

Measurement Note

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On the fabrication of model
proton exchange membrane
fuel cell catalyst films

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ABSTRACT

A key requirement for the commercialisation of proton exchange membrane fuel cells (PEMFCs) is the improvement of the performance and durability of the catalyst layer. In order to support the search for new catalyst materials, there is a need for a reliable ex-situ screening test using model catalyst films that mimic those present in PEMFCs. This measurement note describes a procedure for producing model catalyst films by spin casting and provides insight into the effect of processing variables on the nature of the films.

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Approved on behalf of the Managing Director, NPL,
by Dr M G Cain, Knowledge Leader, Materials Team

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1. INTRODUCTION

Extended use of proton exchange membrane fuel cells (PEMFCs) requires a combination of increased fuel cell efficiency and reduced cost. Improvements in system management are being made but a key component is to reduce the catalyst loading per unit power output. Whilst new catalyst materials are being sought¹, processing still has an impact on catalyst performance for the carbon-supported platinum nanoparticle (NP) catalysts used in most PEMFCs. Ultimately, meaningful testing of efficiency currently requires evaluation in an operational fuel cell in response to current demand. However, there are many factors that affect the overall performance of a fuel cell and decoupling the performance of the catalyst from factors associated with water management, transport through the diffusion and agglomerate layers, the extent of three-phase (fuel, catalyst, electrolyte) boundaries (TPBs) and transport through the membrane can be difficult. Furthermore, evaluation of catalyst performance in a fuel cell is costly and time consuming. Thus, there is merit in establishing a preliminary experimental approach that allows ready ex-situ discrimination of catalytic activity with respect to catalyst composition, loading and processing route in an environment that mimics that found in a PEMFC.

There has been intense research effort in developing rapid screening methods for use in combinatorial approaches towards the discovery of new catalyst materials²⁻¹⁵. However, these samples tend to be very idealised (with respect to carbon support, substrate material and TPB conditions) and may not reflect the environment present in a PEMFC catalyst layer.

This measurement note presents guidelines for the fabrication of spin cast carbon-platinum NP catalyst films. The effect of several variables is investigated and suggestions are made for the fabrication of model catalyst films that mimic the composition and structure present in the catalyst layer in an actual fuel cell. These films are ideally suited for scanning electrochemical microscopy investigations, where appropriate TPB conditions can be simulated, and direct spatial electrocatalytic activity heterogeneity can be resolved¹⁶.

2. SPIN CASTING VARIABLES

The spin casting of Pt:C NP inks was chosen as the route to make catalyst films as it allows the fast and reproducible fabrication of uniform films not unlike those produced by large-scale production methods (involving Pt dispersion, Pt reduction and thermal activation steps). A Speciality Coating Systems, G3-8 spin coater was used to fabricate the films.

The catalyst powder used (provided by Dart Sensors Ltd) consisted of Pt NPs supported by C Black NPs (Vulcan XC72R) in a 6:4 Pt:C weight:weight ratio. 60% Pt loading is in the upper range of typical loadings used in PEMFCs³. A 100% C Black powder (Fuel Cell Store) was also used. All chemicals and solvents (from Fisher Scientific unless stated) used in the studies herein were of the highest purity available and used as received.

An important issue that must be addressed prior spin coating of the film is the cleanliness of the substrate surface. For these studies silicon, glass and highly

orientated pyrolytic graphite (HOPG) were used as substrates. The HOPG was of type ZYB and was supplied (by NT-MDT Ltd) in the form of 1 cm × 1 cm × 1.2 mm pieces. The silicon was in the form of polished, single crystal (CZ), highly doped (n-type, P dopant, resistivity = $0.010 \pm 0.001 \text{ } \Omega\text{cm}$) 4 inch diameter, $525 \pm 25 \text{ } \mu\text{m}$ thick, silicon wafers (obtained from Siltronix). Prior to spin coating, the silicon was cleaved into *ca.* 1 cm × 1 cm pieces with a diamond tip pen. The glass was in the form of polished borosilicate microscope slides (from Fisher Scientific) which were cut, using a diamond tip pen, into *ca.* 1 cm × 1 cm pieces. HOPG is natively hydrophobic, while silicon and glass are hydrophilic (mostly due to alcohol groups at the surface). A number of silicon and glass pieces were rendered hydrophobic by immersion for 60 minutes in a 5% vol:vol. solution of 1,1,1,3,3,3-hexamethyldisilazane (98% Acros Organics) in acetone and then rinsing with copious amounts of acetone.

