

УДК 537.632.3

Synthesis, Morphology, and Visible Magnetic Circular dichroism of Ni-C nanoparticles

Dmitriy A. Petrov*

Ruslan D. Ivantsov†

Kirensky Institute of Physics SB RAS
Akademgorodok, 50/38, Krasnoyarsk, 660036

Russia

Sergey M. Zharkov‡

Kirensky Institute of Physics SB RAS
Akademgorodok, 50/38, Krasnoyarsk, 660036

Siberian Federal University
Svobodny, 79, Krasnoyarsk, 660041

Russia

Chun-Rong Lin§

Fu-Ren Shu

Yaw-Teng Tseng

National Pingtung University
Pingtung City, Pingtung County, 90003

Taiwan

Received 10.09.2016, received in revised form 10.10.2016, accepted 12.11.2016

Ni-C nanoparticles powder (NP) was synthesized using high-temperature pyrolysis of nickel nitrate in an organic solvent. The NP was characterized by transmission electron microscopy (TEM), energy dispersive x-ray spectroscopy (EDXS) and selected-area electron diffraction (SAED). Structural characterization of the samples confirmed the formation of small, medium and large Ni nanoparticles with face-centered cubic structure (fcc) and enlarged lattice parameter. Magnetic circular dichroism (MCD) of samples with different mean particle sizes showed a red shift with decrease of mean particle size.

Keywords: Ni-C, nanoparticles, MCD, TEM.

DOI: 10.17516/1997-1397-2016-9-4-481-484.

1. Preparation

Ni-C NP was prepared using high-temperature pyrolysis of nickel nitrate in an organic solvent. In the typical process, 4 mmol nickel (II) nitrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ was dissolved in a mixture solution of 10-ml oleylamine and 20-ml oleic acid in a three-neck flask equipped with an inlet of argon gas, condenser, magnetic stirrer, thermocouple, and heating mantle. All chemicals and

*irbiz@iph.krasn.ru

†ird@iph.krasn.ru

‡zharkov@iph.krasn.ru

§crlin@mail.nptu.edu.tw

solvents are used as received. After the argon gas was introduced into the system for 10 min, the mixture was heated to 240°C and maintained at this temperature for 1 hr. It took ~ 50 min to heat the reaction system to boiling at 360°C. After this temperature remained for 1 hr, it started to increase steadily. After reaching $\sim 380^\circ\text{C}$, the reaction solution burst out to form dense gas clouds and black products, accompanying a fast increase in temperature up to 395°C. The reaction temperature then fell down instantly and dwelled at 140°C for 1 hr before removing heating mantle for cooling. With the addition of hexane, the black precipitates were magnetically separated for several times and stored in hexane for further studies.

2. Samples Morphology

Microstructure and phase composition of the NP were investigated by TEM and EDXS using a transmission electron microscope 200 kV JEOL JEM-2100 (LaB₆).

Using the TEM, three typical sizes of Ni-C nanoparticles were revealed (Figs. 1, 2): small (~ 5 nm), medium (20-30 nm) and large (50–200 nm) particles. The elemental composition was determined by EDXS supports the presence of nickel. The set of diffraction reflections (Fig. 3) in the SAED pattern corresponds to the Ni fcc structure 17% enlarged cell parameter. A similar large increase of lattice parameter was observed in Ref. [1].

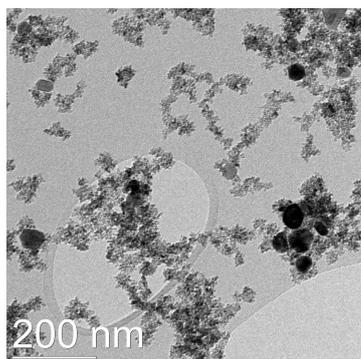


Fig. 1. The TEM of Ni-C NP

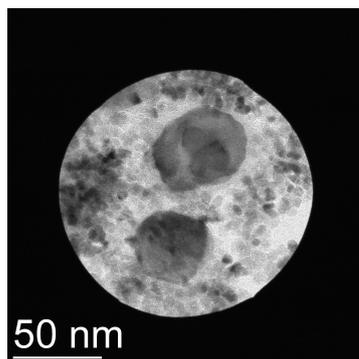


Fig. 2. The TEM of Ni-C NP with higher magnification

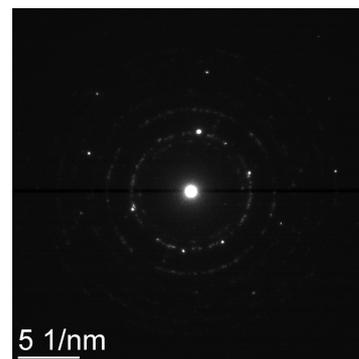


Fig. 3. The set of diffraction reflections

3. Magnetic Circular Dichroism

To investigate the MCD, Ni-C NP were dried and four samples were prepared. The first sample was prepared by mixing of NP with silicone matrix (all particles). Further, NP was placed in an alcohol for the flotation. Then the test-tube was agitated. After 5 min (small and medium) and 20 min precipitation (small) the top layer was taken from the tube and then was dried on the glass. It is worth noting that the terms "small", "medium" and "large" are conventional and do not preclude a finding of large particles in the "small" samples. The procedure was repeated many times. Thereafter particles also were placed in the silicon matrix. The fourth sample was prepared like the last sample but was not mixed with silicon.

