Anal. Bioanal. Electrochem., Vol. 5, No. 2, 2013, 265 - 269

Analytical & Bioanalytical Electrochemistry

Full Paper

Electrochemical Method for Synthesis of Nickel Titanate Nano Particles

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Received: 18 January 2013 / Received in revised form: 14 April 2013 / Accepted: 15 April 2013 / Published online: 30 April 2013

Abstract- Nano sized nickel titanate particles were synthesized by using electrochemical methods. The nickel titanate particles produced are characterized by XRD, TG, UV-Vis absorption and FTIR analysis. Electrochemical method is found suitable for producing nickel titanate particles. The particle size was calculated based on the Scherer equation and the average particle size was found to be 50 nm.

Keywords- Nickel titanate, Electrochemical Method, Band Gap, Photocatalyst

1. INTRODUCTION

Titanate particles have wide application in the field of solid oxide fuel cell [1,2], metal air batteries [3] and sensors [4]. The major application of titanate lies in the field of photocatalysts [5-7]. The use of titanium dioxide in the field of photocatalyst is limited, because it is activated only under UV light irradiation which accounts for only a small fraction (~5%) of the solar irradiance compared to the visible region (~45%) [8]. The band gap of nickel titanate is lower than that of titanium dioxide; it is activated under visible light irradiation [6]. Hence, the development of visible light driven titania related photocatalyst would therefore

be a major advance, not only needs expensive light source but also in efficient in utilizing the sunlight.

Nickel titanate has been investigated as a tribological coating to reduce friction and wear in high temperature applications, without using liquid lubricant [9-10]. There are several different methods, such as solid-state synthesis [11] wet-chemistry synthesis [2] sol-gel method [10, 12] etc has been used for preparation of nickel titanate nano particles. The common solid-state method for the preparation of nickel titanate can produce large $NiTiO_3$ particles with poor compositional homogeneity and uncontrolled structural morphologies. The electrochemical technique produces particles having uniform particle size, high purity and compositional uniformity.

2. EXPERIMENTAL METHOD

500 mL of 0.2 M $TiCl_4$ (solution A) was prepared and the solution is taken in an electrochemical cell having a volume capacity of 1000 mL. A mixed solution of 400 Ml (solution B) is prepared by admixing 20 mL of 30% H_2O_2 solution, 15 ml of 28% aqueous ammonia solution and rest of distilled water. The mixed solution was taken in a burette. A pure nickel electrode (surface area 2 cm^2) immersed in the electrochemical cell act as the anode. Stainless steel (SS-316 L) (surface area 10 cm^2) acts as the cathode. A saturated calomel electrode (SCE) served as the reference electrode for measuring the potential of working electrode. The potential between the working electrodes is suitably set by adjusting the current flowing across the anode and cathode by using rheostats. The multimeters are used to measure the current and voltage in the system. Dry cell batteries are the DC power source in the electrochemical cell. A potential of 0.6 V *vs*. SCE is maintained in the anode material kept in the electrochemical cell containing 0.2 M $TiCl_4$ (solution A). To this electrochemical cell mixed solution (solution B) is added drop wise drop till 400 Ml solution is consumed in the cell. The precipitate deposited in the cathode is cleaned with water and dried. The deposited material is scratched from the metal surface and calcined at 850 ^oC to obtain nano crystalline $NiTiO_3$.

X-ray diffraction was performed on $NiTiO_3$ powders using CuKα radiation (Phillips Analytical). Average crystalline size was calculated from the peak width using Scherer formula. A thermal study was performed for the uncalcined precipitated sample by using TGA Versa thermo gravimetric analyzer. FTIR spectra of $NiTiO_3$ have been recorded in Bruker FTIR instrument. The light absorbance of the films was also measured by UV-Vis spectrophotometer of Shimadzu

3. RESULTS AND DISCUSSION

3.1. X-ray analysis

Table1 shows the XRD results of $NiTiO_3$ powders calcined at 850 ^oC. All the peak values in table are well matched with database in JCPDS for the nickel titanate. The particle size calculated using the Scherer formula for different peaks varies from 40 nm to 60 nm.

