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Electrochemical actuation behaviors and mechanisms of bulk nanoporous Ni-Pd alloy

Jie Zhang^a, Lanfen Lv^a, Hui Gao^a, Qingguo Bai^a, Chi Zhang^a, Zhonghua Zhang^{a,b,*}^a Key Laboratory for Liquid-Solid Structural Evolution and Processing of Materials (Ministry of Education), School of Materials Science and Engineering, Shandong University, Jingshi Road 17923, Jinan 250061, PR China^b School of Applied Physics and Materials, Wuyi University, 22 Dongcheng Village, Jiangmen 529020, PR China

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ABSTRACT

Metallic actuators have recently aroused great interests, and alloying of noble element with earth-abundant metal is essential to lower the cost of actuation materials while keeping a significant strain response. Here, we report the design/fabrication of bulk nanoporous nickel-palladium alloy by dealloying. The alloy with a hierarchically porous structure shows different actuation behaviors in different electrolytes, which are correlated with the adsorption/desorption of hydrogen/hydroxyl and the nature (clean or oxide-cover) of ligament surface. The maximum reversible strain of the nickel-palladium actuator could reach 0.47% in potassium hydroxide solution, which is more competitive compared with other metallic actuation materials.

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High performance actuators can generate mechanical energy efficiently. A series of traditional actuator materials, such as piezoelectric ceramics [1], conducting polymers [2,3], shape memory alloys [4], carbon nanotubes [5], have been explored and present outstanding actuation properties. However, practical applications are constrained by their own disadvantages. In recent years, nano-structured high-surface-area materials have attracted considerable attention in various fields. People have started to think whether nanoporous (np) metals can be applied to actuation materials [6]. Several nanoporous materials have been reported to possess reversible strain response when electrochemical potentials were applied, e.g. np-Pt [6], np-Au [7–9], np-Ag [10], np-Pd [11], np-Au-Pt [12], etc. The np-Pt is a pioneering material for actuators or metallic muscles [6,13]. The reason why nano-structured materials can achieve their functionality as actuators is that the variation of surface charge can induce large compensating stress and strain in their solid skeletons of metallic nanoporous materials [14,15].

At present, most of the reported metallic actuators are noble metals/alloys, and the high cost impedes their practical applications. It is urgent to reduce the cost of metallic actuators and improve their actuation performance. Alloying of noble metals with earth-abundant elements is an effective means to lower the cost of actuation materials for metallic actuators, while maintaining their actuation performance. In this work, we designed and fabricated bulk np-Ni₈₀Pd₂₀ alloy through dealloying

of Al₇₅Ni₂₀Pd₅ precursor. We further explored the actuation behaviors of np-Ni₈₀Pd₂₀ in different electrolytes (KOH and NaF). The related mechanisms have also been rationalized based upon adsorption/desorption of involved species like H and OH.

The Al₇₅Ni₂₀Pd₅ (at%) precursor alloy was prepared from pure Al (99.99 wt%), Ni (99.99 wt%) and Pd (99.99 wt%) using high-frequency induction heating in a quartz crucible. The Al₇₅Ni₂₀Pd₅ precursor with a rod-like shape was fabricated by a blow casting technique in a copper mold. The Al-Ni-Pd rods were annealed at 200 °C for 10 h before dealloying to remove the residual stress. Bulk np-Ni₈₀Pd₂₀ samples were prepared by dealloying the Al-Ni-Pd rods in a 5 M NaOH aqueous solution at room temperature until no obvious bubbles appeared. Dealloying was continuously carried out at 90 ± 5 °C in the 5 M NaOH solution in order to further remove the residual Al. X-ray diffraction (XRD, Rigaku D/max-rB) was served to identify the phase constitution of the dealloyed samples. Scanning electron microscopy (SEM) observations were executed with a field-emission microscope (FEI QUANTA FEG250). Transmission electron microscopy (TEM) was used to characterize the microstructure of the np-Ni₈₀Pd₂₀ samples.

