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ARTICLE

Synthesis of Nickel Powders by Hydrazine Hydrate Reduction of Nickel Hydroxide

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Abstract: Fine nickel powders with a narrow particle size distribution were prepared by reducing nickel hydroxide in aqueous solution. The formation and reduction pathways of nickel powder were analyzed, as well as the effects of the molar ratio of hydrazine hydrate to nickel hydroxide, hydrazine concentration, and the dosage of surfactant PEG6000 on particle size, surface morphology, and dispersion. Results reveal that the nickel particle nucleation occurs on the nickel hydroxide surface, and the nickel hydroxide gradually dissolves during the reaction. With the increase in molar ratio of hydrazine hydrate to nickel hydroxide, the nickel particle size is initially decreased and then increased. Higher hydrazine hydrate concentrations result in smaller particle sizes. A small amount of PEG6000 surfactant can improve dispersion of nickel particles, but a higher amount of PEG6000 surfactant results in the maintenance of the morphology of nickel hydroxide. Adjusting the surfactant dosage can control the average particle size between 1–2 μm.

Key words: nickel hydroxide; liquid-phase reduction; dispersibility; surfactant

1 Introduction

Extensive studies have been performed over the past two decades on the production of micro/nano-nickel powder due to its potential applications in absorbing materials, multilayer ceramic capacitors (MLCCs), catalysts, and electrode materials[1–7]. Several procedures have been used to produce nickel powder, including the vapor phase method[8–9], spray pyrolysis[10], micro-emulsion[11], polyol process[12–15], and liquid-phase reduction[16–22].

Among these methods, liquid-phase reduction has become popular for its ability to precisely manipulate the composition, size, and shape of nickel powder. For example, the reduction of nickel salts (such as $NiCl_2$ and $NiSO_4$) can be conducted through hydrazine hydrate in alkaline conditions[23–24]. Acidic solutions cause the standard reduction potential of hydrazinium ion $N_2H_5^+$ as −0.23 V. The reaction can be represented as follows:

$$N_2+5H^++4e^-\rightarrow N_2H_5^+ \quad (1)$$

Hydrazine (N_2H_4) exhibits a standard reduction potential of −1.16 V in alkaline solutions.

$$N_2H_4+4OH^-\rightarrow N_2+4H_2O+4e^- \quad (2)$$

Conversely, the conventional reduction potentials for Ni^{2+} and $Ni(OH)_2$ as sources of nickel are −0.25 and −0.72 V, respectively[25].

$$Ni^{2+}+2e^-\rightarrow Ni \quad (3)$$

$$Ni(OH)_2+2e^-\rightarrow Ni+2OH^- \quad (4)$$

This result indicates that reducing nickel hydroxide requires a higher overpotential and is more challenging than reducing nickel ions. Although extensive research has been conducted on the effect of hydrazine in alkaline environments on the size and shape of nickel powders obtained from nickel salts, the direct reduction of nickel hydroxide in aqueous solution using hydrazine is rarely reported. In this research, the nickel powder was prepared via the direct reduction of nickel hydroxide with hydrazine. The reduction pathways were discussed.

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2 Experiment

The materials used in this experiment were $Ni(OH)_2$, 80% $N_2H_4 \cdot H_2O$, and polyethylene glycol 6000 (PEG6000). All reagents used were of analytical quality and did not necessitate additional purification.

The molar ratio of hydrazine hydrate to nickel hydroxide is denoted as K, the hydrazine concentration is denoted as C, and the surfactant dosage is denoted as X. To study the reduction process of $Ni(OH)_2$ in hydrazine hydrate, a composition ratio of $K=5$ and $C=1.5$ was chosen. At 60 °C, 1.58 g $Ni(OH)_2$ was mixed with a certain amount of water and stirred. $N_2H_4 \cdot H_2O$ was then added for reaction. Samples were taken out of solution at different time of reaction to elucidate the transformation process of $Ni(OH)_2$. Based on reaction time, the powder color varied from green to dark green and finally to black. To investigate the effects of different K, C, and X values, the mixture was thoroughly stirred for 3–5 h to ensure complete reaction. The resulting powders were then washed three times with deionized water and anhydrous ethanol by centrifugation and filtration, followed by drying in a ventilated oven at 60 °C for 24 h.

The nickel powder was analyzed using X-ray diffractometer (XRD, Rigaku, D/Max−2000). The morphology and dispersity of the powder were analyzed using scanning electron microscope (SEM) coupled with energy disperse spectroscope (EDS). Particle size and size distribution were analyzed using a laser particle size analyzer (Malvern2000, measurement range: 0.01–1000 μm).

