Electronic Supplementary Information

Triggering highly stable catalytic activity of metallic titanium for hydrogen storage in $NaAlH_4$ by preparing ultrafine nanoparticles

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Sample characterization

An X'Pert Pro X-ray diffractometer (XRD, Rigaku, Japan) with Cu Kα radiation (40 kV, 40 mA) was employed to identify the phase structure of the samples. The XRD data were collected in the 2θ range of 10-90° with 0.05° step increments at room temperature. The samples were sealed in a custom-designed container with a window covered by Scotch tape for transmission of X-rays to prevent air and moisture contamination. A scanning electron microscope (SEM, Hitachi, S-4800) and a transmission electron microscope (TEM, FEI, Tecnai G2 F20 S-TWIN) were used to observe the morphology and nanostructure of the samples. For the SEM analysis, the sample was transferred quickly to the SEM facility under Ar atmosphere. For TEM observation, the sample was protected with a double tilt vacuum transfer holder (Gatan 648, USA). The distribution of elemental C and Ti was identified using an energy-dispersive X-ray spectrometer (EDS) attached to the Tecnai G2 F20 S-TWIN TEM facility. X-ray photoelectron spectroscopy (XPS) analyses were carried out using a Kratos AXIS Ultra DLD spectrometer. The powder sample was first compacted into a pellet at room temperature and then mounted on the sample holder in Ar atmosphere. It was then transferred from the glovebox to the XPS facility in a transfer vessel for preventing air contamination. The XPS data were recorded using a monochromatic Al Kα X-ray source with a base pressure of 6.8×10^{-9} Torr. The binding energy spectra were fitted using XPSPEAK software. Raman spectra were recorded using a confocal Raman microscope (Via-Renishawplc, UK) at a laser excitation wavelength of 532 nm.

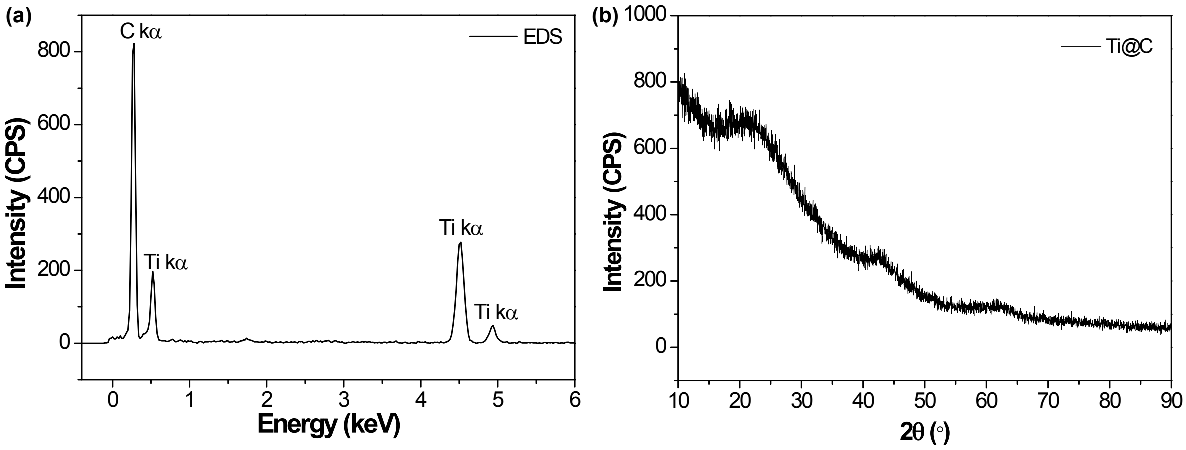

Fig. S1. EDS profile (a) and XRD pattern (b) of prepared nano-Ti@C.