A cleaning protocol was developed for these substrates that relied on solvent cleaning. For the hydrophilic substrates, this consisted of ultrasonication for (at least) 5 minutes each in acetone, propan-2-ol and cyclohexane, followed by drying with filtered compressed air. For the hydrophobic substrates, the order of the solvents was reversed. The exception was HOPG, which could be cleaned by cleaving the basal plane (upper most layer/s) of the surface using 3M Scotch tape. This procedure is well known in the literature¹⁷ and can produce a clean, atomically flat (within each crystal grain boundary) and hydrophobic surface.

The substrates were inspected prior to and following spin casting with an inverted optical microscope (Medline Scientific Ltd, Ferox PL) placed on an optical table and equipped with a 3MP digital camera.

The success of the cleaning procedure can be seen in fig. 1. Image 1 shows a cleaned hydrophobic silicon substrate. The small feature in the top left quadrant of the image is actually due to dust in the camera (hence the blurring). Images 2 and 3 show the surface of a HOPG substrate before and after cleaving with Scotch magic tape. The same feature due to dust in the camera can be discerned in Image 3.

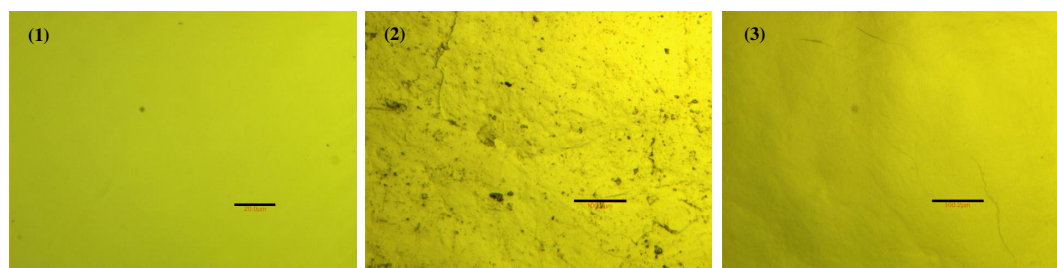


Figure 1. Image 1: Cleaned hydrophobic silicon substrate, scale bar corresponds to 20 μm . Image 2: HOPG surface prior to cleaving. Image 3: HOPG surface after cleaving. For the last two images, the scale bar corresponds to 100 μm .

In order to spin cast a film, the film forming material needs to be dissolved/dispersed in a low boiling point solvent. A small volume of spreading solution is then placed (normally using a micropipette) onto the substrate surface, which is then rotated at a high speed to evaporate/displace the solvent. Thus, the variables to control are: solvent, concentration of solution, substrate hydrophobicity, film forming material, rotation speed and acceleration time. The effect of each of these will be demonstrated in turn below.

Unless stated, standard spin coating conditions were: Pt:C NPs, 1 mg mL⁻¹ propan-2-ol suspension, hydrophobic silicon substrate, 2000 rpm rotation speed, < 0.1 second acceleration time, 1 layer spun.

2.1 SPREADING SOLUTION SOLVENT

Carbon-platinum particles are sparingly soluble in most common laboratory solvents. However, quasi-stable suspensions (inks) can be made from a variety of solvents. Although initial solute-solvent dispersion is very low, it can be significantly improved with an ultrasonication step (> 30 minutes). The resulting suspension is reasonably uniform and stable over minutes (the precise time depends on the solvent and concentration used), which is ample time for spin casting.

The effect of three different solvents (acetone, propan-2-ol and cyclohexane) on the fabrication of C NP spin cast films was investigated. The choice of these was judicious: the boiling points are 56 °C, 82 °C and 81 °C respectively; and the dielectric constants (a measure of the polarity) 21, 18 and 2, respectively. Thus, the influence of boiling point and dielectric constant can be inferred.

