Preparation of Pd membrane on porous stainless steel support modified with Zr for hydrogen purification

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INTRODUCTION

Hydrogen selective membranes have attracted considerable attention due to their potential applications in separation and purification of hydrogen for fuel cell and membrane reactors [1]. Membrane reactors have the advantage of combining chemical reaction and separation in a single unit.

Porous supports such as stainless steel have often been employed as a support for Pd membrane because of its thermal expansion coefficient being close to that of Pd-based films, corrosion resistance, high thermal stability and good mechanical strength.

EXPERIMENTAL

The tubular stainless steel supports (32 mm length, 15 mm o.d., 10 mm i.d.) were supplied by Brats, Brazil. Its composition in wt. is: Si: 7.6%, Cr: 17.5%, Fe: 62.2%, Ni: 11.1%, Mo: 1.6%. The supports were washed with carbon tetrachloride for 1 h in ultrasonic vibration bath, then with ethanol for 0.5 h for removal of contaminants and dried at 393 K for 1 h.

The next step in membrane preparation was the modification by a sol-gel process. The colloidal sol was composed of ZrO_2 and polyvinyl alcohol. After dipping, it was dried at 773 K for 2 h [2].

The deposition of Pd on the modified surface of the porous supports was carried out at 333 K by electroless plating technique [2]. The plating solution consisted of EDTA stabilized Pd-amine metal complexes with hydrazine as reducing agent.

RESULTS AND DISCUSSION

Figure 1 shows SEM pictures of the stainless steel support. As shown in Fig. 1(A), the support without modification had an asymmetric structure with a porous layer on the outside. After a single layer Zr has been deposited on support (Fig. 1B) some pores were plugged by Zr. However, the larger pores are not completely covered yet. The support modified by multilayer Zr derived from three cycles (Fig. 1C) of dip-coating. The surface composition was analyzed by EDS and it shows that the wt of Zr increased of 10.1% (single layer) to 19.5% (multilayer). Figure 1(D) shows the support after six cycles of Pd plating. The surface of the support was covered with a thin and bright Pd homogeneous film.

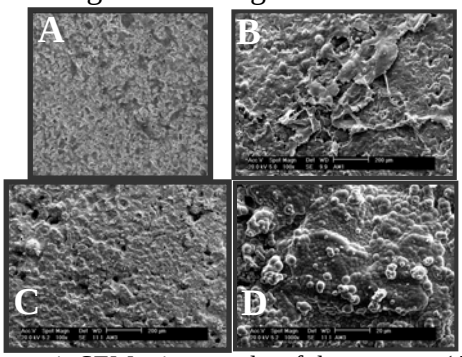

Figure 1: SEM micrographs of the support. (A): without modification; (B): modified by monolayer Zr; (C): modified by multilayer Zr; (D) after Pd plating

Conclusions

The model of hydrazine-based plating solution is in good agreement with the experimental data, and provides accurate predictions of deposition rate as function of plating parameters.

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