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研究課題名（和文）Development of New Mg-Based Hydrogen Storage Materials by Binding-Energy Engineering

研究課題名（英文）Development of New Mg-Based Hydrogen Storage Materials by Binding-Energy Engineering

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研究成果の概要（和文）：室温での水素貯蔵を可能にする低い水素束縛エネルギーによる新しい水素貯蔵材料の開発実験に第一原理計算とCALPHAD法を組み合わせた。高圧下ねじり加工法で指定された材料を合成した結果、様々なMg，Ti、ハイエントロピーベースの水素貯蔵材料を発見した。この中でも、Mg4NiPdは、束縛エネルギーの計算と一致し、0.8 wt%の水素を室温で可逆的に貯蔵した良い例である。２番目の良い例はTiZrCrMnFeNiで、1.7 wt%の容量で高速かつ可逆的な室温水素貯蔵を示した。希土類フリーのTiZrCrMnFeNiは活性化せず水素を貯蔵するが、多くの水素貯蔵合金は追加の活性化プロセスを必要とする。

研究成果の学術的意義や社会的意義
A theoretical/experimental method is reported to design and synthesize new solid materials that can store hydrogen at room temperature. Such materials can potentially be used not only for compact and safe storage of hydrogen in stationary applications but also for energy storage in Ni-MH batteries.

研究成果の概要（英文）：First-principle calculations and CALPHAD methods were combined with experiments to design new hydrogen storage materials with low hydrogen binding energy that can store hydrogen at room temperature. The high-pressure torsion method was used to synthesize the designated materials, resulted in discovery of various Mg-, Ti- and high-entropy-based hydrogen storage materials. Among these materials, Mg4NiPd was one good example which reversibly stored 0.8 wt% hydrogen at room temperature, in agreement with binding energy calculations. The second example was TiZrCrMnFeNi, which showed fast and reversible hydrogen storage at room temperature with 1.7 wt% capacity (30% higher than capacity of commercial rare-earth-based LaNi5). Rare-earth-free TiZrCrMnFeNi stored hydrogen without activation, but many hydrogen storage alloys need an extra activation process. This study showed a clear method to design new room-temperature hydrogen storage materials using the concept of binding energy engineering.

研究分野：Materials Physics and Chemistery

キーワード：Hydrogen Storage　Metal Hydrides　High-Pressure Torsion

１．研究開始当初の背景

Hydrogen is considered as a clean fuel with zero CO_2 emission, but a major issue in development of hydrogen as a fuel is its safe and compact storage. Among different hydrogen storage techniques such as gas-, liquid- and solid-state storage, the solid-state storage in the form of metal hydrides provides the most compact technology. However, the main drawback of metal hydrides is their need to a heating process for hydrogen absorption (activation and kinetic problem) and/or hydrogen desorption (thermodynamic stability and high hydrogen binding energy problem). Although several materials such as $LaNi_5$, TiFe and Ti-V-Cr exhibit good thermodynamic features for room temperature hydrogen storage, these materials have some limitations: TiFe and Ti-V-Cr suffer from poor air resistance and need a thermal or mechanical activation process, $LaNi_5$ which is used commercially for stationary hydrogen storage and for nickel-metal hydride (Ni-MH) batteries suffer from high price of rare-earth La element and low storage capacity as 1.38 wt%. Therefore, design and synthesis of new materials for room temperature hydrogen storage is still a challenging task.

２．研究の目的

The goal of this study was to propose a strategy of designing and synthesizing new compounds which can reversibly store hydrogen at room temperature with fast kinetics and without need to activation process. The main focus of the study was on ternary Mg-based alloys, but the strategy was further extended to the Ti-based alloys and high-entropy alloys.

３．研究の方法

As briefly summarized in Fig. 1(a), to engineer new ternary Mg-based compounds with a low hydrogen binding energy close to zero, our strategy is mixing two different types of binary compounds at the atomic scale: a compound with a negative hydrogen binding energy (such as Mg_2Ni) and a compound with a positive hydrogen binding energy (such as Mg_2Pd). Although many different compositions can be fabricated by mixing the compounds with negative and positive hydrogen binding energies, we initially selected Mg_4NiPd (Mg_2Ni + Mg_2Pd) among many other possible choices because of well-known catalytic effect of Ni and Pd elements on hydrogen storage in Mg-based alloys, which minimizes the effect of kinetic issues on the experimental results. However, the study was further extended to various kinds of Mg-based, Ti-based and high-entropy alloys. For these studies, hydrogen binding energy and the crystal structure of alloys were investigated by theoretical methods such as first-principles calculations and CALPHAD technique.