It is expected for the C NPs to be hydrophobic and hence to preferentially dissolve/disperse in non-polar solvents (*i.e.* low dielectric constant solvents). Also, it follows that better dispersed solutions will contain smaller aggregates and thus be likely to produce more uniform spin cast films containing smaller aggregates.

As shown in fig. 2 however, these assumptions are not borne out by experiment. The film spin cast from a cyclohexane dispersion (image 6) contains the biggest clumps. Although the film spin cast from acetone (image 4) probably contains the most substrate coverage, it is not very uniform, with clusters of varying sizes present. The film spin cast from propan-2-ol (image 5) is the most uniform.

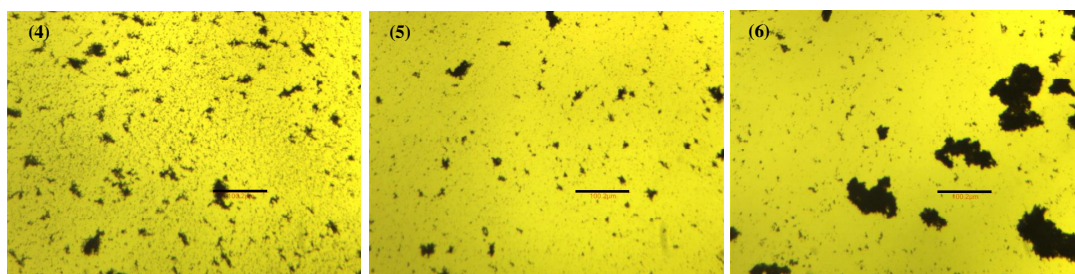


Figure 2. Spin cast films on hydrophobic silicon made from 1 mg mL⁻¹ C NP suspensions, spin cast on hydrophobic silicon at 2000 rpm, < 0.1 second acceleration time. Spreading solution solvent was, from left to right: acetone (image 4), propan-2-ol (image 5), cyclohexane (image 6). The scale bar corresponds to 100 μm for all images.

Cyclohexane and propan-2-ol share virtually the same boiling point but differ in their dielectric constants, whereas acetone and propan-2-ol have very similar dielectric constants but differ in their boiling points (*vide supra*). Thus, for the fabrication of well-dispersed, uniform catalyst-on-substrate coverage, spin cast films, a solvent with relatively high dielectric constant and boiling point is preferable.

2.2 CONCENTRATION OF SPREADING SOLUTION

The concentration of the spreading solution is perhaps the variable that has the most influence on the films. Fig. 3 shows C NPs films made from 0.5, 1.0 and 2.0 mg mL⁻¹ spreading solutions.

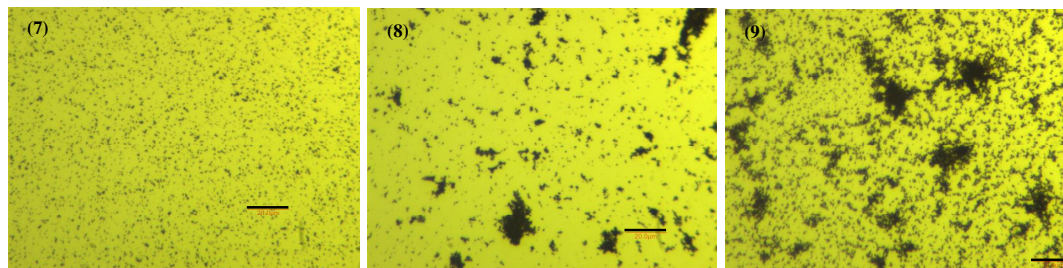


Figure 3. Spin cast films, rotated at 2000 rpm with < 0.1 second acceleration time on hydrophobic silicon, made from, left to right: 0.5 mg mL⁻¹ (image 7), 1.0 mg mL⁻¹ (image 8), 2.0 mg mL⁻¹ (image 9) C NP propan-2-ol spreading solutions. The scale bar corresponds to 20 μm for all images.

As expected, as the concentration of spreading solution increases, so does the film substrate coverage. It is interesting to note that as the spreading solution concentration doubles from 0.5 to 1.0 mg mL⁻¹, cluster sizes increase significantly (image 7 *vs.* image 8). In contrast, as the concentration doubles again, from 1.0 to 2.0 mg mL⁻¹, cluster sizes remain largely unchanged (image 8 *vs.* image 9).

