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High-energy ball milling routes for preparing hydrogen storage TiFe-Nb alloy

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In this study TiFe-Nb alloy (6.8 at.% of Nb), utilized in hydrogen storage applications, was produced using three different routes involving high-energy ball milling followed by synthesis reaction heat treatments. The starting materials used in each of these milling routes were varied: Route 1 (TiH2 + Fe + NbH), Route 2 (TiFe + NbH), and Route 3 (TiFe + Nb). The millings, for 2h, were perfomed in a SPEX shaker mill under a purified argon atmosphere. The milled samples were characterized before and after the synthesis reaction treatments using X-ray diffractometry (XRD) and scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX). Differential exploratory calorimetry (DSC) and thermogravimetry (TG) analyses were performed to evaluate the behavior of the milled samples during heating up to 1273 K, with a heating rate of 10 K.min-1. The synthesis reactions under vacuum were performed with a heating rate of 10 K.min-1 up to 888 K for Route 1 and 823 K for Routes 2 and 3. Route 1 provided the best milling yield, with 65.4%. Pressure-composition isotherms for hydrogen absorption and desorption were obtained with homemade Sieverts-type equipment operating in dynamic mode (constant flow). The Route 1 milled samples reached hydrogen absorption and desorption at room temperature under pressure plateaus of approximately 0.93 and 0.21 MPa, respectively. The hydrogen storage capacity was 0.75 wt.% of H2 at 2.1 MPa, with a reversal of 0.73 wt.% of hydrogen at 0.04 MPa bar. The Route 2 milled samples obtained better reversibility, with 0.55wt.% absorption and desorption of hydrogen for pressures of 0.03 to 2.1 MPa and 1.1 to 0.04 MPa, respectively.