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M.Lototskyy Metal Hydrides – from Basic Materials Development to Applications Overview of MH-related R&D activities of SAIAMC / HySA Systems MH team



Hydrogen South Africa (HySA): Motivation

	Reserves			
Product	% of world total	Rank	Applications	
Platinum Group Metals (PGM)	>75%	1	Hydrogen & Fuel Cell Technologies	
Rare-Earths	0.7%	9		
Titanium	7.9%	4		
Zirconium	18.9%	2	Metal Hydride	
Manganese	29.4%	1	Materials &	
Chromium	39.2%	2	Technologies	
Vanadium	17.5%	3		
Nickel	5.0%	7		









Hydrogen South Africa (HySA): Organization









HySA Systems: What we do

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HySA Systems: resources





Total 3000 m² usable floor space

- 250 m² testing of fuel cell systems
- 150 m² MEA processing and fuel cell preparation
- 1000 m² of MH pilot plant





Director, 2 Programme Managers, 3 Key technology specialists, 3(1) engineers

- 10(2) post-doctoral, 5(1) PhD and (4)MSc students
- 3(1) technicians and 2 research assistants
- 1 administrative staff, 6 Support Staff





MH activities at SAIAMC and HySA SYSTEMS

Background

- Strong expertise in synthetic procedures:
 - Melting & annealling
 - Surface modification / electroless plating (PGM)
 - Carbon nanomaterials (CNT)
 - Reactive Ball Milling
- Various analytical facilities + necessary skill
 - XRD, SEM, TEM
 - BET, DSC
 - Gas analysis (GC, MS)
 - Sieverts apparatus
- Computer modelling (PCT, kinetics, heat-and-mass transfer)
- Infrastructure + necessary skill for components & systems manufacturing and testing
- Strong international links with MH and HFCT teams (Norway, Germany, Croatia, China, Russia, etc.: both MH materials and systems)

MH activities at SAIAMC started in 2004













Recently Completed and Actual MH Related Activities at SAIAMC and HySA Systems

- Poisoning-tolerant surface modified MH materials. MH systems for H₂ separation & purification
- AB₅- and AB₂-type MH alloys: preparation (up to 15 kg/load) & characterisation
- Mg-based nanocomposites for H storage and heat management
- Thermally-driven MH H₂ compressors
- MH system integration:
 - H storage systems for LT PEM FC applications
 - H storage (+ refuelling) systems for utility vehicles















Funding Sources

- South African Government (incl. co-funding of international collaborative projects):
 - Department of Science and Technology (DST);
 - National Research Foundation (NRF);
 - Department of Trade and Industry (DTI) via THRIP programme.
- South African industry:
 - Eskom Holdings Limited;
 - Impala Platinum.
 - International funding:
 - EU via H2020 / RISE program.





Total budget of MH-related projects at SAIAMC – ~ R5m / y







MH projects (HySA funded)

- Key Programme 2 Portable Power Systems:
 - KP2-S01 MH H2-Storage for LT PEMFC Power Systems (2008–2010).
- Key Programme 3 Hydrogen Fuelled Vehicles:
 - KP3-S02 On Board Use of Metal Hydrides for Utility Vehicles (2008–present);
 - KP3-S04 On-Board Hydrogen Storage (2008–2012).
- Key Programme 8 Special Projects:
 - KP8-S02 Metal Hydride Integrated Energy Systems (2008–2011);
 - KP8-S05 Metal Hydrides for underground Mining Applications (2015–present).







HySA Systems projects (Additional funding)

South African Government:

- DST Innovation Project South African Metal Hydride Alloys: Proof of Concept (2012–2014);
- DTI / NRF THRIP project Advanced Materials and Technologies for Hydrogen-Powered Utility Vehicles (2013–2015).
- South African industry:
 - Eskom Materials and Processes for UCG product gas, clean up and storage (MH materials and systems for hydrogen separation, purification and compression; 2008–2015);
 - Impala Platinum Development of a Fuel Cell Powered Forklift Utilising Advanced Metal Hydride Hydrogen Storage and Refuelling (2012–present);
 - Anglo American Platinum Underground use of FC Technologies (2014–2015).
- International funding:
 - International Science and Technology Agreements South Africa Norway Industrial Applications of Metal Hydrides for Hydrogen Extraction, Storage and Compression / HYDROTECH (co-funded by NRF, 2007–2010, prolonged to 2013);
 - ERAfrica FP7 Programme Advanced Hydrogen Energy Systems / HENERGY (co-funded by DST, 2014–2018);
 - Horizon 2020 / RISE Programme Hydrogen fuelled utility vehicles and their support systems utilising metal hydrides / HYDRIDE4MOBILITY (started in February 2018).







