Project: **Chip Integrated Hydrogen Generation-Storage-Power Microsystem**

Acronym: **HyGenMEMS Grant Agreement N0.236667**

Host Organisation: **RWTH Aachen University, Germany**

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PROJECT SUMMARY

The main objective of the HyGenMEMS project carried out in the frame of Marie Curie Action Intra-European Fellowships was the career development of the researcher through acquiring of additional knowledge, expertise, and experimental skills in the modern, interdisciplinary research field of hydrogen energy converting microsystems. The primary technical objective was the exploration of the possibilities and proof of the concept for development of a chip integrated hydrogen generator based on polymer electrolyte membrane water electrolysis, capable of a reverse operation as a hydrogen microfuel cell. The long term goal is the technical realization and fabrication of an integrated hydrogen generation–storage–power micro system for autonomous energy supply of wireless electronic devices. The competence of the host organisation and the researcher complemented each other in an ideal way, building a strong basis for successful realization of these goals. The main achievements of the project are summarized as follows: 1). broadening and enhancement of researcher's knowledge, competence, and expertise; 2). improved perspectives for career development in terms of taking a leading position, establishment of research group and laboratory of micro electrochemistry, thus contributing to the strengthening of ERA in accordance with 7FP goals; 3). scientific and technical input to the current state-of-the-art of the hydrogen micro energy systems.

The realization of the personal development plan was organized following the principles of ***training by learning*** and ***training by doing***. The main activities included attendance of variety of specialized lectures courses and a practical training aimed to increase the competence of the researcher in the interdisciplinary field of microsystem technology. The specialized lectures covered various scientific topics, including: surface and bulk analytical techniques, energy storage systems, sensors and actuators based on silicon microtechnology, etc. Another aspect of the ***training by learning*** approach was focused on enhancement of the competence and acquiring of systematic knowledge on project-, quality-, and staff management as well as improvement of the social awareness and capabilities for solving various team work issues and problems, encountered in the research practice.

The ***training by doing*** activities were dedicated mainly to experimental work carried out in the Clean Room Technology Center of IWE1. The researcher had an access to the modern research facilities and support from highly competent scientists and engineers, receiving an intensive training in all technological processes (lithography, wet-bench processes, wet and dry etching, micro electroplating, screen printing, micro assembling, etc.) and acquiring the necessary practical skills to work independently with the available machines and the specific equipment (PVD machines, etchers, flip-chip bonders, die bonders, 4-point resistance measurement station, etc.).

In parallel, the researcher made serious efforts for ***networking in accordance with the EC strategy for strengthening of the European Research Area***. She realized new professional contacts and collaborative links with leading scientists and research organisations in the field among which: Juelich Research Center, Juelich; Fraunhofer Institute, Oldenburg; and Fuel Cell Technology, GmbH, Duisburg, Germany; Norwegian University of Science and Technology, Trondheim, Norway; Gebze Institute of Technology, GYTE, Turkey; Trinity College, University of Dublin, Ireland.

The scientific and technical realisation of the project included:

- ***Development and testing of electrocatalysts for hydrogen energy conversion***

The production of a single chip integrated hydrogen micro energy system requires highly active cost efficient catalysts, deposited via preparation technique compatible with the other microsystem technology processes. In such a respect the method of magnetron sputtering is of particular interest.

During the HyGenMEMS project several mono- and bimetallic systems (*Pt, PtTi, PtIr*) were deposited as thin sputtered films and investigated in details. The performed parametric study was focused on the changes in the film morphology including its depth profile at varying dc-sputtering power, argon pressure, and film thickness. The corresponding effects on the electrochemical active surface area and the catalytic efficiency toward the electrode reactions on the hydrogen and oxygen electrodes were followed. It was found that high sputtering pressure and low sputtering power are beneficial, ensuring large active surface and increased catalytic activity. The performed morphological and electrochemical investigations revealed superior catalytic efficiency of the co-sputtered PtIr films with best performance for the sample deposited at power ratio P_{Pt}:PIr = 100:30W containing 11 at.% Ir, that has also the most developed active surface. The observed effects were explained with the enhanced affinity of Ir to formation of $IrOH_{ads}$ surface coverage, electronic interaction between both metals, and the established changes in the morphology of the bimetallic films.

- ***Deposition of Pd films as hydrogen storage by dc magnetron sputtering***

The hydrogen storage material of choice was palladium deposited by magnetron sputtering. In view of the device compatibility, it was very important to grow high quality nanostructured Pd thin films on lattice non-matching substrates. The research allowed to establish an optimal sputter regime, resulting in fabrication of fully reproducible Pd layers with highly developed surface, moderate porosity, and long term mechanical stability which sustained in the whole range of film thicknesses tested (from 100 nm up to several μm).

- ***Formation of the polymer electrolyte membrane (PEM)***

The PEM was fabricated by spin coating of Nafion (20 wt. % solutions, ETEK) directly on the sputtered thin catalytic film. A set of coating parameters was found, allowing fabrication of mechanically stable homogeneous PEM with controllable thickness and proton conductivity of around 5.10^{-3} $S.cm^{-1}$ at ambient temperature and relative humidity that is in accordance with the conductivity of the commercially available Nafion PEM. The PEM thickness was in the order 10-20 μm and the voltage drop over the membrane at optimal electrolysis conditions was in the range of only a few mV.

- ***Chip integrated hydrogen generation–storage–power***

All components of the developed hydrogen energy converting microsystem were built up on a single monocrystal Si (111) wafer using 5 different lithography masks. The technological scheme consisted of numbers of consecutive steps including metallization, photolithography, wet chemical and dry plasma etching, magnetron sputtering, dicing, and spin coating. The contact pads (Au) and the adhesive metal layers (Ti, Cr) were deposited by electron beam deposition/evaporation, while the catalytic films and the hydrogen storage material were fabricated by dc magnetron sputtering. The PEM was formed by spin coating. The HyGenMEMS performance characteristics were investigated on a single cell, using a self designed laboratory testing device with possibilities to investigate the individual properties of the active and passive components as well the functioning of the whole system both in an electrolyser (H_2-charging) and in a fuel cell (discharging) mode. The functioning of the HyGenMEMS in charging regime was optimised in view of the electrode processes overpotential, efficiency, rate, duration, thickness and mechanical stability of the hydrogen storage layer. The best performance (10 $mA.cm^{-2}$ at cell voltage of 1.74V, energy efficiency 78-80 %) was achieved with a single cell having 500 nm thick Pd layer, 150 nm thick Pt as a hydrogen electrode catalyst, and a sputtered IrO_2 as an anode (oxygen electrode) catalyst. The reverse FC operation of the chip-integrated microsystem showed worse characteristics compared to the electrolysis mode. The open circuit potential was around 0.5V and the maximum charge gained from the fuel cell after charging of the system both to the maximum theoretical capacity of 0.67 $C.cm^{-3}$ and to 0.167 $C.cm^{-3}$ was 0.0034$C.cm^{-3}$ which is one fifth of its theoretical capacity. The maximum energy density of the HyGenMEMS single cell was 120 $\mu Wh.cm^{-2}$. The round trip efficiency of the system was around 40%.

The project addressed a broad interdisciplinary research field. The researcher acquired expertise in the thin film processing and microsystem technology. The fulfillment of the technical part of the project resulted in a functioning prototype proving the concept of the proposed reversible hydrogen microenergy system. The further research will be focused on development, design, and integration of different numbers of HyGenMEMS single cells in stacks to fulfill specific energy needs and applications.