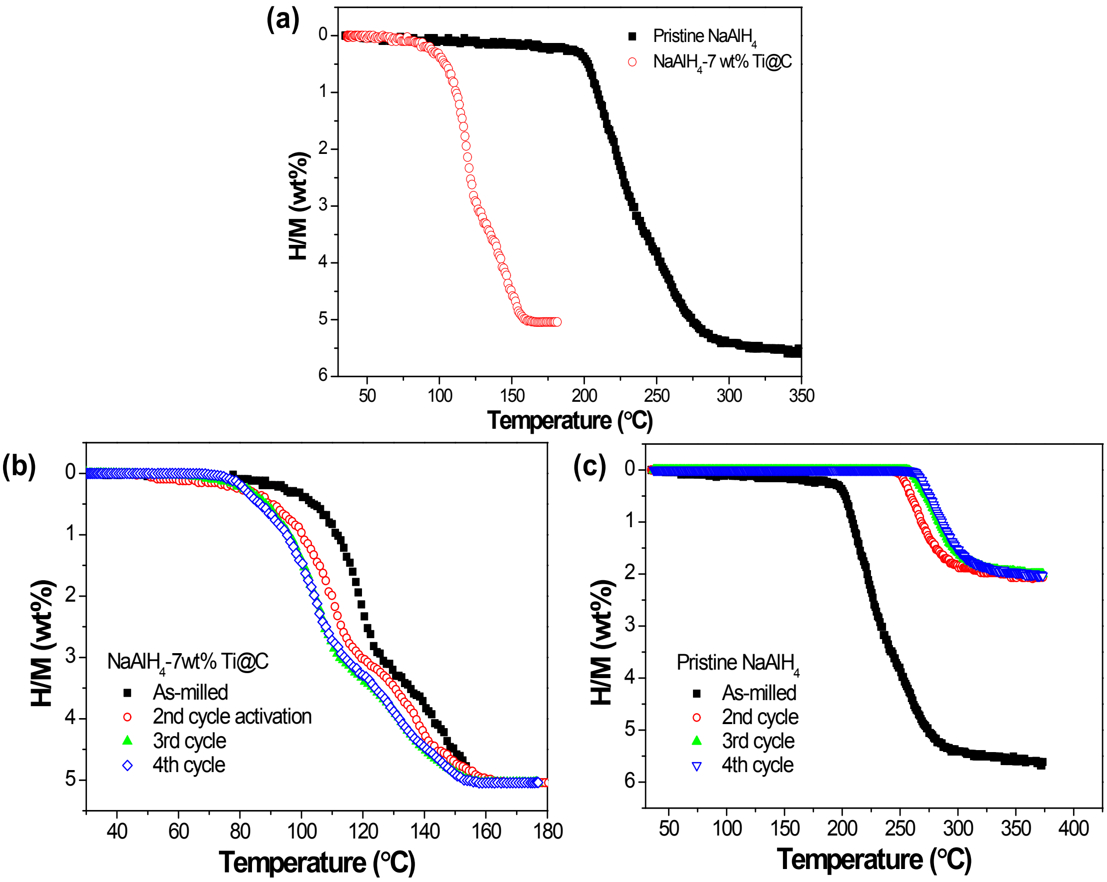

Fig. S2. Temperature-dependent dehydrogenation curves of $NaAlH_4$ with and without Ti@C (a), and cycling dehydrogenation curves of nano-Ti@C-containing (b) and pristine (c) $NaAlH_4$.

