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Synthesis and electrochemical double layer characterization of nanoporous gold

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Abstract

The present article highlights synthesis and electrochemical double layer characterisation of nanoporous Au. The electrochemical double layer studies were carried out on nanoporous Au samples immersed in a weakly adsorbing HClO₄ electrolyte in-situ in three-electrode cell geometry. The specific-volumetric capacitance value obtained from the double layer charging experiments are discussed in terms of the variation in ligament diameter.

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1. Introduction

Nanoporous (np-) Au electrode and its properties has been investigated in detail over the past two decades [1-5]. Due to its extremely small structure size and the formation of capacitive double layer at the electrolyte impregnated pore surfaces, np-Au electrodes have generated a high interest. This involves fundamental understanding about the electrified pore - electrolyte interface and its functional character due to potential controlled accumulation of ions in

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the electrolyte. As evidenced by the rapid increase in its surface controlled properties, np - Au metal behaves as a device material and there by constitutes an affordable material for use as mechanical actuator,

sensors, super-capacitors, catalysts, energy converters and membranes [3,6-10]. It is noting that the magnitude of charge density acquired at the porous Au surfaces depend up on its electron density, ion density adjacent to electrolyte and charge separation between the pore surfaces and the ions in the electrolyte [11,12].

The nanoporous Au electrodes are prepared either by using electrochemical dealloying method [13] in a controlled anodic potential or by following a free-corrosion route [14]. Among these two preparation methods, the nanoporous Au produced by the electrochemical dealloying is reportedly monolithic in nature. It exhibits superior elastic and plastic deformation properties. The structure size in nanoporous Au can be varied depending upon a number of preparation parameters like dealloying potential, electrolyte bath temperature and alloy composition. Jin et al., [1] reported that the electrochemical dealloyed nanoporous Au exhibits remarkable ductile behaviour with reversible yielding up on polarizing the electrolyte wetted porous Au surfaces.

What is not known till date in the area of research on nanoporous Au is about the behaviour of specific capacitance of np-Au with the variation of ligament diameter d . To gain knowledge on this issue, we were motivated to study the ligament size dependent variation in specific capacitance value in np-Au up on double layer charging. The double layer charging experiments were carried out on np-Au by varying the ligament-pore microstructure with temperature. The study is of important to understand the effect of ions in the fluidic electrolyte to the double layer capacitive behaviour on metal surfaces and to find the nature of np-Au electrode surfaces in contact to the electrolytes. The state - of - art issues related to the double layer charging in the followed determination of specific capacitance on nanoporous Au surfaces are also discussed.

2. Experimental Procedure

The preparation method of nanoporous Au, electrochemical set-up used, and the measurement protocol have been discussed in our earlier publications [15,16]. For sake of completion only a brief summary will be furnished here. Small pieces of specimen were cut from the arc-melted $\text{Ag}_{75}\text{Au}_{25}$ alloy ingots were used for electrochemical dealloying experiments. The arc-melted $\text{Ag}_{75}\text{Au}_{25}$ ingots were shaped in to discs using a mechanical rolling machine. The disc specimens were polished on fine emery sheets of about quarter micrometer finish in oxygen free silicon fluid and finally cleaned in an ultrasonic bath using ethanol solution. Then, the specimens were subjected to heat treatment in a sealed quartz ampoule at 800°C to remove the residual stress and for chemical homogenization.

The dealloying experiments were performed on the defect free $\text{Ag}_{75}\text{Au}_{25}$ samples at room temperature under a potentiostatic control potential of 850 mV versus Ag/AgCl pseudo reference electrode. The electrolyte solutions were prepared from analytical reagent grade chemicals and 18.2 M Ω Millipore water free of organic impurities. Since we maintain large electrode surface area - to - solution volume ratio, the problem related to the organic contamination is expected to be reduced during the cyclic potential scans. Cyclic voltammograms of pure np-Au samples immersed in 0.5 M HClO_4 electrolyte were recorded in a wider potential interval for pin pointing the dominant double layer potential region. Subsequent to the CVs recorded across the wider potential intervals, the cyclic scans were performed around the double layer dominant region to determine the specific-volumetric capacitance C_v value in np-Au with a ligament diameter of d . Scanning electron microscopy images of np-Au acquiring from the temperature dependent double layer experiments were used to determine the ligament diameter d . All the electrochemical experiments were carried out with an Autolab PGSTAT 302N.

3. Results and Discussion

Figure 1 a) illustrates the electrochemical cell configuration before and after dealloying of $\text{Ag}_{75}\text{Au}_{25}$ at 850 mV in 1M HClO_4 solution. Noticeable changes were observed in the cell during the dealloying process. The electrochemically active Ag dissociated from the master alloy and dissolved in to the solution as Ag^+ ions and accumulated near the surface of Ag spiral counter electrode. The most important observation was the change in colour of the sample after Ag dissolution. The dealloyed sample turns into brownish black from the original silvery

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