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ARTICLE

Preparation of Submicro-porous Nickel Wafers by Molding-Decomposition-Sintering Method Using Nickel Oxalate Nano-Rods as Precursors

Li Tao[1], Liu Ying [2], Ma Guohua [3]

[1] Center of Engineering and Technology, Southwest University of Science and Technology, Mianyang 621010, China; [2] College of Materials Science and Engineering, Sichuan University, Chengdu 610065, China; [3] Analytical and Testing Center, Southwest University of Science and Technology, Mianyang 621010, China

Abstract: Submicro-porous nickel wafers were prepared by a molding-decomposition-sintering method, using nickel oxalate nano-rods synthesized via a liquid phase precipitation process as precursors, keeping the decomposition temperature of 360 °C for 10 min, and sintering temperature of 420, 450，480 and 510 °C for 10 min, in a tubular oven filled with Ar gas. The study of Infrared spectroscopy (IR) indicates that the as-prepared precursor was pure nickel oxalate. The results of X-ray diffractions (XRD) indicate that the as-treated grey wafers were nickel, which has face-centered cubic crystal structure. The images of scanning electron microscopy (SEM) and atomic force microscopy (AFM) show that the morphology of metal nickel wafers exhibit an obvious porous structure. The structure of pores is irregular with 100~1000 nm in diameter, and the pore walls are composed of fiber-like nickel with 100~300 nm in diameter.

Key words: submicro-porous; nickel; molding; decomposition; sintering

Nickel-based porous materials with lager specific surface area and larger electro-catalytic activity[1], are widely used as catalysts[2-5], electrodes[6-9], and magnetic materials[10], etc. Their electrochemical properties are influenced by the porous size, the structure and the distributions which depend on the preparing technology.

Many methods are used for preparing nickel porous materials with different sizes and structure. Using electrochemical deposition method, Zhuo Kai et al[7] obtained nano-porous Ni-Sn alloy with dendritic structure and 20~50 nm in pores diameter. Li Jing et al[11] prepared NiTi biological porous materials using the polymer sponge impregnation technique with pore sizes in the range of 150~500 μm. D. Chade et al[12] obtained micro-sized porous Raney nickel adopting atmospheric plasma spraying for alkaline water electrolysers. Li Meng[13] prepared micro-sized porous Ni bulk catalyst adopting spark plasma sintering (SPS) using spherical atomized Ni powder as the starting material. Li Kaihua et al[14] prepared foam nickel with 400~500 μm in pore diameter using the method of low temperature electroless plating nickel on the polyurethane foam and thermal decomposition. Zhu Qingwen et al[10] obtained hollow and porous nickel microspheres adopting low temperature molecular self-assembly in an aqueous medium in which hydrazine hydrate acted as the reducing agent, and the average diameter of the spheres was 1.4 um and mean thickness of the sphere wall was 120 nm. J. H. Choi et al[15] synthesized porous nickel powder with micro-sized pores and reticular structure, using hydrazine monohydrate as reducing agent and polyvinylpyrrolidone (PVP) as a soft template through a polyol process. Liu Zhiguo et al[16] synthesized nano-

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Corresponding author: Li Tao, Ph. D., Associate Professor, Center of Engineering and Technology, Southwest University of Science and Technology, Mianyang 621010, P. R. China, Tel: 0086-816-2419090, E-mail: ltao74@163.com

porous nickel rods with 50 nm in pore diameter, using p-phenylenediamine (PPD) and $NiCl_2 \cdot 6H_2O$ as reducing agent and oxidizing agent, respectively in aqueous solution. Qi Zhen et al[17] prepared heterogeneous micro-porous Ni catalysts adopting a de-alloying chemical method, via treating Al-Ni alloys in a NaOH solution at a temperature of 95 ± 5 °C. Li Sun et al[18] also prepared heterogeneous submicro-meter sized porous Ni film using an electrochemical de-alloying method, using Ni_xCu_{1-x} alloy film as a precursor.

To date, however, less attention has been paid to the preparation of nano-porous nickel via a process of molding, decomposition and sintering of nickel-organic precursors, while the method of liquid phase precipitation was widely used for preparing nickel-organic precursors [19-21].

In the present work, a simple novel approach was introduced to obtain nano-porous nickel at a low temperature within a relatively short time. Since this method was simple and controllable, it could be used for commercial applications. The effect factors during the process of preparation of porous nickel were analyzed. Subsequently, the phase and the morphology of porous nickel were investigated.