3 Results and Discussion

3.1 Formation of nickel particles and their reduction pathways

According to traditional methods, the reaction of hydrazine reducing nickel salts in an alkaline environment is as follows[26]:

$$2Ni^{2+}+N_2H_4+4OH^- \rightarrow 2Ni+N_2+4H_2O \tag{5}$$

In addition, two side reactions also occur[26]:

$$N_2H_4 \rightarrow N_2+2H_2 \tag{6}$$

$$3N_2H_4 \rightarrow N_2+4NH_3 \tag{7}$$

Nevertheless, the mechanisms of hydrazine reduction and nickel nucleation with nickel hydroxide serving as the source of nickel are still obscure. XRD patterns of samples obtained at reaction time of 30, 45, 60, 75, and 90 min are shown in Fig. 1. When the reaction time is 30 min, there is no evidence of nickel nucleation, indicating that there is an induction period in the reduction of nickel hydroxide by hydrazine. After reaction for 45 min, the diffraction peak of nickel becomes perceptible with a rather weak intensity. This phenomenon suggests that some nickel particles enter the induction period and nucleation begins. After reaction for 60 min, the diffraction peak intensity of nickel particles increases, whereas that of nickel hydroxide decreases. This result indicates the significant nickel nucleation. After reaction for 75 min, only nickel diffraction peaks can be observed, whereas those of nickel hydroxide cannot be observed. This result suggests that the nickel particles generated in the earlier stage are increased in size, and the nickel hydroxide is entirely reduced. After reaction for 90 min, a distinct nickel phase appears without impurities.

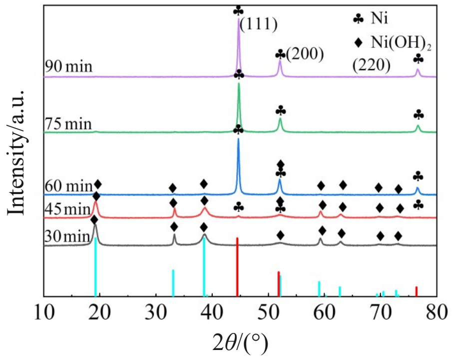

Fig.1 XRD patterns of samples at different reaction time

Fig. 2 depicts SEM microstructures of nickel powders produced through the direct reduction of nickel hydroxide using hydrazine hydrate. After reaction for 30 min, the nickel is still in the induction period and has not yet formed nuclei. Nickel hydroxide can be observed. With the prolongation of reaction time, nickel particles start to form on the surface of nickel hydroxide. During the reaction, additional nickel particles are produced. The majority of these particles stick to the surface of the nickel hydroxide, creating a core-shell structure composed of nickel and nickel hydroxide. However, some particles detach from the surface of the nickel hydroxide. With the reaction further proceeding, no trace of nickel hydroxide exists, suggesting that the nickel hydroxide is fully reduced by hydrazine.

Additionally, the nickel particles suffer agglomeration to a certain extent. The agglomerates maintain some features of the nickel hydroxide in terms of shape and particle size, as a result of the formation and enlargement of nickel particles on the surface of the nickel hydroxide, followed by the aggregation caused by van der Waals forces.

This phenomenon implies that the process of nickel hydroxide reduction by hydrazine involves the interaction between hydrazine and nickel hydroxide on the surface of nickel hydroxide. Nickel atoms form clusters on the surface of the nickel hydroxide instead of being dispersed in the solution. Throughout the reaction, the nickel hydroxide gradually dissolves and disappears, whereas the nickel nuclei undergo growth and agglomeration as a result of van der Waals forces and magnetic attraction. The reaction between hydrazine and nickel hydroxide is as follows:

$$2Ni(OH)_2+N_2H_4 \rightarrow 2Ni+N_2+4H_2O \tag{8}$$

To further investigate the product variations during the reduction process, the mixed powder after reaction for 60 min was analyzed, as shown in Fig. 3. Element Ni represents the reduced Ni, whereas the element O corresponds to nickel hydroxide. The results indicate distinct enrichment and

