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Deliverable Report

D4.3: Demonstration of a catalytic entity showing 0.7 A/mg_{Pt}, in an MEA test and a surface area > 40 m^2/g_{Pt} after 30,000 cycles from 0.6 to 0.925 V

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Dissemination Level

PU	*Public*	***X***
PP	*Restricted to other programme participants (including the Commission Services)*	
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Nature of the Deliverable

R	*Report*	***X***
P	*Prototype*	
D	*Demonstrator*	
O	*Other*	

SUMMARY

Keywords	*MEA testing, catalyst mass activity, catalyst surface area, oxygen reduction reaction.*
Abstract	Pt and Pt alloy oxygen reduction reaction (ORR) electrocatalysts have been further advanced in the first 18 months of the GAIA project. The project targets for the ORR catalyst were to achieve a mass activity (MA) of at least 0.7 A/mg_{Pt} within an MEA and to maintain a surface area of at least 40 m^2/g_{Pt} after 30,000 cycles from 0.6 to 0.95 V. At the start of the project, de-alloyed PtNi/C, octahedral PtNiIr/C and Pt-Rare Earth (RE) nanoparticle catalysts were identified as candidates to reach the project mass activity and surface area stability targets. TUB scaled up three octahedral PtNiIr/C catalyst variants from 20 mg to about 800 mg, at Pt loadings of 8% and 15% by weight on Vulcan XC72R carbon. The scaled octahedral PtNiIr/C alloys catalysts were evaluated in the rotating disk electrode (RDE) and demonstrated extremely high initial mass activities of up to 2.3 A/mg_{Pt} and electrochemical surface areas above 40 m^2/g_{Pt}. Three of these catalyst variants were sent to JMFC for testing in 50 cm^2 single cells but, unfortunately, in MEAs the MA was only about 0.30 A/mg_{Pt}. Work at JMFC on carbon-supported de-alloyed 50% PtNi catalysts led to material with an average particle size of 4.5 nm, as measured by transmission electron microscopy (TEM), and a surface area of about 65 m^2/g Pt. Performance testing in 50 cm^2 single cells gave a MA of 0.44 A/mg_{Pt} using a cathode loading of 0.20 mg Pt/cm^2, but a formulation tested at a reduced cathode loading of 0.10 mg Pt/cm^2 yielded an excellent MA of 0.89 A/mg_{Pt}. When this same catalyst was tested for durability in a 50 cm^2 single cell using 30,000 cycles (0.6 – 0.925 V) at 80°C, the end of life surface area was 39 m^2/g_{Pt}, only marginally below the end of test target of 40 m^2/g_{Pt}. This catalyst has therefore been considered to meet the targets set for this deliverable D4.3 (demonstration of a catalytic entity showing 0.7 A/mg_{Pt} in an MEA test and a surface area > 40 m^2/g_{Pt} after 30,000 cycles from 0.6 to 0.925 V) and will be progressed to WP5 for further evaluations. The Pt-RE catalysts being developed at CNRS, TUM and JMFC are also showing promise, with increased surface areas, but are at an earlier stage of development, and are therefore not included in this report.
Public abstract for confidential deliverables	

REVISIONS

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D4.3: DEMONSTRATION OF A CATALYTIC ENTITY SHOWING 0.7 A/mg$_{Pt}$, IN AN MEA TEST AND A SURFACE AREA > 40 m^2/g$_{Pt}$ AFTER 30,000 CYCLES FROM 0.6 TO 0.925 V

