X-ray diffraction analysis of tin metal powder produced by high energy ball milling

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Ingot commercial Sn was sealed together with 8 steel balls of 10 mm in diameter into a cylindrical steel vial under argon atmosphere. Due to the notorious ductility of tin, 3wt% of stearic acid was added, as a process control agent (PCA), to prevent an excessive cold-welding among particles and among particles and milling media during ball-milling. The ball-to-powder weight ratio was 8:1. The vial was mounted on a SPEX Mixer/mill, model 8000, and milling was performed at room temperature. The structural changes occurring in the sample with milling time were followed by recording the X-ray diffraction (XRD) patterns on a Bruker D2 Phaser powder diffractometer, using the CuKα radiation (λ =0.15406 nm). Using the GSAS package[1] the Rietveld method[2] was used to refine the structural parameters from the XRD patterns, following the guidelines recommended by the IUCr[3] Chebyschev polynomials were used to fit the background, while a convolution of the modified Thompson-Cox-Hasting pseudo-Voigt was used to fit the peak shapes. A silicon standard sample was used to take into account the broadening due to instrumental effects, and it was assumed that the thermal parameters were isotropic. The nanometric structure is formed by crystallites having dimensions of few nanometers (from 2 up to 100 nm) and the mean size peaks of the crystallites can be satisfactorily estimated by Scherrer's equation. The table 1 shows the refined crystallographic Sn parameters of the three samples, where we can see an increase in the c lattice parameter resulting in a decreasing in the Sn cell volume and crystallite size.

Table 1 – Results of crystallographic analysis by the Rietveld method

Time Ball Milling	*a = b (Å)*	*c (Å)*	*V(Å^3)*	*ρ (g/cm^3)*	*d (nm)*	*Rwp (%)*
1 h	5.8302	3.1809	108.120	7.292	97	9,5
3 h	5.8304	3.1810	108.133	7.291	85	6,8
6 h	5.8308	3.1811	108.151	7.289	62	7,6

Keywords: High Energy Ball milling, Rietveld method, Tin metal powder.

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