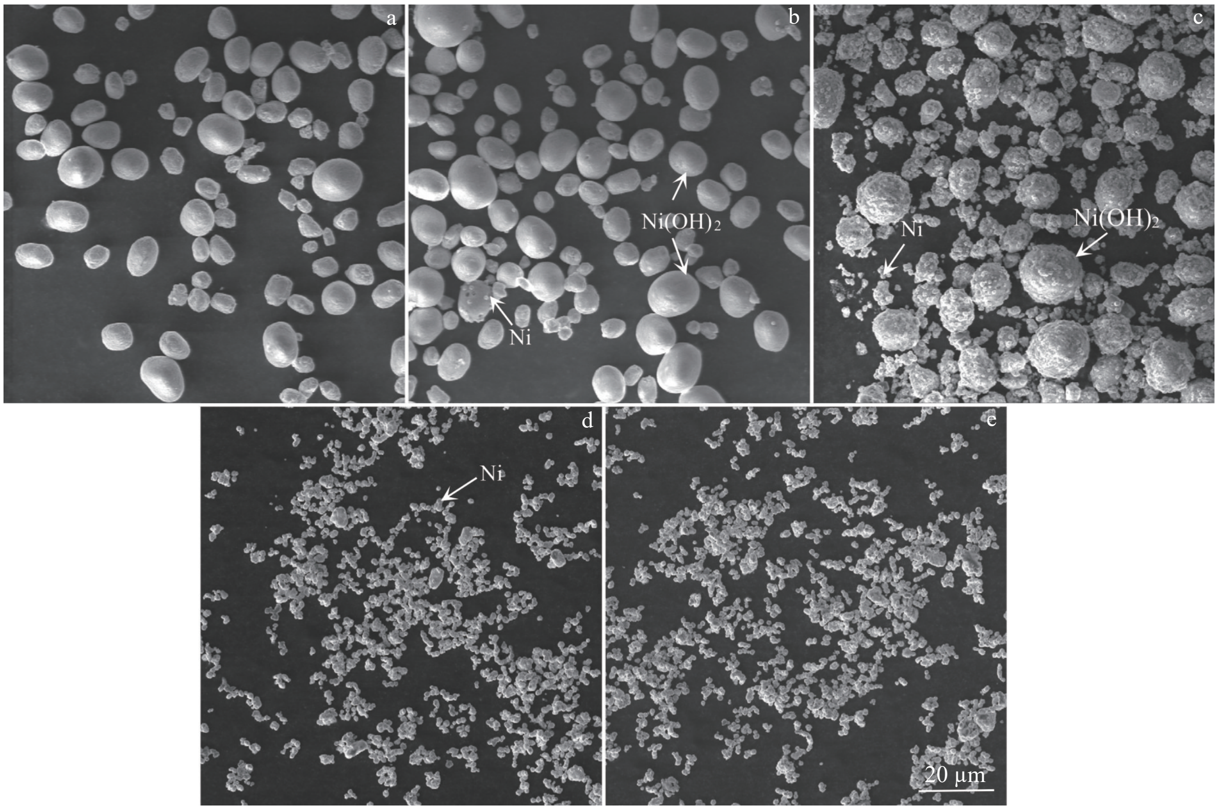

Fig.2 SEM microstructures of samples after reaction for 30 min (a), 45 min (b), 60 min (c), 75 min (d), and 90 min (e)

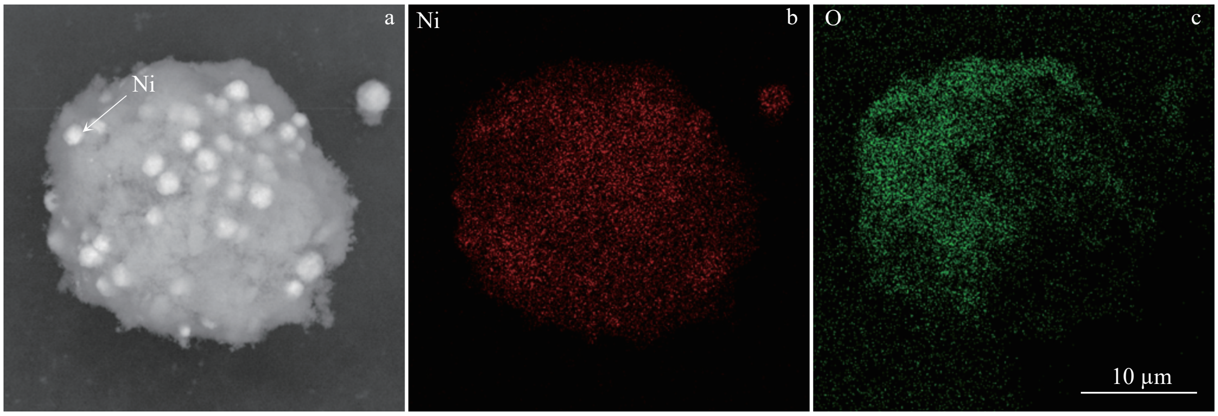

Fig.3 SEM image (a) and corresponding EDS element mappings (b–c) of sample after reaction for 60 min

depletion regions for elements Ni and O with Ni-enriched regions aligning with O-depleted areas. Combined with XRD and SEM analyses, it is confirmed that nickel nucleates on the surface of nickel hydroxide rather than in the solution.

