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Investigation of PEM fuel cell electrodes by transmission electron microscopy, scanning electron microscopy and x-ray diffraction

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Abstract

PEM fuel cell electrodes were investigated by transmission electron microscopy (TEM), scanning electron microscopy (SEM) and x-ray diffraction (XRD). As one example of the application of these techniques, catalyst nanoparticles were characterized before and after desorption experiments up to 150°C, in the frame of temperature-dependent CO desorption kinetics experiments, which are of importance for a better understanding of the fundamental physicochemical processes involved in improving CO tolerance. Particle diameter distributions of a novel Pt-on-Au based catalyst nanomaterial were determined from TEM data and compared with average sizes calculated from XRD data.

Introduction

PEM fuel cells can already fulfill the performance requirements for automotive applications, while cost and durability are still very relevant issues [1]. In this context, the mechanistic understanding of the fuel cell tolerance against CO is very relevant [2, 3].

The micro- and nanostructure of a novel Pt-on-Au/C fuel cell anode catalyst obtained by electrochemical deposition of platinum on carbon-supported gold nanoparticles was characterized before and after the exposure of the catalyst to temperatures up to 150°C during desorption experiments.

Results and Discussion

The desorption kinetics of adsorbed isotopically labelled ^{13}CO in the presence of natural ^{12}CO were studied as a function of the temperature. Materials scraped from the catalyst layer of samples before and after desorption experiments were characterised using a FEI Tecnai 20 transmission electron microscope operated at 200kV with a LaB_6 filament. The catalyst dispersion and particle size distribution were assessed by the observation of a large representative number of catalyst nanoparticles (more than 200). The surface structure of the Pt-on-Au/C system, both before and after the desorption experiments, was investigated using a Zeiss Supra 50 field-emission gun scanning electron microscope. Characterisation of the bulk composition of the synthesised Pt-on-Au catalyst nanoparticles was performed using a Philips PW 3830 X-ray diffraction apparatus, both before and after the desorption experiments.

Figure 1 shows a transmission electron microscopy micrograph of Pt-on-Au catalyst nanoparticles and the corresponding particle diameter distribution of as received catalyst material. The average diameter is 4.1 nm ± 0.8 nm.

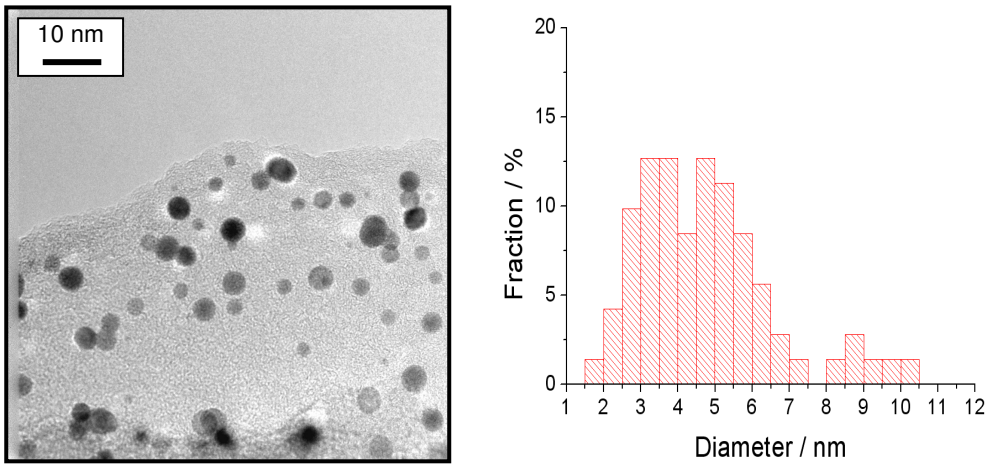

Figure 1: TEM photo of as received carbon-supported Pt-on-Au catalyst nanoparticles and corresponding diameter distribution

XRD characterization has been used in addition to TEM. This combination of methods is beneficial as TEM and XRD are complementary methods for the evaluation of catalyst nanoparticles size. TEM allows direct imaging while XRD allows averaging of relatively large sample volumes. Good agreement between TEM and XRD data was obtained and no significant differences in micro- and nanostructure before and after CO desorption experiments were found.
SEM allows the investigation of larger surfaces of the electrode while catalyst nanoparticles are still detectable.
Figure 2 shows a scanning electron micrograph of the as received material. The catalyst particles are visible.

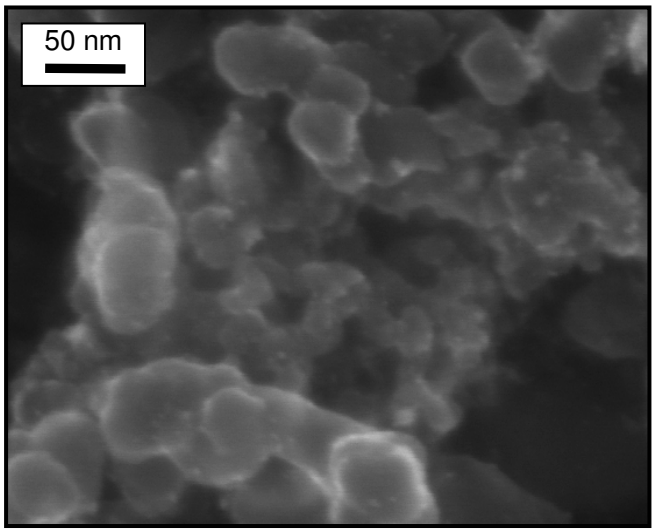

Figure 2: Scanning electron micrograph of the Pt-on-Au/C surface as received

XRD is also of interest for characterization of the catalyst nanoparticle composition. The Au peaks in the XRD pattern of the as received sample are shifted to lower 2θ values with respect to pure Au and show a relatively low intensity when compared with Au peaks observed on pure crystalline Au nanoparticles. This suggests the formation of an alloy involving the incorporation of Pt atoms into the fcc structure of Au. Additionally, unshifted Pt peaks are also observed which suggests the presence

of an additional crystalline Pt phase. This suggests that the real catalyst structure deviates from core/shell particles which might be expected when only considering the preparation method. Instead the synthesised Pt-on-Au catalyst nanoparticles might be constituted of a core alloy phase rich in Au, in which Pt atoms have been incorporated, and of a thin Pt film at the surface.

Conclusions

A novel Pt-on-Au/C fuel cell anode catalyst was characterized before and after CO desorption experiments up to 150°C by SEM, TEM and XRD. Insight into the composition, structure and particle diameter distribution of the catalyst nanoparticles was obtained. The influence of the temperature on the properties of the catalyst nanoparticles was also assessed. No significant differences in micro- and nanostructure before and after CO desorption experiments up to 150°C were observed.

References

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