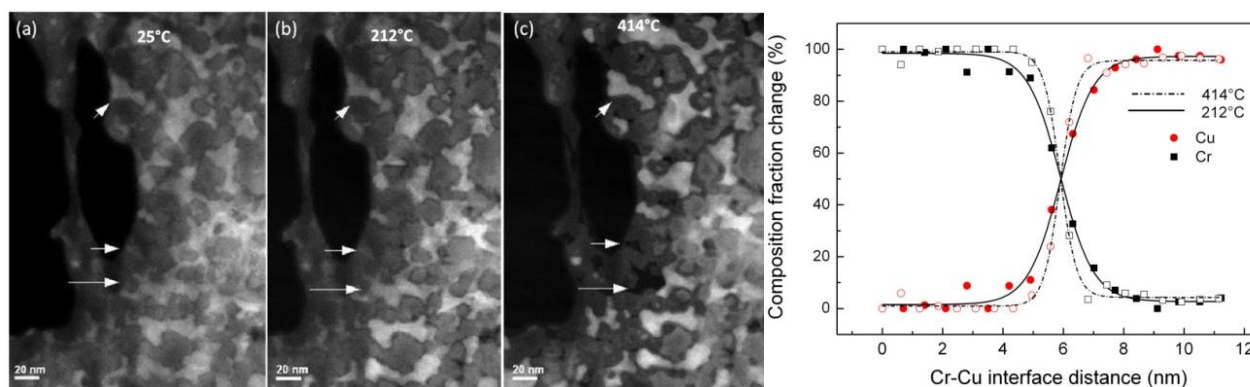
The evolution of the structural and chemical composition in the nanostructured materials with temperature was tracked in *real-time*. It demonstrates that the nanostructured materials are not only subjected to a structural change but also to obvious chemical composition fluctuations upon annealing. The destabilization process in the nanostructured materials starts at a quite low temperature (Figure 1). *Real-time* imaging and composition determination reveal the concentration changes with temperature, and allows further analyzing the dynamic behavior in nanocrystalline materials in details, i.e. deducing the instantaneous diffusion coefficients and excess vacancy concentration generated by severely deformation, and interface sharpening with annealing is observed. The experiments demonstrated a direct approach to measure the excess vacancy concentration via measuring elemental profiles, except visualizing the morphology evolution. Essentially, the study uncovers the interplay between the thermal stability and chemical decomposition process of bulk nanostructured materials in *real-time*. [2]

On the other hand, the dynamic process in nanocrystalline materials is modified when the impurity level (i.e. oxygen) in nanostructured materials is higher. One example, such as Cu-Fe (25%wt Fe) deformed using powders precursors which contain a different content of oxygen, is given, demon-

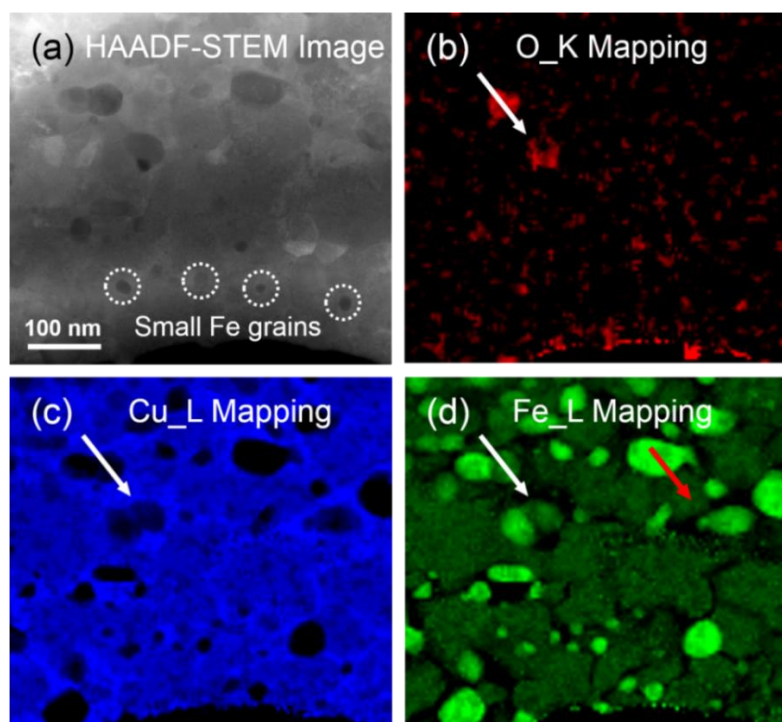
strating a total different behavior when annealed. Surprisingly, nano-oxides form firstly (Figure 2), and then followed by a decomposition of supersaturated solid solution upon heating [3, 4].

#### References:

- [1] RZ Valiev *et al*, *Mater Res Lett* **4** (2016), 1.  
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 [3] Jinming Guo *et al*, *Nature communications* **9** (2018), 946.  
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**Figure 1.** Left (a) (b) and (c): HAADF STEM image demonstrating the nanocrystalline structural evolution process; (a) 25°C, (b) 212°C, (c) 414 °C. Particular locations labeled by arrows. Some pores are formed immediately upon 414 C. Right panel: Elemental profile crossing one CrCu boundary.



**Figure 2.** EELS elemental mapping of annealed sample (CuFe nanostructured materials) at 420°C. (a) HAADF-STEM image. (b) O\_K mapping. (c) Cu\_L mapping. (d) Fe\_L mapping.