2.3 SUBSTRATE HYDROPHOBICITY

During the spin coating process, the film forming material undergoes a transition from a disordered liquid environment to a dry, more ordered, environment. A key factor to be controlled is the adherence of the film towards the substrate. This can be achieved by controlling the hydrophobicity of the substrate surface. Borosilicate glass and silicon containing solely an ambient-produced oxide top layer are natively hydrophilic (*i.e.* advancing contact angles < 90°). However, these surfaces can be turned hydrophobic (*i.e.* advancing contact angles > 90°) by a simple silanising substitution reaction (*vide supra*).

Fig. 4. shows films made on hydrophilic (native) silicon substrates from the same spreading solution concentration of C NPs as those in fig. 3, *viz.* 0.5 mg mL⁻¹ (image 10), 1.0 mg mL⁻¹ (image 11), 2.0 mg mL⁻¹ (image 12), albeit at marginally higher magnification (*cf.* scale bars).

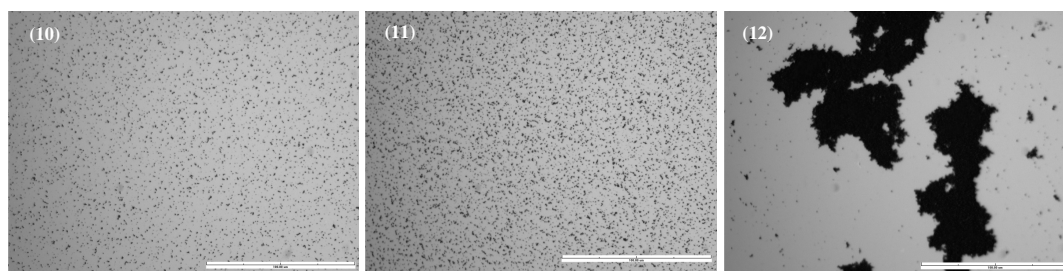


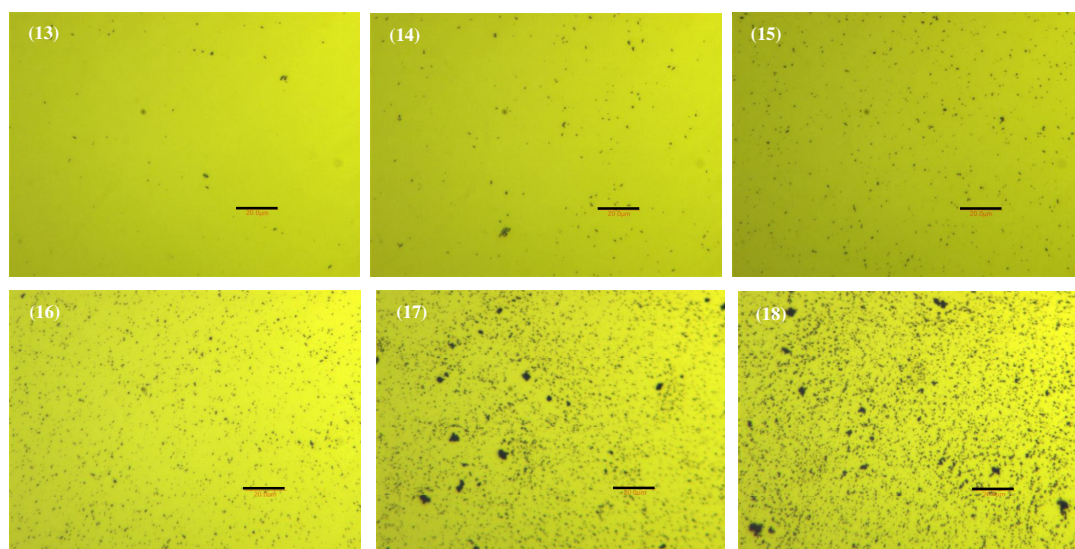
Figure 4. Spin cast films, rotated at 2000 rpm with < 0.1 second acceleration time on hydrophilic silicon, made from, left to right: 0.5 mg mL⁻¹ (image 10), 1.0 mg mL⁻¹

(image 11) and 2.0 mg mL^{-1} (image 12) C NP propan-2-ol spreading solutions. The scale bar corresponds to $100 \text{ }\mu\text{m}$ for all images.

