Challenges on the microstructural characterization of nanocrystalline alloys produced by Mechanochemistry

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Almost two decades ago our research group has performed synthesis and characterization of nanocrystalline alloys looking for green and efficient way to get technological materials for several kinds of applications [1 - 3]. This work aims to show a microstructural analysis of the nanocrystalline phases produced by mechanical alloying taking into account its peculiarities due to high level of defects expected ball milling elemental powders. For that we carried out X-Ray Powder Diffraction (XRPD) experiments in laboratory (LDRX-UFSC) and Wide Angle X-Ray Total Scattering (WAXTS) measurements in synchrotron light sources (XRD1 beamline at LNLS – Brazil [4] and MS beamline at SLS - Switzerland). A critical comparison of two computational tools will be presented, the TOPAS [5] and DebUsSy [6] (Debye User's System) suite. Here, the first two uses the well-known Rietveld refinement procedure [7] and are able to furnish several very useful structural information as well phase percentages, being the microstructural characterization quite limited to average crystallite size, microstrain, stacking fault, etc. The last tool (DebUsSy) makes use of a different approach where the Debye's equation is used to calculate a theoretical WAXTS pattern from a modeling of the microstructure consisting of virtual sets of nanoclusters of different sizes and shapes. It is very powerful to show how the information about the diffuse scattering of nanocrystals affects the baseline of the diffraction pattern and do not include it in the background modeling, as usual in Rietveld-like approach (modeling of the diffraction pattern). Moreover, DebUsSy allows to take into account structural (lattice size, occupancy and atomic dislocation) deformations as a function of the crystallite size, considering different crystallite shapes (spherical, cylindrical, cubical, etc.) and its size distribution (monomodal, bimodal, etc.). Here we plan to critically discuss about the procedures to obtain diffraction data free of experimental contributions (such as instrumental's, air scattering's, sample holder's, capillary's, etc.) and taking into account absorption and packing (density) effects from real samples [8]. To illustrate our results on the microstructural analysis some of the diffraction data collected from the mechanical alloyed $Ni_{33}Te_{66}$ sample (containing a trigonal $NiTe_2$ phase, space group *P-3m*) will be presented, as well as the experimental procedures of the samples preparation using a high-energy ball mill.

REFERENCES

1. C.E.M. Campos, J.C de Lima ; K.D. Machado, T.A. Grandi, P.S. Pizani, Structural studies of iron selenides prepared by mechanical alloying. solid state communications, v. 123, n.3, (2002) 179-184.

2. K.F. Ulbrich, F. Bertolotti, N. Masciocchi, A. Cervellino, A. Guagliardi and C.E.M. Campos, A comprehensive structural and microstructural investigation of a new Iron-Telluride nano phase. Journal Materials Chemistry C, vol.6, (2018) 3047-3057.

3. K.F. Ulbrich, J.P. Winiarski, C. L. Jost, C.E.M. Campos. Mechanochemical synthesis of a Ni3-xTe2 nanocrystalline composite and its application for simultaneous electrochemical detection of dopamine and adrenaline. Composites Part B-Engineering, v. 183, (2020) 107649.

4. A. M. G. Carvalho, D. H. C. Araújo, H. F. Canova, C. B. Rodella, D. H. Barrett, S. L. Cuffini, R. N. Costa, R. S. Nunes, X-ray powder diffraction at the XRD1 beamline at LNLS, J. Synchrotron Rad., 23, (2016), 1501-1506

5. A. A. Coelho, TOPAS and TOPAS-Academic: an optimization program integrating computer algebra and crystallographic objects written in C++, J. Appl. Cryst. 51, (2018), 210-218.

6. A. Cervellino, C. Giannini, A. Guagliardi, DEBUSSY: a Debye user system fornanocrystalline materials, J. Appl. Cryst, 43, (2010), 1543-1547.

7. L. B. McCysker, R. B. Von Dreele, D. E. Cox, D. Louër, P. Scardi, Rietveld Refinement Guidelines, J. Appl. Cryst., 32, (1999), 36-50.

8. M. Bowden, M. Ryan, Absorption correction for cylindrical and annular specimens and their containers or supports, J. Appl. Cryst 43, 69, (2010), 693-698.