3.2 Effect of molar ratio of hydrazine hydrate to nickel hydroxide

Eq.(3−4) demonstrate that hydrazine hydrate necessitates a lesser potential for the reduction of nickel hydroxide in comparison to nickel salts.

Fig. 4 displays XRD patterns of nickel powders prepared under different molar ratios of hydrazine hydrate to nickel hydroxide, namely K=3−7. The diffraction peaks labeled with ♣ correspond to the face-centered cubic (fcc) metallic nickel (Ni) phase. Eq. (8) indicates that the minimal molar ratio should be 0.5. However, the presence of side reactions Eq.(6–7), which are catalyzed by nickel and cause the decomposition of hydrazine hydrate, substantially raises the value of K. The peaks at 44.35° (111), 51.91° (200), and 76.42° (220) in Fig.4 all correspond to the standard diffraction data, which confirm the formation of pure nickel powder with K=3–7.

Fig. 5 depicts microstructures of nickel particles prepared under different molar ratios of hydrazine hydrate to nickel hydroxide. The Ni powder formed by direct reduction of nickel hydroxide with hydrazine hydrate has a primary particle size of 0.5–2 μm.

The primary particles agglomerate to generate secondary particles. With the increase in K value, the primary particle size

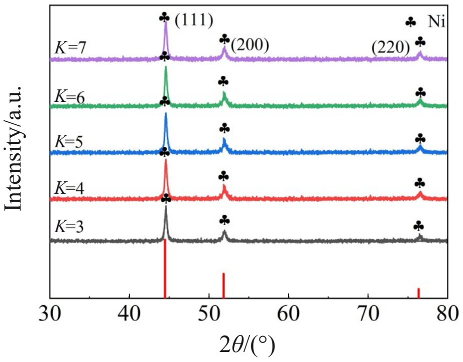

Fig.4 XRD patterns of samples prepared under different molar ratios of hydrazine hydrate to nickel hydroxide

of the resultant Ni powder is initially decreased and subsequently increased. When $K\leqslant 4$, both the nucleation rate and the number of nuclei of Ni are low, leading to a comparatively large primary particle size. Simultaneously, the slow reaction rate prevents the Ni particles from detaching from the nickel hydroxide surface and forming connections with other particles during particle development. When $K\geqslant 5$, the reaction proceeds rapidly, resulting in the formation of a significant number of nickel nuclei during the initial stage of the reaction. These nuclei detach from the surface of nickel hydroxide. After that, the size of the primary Ni particles reduces, and the process of agglomeration becomes less pronounced.

Fig. 6 depicts the trends in the average particle sizes D_{10}, D_{50}, and D_{90} of nickel powder prepared under different molar ratios of hydrazine hydrate to nickel hydroxide. The average size of the nickel particles is initially decreased and subsequently increased with the increase in molar ratio. The minimum values of D_{10}, D_{50}, and D_{90} are achieved when K=5.

3.3 Effect of hydrazine hydrate concentration

With a fixed reaction molar ratio of 5 (K=5), Fig. 7 illustrates XRD patterns of nickel powders prepared with hydrazine hydrate concentration of 1, 1.5, 2, 3, and 4 mol/L, namely C=1–4 mol/L. The diffraction peaks marked with ♣ correspond to fcc metallic Ni phase. According to Fig. 6, it is evident that each reaction yields single-phase nickel powders. This result indicates that varying the hydrazine hydrate concentration does not affect the endpoint of the reaction. When the hydrazine is sufficient, the final reaction product is independent of the relative concentration.