The films made from the lower concentration of spreading solution appear reasonably similar on both types of substrates (image 7 vs image 10), with perhaps marginally higher film coverage on the hydrophobic surface. Using a concentration of 1 mg mL^{-1} of spreading solution, a more uniform film, with less clumping present, is achieved if a hydrophilic surface is employed (image 8 vs image 11). However, the biggest difference is found when a 2.0 mg mL^{-1} spreading solution is used. The film fabricated on the hydrophilic surface exhibits much larger clusters; indeed most of the film seems concentrated in two large aggregates (image 12), compared to that on the hydrophobic substrate (image 9) where a more or less uniform distribution of considerable smaller aggregates can be seen. Thus, although the difference seems small at low concentrations, as these are increased, a hydrophobic substrate is favoured.

2.4 FILM FORMING MATERIAL

Obviously the film forming material will have a direct influence on the spin cast film. However, the focus here is narrow: Pt NPs supported on C NP networks. Although the films investigated so far were made with 100% C NPs powders, these are not likely to behave in a significantly different manner (with respect to the variables studied) from Pt:C NP powders. Moreover, as the drive towards decreasing Pt loading continues (10 % by weight and even lower loadings are of increasing interest) the behaviour of the films should be increasingly dominated by the C content. However, as a check, films were also made from a wide range of Pt:C concentrations (*viz.* $10^{-3} \text{ mg mL}^{-1}$, image 13; $10^{-2} \text{ mg mL}^{-1}$, image 14; 0.1 mg mL^{-1} , image 15; 0.5 mg mL^{-1} , image 16; 1 mg mL^{-1} , image 17; 2 mg mL^{-1} , image 18; 4 mg mL^{-1} , image 19; 8 mg mL^{-1} , image 20), and are shown in fig. 5.



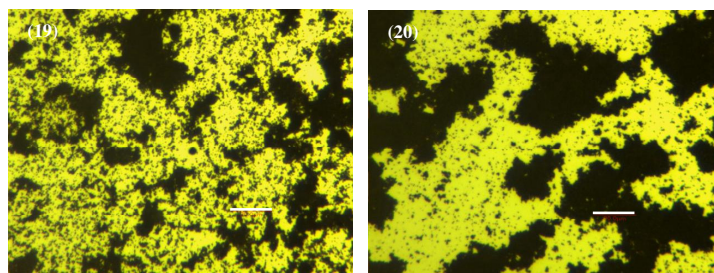


Figure 5. Spin cast films, rotated at 2000 rpm with < 0.1 second acceleration time on hydrophobic silicon, made from, 10^{-3} mg ml $^{-1}$ (image 13); 10^{-2} mg ml $^{-1}$ (image 14); 0.1 mg ml $^{-1}$ (image 15); 0.5 mg ml $^{-1}$ (image 16); 1 mg ml $^{-1}$ (image 17); 2 mg ml $^{-1}$ (image 18); 4 mg ml $^{-1}$ (image 19); 8 mg ml $^{-1}$ (image 20), Pt:C NP propan-2-ol spreading solutions. The scale bar corresponds to 20 μ m for all images.

As observed for the C NP films (fig. 3), as the concentration of spreading solution increases, film substrate coverage increases. It is interesting to note that even when using very dilute concentrations (10^{-3} mg ml $^{-1}$, image 13), particles can be discerned on the substrate (as well as a few blurred artefacts, due to dust specks on the digital camera, that are always present). Also noteworthy is the fact that up to modest concentrations (from 10^{-3} mg mL $^{-1}$ to 2 mg mL $^{-1}$, images 13 – 18), film coverage increases without a concomitant increase in aggregate size. At greater concentrations (*i.e.* 4 mg mL $^{-1}$ and 8 mg mL $^{-1}$) there is a sudden jump in the size of the agglomerates. Interestingly, whereas in the 4 mg mL $^{-1}$ image, the film is made up by a network of interconnected clusters of varying size (image 19), the film made up from 8 mg mL $^{-1}$ concentration of spreading solution is mostly concentrated in large clumps (image 20). 8 mg mL $^{-1}$ is close to the maximum concentration limit that allows a more or less uniform spreading solution dispersion.