CONTENTS

1. INTRODUCTION

Design and development of high mass activity (MA) and high stability catalysts is one of the main objectives of WP4, together with the development of highly structured, surface-modified, corrosion resistant supports. The work in Task 4.3 encompasses the synthesis, characterisation and activity evaluation of Pt and Pt alloy nanoparticle catalysts for PEM fuel cell cathodes. Preliminary rotating disk electrode (RDE) screening tests selected two transition metal doped PtNi alloy catalysts from TUB that met Milestone 1 (M1): "Catalyst designs showing promise to reach ECSA/MA targets" (ECSA = electrochemically active surface area), exceeding those of the reference PtNi/C alloy catalyst. Among them, a PtNi(Ir) alloy nanoparticle catalyst showed a well-defined octahedral particle shape, a satisfactory ECSA above 40 m^2/g_{Pt} and much higher MA than the other PtNi(Rh) catalyst,[1] which was attributed to the octahedral shape.[2] This material was therefore chosen to be integrated into a membrane electrode assembly (MEA) and measured in a fuel cell. The synthesis of octahedral Pt alloy nanoparticles at larger scale (hundreds of grams) is challenging, however. Work at JMFC focused on more readily scalable synthesis routes capable of producing high surface area 50% PtNi/C alloy catalysts. This is also challenging, because as the Pt wt% increases on the carbon support, it is intrinsically harder to produce good catalyst dispersions with small particle sizes. The third approach pursued in WP4 to find highly active and stable alloy catalysts has focused on the synthesis of Pt-rare earth (RE) alloy catalysts such as Pt_3Y, Pt_5Ce and Pt_5Gd. Whilst showing great promise, these materials are not yet ready for validation in MEAs, and are therefore not included in this report.

2. EXPERIMENTAL

2.1 Catalyst Synthesis and Characterisation

For the synthesis of the scaled-up octahedral transition metal doped PtNi alloy nanoparticles at TUB, the metal precursors were reduced in a solvothermal synthesis employing a 500 ml Ace pressure flask as the reactor. The synthesised metal nanoparticles were then added to a carbon support suspension, washed and freeze dried. Alloying of the PtNi-based catalysts was confirmed by the presence of a face-centred cubic PtNi phase using XRD analysis with a D8 Advance Diffractometer (Bruker) equipped with a Lynx Eye Detector and KFL Cu 2K X-ray tube. The elemental composition and metal assay were obtained by inductively coupled plasma optical emission spectroscopy (ICP-OES), using a Varian 715-ES-ICP analysis system. Transmission electron microscopy (TEM) images were obtained using a FEI Tecnai G_2 20 S-TWIN with a LaB_6 cathode (200 kV acceleration voltage, 0.24 nm resolution limit). For TEM, the sample powder was first dispersed in ethanol, drop-cast onto a Cu grid (300 mesh), and dried at 60 °C in air. RDE measurements were conducted at TUB following an established protocol developed in the INSPIRE project.[3] Each electrochemical measurement was repeated three times. MEA preparation and single fuel cell measurements of the scaled-up catalysts were performed at JMFC.

The high loading 50% PtNi/C catalysts were made at JMFC using proprietary methods.

2.2 MEA Preparation

Nafion 1100EW (equivalent weight in g polymer/mol H^+) aqueous perfluorosulfonic acid (PFSA) ionomer was used to fabricate thin-layer electrodes as catalyst coated membranes (CCMs). The cathode catalyst layers were prepared at metal loadings of 0.1 mg_{Pt}/cm^2, unless specified otherwise. The anode catalyst layer was kept constant at a metal loading of 0.1 mg_{Pt}/cm^2. The membrane used for these evaluations was a PFSA type, made at JMFC with a thickness of 15 µm.

Catalysts layers were deposited onto a PTFE release substrate and then transferred via hot pressing onto the membrane. Commercially available gas diffusion layers (GDLs), based on wet-proofed carbon fibre paper substrates, were used for this work and were adjusted to optimise gas and water transport at the anode and cathode electrodes. Single cells (50 cm^2 active area) were assembled by sandwiching the CCMs between the appropriate GDLs and applying compression onto the active area.

