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CHARACTERIZATION OF PD-NI THIN FILM BY ANNEALING METHOD

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ABSTRACT

Thin film electrode production and characterization for heat cell studies are described. The objective is two-fold: maximizing excess heat production and insuring a long lifetime and electrolysis. To do this in a reproducible faction, a pre-and post-run analysis of the films was carried out using various probe techniques. In this study, various Pd-Ni thin films were manufactured using with a magnetron sputtering method. A variety of methods for pre-conditioning of substrates were experimented with. The samples were also treated by different annealing methods using inert gas and vacuum annealing processes. The investigations of samples were performed in an UHV system equipped with scanning electron microscopy (SEM), x-ray diffraction (XRD) and atomic force microscopy (AFM). It is shown that the vacuum annealed samples provided the best films. This suggests that vacuum annealing can pull bubbles from pores in the thin film, giving a higher density thin film that performs better. These results and their implications for electrode development will be discussed.

1. INTRODUCTION

In 1989, Professors Pons and Fleischmann[1] created a media storm by announcing their success in causing fusion to take place in an ordinary electrolytic cell containing D_2O. Even though these results remains debatable a number of laboratories are still researching this phenomena[2-4]. But non-reproduciblity has prevented a clear identification that also strongly influences the critical factor required to cause such low energy nuclear reactions. Studies at The University of Illinois have focused on high water reactions in thil film of Pd, Ni, Ti, etc. Hydrogen traveling in Pd-Ni thin films also effect electrical properties of the film, and such effects appear to be related to reaction phenomena. In order to increase electrical properties of thin films, good adhesion, and good lattice formation along with preferred crystal orientation are very important factors. According to basic concepts for the vacuum processes and thin films, the annealing process is a critical method to get good properties. Consequently, we investigated the characterization of Pd-Ni thin films on Al_2O_3 substrate using vacuum annealing methods such as inert gas and vacuum processing.

2. EXPERIMENTS

Ultrasonic cleaner is used for the $A1_2O_3$ substrate cleaning,. The Pd-Ni thin film was fabricated using high purity Palladium and Nickel target, and the magnetron sputtering system shown in figure 1. The base vacuum was $1.9*10^{-7}$ torr. An initial glow discharge treatment was carried out by Oxygen glow discharge with a gas flow rate of 30 sccm, the system pressure of 20 mtorr was maintained for 3 minutes. The 8000 Å thick Palladium thin film was fabricated at a power of 250 W, the thickness, and a system pressure of 5 mtorr, with and an Argon gas flow rate of 20 sccm. A topcoat of 1000 Å Nickel thin film was sputtered at 250 w, at a system pressure of 5 mtorr, and an Argon gas flow rate of 20 sccm. For comparison purposes, the Pd-Ni thin films was treated with inert gas (Argon) and vacuum annealing methods up to 700 ˚C suing step temperature profile for both heating and cooling.

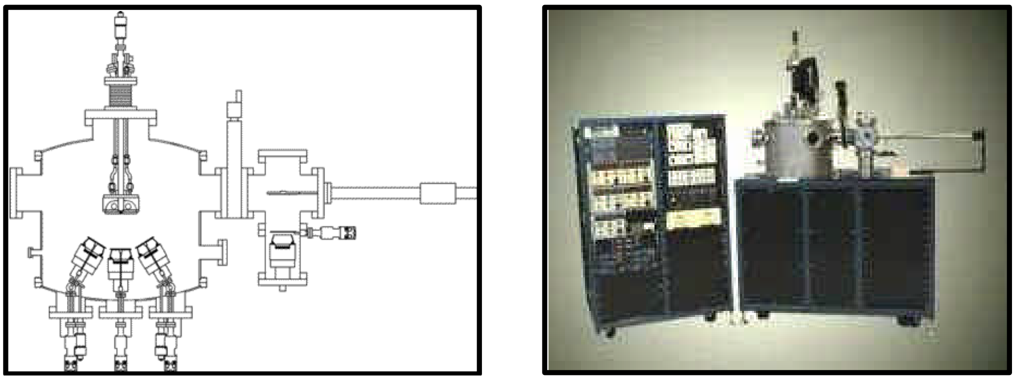


Fig. 1 Schematic diagram and picture of magnetron sputtering system

The surface morphology of Pd-Ni thin film was investigated using secondary electron microscopy (SEM), atomic force microscopy (AFM). X-ray diffraction (XRD) was used to observe the crystal structure of Pd-Ni thin films. Figure 2 shows the flowchart of the overall process.

