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Structure and physicochemical properties of MgB_2 nanosheets obtained via sonochemical liquid phase exfoliation

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ABSTRACT

We have performed ultrasonic liquid phase exfoliation (LPE) of MgB_2 in ethanol and investigated the resulting nano-assemblies as a function of the ultrasound processing time. TEM morphological and structural analysis, along with STM topographic characterization, showed that for short sonication times the exfoliated grains preserve the MgB_2 crystal structure, have a 2D character and produce Moiré patterns corresponding to stacked layers with rotational misalignment. On the other hand, the longest process times result in spheroidal nanoparticles with diameters of the order of 10 nm, which can also coalesce into larger agglomerates. Optical absorbance spectra confirmed that the exfoliated material preserves a metallic nature with a predominant 2D character corresponding to the *ab*-plane, whereas Raman spectra showed the presence of extra-modes induced by 3D symmetry breaking in the exfoliated products, along with indications of some B-H stretching modes. The detection of a positive zeta potential confirms that an active surface hydrogenation process has taken place during sonication. The mechanochemistry of the exfoliation mechanism has been attributed to particle–particle collisions and particle-shockwave interactions originating from the implosive bubble-collapse. The present study provides important information useful for implementing the LPE process in different contexts for MgB_2, like few-layer superconductivity, antibacterial coating and nano-drug preparation.

1. Introduction

Liquid phase exfoliation (LPE) is a simple and versatile processing method to produce atomically thin, scalable quantities of two-dimensional (2D) sheets by direct exfoliation [1–7]. Its primary microscopic mechanism is represented by cavitation bubble collapse, which generates a combination of high energy microjets, interparticle collisions and direct shockwave-solid interactions to yield exfoliated 2D-nanosheets [8–13]. Typically, bulk solids of layered materials are the candidates of choice to undergo the LPE process, but recently also non-layered materials with non-isotropic 3D bonding arrangements (like, for instance, strong in-plane ionic or covalent bonds versus weaker out-of-plane van der Waals or metallic bonding forces) have been successfully treated [14–16].

Generally speaking, 2D-nanostructures have already been obtained from several metal-boride materials and their possible applicability to various fields, like e.g. sensing, electronics, catalysis, energy harvesting, ion transport, hydrogen generation and storage, has been recently discussed [17]. In the specific case of MgB_2, its crystal structure is analogous to that of intercalated graphite, having alternate graphene-like boron basal honeycomb layers (borophene) sandwiched by a close-packed Mg-triangular layer [18,19]. This implies that non-isotropic atomic bonds exist, with strong covalent bonding within each layer and weaker metallic bonding between different layers [20]. For this reason, MgB_2 is expected to preferably cleave at the site of the Mg-B bonds between adjacent layers [21]. In principle, this material could be of interest as a starting point for the production of borophene single layers and indeed several methods have already been used in this direction to obtain MgB_2 nanosheets, including chemical exfoliation by means of chelating agents [22,23], sonication in water [21] or high energy ball milling in an inert atmosphere [24]. However, these methods have shown a deep influence on the MgB_2 structure and properties, resulting in amorphization or high distortion of its crystal structure [21,23], in the heavy reconstruction of the B surfaces [24] and in the

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