Although the above-mentioned strategy for Mg_4NiPd seems feasible from a theoretical perspective, there are some technical limitations for mixing these compounds homogenously and achieve a uniform atomic-scale elemental distribution (i.e. a homogenous single phase) because of the thermodynamic immiscibility of Mg in many different systems. One effective solution for atomic-scale mixing of Mg with other elements at the atomic scale is the application of severe plastic deformation (SPD) using high-pressure torsion (HPT) method. The HPT method, as schematically shown in Fig. 1(b), was used in this study and the synthesized materials were characterized by various methods such as X-ray diffraction (XRD) analysis, in-situ synchrotron examination under hydrogen, scanning electron

microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS) and electron-back scatter diffraction (EBSD), transmission electron microscopy (TEM), aberration-corrected scanning-transmission electron microscopy (STEM), atom-probe tomography (APT), differential scanning calorimetry (DSC), thermogravimetry (TG), hydrogen pressure-composition-temperature (PCT) isotherm measurement and hydrogen kinetic test.

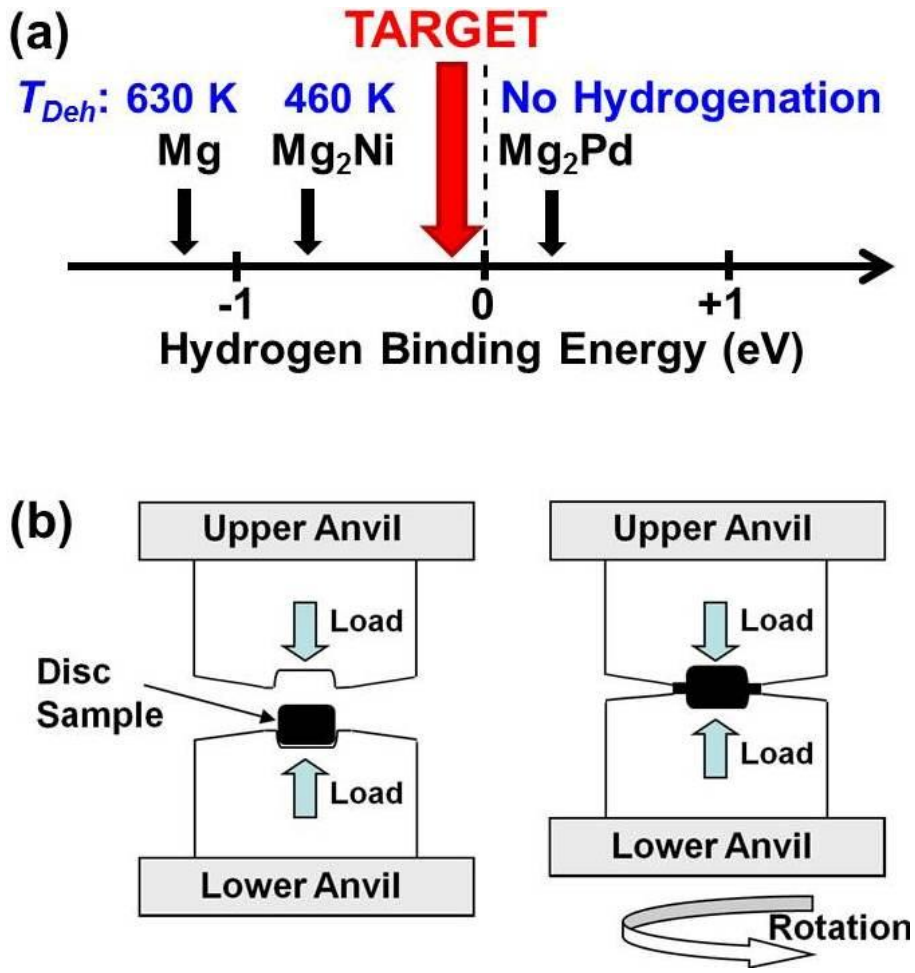

Fig. 1. (a) Schematic illustration of hydrogen binding energy effects on hydrogen storage behavior of materials in the Mg-Ni-Pd system, and (b) schematic illustration of high-pressure torsion (HPT) method [Edalati et al, Acta Mater. 149 (2018) 88-96].