2.3 Fuel Cell Testing

A customised JMFC fuel cell station and cell hardware were used in this work. Pure oxygen and synthetic air were used as cathode reactants and pure H_2 as the anode reactant (all gases of 99.9% purity). Stoichiometric flow rates of anode (s = 2) and cathode (s = 9.5 for O_2 and s = 2 for air) reactants were used at current densities >0.2 A/cm^2 and constant flows, corresponding to 0.2 A/cm^2, were used at <0.2 A/cm^2. The specific details for this protocol are shown in Table 1. Reactant humidification was achieved by water-bubblers, the temperatures of which were calibrated to yield the quoted relative humidity (RH) values. The humidity and cell pressure were measured at the inlet for the anode and cathode. The high frequency resistance of the cell as a function of current density was determined using an AC perturbation of 1 kHz.

The MEAs were conditioned by the application of a constant current density of 500 mA/cm^2 under H_2/air at 100 kPa_{gauge}, 100% RH and 80 °C. The cell voltage was monitored until a stable value was observed. The conditioning step lasted two hours unless specified otherwise. Afterwards the cathode catalyst layer was exposed to a series of cathode starvation steps under H_2/air, as specified in Table 1 and following the US Department of Energy (DOE) Tech Team fuel cell polarisation protocol, but under the pressure and humidity conditions specified in the conditioning step. After the starvation steps, the MEA was ready for testing and a series of measurements were performed for each sample. These consisted of a series of H_2/O_2 polarisation curves for mass activity quantification at different stages of the protocol. The polarisation curves were recorded in the descending direction, from high current density (i.e. 2 A/cm^2) to low current density (i.e. 0.05 A/cm^2), and the ascending direction, low current density to high current density. For each data point, the cell voltage was stabilised over 10 minutes and data averaged over the last minute of the hold. The mass activity value was obtained from the ascending polarisation curve on pure oxygen and the current density maintained for 4 minutes at each step. Details of such conditions are shown in Table 1. The measured mass activity was not corrected for the measured H_2 crossover. For guidance, the H_2 crossover current under the operating conditions (i.e. temperature and H_2-partial pressure for mass activity) was in the order of 2–5 mA/cm^2.

***Table 1.** Test protocol conditions for 50 cm^2 cell.*

	Anode Gas	Anode Stoich.	Cathode Gas	Cathode Stoich.	% RHA	% RHC	Anode Inlet P (kPa$_g$)	Cathode Inlet P (kPa$_g$)	Cell Temperature °C	Current Density mA/cm^2
Initial Conditioning	H_2	2.0	Air	2.0	100	100	100	100	80	500
Cathode Starvation	H_2	2.0	Air	0.0/2.0	100	100	100	100	80	500
Oxygen polar	H_2	2.0	O_2	10.0	100	100	50	50	80	25-500
Air polar	H_2	2.0	O_2/Air	10/2	100	100	100	100	80	25-2000
Air polar	H_2	2.0	O_2/Air	10/2	30	30	100	100	80	25-2000
Cathode CV (CO)	H_2	N/A	N_2 – N_2-CO	N/A	100	100	100	100	80	N/A

2.4 ECSA Measurement in MEAs

The electrochemical surface area was measured by the carbon monoxide (CO) stripping method using the cell in half-cell mode. Under this mode, the anode electrode acts as a pseudo reference electrode. The cathode voltage was controlled at 0.125 V at 80 °C and 100 kPa$_{gauge}$ whilst purging with 1% CO in N_2 at 300 ml/min for 15 min. Afterwards the cathode was purged with N_2 at the same flow rate and for 2 h to ensure that CO was removed from the bubblers and the catalyst layer pores. The CO adsorbed on the Pt surface was oxidised electrochemically by scanning the cathode voltage from 0.125 V to 0.85 V and back to 0.05 V at 20 mV/s, for three cycles. The area under the CO oxidation peak was integrated by subtracting the third scan from the first scan and using 420 µC/cm^2 as the charge constant for a monolayer of CO on Pt.