The electrochemical actuation of np-Ni₈₀Pd₂₀ was measured through a three-electrode system. Fig. S1 shows the measuring apparatus for electrochemical actuation schematically. A saturated calomel electrode (SCE) was used as the reference electrode (RE), and a platinum sheet with an enough large area served as the counter electrode (CE). The np-Ni₈₀Pd₂₀ rod was directly placed as the working electrode (WE) in an electrochemical cell inserted in a dilatometer (KY-PZY-III). The resolution of the displacement sensor was 100 nm and the measuring range was 1000 μm. The sample was connected through a push rod

* Corresponding author at: Key Laboratory for Liquid-Solid Structural Evolution and Processing of Materials (Ministry of Education), School of Materials Science and Engineering, Shandong University, Jingshi Road 17923, Jinan 250061, PR China.

E-mail address: zh_zhang@sdu.edu.cn (Z. Zhang).

to the displacement sensor which recorded the expansion and contraction of np-Ni₈₀Pd₂₀. The electrochemical data were recorded using a CHI 660E potentiostat (Shanghai Chenhua Instruments Co. Ltd, China). The electrochemically induced actuation of np-Ni₈₀Pd₂₀ was measured in 1.0 M KOH or 0.7 M NaF aqueous solution at room temperature. Cyclic voltammetry (CV) and square wave potential (SWP) were used to give the electrochemical stimuli.

The high quality crack-free bulk np-Ni₈₀Pd₂₀ rods with a hierarchically porous structure have been fabricated successfully through the dealloying strategy (inset of Fig. 1a). The low and high magnification SEM images clearly show that the np-Ni₈₀Pd₂₀ rods are composed of dendrite-like blocks and continuous large-sized channels (Fig. 1a and b). The TEM image (Fig. 1c) confirms that the dendrite-like blocks are nanoporous and have ligaments/nanopores of several nanometers. The selected area electron diffraction (SAED, inset of Fig. 1c) verifies the nanocrystalline nature of np-Ni₈₀Pd₂₀. In addition, the XRD pattern shows a new set of broad diffraction peaks located at around 40.8, 47.4, 68.8 and 82.7° (2θ), which shift to higher angles compared to f.c.c. Pd phase. These diffraction peaks can be assigned to the (111), (200), (220) and (311) reflections of f.c.c. PdNi alloy in which Ni atoms exist in the form of solutes.

Electrochemical actuation behaviors of the np-Ni₈₀Pd₂₀ sample with a clean surface in the 1 M KOH solution are shown in Fig. S2. Continuous five CV curves almost overlap, indicating the good reproducibility and the structural stability of np-Ni₈₀Pd₂₀ (Fig. S2a). The strain is given as $\varepsilon = \Delta L/L_0$ (here, ΔL and L_0 denote the length change and the initial length of np-Ni₈₀Pd₂₀, respectively). Fig. S2b presents the corresponding strain with the change of time, and irreversible shrinkage occurs in

np-Ni₈₀Pd₂₀. The reason for the irreversible shrinkage may be the inverse strain response of Pd and Ni in this potential range related to the different adsorption/desorption processes of H and OH species [16,17]. The np-Pd presents reversible expansion and contraction features associated with hydrogen adsorption/absorption-desorption, accompanying the evolution of CVs in the same potential range [16]. In comparison, the np-Ni with the clean surface expands with the OH-adsorption (reversible formation/reduction of α -Ni(OH)₂, $\text{Ni} + 2 \text{OH}^- \leftrightarrow \alpha\text{-Ni(OH)}_2 + 2e^-$) and H-desorption, and reversibly contracts with the OH-desorption (reduction of α -Ni(OH)₂ back to metallic Ni) and H-absorption in the same potential region [17,18]. As a result, the combined effect of H- and OH- adsorption/desorption on Pd and Ni leads to the irreversible shrinkage of np-Ni₈₀Pd₂₀, as schematically shown in Fig. S2c.