Table 1. XRD values of $NiTiO_3$ Synthesized

Peak (2θ value)	31.5	34.1	37.2	42.1	49.4
Intensity, (Counts/Second)	300	1050	1950	1200	1720
Patrice size, Scherer equation (nm)	58.5	57.6	50.2	40.6	48.2

3.2. Thermal analysis

Thermo gravimetric analysis was performed on un-calcined sample and the results are shown in Fig. 1. The first step of weight loss (up to 180 ^oC) was due to the loss of water. The second step of weight loss is due to the elimination of hydrogen peroxide present in the complex, resulting the formation of titanate.

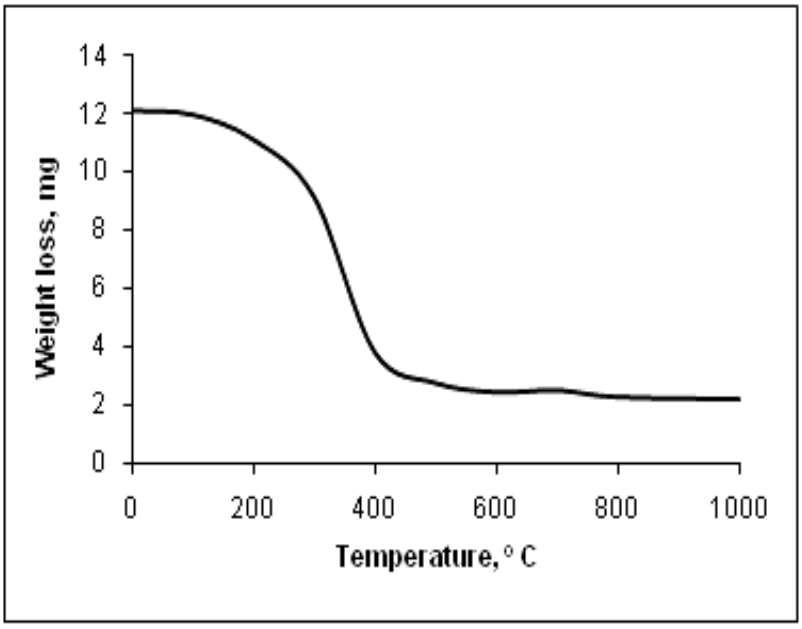

Fig. 1. TG curves of un-calcined $NiTiO_3$

3.3. Infrared spectral analysis

FTIR spectra recorded for $NiTiO_3$ powders calcined at 850 ^oC is shown in Fig. 2.

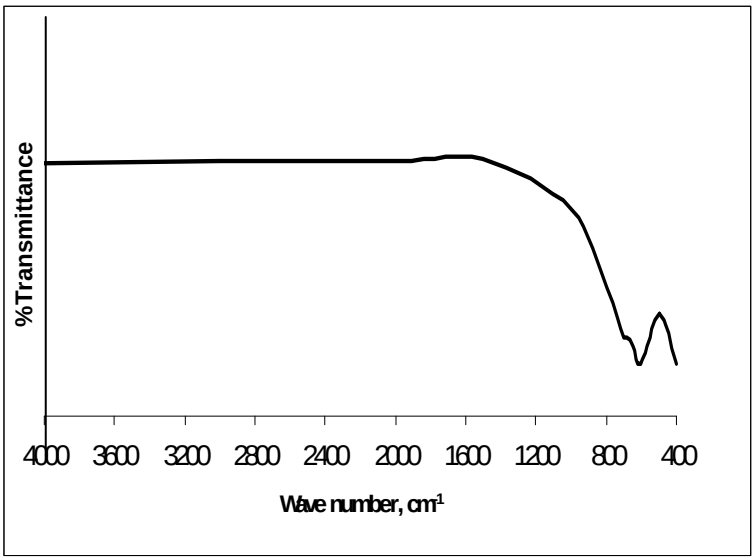

Fig. 2. FTIR spectra of the $NiTiO_3$ powder calcined at 850 ^oC

IR peaks below 800 cm^{-1} were assigned to the M-O (M–Titanium or Nickel) stretching and bending mode of O-M-O bond vibration corresponding to the formation of nickel titanate particles.