3. RESULTS AND DISCUSSION

In WP4 a wide range of different strategies were used for the synthesis of highly active cathode catalysts. This deliverable 4.3, however, only focuses on the validation of the mass activity in MEAs of the two alloy formulations that were showing the highest mass activities: the TUB octahedral PtNiX/C and the JMFC de-alloyed 50% PtNi/C catalysts.

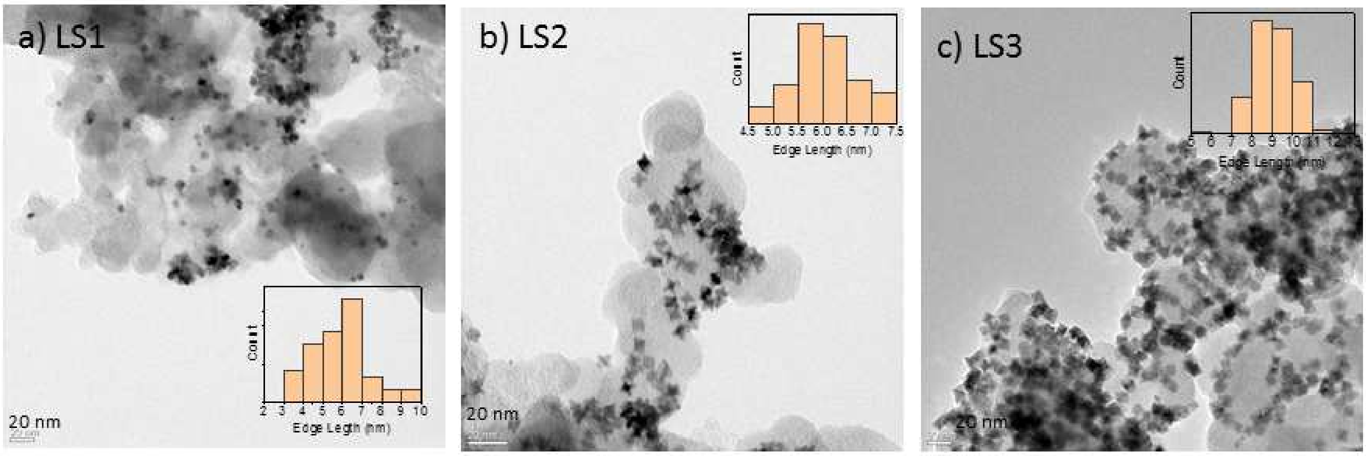
3.1 Octahedral PtNiIr/C Catalyst Properties

The scaling-up of the octahedral PtNiIr/C catalyst synthesis from a single batch of 100 mg to 1 g was not straightforward. The direct scale-up of the precursor amounts was not successful; the nanoparticles did not show the required octahedral shape. Adaptations of the synthesis conditions were made and multiple batches of different catalyst variations were produced at about 800 mg per batch. Of these, three batches were selected, with the elemental compositions provided in Table 2, and labelled as LS1, LS2 and LS3 (LS standing for large scale).

***Table 2. Elemental composition**. Elemental composition by ICP-OES of the scaled-up octahedral PtNiX nanoparticle catalysts, where X is a transition metal dopant.*

	LS1	**LS2**	**LS3**
Atomic compostion	$Pt_{55.6}Ni_{44.4}$	$Pt_{63.6}Ni_{35.5}Mo_{0.9}$	$Pt_{13.4}Ni_{85.5}Ir_{1.1}$
Pt wt%	12.9%	16.7%	8.5%

In LS1, the Ir metal precursor was introduced in the synthesis, but its value in the as-made catalyst was below the detection limit of the ICP-OES and therefore it is not indicated. In contrast, Ir was detected in LS3, in an amount typical for this kind of doping, i.e. the atomic % of Ir in the small-scale PtNi(Ir) was 1.4. LS3 also showed a high Ni content with respect to the other catalysts. Among the catalyst variations, LS2 showed a Pt:Ni ratio matching reasonably closely that of the small-scale PtNi(Ir), which showed the overall composition of $Pt_{67.8}Ni_{30.8}Ir_{1.4}$, *i.e.* Pt:Ni ~2:1. Overall, the scaled-up catalysts showed a lower loading with respect to the small-scale PtNi(Ir) (21.2 Ptwt%), with LS2 still showing the closest value. The TEM and the particle size distributions (PSD) that were calculated by considering the edge of the octahedra are shown in Figure 1.



