Mg-Ni ALLOYS FOR HYDROGEN STORAGE OBTAINED BY BALL MILLING

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Abstract— **The effect of the atmosphere and milling time on the structural, morphological and hydrogen reactivity properties of the mechanically alloyed Mg-Ni system is studied. Nanocrystalline Mg_2Ni formed by ball milling in argon exhibits a higher thermal stability than the metals amorphized by milling during shorter times. It is shown that the initially fast hydrogen absorption kinetics of Mg_2Ni at 250°C strongly depends on the crystallinity degree. When the Mg-Ni mixture is mechanically alloyed under hydrogen atmosphere, MgH_2 is formed at first and Mg_2NiH_4 is obtained later. Samples prepared in argon show a higher hydrogen absorption capacity than those obtained in hydrogen.**

Keywords— **Mg-Ni, metallic hydrides, mechanical alloying, hydrogen absorption.**

I. INTRODUCTION

In the last years, several studies related with mechanically alloyed materials useful for hydrogen storage have been reported. This technique allows obtaining intermetallic compounds that react with hydrogen in a reversible process. In this way, the properties of the materials related with their surface, their particle size and the density of defects are important factors to be considered to improve the rate of the reaction with hydrogen (Abdellaoui *et al*., 1999; Huot *et al*., 1995; Liang *et al*., 1998; Orimo *et al*., 1997). In particular, there are several works on the Mg-Ni system that analyze the product of the mechanical alloying from pure metals (Huot *et al*., 1995; Liang *et al*., 1998), the effect of milling parameters on the formation of the Mg_2Ni intermetallic compound (Orimo *et al*., 1997; Tessier *et al*., 1998) and also the characterization of the reaction with hydrogen.

In this paper we study the effect of milling time on the thermodynamics and the structural properties of Mg-Ni mixtures (2:1 atomic proportion) prepared under argon or hydrogen atmospheres. The hydrogen reaction kinetics is also analyzed.

II. EXPERIMENTAL

Mechanical alloying of Mg-Ni mixtures was performed in a Uni-Ball-Mill-II (Australian Scientific Instruments) apparatus, using ferromagnetic steel balls as milling media and an external magnetic field controlling the movement of balls.

Mg granules (purity higher than 99.9%) and Ni powder (purity higher than 99.9%) in the atomic proportion 2:1 were placed in the milling chamber in the argon atmosphere of a glove box. The ball to powder weight ratio used was 18:1. The milling was performed in two atmospheres: argon (purity 99.995%) at a pressure of 0.1 MPa or hydrogen (purity 99.995%) at a pressure of 0.5 MPa. Samples of the product of milling were taken from the chamber, opened in the argon glove box, at regular intervals of time. Their structures were studied by X-ray diffraction (XRD, PW 1710, Philips Electronic Instruments) using CuK_α radiation and Ni filter, at 40 kV and 30 mA. In order to investigate their thermal stability, samples milled during 108 and 273 hours were thermally treated at 200°C during 10 min under argon atmosphere. The thermal behavior of the samples was studied by differential scanning calorimetry (DSC 2910, TA Instruments) at a heating rate of 25°C min^{-1} and an argon flow rate of 18 ml min^{-1}. The total hydrogen content was determined by fusion and decomposition (LECO RH-404). Scanning electron microscopy (SEM 515, Philips Electronic Instruments) and energy dispersive X-ray analysis (EDX) were used to observe the microstructure.

The samples were placed in a closed system with constant volume under pure hydrogen atmosphere (99.995%) to study the hydrogen absorption kinetics. They were kept in vacuum at 250°C dur-