Poisoning-Tolerant Surface Modified MH Materials

- 2007-2012; completed:
 - ESKOM-funded SAIAMC project;
 - South Africa Norway Research collaboration project;
 - HySA Systems project KP8-S01.



Eskom

- Substrate: AB₅ (A=La;
 B=Ni+Co+Mn+AI
- Surface modification: fluorination + functionalisation + electroless deposition of Pd (2 international patents)
- No significant deterioration of the surface modified MH was observed (CO up to 100 ppm, CO_2 up to 30%)
- Introduction of $CO_2 + CO$ reduces H_2 separation productivity by 20% only, stable performance over ~200 cycles.



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AB₅- and AB₂-type MH alloys for H storage & compression

- 2009 ...; on going:
 - DST innovation project;
 - HySA Systems projects KP3-S02, (KP8-S01), KP8-S05.



MH Alloys Manufactured at HySA Systems





Mixing with Additives

MH Material Powder



The MH manufacturing facilities were built due to SA Government (DST) funding

- Locally developed induction melting and annealing furnaces
- Production capacity up to 15Kg per a load
- Original recipes (AB₅ & AB₂): customized compositions according to customer's specifications, including target PCT performances for H₂ absorption / desorption
- Mostly South African feedstock
- H storage capacities 160 to ≥200 Ncm³/g
- H₂ equilibrium pressures at T=20°C from
 <1 to ~50 bar
- Suitable for H storage and H₂ compression applications
- Patented solution on the preparation of MH composite and its loading in MH container: easy activation, tolerance to gas impurities, low pyrophority, and better heat transfer performances of the MH bed.



Some AB₂-type MH materials manufactured at HySA Systems



Mg-based nanocomposites for H storage and heat management

- 2007 ...; on going:
 - South Africa Norway research collaboration project;
 - EU FP7 "ERAfrica" research collaboration project (NO, TR, EG, ZA);
 - HySA Systems projects KP3-S02, (KP3-S04);
 - THRIP project.



Thermally-driven MH H₂ compressors

- 2008-...; on-going:
 - ESKOM-funded SAIAMC project;
 - HySA Systems projects KP3-S02, (KP8-S01).



Objective / general approach

- To develop prototype MH H₂ compressors potentially satisfying the requirements of industrial consumers:
 - Providing necessary H₂ pressure (up to 200 bar) and productivity (x 1...10 m³/h);
 - Operation using available infrastructure for heating (steam, water; up to 150 °C) and cooling (water; 15...25 °C);
 - Competitive cost / performance / service requirements as compared to available prototypes.







MH containers for H₂ compression



<u>A. Small-scale</u>

- $4x \frac{1}{2}$ OD SS tubes in heat-distributing AI block
- 10 (2 stages) to 20 (1 stage) NL H₂
- Electric heating, water cooling
- Heating / cooling time 5-10 min



B. Medium-scale

- Internal HE with fins
 - Up to 2 $\text{Nm}^3 \text{H}_2$
- Steam (or pressurised water) heating, water cooling
- Heating / cooling time ~35-40 min
- Fully certified according to SA regulations (200 bar @ 200 °C)

C. Industrial-scale

- Extruded internal HE with fins + external heating / cooling jacket
 - $2.5 \text{ Nm}^3 \text{ H}_2$
- Steam heating, water cooling
- Heating / cooling time ~20-25 min
- Fully certified according to SA regulations (200 bar @ 200 °C)

re

MH H₂ compressors developed at SAIAMC





2008: Small-scale prototype

- 2 stage layout $(1 AB_5; 2 AB_2)$
- Electric heating (130 °C), water cooling (15..25°C)
- H₂ compression in the range 7 200 bar
- Productivity up to 60 NL/h

2011: Medium-scale prototype

- 2 stage layout (1 AB₅; 2 AB₂)
- Steam / pressurised water heating (130 °C), water cooling (15..25°C)
- H₂ compression in the range 10 200 bar
- Productivity up to 1 Nm³/h

2014: Industrial-scale prototype

- 3 stage layout $(1, 2 AB_5; 3 AB_2)$
- Steam heating (140 °C), water cooling (15..25°C)
- H_2 compression in the range 3 200 bar
- Productivity up to 5 Nm³/h