$Edge_{PSD}$ = 5.9 ± 1.5 nm
$Edge_{PSD}$ = 6.1 ± 0.6 nm
$Edge_{PSD}$ = 9.1 ± 1.0 nm

***Figure 1.** TEM and corresponding PSDs. TEM images of LS1 (a), LS2 (b) and LS3 (c). PSD histograms in the insets and average edge length of the PtNiX octahedra below the TEM image of the corresponding catalyst.*

All the samples showed octahedral nanoparticles (Figure), and nanoparticle agglomerations were visible for LS1. Both LS1 and LS2 showed similar average sizes for the octahedra (~6 nm), while LS3 showed larger octahedra (~9 nm). The catalyst activity for the oxygen reduction reaction and their ECSAs were evaluated with RDE measurements (Table 3). All samples showed high MA > 1.4 A/mg_{Pt}, characteristic of highly active octahedral PtNi nanoparticles, and ECSA values above 40 m^2/g_{Pt}. Therefore, all three catalysts showed promising properties and performance, confirming that the scale-up of the small batch RDE-pre-screened samples had been successful, and samples were sent to JMFC for MEA-based single fuel cell tests.

***Table 3. RDE results.** Mass activity (MA) at 0.9 V vs reversible hydrogen electrode (RHE), specific activity (SA) at 0.9 V_{RHE}, and ECSA obtained by hydrogen under potential deposition (H_{upd}) of LS1, LS2 and LS3.*

	LS1	**LS2**	**LS3**
$MA_{0.9VRHE}$ (A/mg_{Pt})	1.44 ± 0.40	2.32 ± 0.19	1.46 ± 0.15
$SA_{0.9VRHE}$ (mA/cm^2)	3.05 ± 0.45	5.55 ± 0.66	2.87 ± 0.36
$ECSA_{Hupd}$ (m^2/g_{Pt})	44.63 ± 5.22	41.92 ± 1.56	50.95 ± 1.43

The requirement to meet the MA target of 0.7 A/mg_{Pt} at 0.9 V is particularly challenging considering that the cathode catalyst in the state-of-the-art MEA is at 0.6 A/mg_{Pt}.[4] Nevertheless, the surface areas of the scaled-up octahedral nanoparticles were significantly higher than those reported in the literature. Considering that the JMFC benchmark catalyst, 50% Pt/C2, has a surface area of 55 m^2/g_{Pt}, the surface areas obtained with the shaped nanoparticles are certainly of interest for testing in layers. If the high mass activities observed in the RDE were to be translated to a catalyst layer, then these octahedral nanoparticles would be expected to perform close to the ultimate project target.

3.2 De-alloyed 50% PtNi/C Catalysts

TEM examination of the de-alloyed 50% PtNi/C catalyst prepared at JMFC was found to have a suitably small particle size, for a 50% loading catalyst, and a correspondingly high metal surface area of 68 m^2/g_{Pt}, measured by carbon monoxide chemisorption, which exceeds that of the JMFC benchmark 50% Pt/C2 catalyst (55 m^2/g_{Pt}).

4. ELECTROCHEMICAL EVALUATION IN 50 CM^2 SINGLE CELLS

4.1 Octathedral PtNiIr/C Catalysts

The TUB scaled up octahedral catalysts were tested for mass activity as CCMs in JMFC single cells with 50 cm^2 active area. Identical protocols were used for electrode preparation, MEA fabrication and testing for each catalyst. During the experiments it was found that the TUB catalysts yielded layers with significant crack formation, in particular for the LS3 catalyst.