1 Experiment

The $NiCl_2 \cdot 6H_2O$, PVP (polyvinyl pyrrolidone), NH_4OH, and C_2H_6O were analytically pure regents (AR) which were purchased and used without further purification.

The nickel oxalate precursor was prepared by the method of precipitation and aging process [19]. 0.2 g nickel oxalate precursor round wafer was prepared by pressing nickel oxalate powder into the stainless mould, at a pressure of 3 MPa. The nickel oxalate wafer was 16 mm in diameter. Then, the dried nickel oxalate round wafers were placed into a tubular oven filled with Ar gas, and treated at the temperature of 360 °C for 10 min. Subsequently, the wafers were treated at the temperature of 420, 450 and 480 and 510 °C for 10 min. Finally, the nickel wafer samples were cooled to room temperature quickly.

The X-ray diffraction (XRD) patterns were recorded by an X'Pert Pro (Holland Philips) using Cu radiation (Kα, λ= 0.154 18 nm) operating at 40 kV and 40 mA with 2θ ranging from 20º to 80º and continuous scanning mode. Infrared (IR) was recorded on a Nicolet 5700 spectrometer in the range of 4000–400 cm^{-1} using KBr pellets method. The scanning electron microscope (SEM) images were taken with an Ultra 55 (German ZEISS) field-emission scanning electron microscope. The atomic force microscopy (AFM) images were taken with a SPA300HV (Japan SEIKO). The thermogravimetry analyzer and derivative thermogravimetry (TGA-DTG) curves were recorded by a SDT Q600 (America TA) thermal analyzer, at heating rate of 20 °C /min in Ar gas atmosphere.

2 Results and Discussion

2.1 Characteristics of nickel oxalate nano-rods

Fig.1 shows the IR pattern of as-prepared powder, which indicates that the band at 3400 cm^{-1} corresponds to the stretching vibrations of the hydroxyl groups of H_2O. The most intensive band at 1625 cm^{-1} is due to the anti-symmetric stretching vibration absorption of ν_{as} (C═O─C), indicating the presence of carbonyl groups. The bands of 1315 and 1360 cm^{-1}, in the area of 1300~1420 cm^{-1} are due to the symmetric stretching vibration absorption of ν_s (C═O─C). The band of 810 cm^{-1} is due to the stretching vibration absorption of ν_s (O─C). The band of 490 cm^{-1} is due to the stretching vibration absorption of ν (O─Ni) [20]. IR pattern further indicates that the powder is nickel oxalate hydrate which corresponds to the SDBS card No.17149.

Fig.2 shows the SEM image of as-prepared nickel oxalate nano-rods. The image shows clearly that the powder consists of a large quantity of uniform, well-separated and nano-sized rods with 100~200 nm in diameter, and 1.0~5.0 μm in length, and the surface is a little rough.

Fig.3 shows the TGA-DTG curves of as-prepared nickel oxalate nano-rods. DTG curve shows that there is a weak endothermic peak at about 68 °C which corresponds to the elimination of adsorptive water and ammonia molecules. In

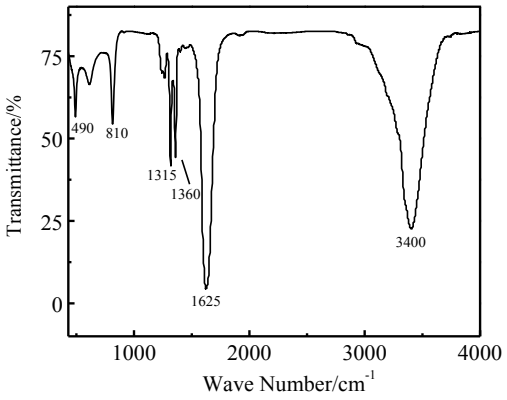

Fig.1 IR pattern of nickel oxalate nano-rods

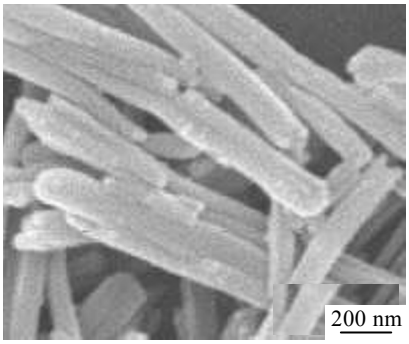

Fig.2 SEM image of nickel oxalate nano-rods