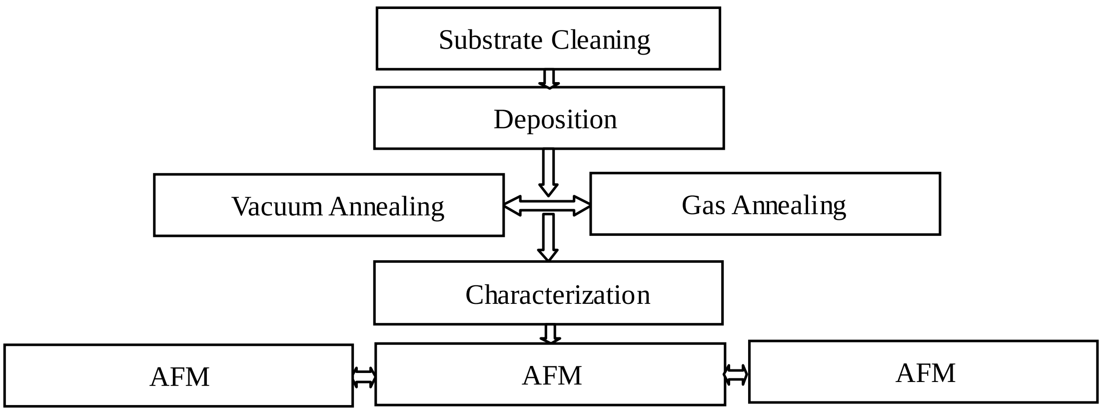
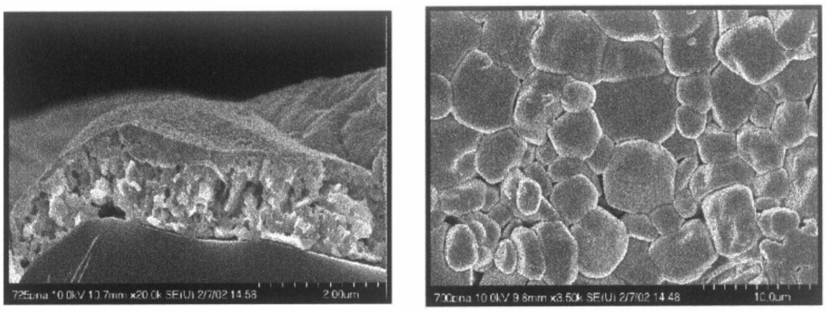


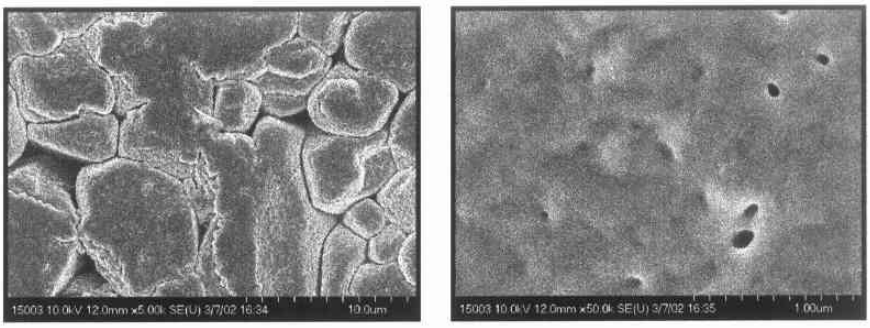
Fig. 2 Flowchart of the overall fabrication and characterization process.

3. RESULTS AND DISCUSSION

Photo. 1 shows surface morphology and cross-sectional image of Pd-Ni thin films fabricated with Argon gas annealing and the vacuum annealing process using SEM. As seen from these photos, the gas annealed Pd-Ni thin films have poor adhesion and highly porous. This indicates that the gas or also air annealing processes were not suitable for the Pd-Ni thin films because both processed left a large density of bubbles and pores. However, the vacuum annealed Pd-Ni thin film formed a connected grain structure. This structure results are increased tensile strength of Pd-Ni thin films. Consequently, when sectioning for the SEM, the film did not fracture but instead curled up along the cut edge.



(b) Argon gas annealed Pd-Ni thin film



(c) Vacuum annealed Pd-Ni thin film

Photo. 1. SEM images of Pd-Ni thin films by different annealing methods

Photo. 2 show the roughness analysis of vacuum annealed Pd-Ni thin film. The roughness of Pd-Ni thin film on $A1_2O_3$ substrate was not changed. The RMS of Pd-Ni thin film was about 751 nm. A smoother surface is ultimately desired, and this will be the focus of future work. Pre-preparation of the substrate and/or use alternate substrate materials may be necessary.

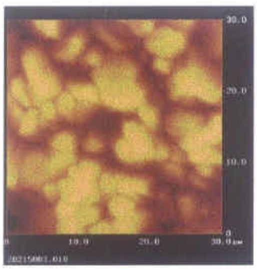


Photo. 2. AFM image of Pd-Ni thin film on Al_2O_3 substrate.

4, CONCLUSIONS

As shown, the vacuum annealing process can provide thin-film properties such as adhesion, tensile strength and fewer pores because this process pull bubbles from pores in the thin film through the surface. Thus, the vacuum annealed samples result in a higher density thin film with improved strength and electrolytic performance.

ACKNOWLEDGEMENT

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