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Surface characterization of $NiTiO_3$ nano powders prepared by coprecipitation method

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ABSTRACT

A process for the preparation of nickel titanate, which comprises adding an aqueous solution containing ions of titanium and nickel, to an aqueous alkaline solution containing hydrogen peroxide. The resulting precipitate is recovered and heated at a temperature of 850 °C to give nickel titanate. The samples were then analyzed using a series of techniques including X- ray diffraction (XRD), Transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR) and Thermo gravimetry (TG). The results indicated that nickel titanate nanopowders with particle size less than 100 nm could be obtained after calcination of precipitate at 850 °C.

Key words: Nickel titanate, X-ray diffraction, $NiTiO_3$ nano powders

INTRODUCTION

Titanate nano particle have caught the attention of researchers due to wide application as capacitors, high performance catalyst electrode for solid oxide fuel cell[1-3], metal air batteries[4] and gas sensors[5]. Nickel titanate has been investigated as a tribological coating to reduce friction and wear in high temperature applications, without using liquid lubricant[6-10]. Nickel titanate has an anti-ferromagnetic structure with M^{2+} spin parallel with in each layer perpendicular to rhombohedral axis, and anti-parallel between adjacent layers[6]. Nickel titanate coating was designed to be economical and applicable to many surfaces due to high durability and high resistant to oxidizing atmosphere[7,11]. 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. However co-precipitation technique have many advantage over other technique, such as homogeneity in the phase of reactant, stoichiometric control, high purity and ease of processing[2]. Thus it is a meaningful work to investigate some route to prepare nano size $NiTiO_3$ with a controlled crystalline morphology, high purity and uniform particle size.

MATERIAL AND METHODS

For preparing $NiTiO_3$, nickel chloride and titanium tetrachloride were used as the starting material. $TiCl_4$ (0.2mol) and $NiCl_2$ (0.2mol) are respectively dissolved in 1000 ml of distilled water. An aqueous solution containing ions of titanium and nickel in an equimolar ratio is prepared by admixing 100 ml $TiCl_4$ solution and 100 ml of $NiCl_2$ solution. A mixed solution is prepared by admixing 20 ml of 30% H_2O_2 solution, 15 ml of 28% aqueous ammonia solution and rest of distilled water. To the mixed solution is added drop wise to the aqueous solution containing ions of titanium and nickel in an equimolar

ratio with stirring to precipitate a complex peroxide corresponding to nickel titanate, the precipitate is washed dried and calcined at 850 °C to obtain nano crystalline $NiTiO_3$.

X-ray diffraction was performed on $NiTiO_3$ powders using CuKá radiation (Phillips Analytical). Average crystalline size was calculated from the peak width using Scherer formula.

The nano nickel titanate particles had been characterized by using transmission electron microscope of model H-600 Hitachi, at a magnification of 80,000. The nano nickel titanate particles were found needle shaped crystals having particle size less than 100 nm.

FTIR spectra of $NiTiO_3$ have been recorded in Bruker FTIR instrument. Thermal studies were conducted using TGA Versa thermo gravimetric analyzer.

RESULTS AND DISCUSSIONS

Thermal analysis

Thermo gravimetric analysis was performed for the dried precipitated sample and the results are shown in figure 1.The first step of weight loss was due to the evaporation of water. The complex peroxide corresponding to nickel titanate can be decomposed by heating at a temperature not lower than 100°C. The second step of weight loss is due to the elimination of hydrogen peroxide present in the complex, resulting the formation of amorphous titanate, being free from impurities and having uniform particle size. On further heating above 600°C will convert amorphous phase to crystalline from.

X-ray analysis

Figure 2 shows the XRD pattern of $NiTiO_3$ powders calcined at 850 °C. The XRD spectra shows sharp intense peak corresponding to well crystalline rhombohedral $NiTiO_3$ phase. All the peak corresponding to rhombohedral phase were well matched with database in JCPDS. The particle size calculated using the Scherer formula showed particle size of 50 nm of $NiTiO_3$ synthesized.