MCD was measured in the normal geometry: the magnetic vector and the light beam were

directed normal to the sample plane. The MCD value was measured in the spectral range 1.2–3.5 eV in the magnetic field 3 kOe at room temperature.

The Fig. 4 shows the normalized MCD spectra for Ni-C nanoparticles and Ni thin film (~ 20 nm). MCD spectra of Ni-C nanoparticles change a sign in the range 1.6–2.25 eV and have the minimum at energy lower 1.5 eV. It is seen that decrease in the mean particle size leads to a red shift of the MCD spectra. The high density of nanoparticles dried on the glass led the spectrum of the nanoparticles to be closer to the spectrum of Ni thin film than to the spectrum of small particles in the silicon matrix.

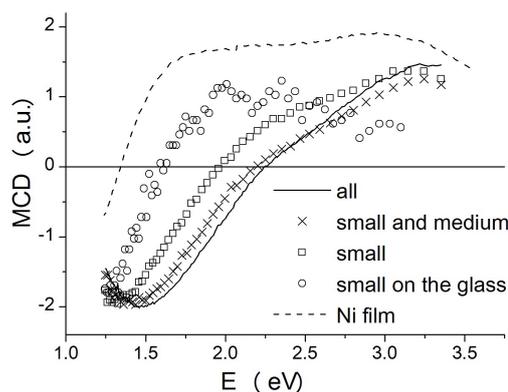


Fig. 4. The MCD spectra of Ni-C nanoparticles and Ni thin film

The MCD spectrum for small Ni-C nanoparticles in the silicon matrix is in a fairly agreement with data on the off-diagonal component of the conductivity tensor for bulk nickel derived from the first-principles calculation based on density-functional theory, Ref. [2]. In this Reference, the Ni band structure was calculated taking into account the contribution of matrix elements originated from p-p and d-d transitions in spin-orbit interaction.

Conclusion

The Ni-C nanoparticles powder prepared by high-temperature pyrolysis of nickel nitrate in an organic solvent was investigated. The powder consists of small (5 nm), medium (20–30 nm) and large (50–200 nm) nickel particles with fcc structure and the 17 % enlarged lattice constant. Nanoparticles, presumably coated with carbon. MCD spectra of the nanoparticles differ from the spectrum of thin nickel film but similar to that they demonstrate the red shift with decreasing the average particle size. Increasing of the concentration of the nanoparticles plays a more significant role in the approximation of the nanoparticles MCD spectrum to the MCD spectrum of the film than the particle size.

This work was supported by the RFBR grant 14-02-01211 and MOST 102-2112-M-153-002-MY3.

References

- [1] A.S.Bolokang, M.J.Phasha, Solid-state transformation in ball milled nickel powder, *Materials Letters*, **64**(2010), 1894–1897.

- [2] N.Mainkar, D.A.Browne, J.Callaway, First-principles LCGO calculation of the magneto-optical properties of nickel and iron, *Physical Review B*, **53**(1996), no. 7, 3692–3701.

Синтез, морфология и видимый МКД наночастиц Ni-C

Дмитрий А. Петров

Руслан Д. Иванцов

Сергей М. Жарков

Институт физики им. Л. В. Киренского СО РАН

Академгородок, 50/38, Красноярск, 660036

Россия

Чун-Ронг Лин

Фу-Рен Шу

Йау-Тенг Цзенг

Порошок наночастиц Ni-C был синтезирован с помощью высокотемпературного пиролиза нитрата никеля в органическом растворителе. Наночастицы были исследованы с помощью просвечивающей электронной микроскопии, энергодисперсионной рентгеновской спектроскопии и дифракции электронов выбранной зоны. Структурная характеристика образцов подтвердила образование маленьких, средних и больших наночастиц никеля с гранецентрированной кубической структурой и увеличенным параметром решётки. Магнитный круговой дихроизм образцов с различными средними размерами частиц показал красное смещение при уменьшении среднего размера частиц.

Ключевые слова: Ni-C, наночастицы, МКД, электронная микроскопия.