Application 2001

Raman spectroscopy on MgHx nanoparticles produced by spark discharge generation

Anca Anastasopol

Delft University of Technology, Delft

Spark discharge generation is a bottom up approach to produce nanoparticles. It is a versatile synthesis method to produce metallic nanoparticles, mixtures of metallic nanoparticles and unlikely alloys. In short, a high voltage power supply charges a capacitor bank which is connected in parallel to a spark gap between two metallic electrodes. A flow of Ar is applied between the electrodes and when the breakdown voltage of the Ar is reached, a conductive channel is created. The charge stored in the capacitor bank is released rapidly, at very high temperatures, causing the evaporation of the electrodesŁ?? surface. The vapour cloud condenses into small particles that can further coalesce and form aggregates. The systems that we work on are usually based on Mg[1] and transition metals, like Ti and Nb[2]. The typical primary particle sizes obtained with this method are about 30nm for Mg and 4-5 nm for Nb as seen from TEM images.

If during the synthesis of our metallic particles we introduce a flow of hydrogen or mixtures of hydrogen and argon we obtain hydrides, mostly MgHx. From X-ray diffraction spectra, we found that with different gas mixtures (H2 and Ar) we get different degrees of crystallinity of the hydrides. These results are supported by transmission electron microscopy (TEM). Moreover, the hydrides synthesized in this way have a reduced enthalpy of formation of 60kJ/molH2 as it is obtained from van't Hoff plots. Such a reduced enthalpy of formation for MgH2 without transition metal or other additives has not been reported and makes the study of this type of materials very interesting for the storage of hydrogen.

These are the type of samples that I would like to measure vibrational spectra using Raman and infrared spectroscopy at the Laboratory for solid hydrogen storage materials (Lasy) at EMPA (Switzerland). With this measurements we hope to understand the binding of hydrogen to magnesium. The samples are air sensitive and a special cell needs to be used during the measurements to protect them from oxidation. Though at TUDelft (The Netherlands), several Raman and infrared spectrometers can be found, the measurement of air sensitive samples in this setup is very problematic. Moreover, the Raman setup at the Lasy allows also in situ hydrogenation and dehydrogenation of the samples. This study could provide some insight in the mechanism of the hydrogenation and dehydrogenation of the samples. The estimated time for measurements is three days for five samples of MgHx produced in different Ar/H2 gas mixtures and two MgH2 reference samples.

[1] V.A. Vons, A. Anastasopol, W.J. Legerstee, F.M. Mulder, S.W.H. Eijt and A. Schmidt-Ott, Acta Mater. 59 (2011) 3070-3080

[2] A. Anastasopol, T. V. Pfeiffer, A. Schmidt-Ott, F.M. Mulder, S.W.H. Eijt, APL, 99, 194103 (2011)