４．研究成果

Theoretical evaluation of Mg_4NiPd by first-principles calculations showed that Mg_4NiPd in a homogenized form should have a BCC-based cubic crystal structure with a hydrogen binding energy as low as -0.12 eV, which is appropriate for room-temperature hydrogen storage. The designated alloy was then fabricated by arc melting and homogenized by the HPT method. Homogenization was confirmed from the micro scale to the atomic scale using SEM, STEM and APT methods. Detailed crystallographic examinations confirmed that the synthesized alloy after HPT processing had a cubic BCC-based crystal structure, in good agreement with the predication of first-principles calculations. The Mg_4NiPd alloy could reversibly absorb and desorb hydrogen at room temperature, as shown in Fig. 2, confirming that the concept of binding energy engineering could be used successfully to design a

new room-temperature hydrogen storage material. Here, it should be noted although the hydrogen storage capacity of this alloy was <1 wt%, the alloy is considered as one of the first Mg-based alloy that could reversibly store hydrogen at room temperature. It should be noted that the Mg-based alloys are the first materials introduced for solid-state hydrogen storage, but long-time attempts to reduce their working temperature to room temperature (without addition of rare-earth elements) has not been successful. Following the successful results on Mg_4NiPd, a similar concept was applied to the Mg-V-Cr, Mg-V-Ni, Mg-Ni-Sn Mg-Ti-V-Cr-Fe systems, but the Mg-V-Cr alloy was the only alloy which could exhibit hydrogen storage at room temperature, but its capacity was smaller than 1 wt%.

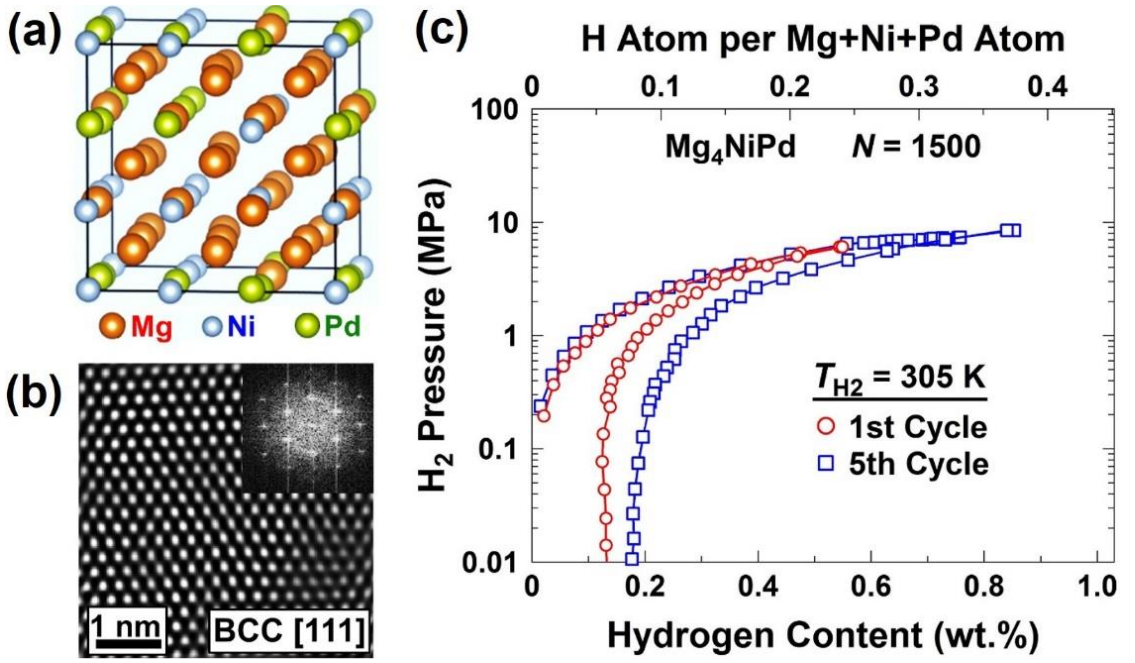

Figure 2. Design of Mg_4NiPd with BCC-based crystal structure using first-principles calculations (optimized structure in a) and its synthesis by the HPT method (high-resolution TEM image in b) with a capability for reversible hydrogen storage at room temperature (PCT isotherms in c) [Edalati et al. Acta Mater. 149 (2018) 88-96].