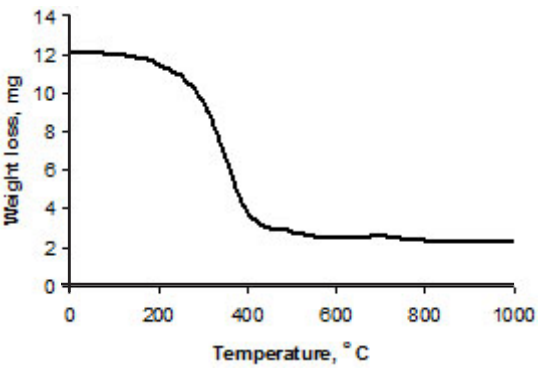

Fig. 1: TG curves of uncalcined $NiTiO_3$

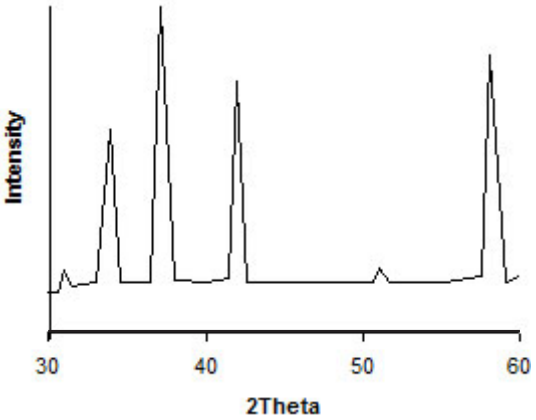

Fig. 2: X-ray diffraction patterns of $NiTiO_3$ calcined at 850 °C

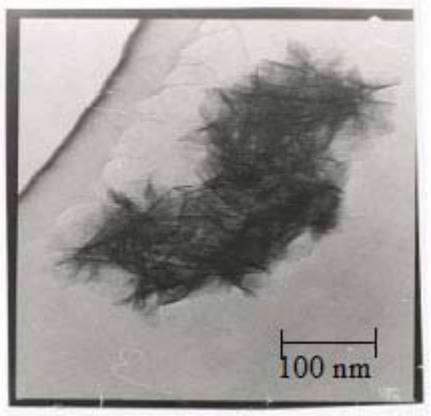

Fig. 3: TEM image of $NiTiO_3$ powder calcined at 850°C

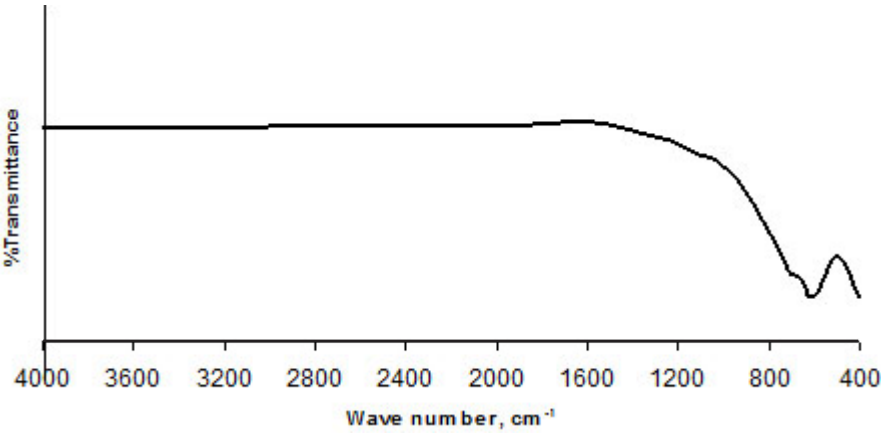

Fig. 4: FTIR spectra of the $NiTiO_3$ powder calcined at 850°C

Morphological analysis

Figure 3 shows the TEM image of $NiTiO_3$ powders calcined at 850 °C. The nano nickel titanate particles were found needle shaped crystals having particle size less than 100 nm.

Infrared spectral analysis

FTIR spectra recorded for $NiTiO_3$ powders calcined at 850 °C is shown in figure 4. 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.

CONCLUSION

The study has demonstrated the feasibility of synthesis of pure $NiTiO_3$ nano powder using a simple co-precipitation process. The $NiTiO_3$ crystalline formation is completed at 850 °C.

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