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The gas-aggregation source technique with a planar magnetron is used for the production of PtX (X = Pd or Au) catalyst nanoclusters on polymeric substrate used in low temperature fuel cell. The target material is sputtered by employing either a DC power supply or a high-power impulse magnetron sputtering power device known to enhance the degree of ionization of the target species. The nanocluster properties are studied as a function of the power characteristics by diagnostics like time-of-flight mass spectrometer and post deposition methods (Grazing Incidence X-Ray Diffraction, Transmission Electron Microscopy) compared to those obtained by standart DC magnetron sputtering.

The gas-aggregation source (GAS) with a planar magnetron may be a convenient technique for the production of pure metallic nanoclusters (NC) [1] compared to wet chemical techniques needing a final calcination step that induces cluster growth and substrate deterioration. Such NC may have important applications such as in heterogeneous catalysis, gas sensor technology and microelectronics. The nanocluster source is already employed for the production of a wide variety of metal NCs; e.g. Co, C, Ti, Pd, Pt… and of a few bimetallic materials. Proton exchange membrane (PEM) fuel cells require the production of such electrocatalysts directly on a polymeric membrane. The properties (shape, structure and size) of these catalytic NCs must be well controlled in order to promote their catalytic activity towards the oxygen reduction reaction.

The present study aims to synthesize PtX (X = Pd or Au) catalysts on the proton exchange membrane by the GAS technique with a planar magnetron. The experimental setup is made of three aligned components : the GAS (0.3 mbar in pure argon), a high vaccum deposition chamber (about 0.003 mbar) and a time-of-flight mass spectrometer. Pt and X atoms are sputtered by argon ions from the PtX target mounted on the two inch magnetron in the GAS. Due to the relative high pressure, PtX NCs are formed. They are ejected from the GAS through a 4 mm in diameter aperture and land on the substrate placed in the high vaccum chamber. When the substrate holder is removed, the mass spectrometer may be used to characterize the NC size. In the past 2 decades, a DC power supply was applied to the magnetron for the production of a temporally stable NC beam. Adjustable parameters such as aggregation length and gas flow rates were used for controlling the beam intensity and the NC size. In this study, these two parameters are kept constant and the target is sputtered by employing a pulsed power supply using low frequency. Such power supply, called high-power impulse magnetron sputtering power (HIPIMS) supply is known to enhance the degree of ionization of the target species [2]. Two recent publications provide insight into the potential benefit of using pulsed power supply for the synthesis of metallic nanoclusters [3,4].

The PtX NCs grown by HiPIMS based GAS are compared to those obtained by conventional GAS employing DC power supply. The size of the PtX NCs is measured by Transmission Electron Microscopy and Mass spectrometer. The nanoclusters have a mean diameter of 5 nm, depending of the operating parameters. The influence of the HiPIMS power characteristics (pulse power, duty cycle and repetition rate) on the NC growth mechanism is studied. For very low frequencies, a unique HiPIMS pulse induces an individual group of nanoclusters. This group is not correlated to the previous and to the following one making possible the control of the NC size. Grazing Incidence X-Ray Diffraction (Soleil synchrotron, SixS) is used to study the cristalline structure of PtPd nanoclusters.

References

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