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Cadmium Sulfide (CdS) preparation by High-Energy Ball Milling

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Cadmium sulfide (CdS) is a type II–VI semiconductor widely used as photosensitizer in various wide band-gap metal oxides and in visible-light photocatalyst for H_2 evolution, due to its satisfactory band-gap (2.4 eV) that matching with the average spectrum of sunlight [1]. Usually, this material has been obtained in the nanocrystalline form by chemical route (bath deposition), using some organic molecules as precursors (hydrazine, thiourea, thioacetamide, dimethylcadmium etc.); however, some reagents commonly used in this route are costly, toxic, flammable and carcinogen [2]. Thus, some alternative routes have been explored, one of them based on processing in solid state, is called mechanical milling (MM) [3,4], where cadmium (Cd) and sulphur (S) are used as precursors for the CdS synthesis. The source of the anion sulphide is S, which has the advantage that is non-toxic for the human body.

In the present work some CdS samples were prepared using a mechanochemical route (based on MM) departing from elemental powders avoiding the use of corrosive and dangerous reagents. Raw materials were: pure elements Cd and S in powder form. Some equiatomic mixtures of precursors were milled in a SPEX high-energy mill using steel balls with a ratio (milling media to powder) of 10:1 (in wt.). The CdS mixtures were mechanically milled during 0, 1, 2, 4 and 8h periods. Structural evolution was followed by X-Rays Diffraction (XRD) in a Panalytical X'pert diffractometer using a Cu cathode (λ = 0.15406 nm). Morphological characterization and chemical analyses were performed with a high-resolution scanning electron microscope JSM-7201F.

The Fig.1 shows the morphological changes of CdS mixtures after milling. The formation of flakes (1 and 2h) and particle comminution (8h) by the milling process is noticed. In the Fig. 2 using the backscattered electron mode a strong contrast between particles at early milling stages (1h) is evident. After 8h of processing, differences in contrast are reduced; this suggests that high level of chemical homogeneity in the milled particles is reached. Also, new types of particles that do not correspond to the elemental powders are visible (indicating formation of CdS compound). EDS analyses show chemical homogeneity, with compositions near the stoichiometric (Cd78S22) with further milling. The previous evidence is corroborated with the Fig. 3, where some diffractograms are showed. After 2h of milling, the diffraction lines of the compound CdS appears, but in form of broad and short peaks, indicating the formation of CdS in form of small crystals, this induced by the milling action of mechanical processing. Deeper experimentation is being carried out in order to obtain CdS particles at nano scale using the described route complemented with the use of different processing additives and milling parameters.

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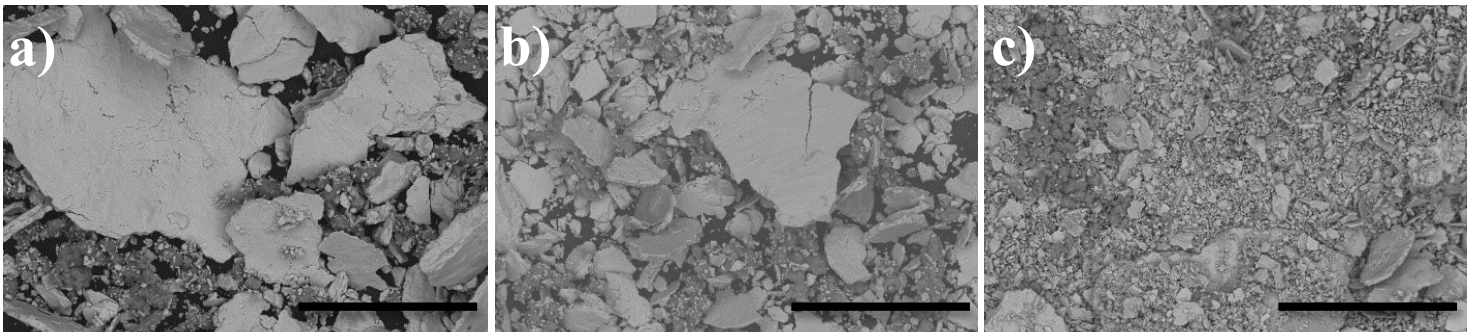

Figure 1. SEM micrographs of samples after: a) 1, b) 2 and c) 8h of milling (bars = 100 μm).

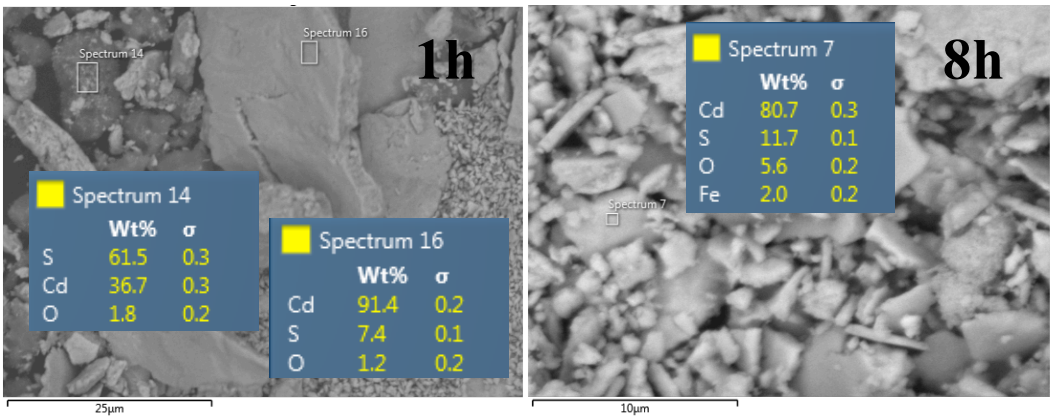

Figure 2. SEM micrographs and EDS elemental analyses of milled CdS mixtures. With further milling (8h), the analysis shows undesirable presence of iron as contaminant from wear and tear of milling media.

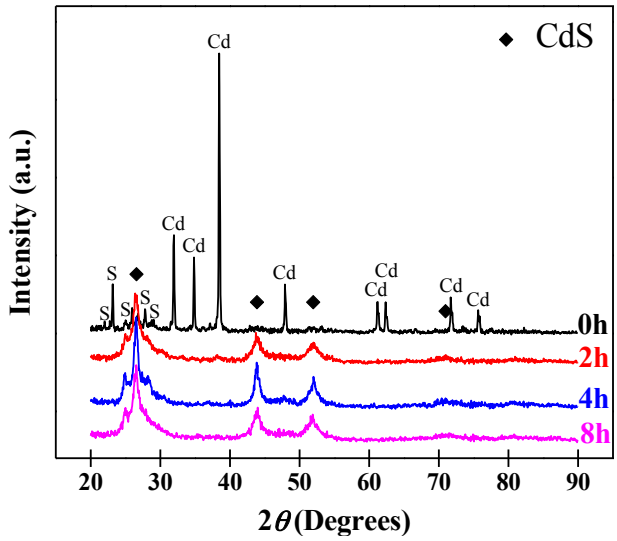

Figure 3. XRD patterns for milled CdS samples.