3.4. UV-Vis absorption Studies

Fig. 3 shows the UV-Vis absorption spectrum of the nickel titanate powder.

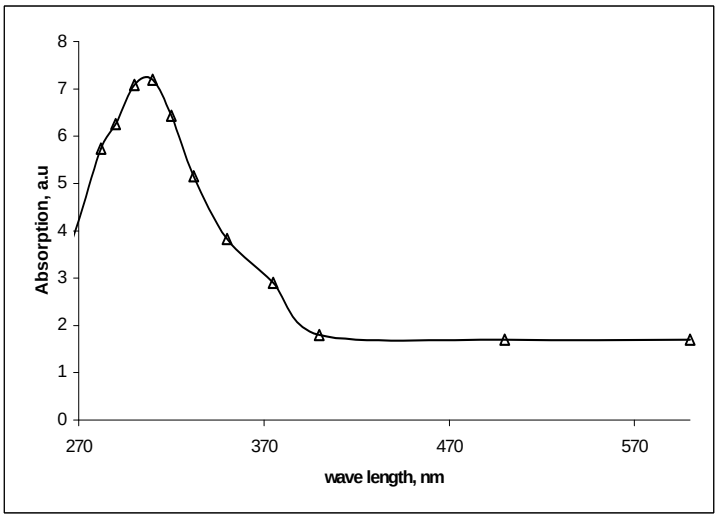

Fig. 3. UV-Vis spectrum of nickel titanate

Nickel titanate exhibit only one characteristic absorption band at 310 nm, which is assigned to the intrinsic transition from the valence band (VB) to the conduction band (CB).

4. CONCLUSION

A new electrochemical method was developed for the synthesis of nickel titanate particles. Thermo-gravimetric studies showed that nickel titanate formation was completed at 600 oC. The IR peaks below 800 cm^{-1} were assigned to the Ti-O stretching and bending mode of O-Ti-O bond vibration corresponding to the formation of nickel titanate particles. The absorption band at 310 nm, in UV-Vis spectrum corresponds to the characteristic absorption band of nickel titanate particles. The average particle size of nickel titanate particles produced is of 50 nm.

REFERENCES

[1] N. Dharmaraj, H. C. Park, C. K. Kim, H. Y. Kim, and D. R. Lee, Mater. Chem. and Phys. 87 (2004) 5.
[2] M. S. Sadjadi, K. Zare, S. Khanahmadzadeh, and M. Enhessari, Mater. Lett. 62 (2008) 3679.
[3] Y. Shimizu, K. Uemura, N. Miura, and N. Yamzoe, Chem. Lett. 67 (1988) 1979 .
[4] X. Shu, J. He and Dong Chen, Industrial Engineering Chemistry Research 47 (2008) 4750.
[5] H. Obayashi, Y. Sakurai, and T. Gejo, J. Solid State Chem. 17 (1976) 299.
[6] K. S. Beenakumari, J. Exp. Nanosci. 8 (2013) 203.
[7] K. S. Beenakumari, Mater. Sci. Res. India 6 (2009) 235.
[8] K. Prabakara, T. Takahashia, T. Nezukaa, K. Takahashia, T. Nakashimab, Y. Kubotac and A. Fujishima, Renew. Energ. 33 (2008) 277.
[9] R. S. Singh, T. H. Ansari, R. A. Sing and B. M. Wankyln, Mater. Chem. Phys. 40 (1995) 173.
[10] D. J. Taylor, P. F. Fleig and R. A. Page, Thin Solid Films 408 (2002) 104.
[11] Y. J. Lin, Y. H. Chang, W. D. Yang and B. S. Tsai, J. Non-Cryst. Solids 352 (2006) 789.
[12] A. R. Phani and S. Santucci, Thin Solid Films 396 (2001) 1.