Eskom



Testing facilities (industrial scale MH compressors) SAIAMC H₂ storage compartment **Eskom MH** Compressor **Testing Facility** Steam generator SAIAMC Eskom Recently commissioned; Will be used to fill H_2 MH Cooling cylinders from electrolyser; compressor tower On-going tests. A. A. Birtheat . TE TOWER f the HyS PE

Prototype 3-stage MH hydrogen compressor: test results



 $C_{\mu}[NL/kg]$ PCT for H absorption at T_L =298 K (a) and H desorption at T_H =403 K (d) for: LaNi_{4.9}Sn_{0.1} (1), La_{0.8}Ce_{0.2}Ni₅ (2) and C14-Ti_{0.65}Zr_{0.35}(Mn,Cr,Fe,Ni)_{2+x} (3).

- 3 stage layout (1,2 AB₅; 3 – AB₂)
- T_L ~20 °C (water), T_H ~130 °C (steam);
- $P_L \ge 3$ bar, $P_H \le 200$ bar
- Productivity up to 5 Nm³/h







Simplified gas piping diagram (a) and general view (b).



Typical operation: (a) temperatures of the compression modules; (b) H_2 pressures and input flow rate.

Eskom





MH system integration

- 2011-...; on-going:
 - HySA Systems projects KP3-S02, KP3-S03, KP8-S05;
 - Impala Platinum funded HySA Systems project;
 - THRIP project;
 - H2020 / RISE project



Metal hydride containers (1)



Metal hydride containers (2)



Liquid heated / cooled
H storage for FC systems (1 to 15 kW)
H₂ compression



1 – containment; 2 – MH powder, 2a – MH/TEG compacts; 3 – gas filter; 4 – gas connector; 5 – transversal fins; 6 – longitudinal fins; 7 – core of inner heat exchanger; H_2 – hydrogen flow; Q – heat supply / removal; H_2O – flow of heating / cooling water.

Layouts for ## 2-5, 8: Patent application WO 2015/189758 A1

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MH for forklift: Project Highlights

- The objective of this project co-funded by Impala Platinum Ltd and the DST is to integrate metal hydride (MH) technologies for on-board hydrogen storage and related applications into utility vehicles providing their efficient system solutions, through the development of MH-based system components.
- The specific activities in the realising this objective included:
 - Integration of MH H storage and supply system in fuel cell powered forklift;
 - Development of the prototype forklift refuelling system with 50-200 bar 7-12 Nm³/h MH compressor;



Start-up and field tests of the hardware at the site of industrial customer (Impala Platinum Refineries).









System Concept



More details to be given in HYDRIDE4MOBILITY overview presentation





- STILL RX-60-30 electric forklift, 3 ton lifting capacity, 80 VDC bus voltage.
- Commercial on-board fuel cell power module (A; GenDrive 1000 160X-80CEA / Plug Power) replacing the forklift battery and equipped with:
 - built-in CGH2 hydrogen storage system and
 - MH hydrogen storage extension tank (**B**).
- Stationary hydrogen refuelling system (C) consisting of a lowpressure H₂ supply and a MH hydrogen compressor.



Future plans

- MH materials:
 - Optimising compositions and manufacturing procedures of MH alloys for H_2 compression (AB₅ \leq 1 bar; AB₂ >400 bar).
- Integrated systems:
 - Forklifts, mine locomotives (FC power modules with integrated MH storage);
 - Small-to-medium scale MH H storage and supply systems for various applications.
- MH H₂ compressors:
 - Lowering suction pressure to ≤1 bar while maintaining productivity x10 Nm³/h;
 - Increase of discharge pressure to 400–700 bar (CGH2 refuelling systems).
- Other MH based systems:
 - Heat management applications;









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Thank you for listening!