Compared to the C NP films, (images 7 vs 16, 8 vs 17 and 9 vs 18) a similar trend in film coverage is seen, although the coverage of the Pt:C films is always lower. This is due to a greater amount of the spreading solution being made up of higher density Pt with respect to C.

2.5 ROTATION SPEED

Rotation speed is obviously an important variable that can affect the uniformity and coverage of spin cast films. However, if the aim is to produce highly uniform film coverage (as it is here), the usable range is usually reasonably small (for a given spreading solution viscosity). Too low a rotation speed and the film will be very non-uniform (as different rates of evaporation and edge material accumulation effects come into play). Too high a rotation speed and very sparse film coverage will result. For the fabrication of the films of interest herein, the density of the spreading solutions used was low and hence only relatively slow spin speeds were considered. Pt:C films rotated at 3000 rpm using spreading solution concentrations of 1 mg ml $^{-1}$ (image 21) and 2 mg mL $^{-1}$ (image 22) are shown in fig. 6.

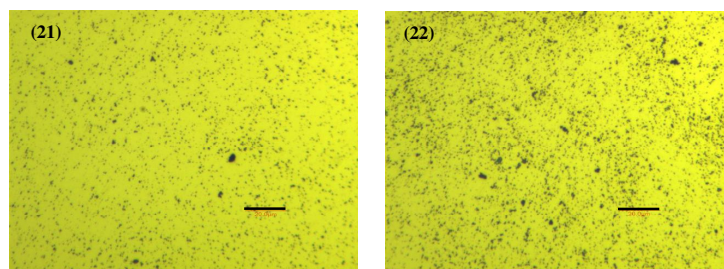


Figure 6. Spin cast films, rotated at 3000 rpm with < 0.1 second acceleration time on hydrophobic silicon, made from 1 mg mL⁻¹ (image 21) and 2.0 mg mL⁻¹ (image 22) Pt : C NP propan-2-ol spreading solutions. The scale bar corresponds to 20 μm for both images.

The expected increase in film coverage as the concentration increases is observed. Compared to the films spun at the same conditions, but at 2000 rpm (images 21 vs 17 and 22 vs 18), the films exhibit less film coverage, as might be expected. However, the degree of film clumping is also significantly less than in the slower spun films.

2.6 CO-DEPOSITED LAYERS

Unlike most film-forming materials usually employed for spin casting, catalyst powders are very insoluble in the majority of solvents normally used. This can be favourably exploited for the fabrication of films with good film substrate coverage: additional layers can be sequentially deposited to increase coverage. For typical spin cast film-forming materials, if a second layer of the same material is co-deposited over an existing film, the solvent will partially dissolve the underlayer and thus affect its uniformity, coverage and structure.

Spin cast films of Pt:C NPs, spun from highly concentrated spreading solutions (8 mg mL⁻¹) made from the sequential deposition of 1 (image 23), 2 (image 24) and 4 (image 25) layers onto HOPG substrates, are shown in fig. 7.

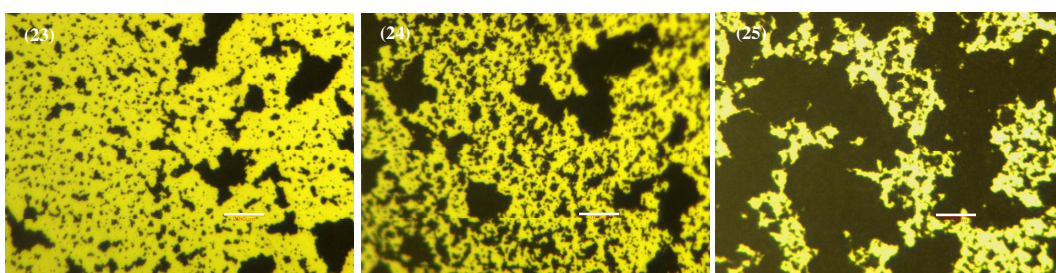


Figure 7. Spin cast films, rotated at 2000 rpm with < 0.1 second acceleration time on HOPG substrates, made from a 8 mg mL⁻¹ Pt : C spreading solution. From left to right: 1 layer (image 23), 2 layers (image 24) and 4 layers (image 25) sequentially deposited. The scale bar corresponds to 20 μm for all images.