Fig. 8 shows microstructures of nickel particles prepared with different hydrazine hydrate concentrations. It can be seen that the primary particle size of nickel powder produced by direct reduction of nickel hydroxide with hydrazine ranges from approximately 0.5 μm to 5.0 μm. These primary particles tend to agglomerate into secondary particles. As the hydrazine hydrate concentration increases, the primary particle size decreases. This is because at lower concentrations, fewer nickel particles nucleate after the induction period, allowing subsequent nucleation and growth more readily on existing

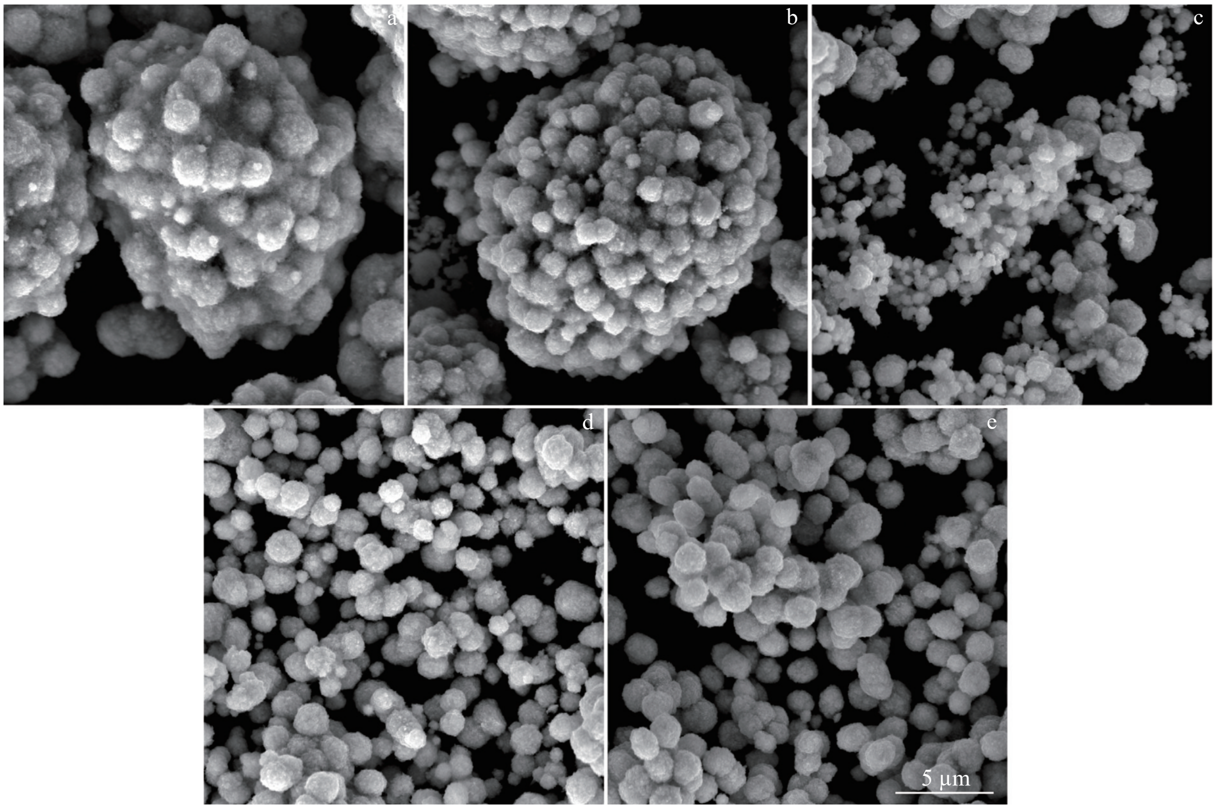

Fig.5 SEM micrographs of samples prepared under different molar ratios of hydrazine hydrate to nickel hydroxide: (a) K=3; (b) K=4; (c) K=5; (d) K=6; (e) K=7

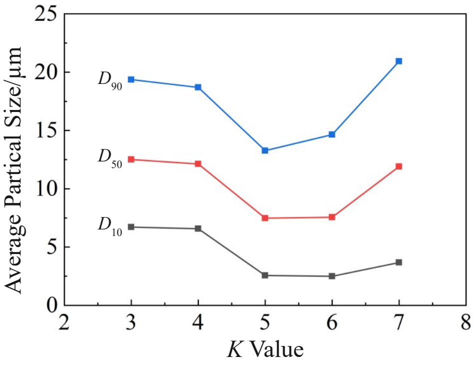

Fig.6 Trends in average particle sizes of nickel powder prepared under different molar ratios of hydrazine hydrate to nickel hydroxide

particles, and resulting in larger primary particles. When C=1.5 mol/L, the slower reaction rate allows nickel particles to grow and fuse on the nickel hydroxide surface, forming larger secondary agglomerates. When C=2 mol/L, the reaction rate is high, causing many nickel nuclei to form early and detach from the nickel hydroxide surface. These detached nickel particles cannot grow effectively, leading to smaller primary particle sizes and reduced agglomeration, and only van der Waals force and magnetic force contribute to the aggregation.

