Efficient Hydrogen Release from Perhydro-N-ethylcarbazole Using Catalyst-Coated Metallic Structures Produced by Selective Electron Beam Melting

W. Peters,^a M. Eypasch,^b T. Frank,^b J. Schwerdtfeger,^c C. Körner,^c A. Bösmann,^a P. Wasserscheid^a*

Received (in XXX, XXX) Xth XXXXXXXX 20XX, Accepted Xth XXXXXXXX 20XX DOI: 10.1039/b000000x

SUPPORTING INFORMATION

Hydrogen Release Unit (HRU) consisting of ten parallel reactors - set up and testing unit

The tenfold, parallel reactor setup was heated by an air stream of up to 450°C and a volume flow rate of 200 NL min⁻¹. For placing the hydrogen-filled reactors directly into the hot air stream, the reactor walls were designed according to the German Pressure Vessel Construction Regulation with a minimum thickness of 2 mm. Due to the test character of the set-up, all connections were realised in a detachable manner. The total volume of the HRU is 4 liter while the total reactor volume of the ten reactors is only 250 mL. The pre-heated H12-NEC is fed into the ten reactors through the unit bottom. The dehydrogenated products as well as the released hydrogen leave the reactor at the top where in the reactor head gas/liquid separation takes place (see Figure S1).



Fig. S1 Hydrogen release unit (HRU) consisting of ten parallel reactors mounted in vertical direction – photograph of the open HRU unit with ten SEBM-manufactured and catalytically functionalized reactor tubes. The unit is surrounded by a box to guide the hot air flow.

Test bench set-up and experimentation

Figure S2 shows the continuous dehydrogenation rig applied for the validation tests of the Hydrogen Release Unit developed within this work. The main components of the applied rig as indexed in Figure 6 include: 1) A storage container for hydrogenated LOHC; 2) a dosing pump for the delivery of the LOHC; 3) the Hydrogen Release Unit (HRU) for the generation of the stored hydrogen as described in the previous sub-chapter; 4) a hold-up tank for the partially dehydrogenated LOHC including a hydrogen outlet for gas/liquid separation; 5) a hot air gun to mimic hot exhaust gas from a hydrogen combustion process as source for the dehydrogenation reaction enthalpy; 6) a system control device. Subsequently to the gas/liquid separation the hydrogen flow rate is measured via a hydrogen mass flow meter and fed into a fuel cell for electricity production including two headlights as electric consumer.

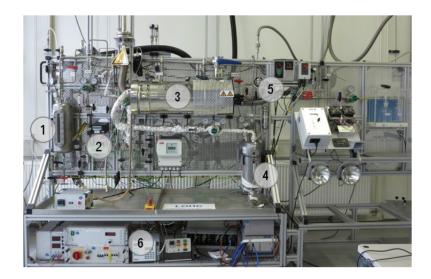


Fig. S2 Continuous test rig for the here described H12-NEC dehydrogenation; the box indexed with "3" contains the Hydrogen release unit (HRU) described in the previous sub-chapter.

The dosing pump 2 transfers hydrogenated LOHC from tank 1 into the HRU 3, where the catalytic dehydrogenation takes place. Before entering the HRU, the H12-NEC is preheated to enter the HRU at the intended reaction temperature. After releasing the hydrogen, the liquid carrier and the gaseous hydrogen product flow through the heated outlet pipe into the hold-up tank where the final gas/liquid separation takes place. The released hydrogen leaves the hold-up tank through an activated charcoal adsorber into the H2-flow meter and from there to either the fuel cell or to the exhaust gas line. The hot air supply for reactor heating is realized by a combination of a blower and a heater. Inlet and exit temperatures of the hot air flow are monitored and controlled in the experiment are the inlet and outlet temperatures of

the LOHC flow at the HRU, the reaction temperature inside the reactor tubes (six different measurement points in six different reactor tubes), the hydrogen exit flow and the system pressure. The latter is measured at two different test points, one between the dosing pump and the HRU and another behind the HRU before the dehydrogenated LOHC enters the hold-up tank. The system pressure of the whole test-rig is 1.6 bar, absolute pressure in all experiments reported here. By adjusting the hot air temperature and the volumetric flow rate, we measured three different isotherms of 230, 250 and 260°C for variable H12-NEC flow rates between 10 and 40 mL min-1. The goal is to monitor hydrogen production rates and catalyst productivities under different reaction conditions to identify suitable operation points of the HRU.