Table 4 shows the mass activity obtained in 50 cm^2 cells for the TUB catalysts.

***Table 4**. Mass activity obtained in 50 cm^2 cells using identical protocols as the one shown for de-alloyed PtNi/C cathode layers. Cathodes printed at 0.10 mg Pt/cm^2.*

Catalyst Code	Pt wt%	Ni wt%	Mo or Ir at%	Atomic Composition	Mass Activity (A/mgPt)
(LS1)	12.6	3.2	0	$Pt_{55.6}Ni_{44.4}$	Performance too low for measurement
(LS2)	16.7	2.8	0.9 (Mo)	$Pt_{63.6}Ni_{35.5}Mo_{0.9}$	0.30
(LS3)	8.5	16.4	1.2 (Ir)	$Pt_{13.4}Ni_{85.5}Ir_{1.1}$	Performance too low for measurement

Out of the three scaled up catalysts, only the LS2 catalyst was capable of producing enough current to measure catalyst layer mass activity. The reason for the low mass activity is under investigation and ex-situ TEM analysis is being carried out after testing in 50 cm^2 single cells to see if the octahedral shape is retained after the protocol used at JMFC.

4.2 De-alloyed PtNi/C Catalysts

Figure 2 shows the mass activity of JMFC de-alloyed 50% PtNi/C catalyst, at different cathode loadings, using the protocol described in the experimental section.