The np-Ni₈₀Pd₂₀ sample was then subjected to a CV treatment in the wide potential range from -1.1 to +0.6 V vs. SCE in the 1 M KOH solution in order to obtain the oxide-covered surface. Fig. S3a shows the successive CV curves of the np-Ni₈₀Pd₂₀ rod at 1 mV s⁻¹. The CV curves exhibit obvious redox peaks in this potential region, which is in agreement with the adsorption/desorption of involved species (H or OH). On the whole, the CV profile of np-Ni₈₀Pd₂₀ is roughly the same with that of np-Pd [16]. The np-Ni₈₀Pd₂₀ sample shows partial reversible expansion and contraction features accompanying the evolution of CVs (Fig. S3b). In such a potential window, the ligament surface of np-Ni₈₀Pd₂₀ will form a nickel oxide layer (β -NiO), which cannot be removed even at quite negative potentials (-1.1 to -0.7 V vs. SCE) [18]. In the potential region of -1.1 to -0.7 V vs. SCE, the np-Ni with the NiO-covered surface shows almost no strain response. In the

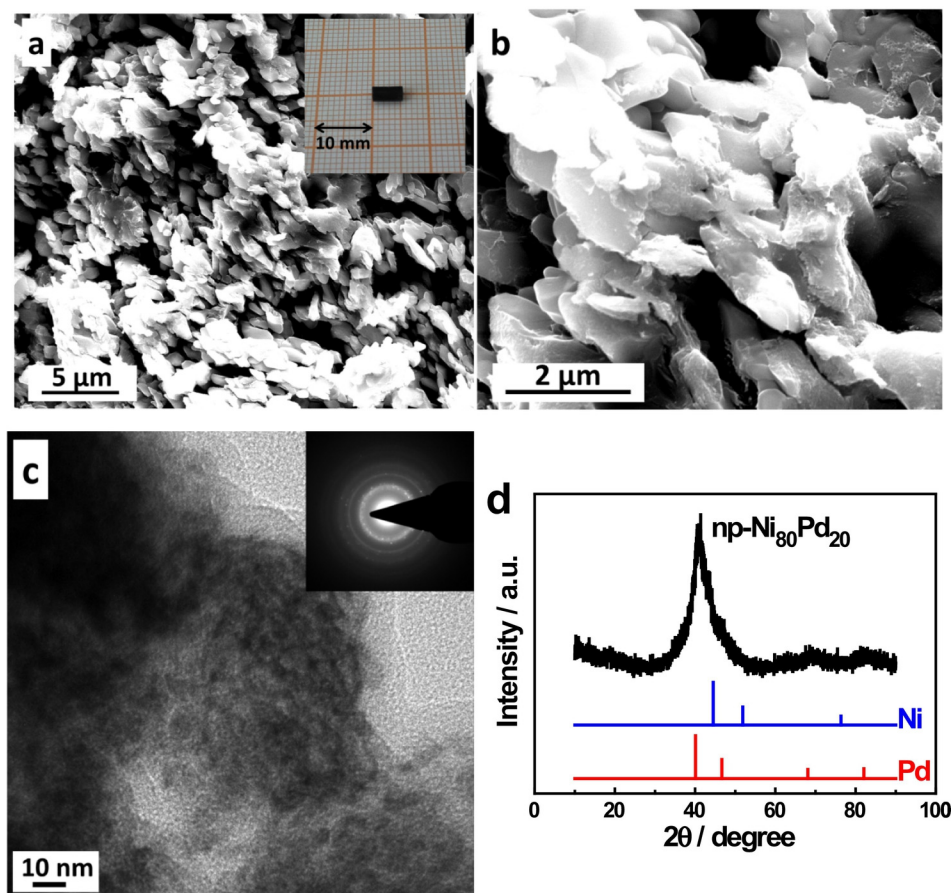


Fig. 1. Structural characterization of bulk np-Ni₈₀Pd₂₀. (a) Section-view SEM image of the np-Ni₈₀Pd₂₀. (b) High-magnification SEM image showing the porous structure. (c) TEM image and (inset) corresponding SAED pattern. (d) XRD pattern of np-Ni₈₀Pd₂₀.

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