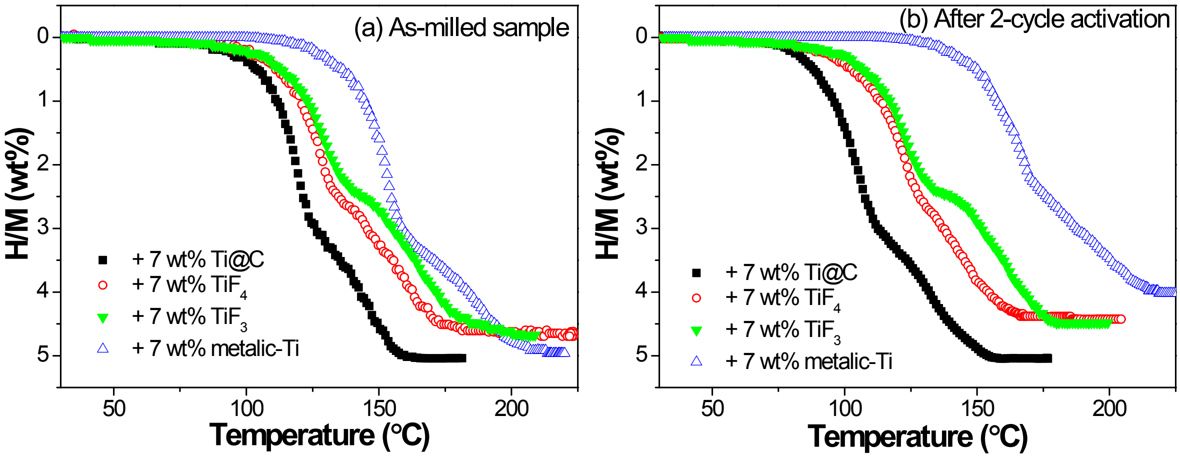

Fig. S3. Temperature-dependent dehydrogenation curves of $NaAlH_4$ with different additives after milling (a) and 2 cycle-activation (b).

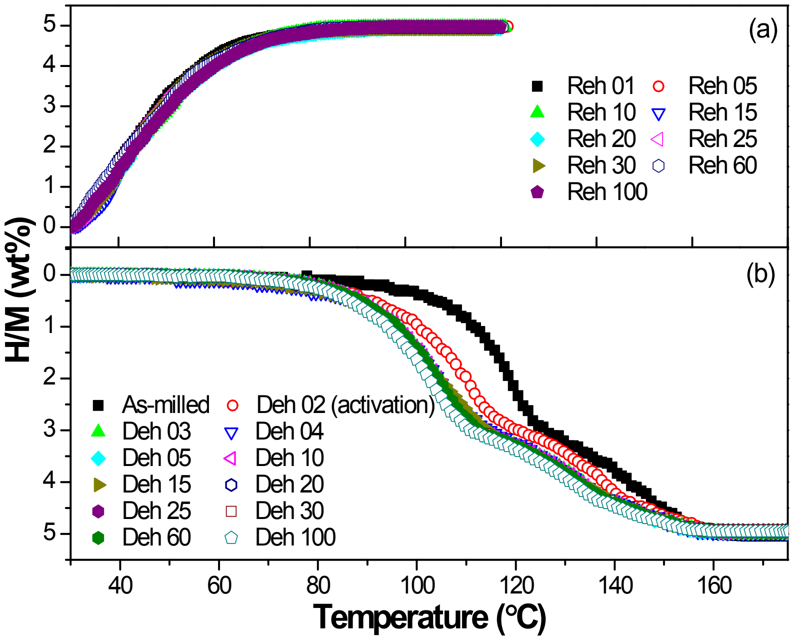

Fig. S4. Non-isothermal hydrogenation (a) and dehydrogenation (b) curves of $NaAlH_4$-7 wt% nano-Ti@C sample upon cycling.

