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Electron beam induced dehydrogenation of nanocrystalline MgH_2 investigated by VEELS

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There is an on-going quest for finding a hydrogen storage material that provides both high H_2 storage density and good H_2 absorption as well as desorption properties. Here, nanosized or nanoconfined hydrides promise improved thermodynamics and reaction kinetics. As for the structural characterization utilizing transmission electron microscopy (TEM), however, most materials degrade fast upon the irradiation with the imaging electron beam due to radiolysis. MgH_2 is one of the best studied binary hydrides due to its relatively high storage capacity of 7.6 wt.% H_2. Therefore, ball milled MgH_2 is used as a reference material for in-situ TEM experiments on submicron particles which are comprised of nano-crystals as revealed by XRD measurements. Special care is taken that the sample is not exposed the oxygen or water vapour from ambient air during the transfer from the glove box to the electron microscope. Therefore a vacuum transfer holder is used which successfully prevents the oxidation of the specimen. Valence electron energy loss spectroscopy (VEELS) is conducted using a monochromated FEI Titan[3] 80-300 microscope. From the observation of the plasmonic absorptions in the low loss regime it is found that MgH_2 successively converts into Mg upon electron irradiation (see "Figure 1.") as it was observed in other studies [1,2]. After removing plural scattering using Fourier-log deconvolution the temporal evolution of the spectra is analysed quantitatively by fitting a function composed of two Lorentzians to each spectrum. As a result it can be shown that the signal of the MgH_2 plasmon follows an exponential decay which allows to determine a characteristic electron dose for the electron beam-induced dehydrogenation. From measurements for incident electron energies of both 80 keV and 300 keV characteristic electron doses are deduced. These characteristic doses are roughly 10^4 electrons/nm^2 and 10^6 electrons/nm^2 for 80 keV and 300 keV electrons, respectively (see "Figure 2."). This observation confirms that radiolysis is the major damaging mechanism. By a comparing the characteristic doses of individual particles a dependence on their thickness is found. This indicates that the electron beam-induced dehydrogenation is diffusion limited and as a consequence, reaction kinetics for individual particles can be investigated. This understanding is also crucial for TEM studies on other hydrides such as AlH_3.

1. M. Danaie, S.X. Tao, P. Kalisvaart, D. Mitlin, Acta Materialia 58 (2010), 3162–3172
2. K.-J. Jeon, H.R. Moon, A.M. Ruminski, B. Jiang, C. Kisielowski, R. Bardhan, J.J. Urban, Nature Materials 10 (2011), 286–290

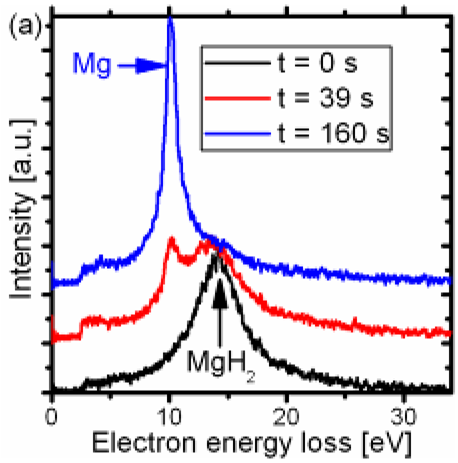

Figure 1. Temporal sequence of three subsequently obtained VEEL spectra of a submicron MgH_2 particle at three different stages during the exposure to the imaging electron beam in the electron microscope.

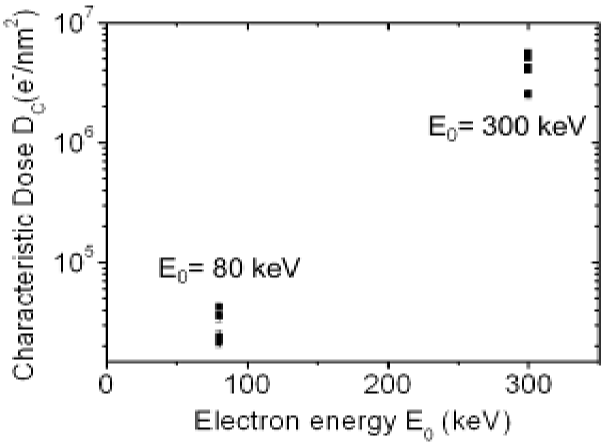

Figure 2. Characteristic electron doses for the electron beam induced dehydrogenation of 14 different MgH_2 particles with incident electron energies of 80 keV and 300 keV.