In another successful attempt, high-entropy alloy TiZrCrMnFeNi with a crystal structure of 95% C14 Laves phase and 5% BCC-type cubic phase was designed by using the CALPHAD method and synthesized by arc melting. The alloy could reversibly absorb and desorb 1.7 wt% hydrogen at room temperature with a fast kinetics and without any need to thermal or mechanical activation treatment, as shown in Fig. 3. The main advantage of this alloy compared to commercial $LaNi_5$ alloy is its higher capacity without including any rare-earth elements in its composition. Moreover, its advantages compared to TiFe and Ti-V-Cr alloys is its high air resistance and no need to the activation process.

In summary, this project confirmed that the concept of "binding energy engineering" can be employed to produce new room-temperature hydrogen storage materials. Such rare-earth-free materials not only can be used for stationary applications but also they have a high potential for application in Ni-MH batteries. The later issue should be examined in future by detailed electrochemical hydrogen storage studies and using the materials for negative electrode of Ni-MH batteries.

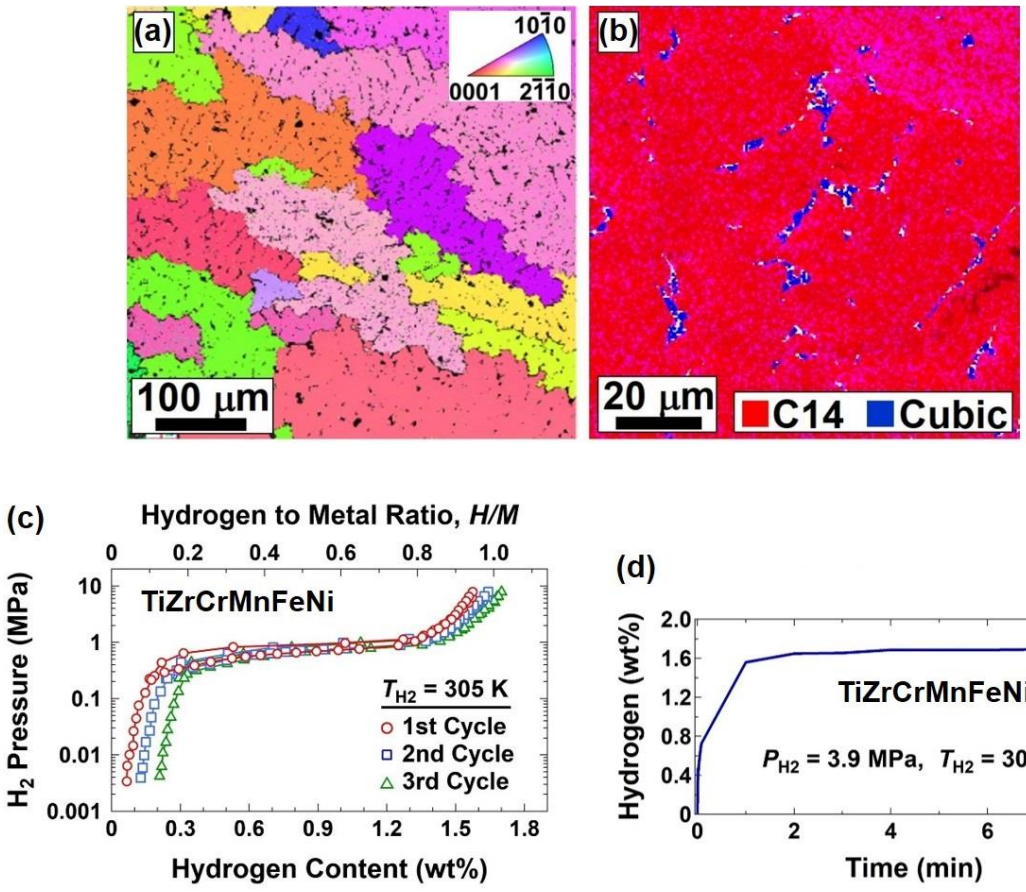

Figure 3. Fast and reversible room-temperature hydrogen storage in high entropy alloy TiZrCrMnFeNi with mainly C15 Laves phase crystal structure. (a) EBSD orientation map, (b) EBSD phase map, (c) PCT absorption/desorption isotherms at 303 K, and (d) hydrogenation kinetic curve at 303 K [Edalati et al. Scr. Mater. 178 (2020) 387-390].

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〔図書〕　計0件

〔産業財産権〕

〔その他〕

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