Experimental report

07/03/2018

Proposal: 5-24-592 **Council:** 10/2016

Title: In-Situ Neutron Diffraction and Phase Transformation of an Hydride Forming High-Entropy Alloy

Research area: Materials

This proposal is a new proposal

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Samples: TiVZrNbHf

Instrument	Requested days	Allocated days	From	To
D1B	0	8	14/02/2017	22/02/2017
D20	4	0		

Abstract:

Hydrogen has a strong potential as an alternative fuel, providing that it can be stored in a safe and efficient way. BCC and laves phase materials show a promising hydrogen capacity. In this proposal a high-entropy alloy (HEA) of TiVZrNbHf crystallizing in a BCC structure will be investigated. The material transforms into a FCT structure upon hydrogen absorption. The alloy has shown large hydrogen storage capabilities, up to 2.5 hydrogen atoms per metal atom. Here we would like to examine, by in-situ neutron diffraction, the mechanism of this phase transformation, as well as the hydrogen position in the crystal structure.

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BCC and laves phase materials show a promising hydrogen capacity, which could be used to store hydrogen for energy usage. In the performed experiment a high-entropy alloy (HEA) of TiVZrNbHf crystallizing in a BCC structure was investigated. The material transforms into a BCT structure upon hydrogen absorption. The alloy had previously shown large hydrogen storage capabilities, up to 2.5 hydrogen atoms per metal atom. The *in situ* experiment performed at the D1B instrument at ILL gave information about structural changes and stability of the material upon hydrogenation at different temperatures.

Experimental

A steel tube, with an inner diameter of 6 mm, was filled with the powdered material and connected to a deuterium tube with possibilities of maximum 50 bar deuterium pressure. The tube was pumped and filled with 50 bar D_2-gas before starting the experiment. Diffraction patterns were recorded for 12 h during heating and subsequent pumping and refilling of deuterium gas. Rietvield analysis [1] on the obtained diffraction patterns was carried out in the program FullProf [2].

Preliminary results

The samples were cycled between vacuum and 50 bar deuterium pressure at 500 °C. The material absorbs deuterium at relative low temperatures, around 200 °C, but higher temperatures are necessary for the deuterium to be desorbed. As can be seen in Figure 1, the information that can be extracted from the *in situ* neutron diffraction is very limited due to the high neutron absorption in the sample. This makes a complete structure refinement very difficult, but some qualitative analysis was performed to determine the deuterium occupancy on the different interstitial sites in the BCT metal lattice. A preferred occupancy of the tetrahedral sites was found, however the difference compared to the octahedral site is very small (53:47). The similar occupancy of the different sites suggests that both the tetrahedral and octahedral interstitial sites are filled simultaneously, rather than filling the tetrahedral sites first. This gives further understanding of the reaction mechanisms for the formation of high entropy hydrides.

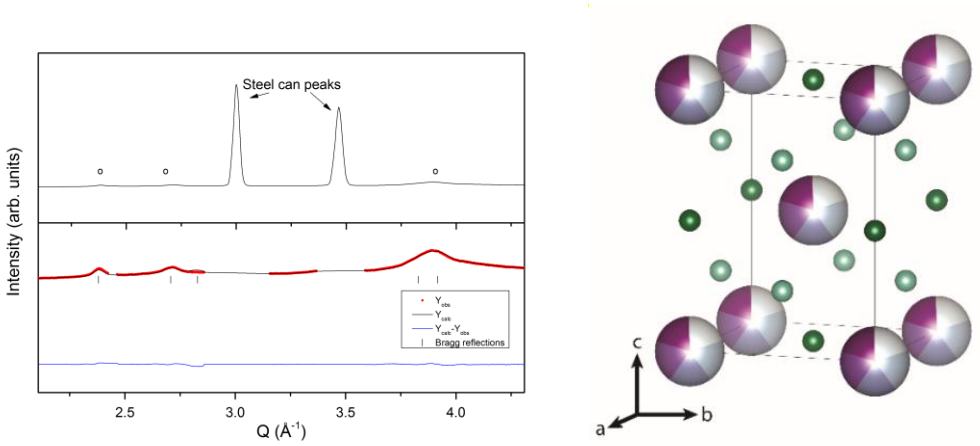

Figure 1. Extracted diffractogram of the hydride phase at 500 °C are shown to the right. The upper pattern shows the total data and the lower is a magnification at the low intensity peaks coming from the hydride. The high absorption of neutrons in the sample, especially from the Hf makes a quantitative analysis difficult. The obtained structure model is presented to the right.

References

[1] Rietveld, H.M., A Profile Refinement Method for Nuclear and Magnetic Structures. Journal of Applied Crystallography, 1969. 2: p. 65-71.

[2] Rodríguez-Carvajal, J., Recent advances in magnetic structure determination by neutron powder diffraction. Physica B: Condensed Matter 192(1), 1993: p. 55-69.