Structural characterization of equiatomic MgAlVCrNi high entropy alloy for hydrogen storage application.

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Developing safe, reliable, efficient and cost-effective materials for hydrogen storage is a scientific and technological challenge that must be overcome to leverage a clean and renewable hydrogen-based energy system. High entropy alloys (HEAs) are unconventional metallic materials that have at least five major elements and can form multicomponent solid solutions. Recently, it was shown that the equiatomic TiVZrNbHf HEA, which crystallizes as single BCC solid solution, is capable to store 2.5 hydrogen atoms per metal atom, which is considerably higher than conventional metal hydrides such as MgH_2. This work presents the characterization of the novel MgAlVCrNi HEA produced by reactive milling (under 3 MPa of H_2 pressure) from pure elements. Transmission Electron Microscopy (TEM) is one of the most important tools to determine the crystallite size and the chemical homogeneity of reactive milled nanostructured powders. Figure 1 shows the TEM images of a single powder particle of the reactive milled MgAlVCrNi. The electron diffraction pattern (figure 1(c)) reveals that this single particle is composed only of a BCC phase with lattice parameter of 2.94 Å. The dark field image shows that the mean crystallite size is about 15 nm. The EDS chemical map presented in figure 2 (b) shows that the chemical composition of the single particle is quite uniform. It can be seen in table 1 that there is a deviation from the theoretical chemical composition of the alloy, which can be related to either chemical inhomogeneity between different particles or to the loss of some elements (mainly Mg) during the milling process. When subjected to 100 C and 60 bar of hydrogen pressure (in a Sieverts-type apparatus) reactive milled alloys is capable to absorb up to 1 %wt. of hydrogen (Figure 3 (a)). Figure 3 (b) shows the X-ray diffraction (XRD) results (only the 110 peak) of (i) the as-reactive milled sample; (ii) sample after a differential scanning calorimetry (DSC) run up to 600 C (hydrogen desorption procedure); and (iii) sample after hydrogen absorption at 100 C and 60 bar. The XRD results demonstrated that the hydrogen absorption takes place without any phase change. As can be seen in figure 3 (b), the hydrogenated sample presents a peak shift to the left when compared to the desorbed sample, indicating an increase of the lattice parameter due to the presence of hydrogen in the interstitial sites of the BCC structure.

Table 1: Theoretical and measured (EDS) chemical composition a single powder particle of the reactive milled MgAlVCrNi alloy.

%at.	Mg	Al	V	Cr	Ni
Theoretical	20	20	20	20	20
EDS	13.1	18.4	22.1	24.5	22.0

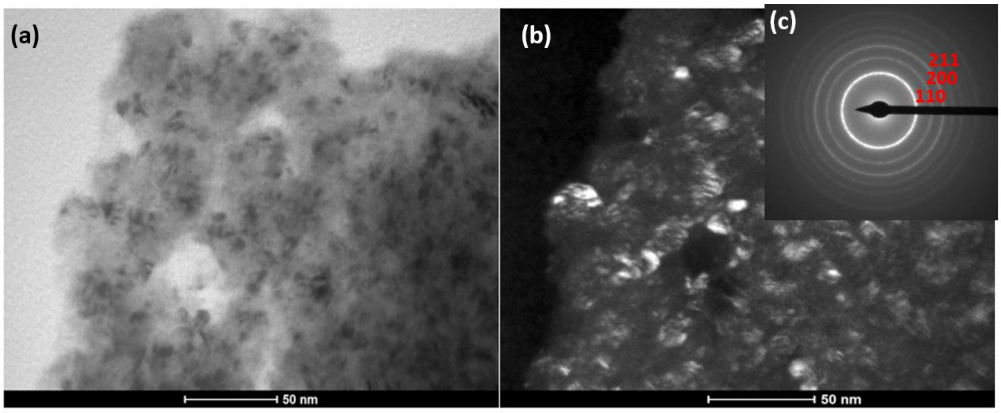

Figure 1: TEM **(a)** Bright Field, **(b)** Dark Field using the 110 reflections ring, and **(c)** Electron Diffraction Pattern of the reactive milled MgAlVCrNi alloy.

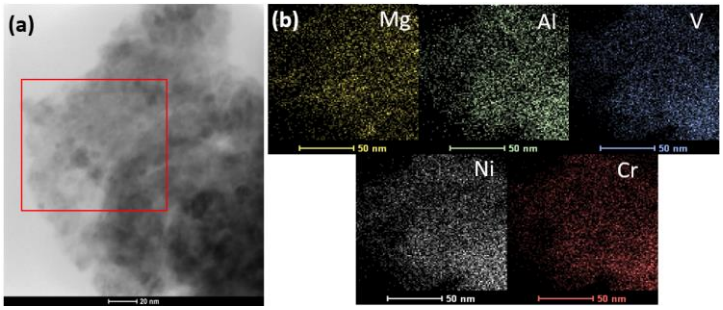

Figure 2: (a) Bright Field STEM image and **(b)** EDS composition map of the marked region of the reactive milled MgAlVCrNi alloy

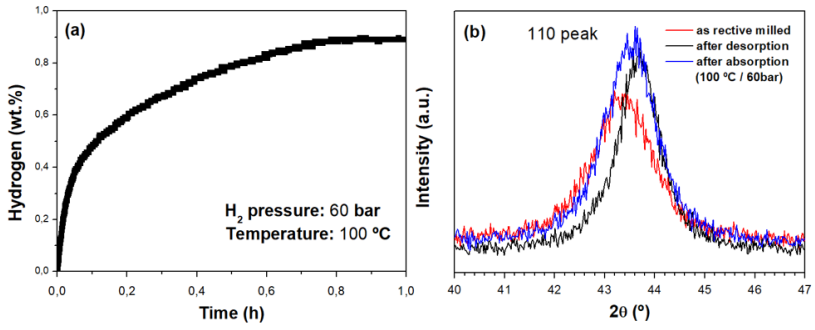

Figure 3: (a) Hydrogen absorption kinetic curve of the reactive milled MgAlVCrNi alloy and (b) XRD patterns showing the peak shift (110 reflection) caused by lattice distortion as result of hydrogen absorption.