

Aerosol Cu Ultra Fine Nanoparticles Size by XRD vs Bundle Diffusion Batteries

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Keywords: Copper, ultra-fine nanoparticles, XRD, Diffusion Batteries

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The synthesis of very small nanoparticles (NPs) with size below 100 Å, realized by using gas flow-reactors working at high temperatures, is one of the few experimental technique that can avoid or minimize phenomena like agglomeration, partial coagulation, coalescence and coating of NPs by impurities, whilst, alas, is characterized usually by very scarce yield. Thus, gas-flow aerosol routes in the technology have been a natural choice in those processes aimed to the generation of standard aerosol, f.i., for calibration, etherogeneous nucleation or to the deposition of very thin substrates, which can work as selective gas sensors. These last nanostructure gas sensors sometimes can detect selectively, and in reversible way, even sub-ppm traces of surrounding gas under test such as NO₂ (di Stasio 2004; Baratto et al 2004; di Stasio and Dal Santo, 2006). In this work we use a quartz flow reactor with a two-furnace arrangement. A mixture of Argon and Hydrogen carrier gases is input of the first furnace, under which, at central longitudinal abscissa, is positioned a quartz boat containing Cu-ACAC powder used as precursor species. The Cu-ACAC foodstock grains are thermally decomposed at a relatively low temperature (about 200 °C) and with short residence-transit times (adjusting Ar flux). Immediately after (with respect to the direction of the gas flux) the first oven, the vapours are input into the second oven, which is positioned just adjacently to the first one. This second is significantly longer with respect to the first one in order to obtain longer transit times. Temperature in the second furnace is ranging from about 400 to 700 °C. Inside the reactor under the second furnace the vapours undergo further chemical decomposition, thus producing at output of second furnace supersaturation burst of copper metal vapours, which quickly nucleate and form metal solid very fine copper nuclei (about 5 nm size).

We sample the NPs obtained with aerosol quartz filters and we perform the XRD measurements. Subsequently, the rough data are stripped, smoothed, subtracted from quartz filter background and corrected in order to account for the diffractometer instrumental line broadening. This last operation is made by a contemporary measurement on the same instrument of diffractograms from standard LaB6 powder, which is both a standard for peak positions and for line width characteristic of the diffractometer used.

From theory of X-ray diffraction and applied crystallography, we learn how to infer from XRD

experimental data, the size of the copper crystallites averaged over all the directions represented by the Miller's crystal indices (i, j, k).

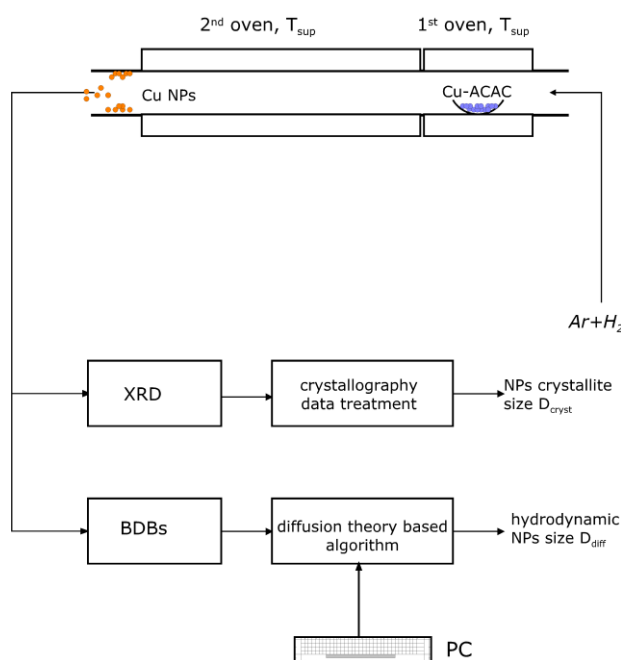


Figure 1. Scheme of the experimental set-up for copper NPs generation and of *a-posteriori* data treatment.

Finally we compare the NPs sizes so obtained via XRD with respect to the modal values returned from the measurements of the Cu aerosol flux, at output of the second furnace, performed with a Bundle Diffusion Battery (BDBs) and we show that the two kinds of physical data (XRD vs BDBs NP size) do compare surprisingly well, within the experimental error.

Detailed results will be shown at EAC 2016 conference presentation.

This work was supported by the international Cooperative Scientific Agreement between Italian National Research Council (CNR) and Siberian Branch of Russian Academy of Science (SB-RAS) for the years 2011-2013.

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