Fig.9 illustrates the trends in average particle size (D_{10}, D_{50}, and D_{90}) of nickel powders prepared with different hydrazine hydrate concentrations.

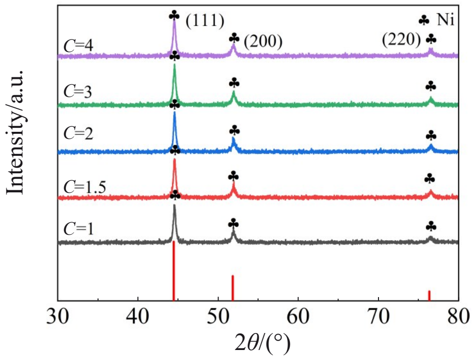

Fig.7 XRD patterns of samples prepared with different hydrazine hydrate concentrations

The average particle size is decreased initially and then increased with the increase in hydrazine hydrate concentration, reaching a minimum value at C=3 mol/L.

3.4 Effect of surfactant addition dosage

The amount of aggregate of nickel powder formed during the reaction can be modified by adjusting K and C values in the process. Huang et al[27] studied the influence of several surfactants on the reduction of nickel salts using hydrazine hydrate in alkaline conditions. It was revealed that the nonionic surfactant PEG6000 showed effective dispersion capability in the reaction system. The following analysis is based on the