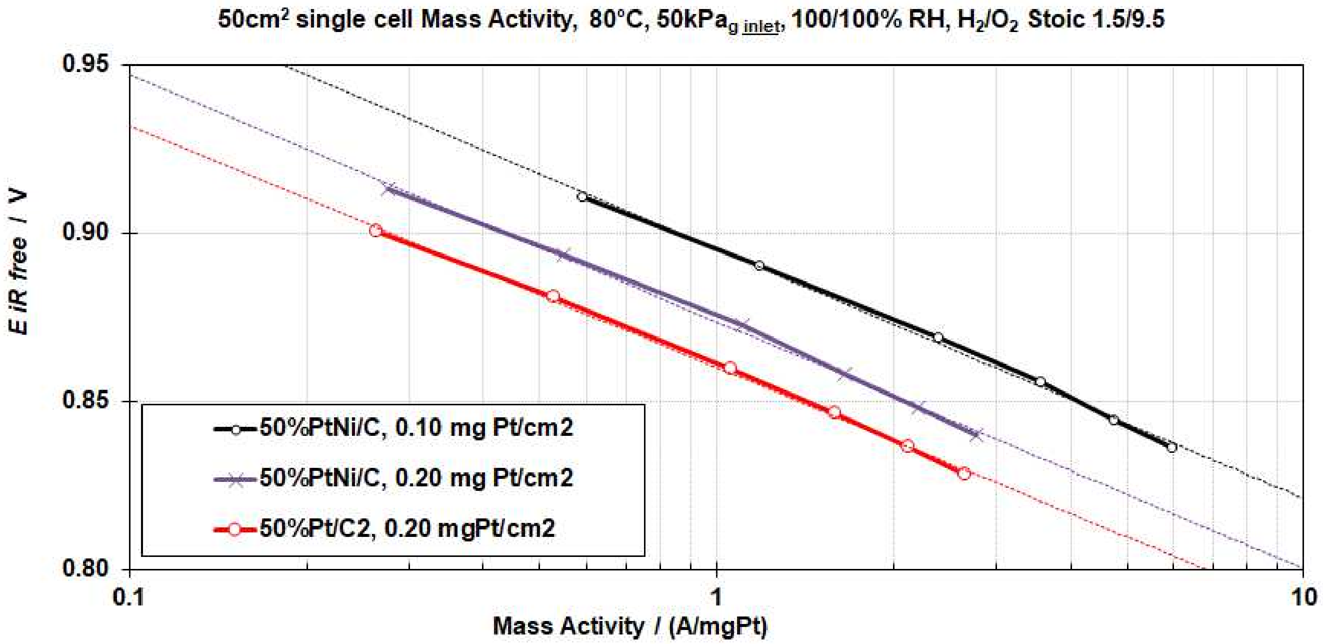


***Figure 2**. Mass activity of a set of JMFC cathode catalysts, 50%Pt/C2 benchmark (red line, 0.20 mg Pt/cm^2), and 50% de-alloyed PtNi/C (purple line at 0.20 mg Pt.cm^2 and black line 0.10 mg Pt/cm^2). Catalyst layers printed at 0.10 or 0.20 mgPt/cm^2 loading as indicated in the legend. All experiments were carried out under identical conditions using a JMFC 50 cm^2 single cell.*

As observed from Figure 2, the JMFC de-alloyed 50% PtNi/C catalyst performs better than the JMFC 50% Pt/C2 benchmark catalyst when tested at the same cathode loading of 0.20 mg$_{Pt}$/cm^2. With mass activities of 0.44 and 0.28 A/mg$_{Pt}$ respectively, this is a 1.6-fold improvement for the alloy versus Pt only. Interestingly when the same de-alloyed 50% PtNi/C catalyst was tested at a lower loading of 0.10 mg$_{Pt}$/cm^2, the mass activity was significantly higher at 0.89 A/mg$_{Pt}$. Results are summarised in Table 5.

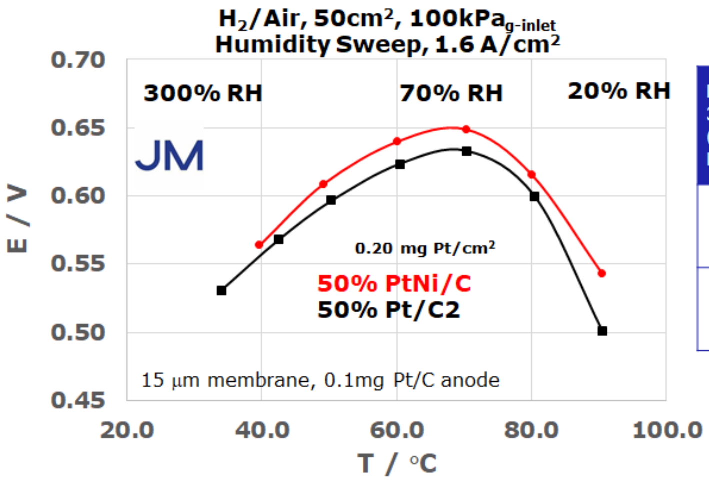
***Table 5**. Catalyst mass activities obtained in 50 cm^2 single cell.*

Catalyst	**Cathode loading (mg$_{Pt}$/cm^2)**	**O$_2$ mass activity in MEA (A/mg$_{Pt}$) at 0.9V iR free** **80°C, 50kPa$_{g\ inlet}$, 100% RH**
50% Pt/C2	0.20	0.28
50% PtNi/C	0.20	0.44
50% PtNi/C	0.10	0.89

With reference to the mass activity target for D4.3 of 0.70 A/mg$_{Pt}$, the JMFC de-alloyed 50% PtNi/C at a cathode loading of 0.10 mg$_{Pt}$/cm^2 exceeded this target, with an extremely high mass activity of nearly 0.9 A/mg$_{Pt}$. The reasons for the sensitivity to layer loading will be investigated in the second half of the project, but this result bodes well for high performance from low loading layers.