As expected, as the number of deposited layer increases, so does the film coverage. The size of the aggregates also increases with the number of deposited layers. The 4 layer film (image 25) exhibits good film coverage (on a micrometer scale). If it is of interest to have complete film coverage over the entirety of the substrate surface, further layers could be deposited. Comparing the single layer film (image 23) with the

single layer film deposited under the same conditions but on hydrophobic silicon, it would appear that coverage is less on HOPG substrates.

3. CONCLUSIONS

Spin coating can be used to fabricate uniform catalyst films similar in structure and composition to those present in PEMFCs. In order to achieve this, the following recommendations are made with respect to spin coating variables.

- Pt:C powders should be dispersed in a concentration of *ca.* 8 mg ml⁻¹. This is close to the maximum concentration that will allow a uniform dispersion to be formed. An ultrasonication step (> 30 min) is essential to increase solution dispersion and must be carried out just prior to spin coating. A high concentration is desirable to maximise film coverage.
- Propan-2-ol should be used as the solvent as it has a tendency to minimise the degree of particulate coagulation.
- Rotation speeds of *ca.* 2000-3000 rpm should be used to ensure good film coverage and uniformity.
- Hydrophobic substrates are preferable since they tend to offer better adhesion for the catalyst films, especially when concentrated spreading solutions are used. In particular, HOPG is recommended since it offers an easily cleaned, (atomically) flat, hydrophobic conductive carbon underside for the catalyst films, akin to the carbon cloth commonly used as a backing layer in PEMFCs.
- Spin acceleration time has to be kept to a minimum. Acceleration time can influence film uniformity (thickness). Here, a high degree of uniformity is of interest and thus the effect of this variable has not been investigated but instead kept to the minimum that the instrument allowed (< 0.1 sec).
- Sequential deposition of spin coated layers can be used to increase film coverage. At least 4 layers are needed for good (continuous - depending on length scale) substrate coverage.

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5. APPENDIX 1

Table 1. Summary of variables studied.

Image No.	Material	Substrate	Solvent	Spin Speed (rpm)	Concentration (mg / mL)	Layers
1	/	phobic Si	/	/	/	/
2	/	uncleaved HOPG	/	/	/	/
3	/	cleaved HOPG	/	/	/	/
4	C NPs	phobic glass	acetone	2000 rpm	1.0 mg/mL	1
5	C NPs	phobic glass	cyclohexane	2000 rpm	1.0 mg/mL	1
6	C NPs	phobic glass	propan-2-ol	2000 rpm	1.0 mg/mL	1
7	C NPs	phobic Si	propan-2-ol	2000 rpm	0.5 mg/mL	1
8	C NPs	phobic Si	propan-2-ol	2000 rpm	1.0 mg/mL	1
9	C NPs	phobic Si	propan-2-ol	2000 rpm	2.0 mg/mL	1
10	C NPs	philic Si	propan-2-ol	2000 rpm	0.5 mg/mL	1
11	C NPs	philic Si	propan-2-ol	2000 rpm	1.0 mg/mL	1
12	C NPs	philic Si	propan-2-ol	2000 rpm	2.0 mg/mL	1
13	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	0.001 mg/mL	1
14	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	0.01 mg/mL	1
15	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	0.1 mg/mL	1
16	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	0.5 mg/mL	1
17	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	1.0 mg/mL	1
18	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	2.0 mg/mL	1
19	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	4.0 mg/mL	1
20	6-4 C-Pt	phobic Si	propan-2-ol	2000 rpm	8.0 mg/mL	1
21	6-4 C-Pt	phobic Si	propan-2-ol	3000 rpm	1.0 mg/mL	1
22	6-4 C-Pt	phobic Si	propan-2-ol	3000 rpm	2.0 mg/mL	1
23	6-4 C-Pt	phobic HOPG	propan-2-ol	2000 rpm	8.0 mg/mL	1
24	6-4 C-Pt	phobic HOPG	propan-2-ol	2000 rpm	8.0 mg/mL	2
25	6-4 C-Pt	phobic HOPG	propan-2-ol	2000 rpm	8.0 mg/mL	4