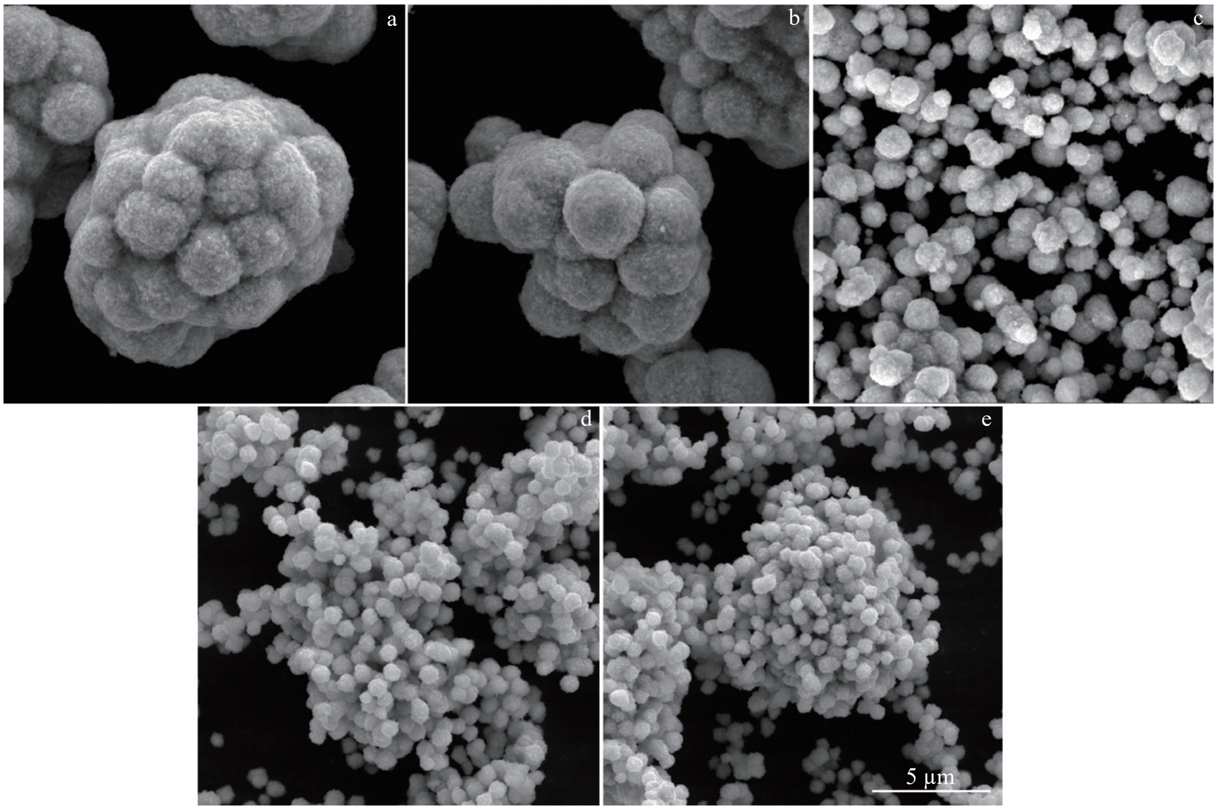

Fig.8 SEM microstructures of samples prepared with different hydrazine hydrate concentrations: (a) C=1 mol/L; (b) C=1.5 mol/L; (c) C=2 mol/L; (d) C=3 mol/L; (e) C=4 mol/L

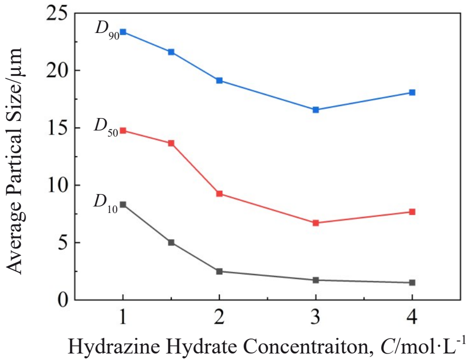

Fig.9 Trends in average particle sizes of nickel powder prepared with different hydrazine hydrate concentrations

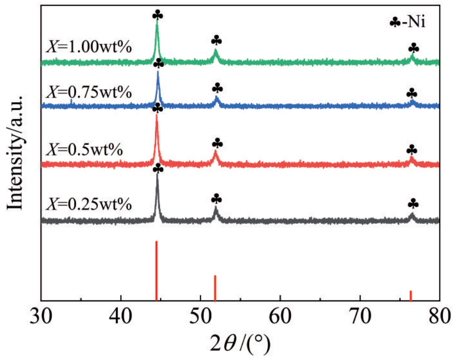

Fig.10 XRD patterns of samples prepared with surfactant of different dosages

conditions of K=5 and C=3 mol/L. Fig. 10 displays XRD patterns of nickel powders generated with different dosages of surfactant addition, namely X=0.25wt%, 0.50wt%, 0.75wt%, and 1.00wt%. Fig. 10 illustrates that the addition of surfactants has no impact on the phase composition of the reaction product. Fig. 11 illustrates SEM microstructures of nickel powders prepared with surfactant of different dosages. Fig. 12 illustrates the effect of dosage of PEG6000 surfactant on the product. When X⩽0.50wt%, a minimal quantity of surfactant is found in the solution system with some surfactant attaching to the surface of nickel hydroxide. During the initial stage of the reaction, a large number of nickel particles detach from the surface of nickel hydroxide due to fast reaction rates. These particles are then isolated by PEG6000 in the solution system, which effectively prevents them from aggregating due to van der Waals force and magnetic force. When X⩾0.75wt%, a significant quantity of PEG6000 is present in the solution system, forming connections with others. Before the reaction, every particle of nickel hydroxide is surrounded by PEG6000. Upon initiation of the reaction, nickel particles promptly emerge within the nickel hydroxide coating and are unable to disengage and enter the solution. Consequently, the aggregates maintain the size and shape properties of the original nickel hydroxide, leading to smaller individual particles and considerably bigger aggregate particles.

Fig. 13 depicts the variations of the average particle size (D_{10}, D_{50}, and D_{90}) of nickel powder with different dosages of surfactant during preparation. The average diameter of nickel