5. DURABILITY TESTING IN 50 CM^2 SINGLE CELL

In addition to addressing the mass activity target, D4.3 also focused on the retention of catalyst surface area after voltage cycling. Of the catalysts developed in WP4, the JMFC 50% PtNi/C catalyst is currently the best performing alloy catalyst in 50 cm^2 single cells. Figure 3 shows a summary of the beginning of life performance of JMFC de-alloyed 50% PtNi/C under a wide range of humidity conditions and the table included in Figure 3 shows the decay in catalyst surface area after voltage cycling.



Durability 30,000 H2/N2 (0.6-0.925)V LSV, 50 mV/s	BOL ECSA m²/g Pt	EOL ECSA m²/gPt	BOL MA A/mg Pt	EOL MA A/mgPt
50% Pt/C2	47	28	0.23	0.18
50% d-PtNi/C	58	39	0.42	0.35

Figure 3. *Summary of performance in a 50 cm^2 single cell of JMFC de-alloyed 50% PtNi/C cathode catalyst. The left plot shows a beginning of life humidity sweep in H_2/air at 1.6 A/cm^2. The table on the right shows the decay in catalyst mass activity and surface area after voltage cycling from 0.6 – 0.925 V for 30,000 cycles in H_2/N_2 at 80 °C. The cathode loadings were 0.20 mg$_{Pt}$/cm^2.*

As can be seen from Figure 3, the de-alloyed 50% PtNi/C catalyst layer has better beginning of life performance over the whole humidity range, compared to the JMFC 50% Pt/C2 benchmark at 1.6 A/cm^2, at the same cathode loading. Further work is in progress in Tasks 4.4 and 4.5 to further improve the performance of this de-alloyed catalyst in catalyst layers. In addition, it is important to observe that the surface area after the durability cycling is circa 39 m^2/g$_{Pt}$, which only just falls short of the D4.3 end of test surface area target of 40 m^2/g$_{Pt}$. Most importantly, however, the de-alloyed catalyst layer has better end of life surface area and mass activity than the JMFC 50% Pt/C2 benchmark. As a result, the de-alloyed 50% PtNi/C catalyst will be transferred to WP5 for further evaluation and optimisation in CCMs.

6. CONCLUSIONS AND FUTURE WORK

The work at TUB focused on three different variations of octahedral transition metal doped PtNi alloy nanoparticle catalysts, which were synthesised at TUB in large batches (~800 mg, which is large for this type of material). The large batch materials showed catalytic activities and ECSAs in RDE tests that were comparable with the small-scale batches and the mass activities were extremely high. This successful production of scaled-up catalysts that met the GAIA Milestone 1 target in the RDE was an important achievement for the project, since relatively large amounts of catalyst are necessary for the in-cell MEA-based tests. Following these results, the three catalysts were tested in MEA-based single fuel cells, but they were unable to reach high current densities. The reasons for this will be investigated, but it is certainly desirable to increase the metal loading on the carbon support. The focus for TUB in the next period will be on the synthesis of transition metal-doped PtNi octahedra with at least 30 Pt wt% loading on carbon and a Pt:Ni atomic ratio of 2 or higher.

The catalysts investigated at JMFC identified de-alloyed 50%PtNi/C as a strong candidate for the project ultimate power density target of 1.8 W/cm^2 at 0.6 V. The performance of this catalyst in 50 cm^2 layers gave very promising performance with 1.6 times higher mass activity compared to the JMFC benchmark 50% Pt/C2 catalyst at 0.10 mg$_{Pt}$/cm^2 and a mass activity at 0.10 mg$_{Pt}$/cm^2 of 0.89 A/mg$_{Pt}$. This value exceeds the D4.3 mass activity target of 0.7 A/mg$_{Pt}$, and further confirmation of the potential of this catalyst will be gleaned from repeat measurements. When this catalyst was tested for stability by voltage cycling, its end of test surface area was 39 m^2/g$_{Pt}$, just marginally short of the D4.3 target of 40 m^2/g$_{Pt}$. Future work will focus on the scale-up and transfer of this alloy variant for evaluation in WP5.

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