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Extra slides







HySA Strategic Goals

- Develop local cost competitive hydrogen generation solution based on renewable resources
- Wealth creation through value added manufacturing of PGM catalysis, goal- supply 25% of PGM catalysts demand by 2020
- Promote equity and inclusion in the economic benefits of South Africa's resources, SMEs to play an important role

From: Dr Phil Mjwara, DG-DST: "Vision 2030: Hydrogen and Fuel Cells in SA", IPHE Meeting, Cape Town, 03 May 2012





HySA Implementation Plan









HySA Systems: Academic Collaborations









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Zentrum für Material- und Küstenforschung

Hydrogen South Africa



भारतीय प्रौद्योगिकी संस्थान गुवाहाटी INDIAN INSTITUTE OF TECHNOLOGY GUWAHATI

MH related projects:

- ✓ Completed: ZA–NO research collaboration program
- ✓ Ongoing: EU FP7 international collaboration project (NO, TR, ZA, EG; ERAfrica program) – PhD project co-supervised
- ✓ Recently granted: Horison 2020 RISE project (NO, DE, HR, ZA, ID);
- ✓ <u>Submitted</u>: BRICS project proposal (ZA, CN, RU, IN)





ИΠΧΦ ΡΑΗ

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MH H₂ compressors: *patents* and main publications

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Mg – BCC-V Alloy: Combustion-Type Hydrogenation



Two re-hydrogenation modes were observed:

- 1. In slow mode hydrogenation takes place even at RT and is completed in 10–15 hours; 80–90% of MAX H capacity is achieved.
- 2. In fast (combustion) mode hydrogenation completes in 5– 60 seconds and is accompanied by a significant heat release; sample temperature, Ts, approaches equilibrium value (Mg \leftrightarrow MgH₂) for the operating H₂ pressure; 60–65% of MAX H capacity is achieved (80% for Mg / 10V / 5 C).



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Mg – C



XRD of re-hydrogenated samples





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Quality Assurance – Composition (SEM / EDS)

25µm	Maj	or phase Imp rity 1 (side) Impuri	Significant in the import graphite m	Significant amounts of C and O in the impurity phases (casting in a graphite mould)			
Component	ent Major phase (7 spectra)		Impurity 1 (3 spectra)	Impurity 2 (1 spectrum)	Impurity 3 (1 spectrum)	More "pure"	
	Nominal composition [at.%]	Results of analysis [at.%]	Deviation from nominal composition [at.%]	Results of analysis [at.%]	Results of analysis [at.%]	Results of analysis [at.%]	casting achieved by the installation of water
Ti (A)	18.31	18.04 ± 0.72	-0.27	19.47 ± 0.49	17.52	11.22	cooled Cu
Zr (A)	14 98	15 15 + 2 05	+0 17	39 45 + 0 95	24.77	86.55	mould
0 / - 1	14.50	13.13 ± 2.03	10.17	55.15 - 0.55			moulu
Cr (B)	27.96	28.91 ± 1.22	+0.95	22.47 ± 0.18	20.10	1.71	moula
Cr (B) Mn (B)	27.96 12.85	28.91 ± 1.22 12.91 ± 0.65	+0.95	22.47 ± 0.18 1.58 ± 0.79	20.10 22.57	1.71 0	mould
Cr (B) Mn (B) Fe (B)	27.96 12.85 18.31	28.91 ± 1.22 12.91 ± 0.65 18.07 ± 0.72	+0.95 +0.06 -0.24	22.47 ± 0.18 1.58 ± 0.79 13.13 ± 1.18	20.10 22.57 11.03	1.71 0 0.52	mould
Cr (B) Mn (B) Fe (B) Ni (B)	27.96 12.85 18.31 6.66	28.91 ± 1.22 12.91 ± 0.65 18.07 ± 0.72 6.19 ± 1.23 0 74 ± 0.22	+0.95 +0.06 -0.24 -0.47	22.47 ± 0.18 1.58 ± 0.79 13.13 ± 1.18 3.9 ± 0.24	20.10 22.57 11.03 4.02	1.71 0 0.52 0	modia







MH materials for H₂ compression



Testing facilities (small- to medium scale)



•Possibility to test both MH containers and MH compressors (1-200 bar, up to 6 m³/h);

•Heating:

- •Steam (up to 140°C / 10 kg/h);
- •Pressurised water (up to 180 °C / 20 L/min);
- •Cooling: tap water (~20 °C, up to 20 L/min);
- •Data acquisition (P, FR, T) using LabView.





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MH material



- AB₂: A=(Ti,Zr),
 B=(Fe,Cr,Mn,Ni,V...).
- Tuning of PCT properties by the variation of the component composition.
- H₂ pressures >10 bar at the temperatures above 0 °C.
- At T > 125 °C most of hydrogen is desorbed at P > 200 bar.
- Suitable for both H₂ storage (T=0–50 °C, P>10 bar) and compression (T=20–140 °C, P=30–200 bar).
- Same material for H₂ storage (MH extension tank) and H₂ compression (refuelling).



Intensity