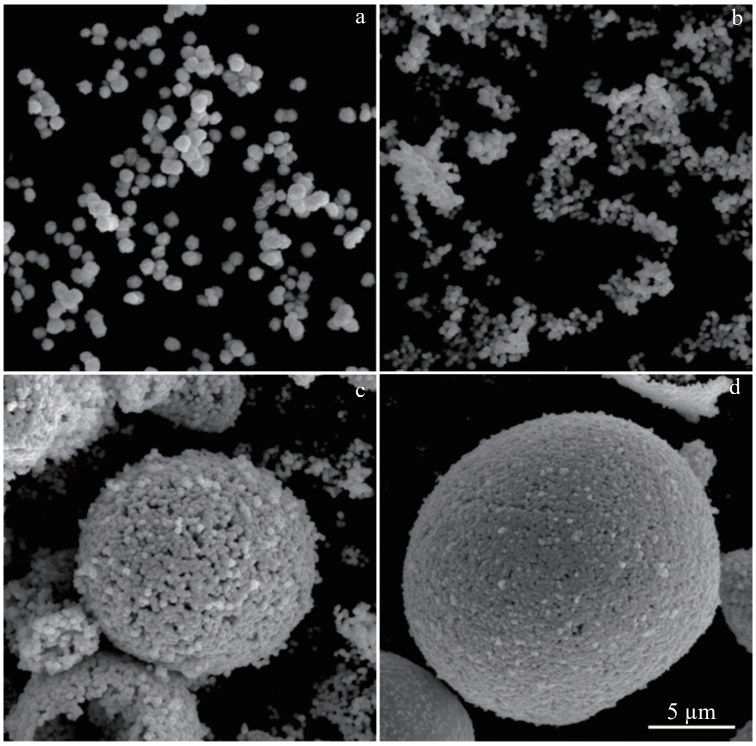

Fig.11 SEM microstructures of samples prepared with surfactant of different dosages: (a) X=0.25wt%; (b) X=0.50wt%; (c) X=0.75wt%; (d) X=1.00wt%

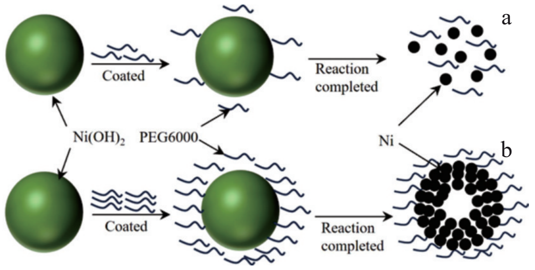

Fig.12 Schematic diagrams of effect of 0.25wt% (a) and 1.00wt% (b) PEG6000 surfactant on prepared nickel powder

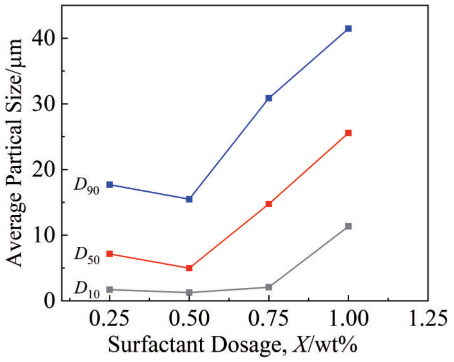

Fig.13 Trends in average particle sizes of nickel powder prepared with different surfactant dosages

powders is initially decreased and subsequently increased with the increase in surfactant dosage. When X=0.50wt% , the values of D_{10}, D_{50}, and D_{90} all reach their minimum.

Briefly, the optimum process parameters for the preparation of ultrafine nickel powder with high purity and spherical shape are obtained, as follows: the reaction temperature is 60 °C; the molar ratio of hydrazine hydrate to nickel hydroxide is 5; the concentration of hydrazine hydrate is 3 mol/L; the PEG6000 surfactant dosage is 0.50wt% . Besides, adjusting the surfactant dosage can control the average particle size between 1–2 μm.

4 Conclusions

1) Nickel is nucleated on the surface of nickel hydroxide by reduction with hydrazine hydrate.

2) The optimum process parameters for the preparation of ultrafine nickel powder with high purity and spherical shape are as follows: the reaction temperature is 60 °C; the molar ratio of hydrazine hydrate to nickel hydroxide is 5; the concentration of hydrazine hydrate is 3 mol/L; the PEG6000 surfactant dosage is 0.50wt%.

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水合肼还原氢氧化镍合成镍金属颗粒

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摘　要：用氢氧化镍在水溶液中被还原制备了窄粒度分布的微细镍粉，分析了镍颗粒的形成和还原途径，讨论了水合肼与氢氧化镍摩尔比、水合肼浓度和表面活性剂PEG6000的添加量对镍颗粒的粒径大小、表面形貌和分散性的影响。结果表明：镍颗粒在氢氧化镍表面完成形核，反应过程中氢氧化镍逐渐溶解。随着水合肼与氢氧化镍反应摩尔比的提高，镍颗粒的粒径先变小后变大；随着水合肼浓度的提高，镍颗粒的粒径变小；少量添加表面活性剂PEG6000使镍颗粒的分散性得到改善，更高的表面活性剂添加量使镍一次颗粒趋于保留氢氧化镍的形貌。通过调节表面活性剂添加量可以使平均粒径控制在1~2 μm。

关键词：氢氧化镍；液相还原；分散性；表面活性剂

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