The catalytic effect of Fe and Cr on deuterium absorption in thin Mg films

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We used Neutron Reflectometry (NR) to study the deuterium absorption process in 55 nm thick Mg films alloyed with Fe and Cr. The reflectometry experiments were performed on the D3 reflectometer at NRU in Chalk River. NR enabled us to determine the deuterium content and deuterium distribution in these thin Mg alloy films.

The kinetics of the deuterium absorption can be easily and quickly monitored by measuring the NR curve in a narrow q-range around the critical scattering vector q_c up to which total reflectivity is observed. This is displayed in figure 1, where NR curves of a 55 nm thick MgFeCr alloy film capped with a FeCr/Pd catalyst bilayer are plotted at different times after a deuterium pressure of 8 mbar was introduced into the sample cell at t = 0 at 300 K. The critical scattering vector q_c increases continuously with time until reaching saturation after about 4 hours. This shift of q_c can be taken as a direct measure of deuterium absorption because the deuterium absorption leads to an increase of the Scattering Length Density (SLD) in the Mg alloy layer due to the large scattering length of deuterium. The reflectivity curve measured after 222 min. exposure time corresponds to a D/M ratio of c = 1.5, very close to the maximum expected saturation value of 1.6, calculated under the assumption that all Mg atoms react to form MgD_2.

The continuous shift of q_c as a function of deuterium exposure time nicely proves that the film continuously absorbs deuterium. However, more details on the deuterium concentration profile can be learned by measuring the NR curve in a larger q-range as shown in figure 2, where NR curves are shown after various exposure times as indicated in the graph. The open circles are the experimental data, the solid lines are fits, and the corresponding SLD profiles are shown as insets.

The SLD profiles reveal that there is a deuterium concentration gradient in the Mg-Fe-Cr film with a higher deuterium concentration towards the catalyst layer. The deuterium diffuses through the whole layer as evidenced by the continuous increase of the deuterium concentration at the Mg-Fe-Cr / Ta interface and the fact that deuterium is being absorbed into the bottom Ta layer. Unlike observed in an earlier study on pure Mg films [2], a blocking MgD_2 layer is not formed. So, similar to our previous study on Mg-Cr-V alloys [2], we can conclude that the catalytic effect of the alloying is the prevention of the formation of a hydride diffusion barrier by the segregation of the FeCr.

References

[1] H. Fritzsche, W. P. Kalisvaart, B. Zahiri, R. Flacau, and D. Mitlin, Int. J. Hydrogen Energy **37**, 3540 (2012)

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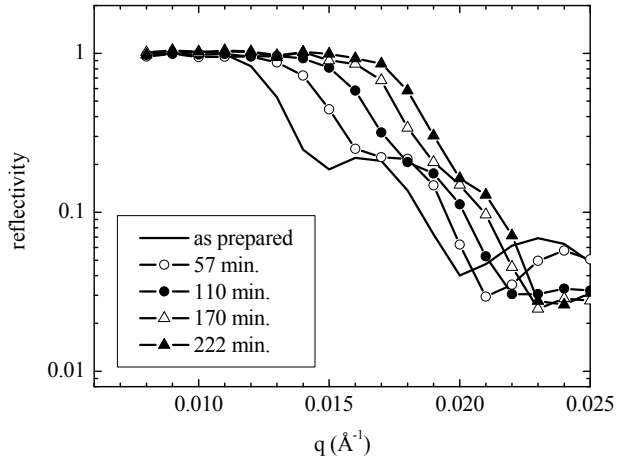

Fig. 1 Neutron reflectivity curves close to the critical scattering vector q_c for a 55 nm thick $Mg_{0.8}Fe_{0.1}Cr_{0.1}$ film capped with a FeCr/Pd bilayer, measured at room temperature in a deuterium pressure of 8 mbar for different exposure times. Reprinted from [1].

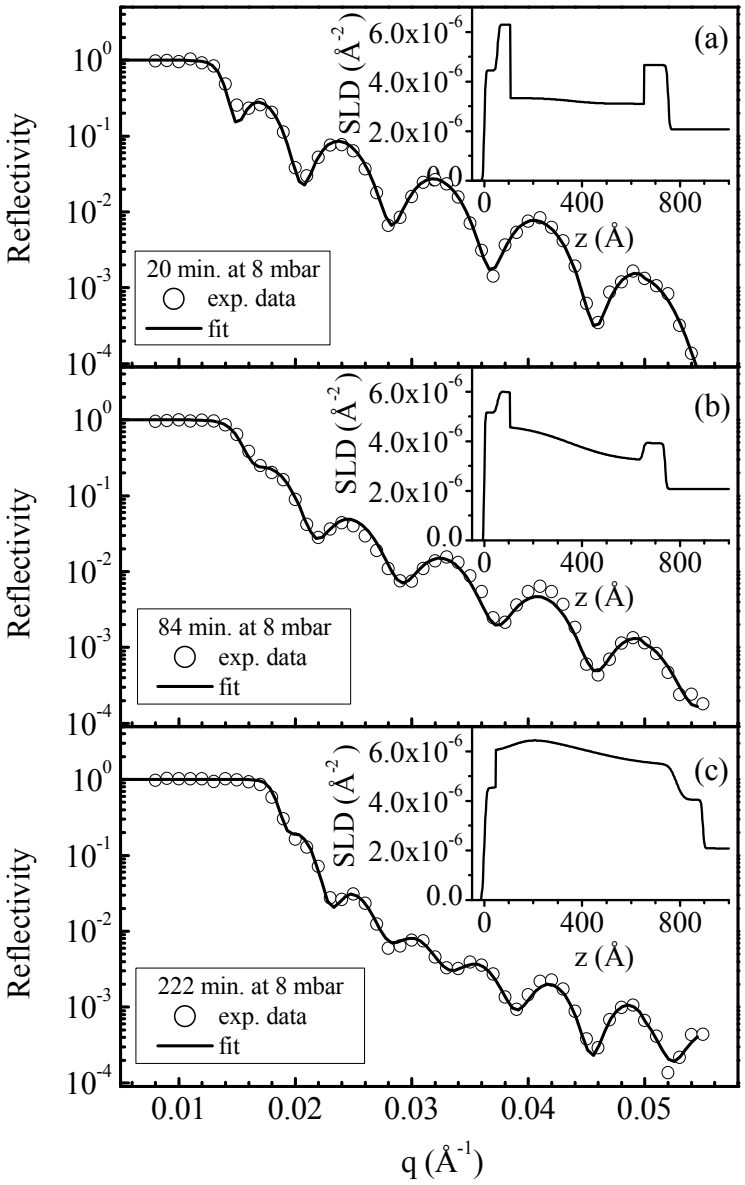

Fig. 2 Measured neutron reflectivity curves (open circles) for a 55 nm thick $Mg_{0.8}Fe_{0.1}Cr_{0.1}$ film capped with a FeCr/Pd bilayer in a deuterium pressure of 8 mbar after exposure of a) 20 min., b) 84 min., and c) 222 min. The solid lines represent fits corresponding to the SLD profiles shown in the inset.