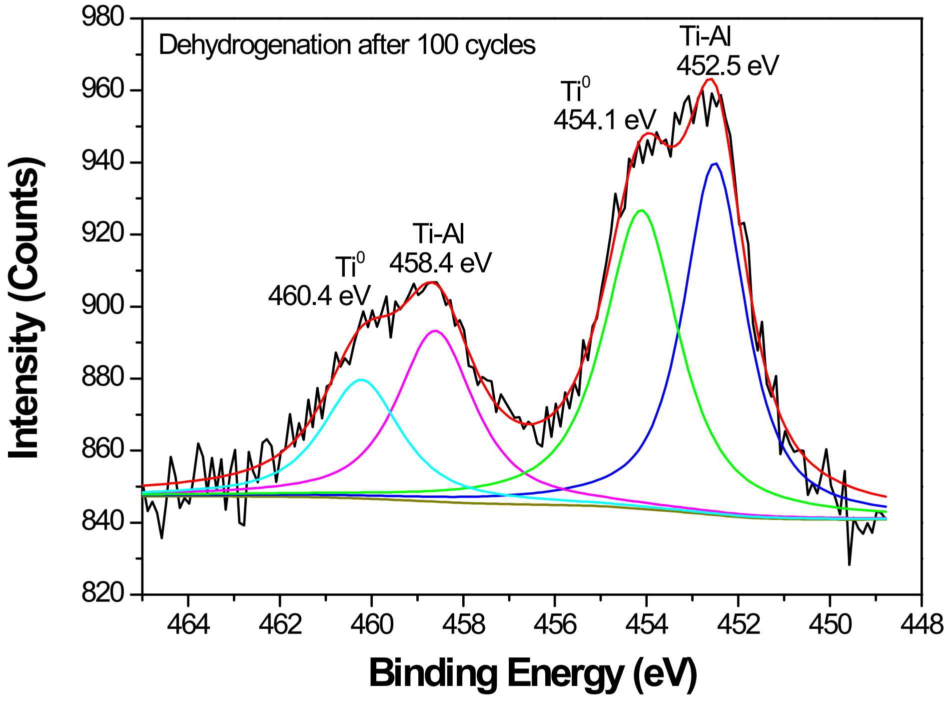

Fig. S5. XPS spectrum of dehydrogenated nano-Ti@C-containing $NaAlH_4$ after 100 cycles.

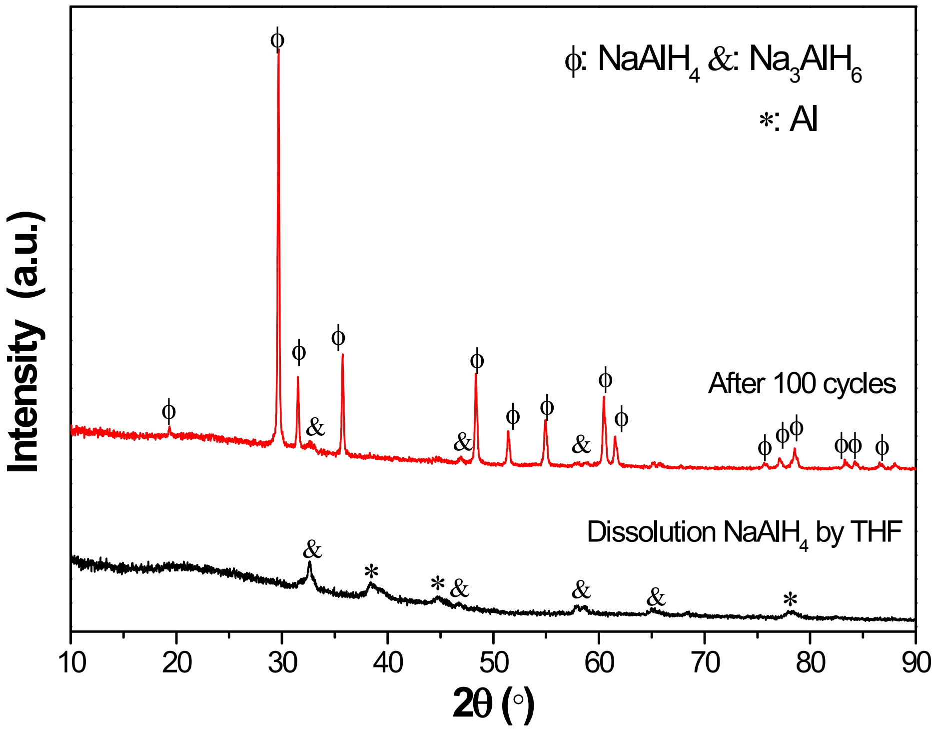

Fig. S6. XRD patterns of post-100 cycled nano-Ti@C-containing $NaAlH_4$ before and after washing with THF.