



Annealing effect on nickel oxide nanoparticles synthesized by sol-gel method

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ABSTRACT

NiO nanoparticles were synthesized by Sol-Gel method and the synthesized nanoparticles were annealed at different temperatures. The powders were investigated with x-ray diffraction, scanning electron microscopy and TGA. The structural characterization was carried out by x-ray diffraction which confirms the polycrystalline nature of the films with a cubic structure. SEM analysis of the films enabled the conclusion that the prepared films are uniform, rough, large crystals and agglomeration of particles were observed. The thermal stability of the sample was examined by TGA analysis which showed NiO nanoparticles formed between 130-720 °C.

Keywords: Nickel Oxide nanoparticles; XRD; SEM; TGA-DTA

1. INTRODUCTION

Metal oxide Nanomaterials exhibit significantly chemical, mechanical, electronic, thermal, magnetic, catalytic properties, and optical properties in comparison with their bulk counterparts, and have extensively attracted applications [1,2]. In past decade, there has been an increasing interest in the synthesis of nanosized crystalline metal oxides because of their large surface areas to volume ratio, high chemical reaction rate, unusual adsorptive properties, surface defects, and fast diffusivities [3]. Nickel is a Block D, Period 4 element, while oxygen is a Block P, Period 2 element.

Nickel oxide is a kind of transition metal oxide and has received a great deal attraction of due to its applications in various fields such as electrochromic films [4-6], fuel cell electrodes [7] and gas sensors [8-10], battery cathodes [11-15], pn heterojunctions [16], magnetic materials [17-20], photovoltaic devices [21], electrochemical supercapacitors [22], smart windows [23] and dye-sensitized photocathodes [24].

Most of these applications require particles with a small size and a narrow size distribution. With the volume effect, the quantum size effect and the surface effect, NiO Nanoparticles are expected to possess many improved properties and even more attractive applications than those of bulk-sized NiO particles. For example, NiO nanostructures are p-type semiconductors with particular magnetic behaviors such as superparamagnetic, superantiferromagnetic, and ferromagnetic order depending on the particle size, particle shape, and synthesis route, whereas bulk-sized NiO is an antiferromagnetic insulator with a Neel temperature (TN) of 523 K. Preparation of nickel involves the solution phase chemistry route, which in theory should provide multiple, simple ways to control the morphology, particle size and desirable crystalline phase. It is very important to develop methods for the synthesis of nickel oxide nanoparticles in which the particle size and the crystal structure of the products can be controlled. The Nickel Oxide nanoparticles have been prepared by sol gel method and the prepared Nickel Oxide nano particles were annealed at two different temperatures 500 °C and 700 °C. The effect of annealing on synthesized Nickel Oxide nanoparticles has been analyzed through XRD, SEM, TGA-DTA and presented in detail.

2. EXPERIMENTAL METHOD

Sol gel preparation is one of the simplest, environmental safe and lowest-cost techniques for preparing pure transition metal oxides with relatively high specific surface area at low temperature. This method also has potential advantages, including operational simplicity, and high yield of product, low energy consumption and no special equipment required. The process of creating materials by Sol-Gel process consists of four main stages:

- Creating solution (sol)
- Gelled and shaped
- Drying
- Conglomeration

The schematic representation of Sol-Gel process is represented in Fig. 1.

Step: 1 0.1M of nickel acetate is dissolved in 25ml of ethanol and isopropyl alcohol.

Step: 2 The precursor solution is allowed to stir until clear solution is formed.

Step: 3 Then the Sodium Hydroxide was added drop by drop to reach the pH value of 9.

Step: 4 The sol is formed and the temperature was set above 70 °C in the magnetic Stirrer.

Step: 5 After 2 hours the green color gel was formed and it is washed with ethanol for 3-5 times to remove by products and unreacted compounds.

Step: 6 The Gel is then placed in hot air-oven at 100 °C for 24 hours.

Step: 7 The dried powder is then annealed in muffle furnace for different annealing temperatures (500 °C & 700 °C).

Step: 8 After annealing process, the green coloured NiO nanoparticles changes into Black color.

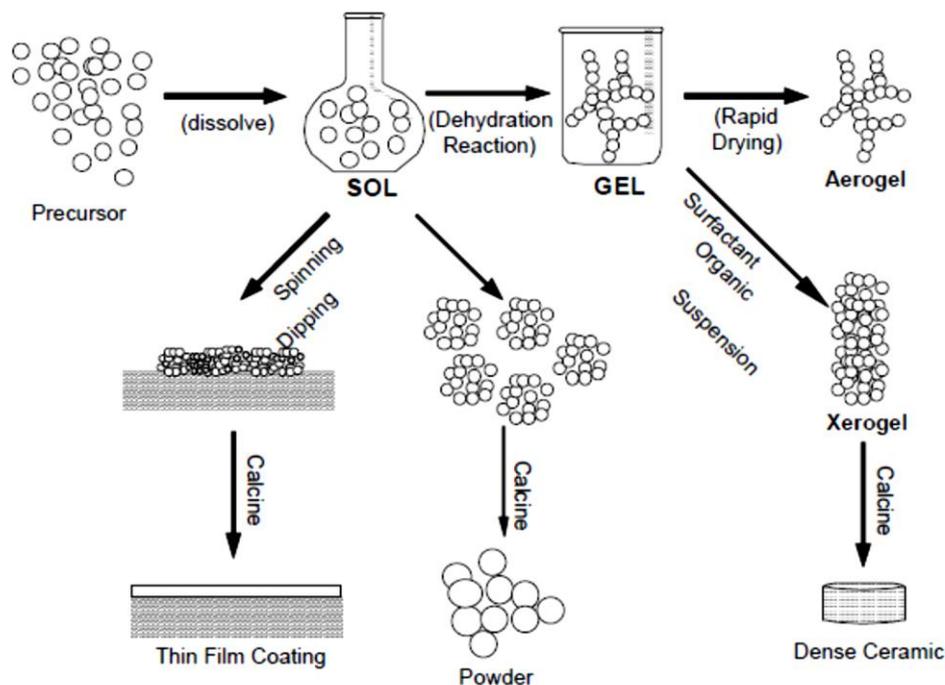


Figure 1. Schematic representation of sol-gel process for the synthesis of nanomaterials Preparation of NiO nanoparticles by Sol-Gel method.



Figure 2(a). Before annealing NiO Nanoparticles



Figure 2(b). After annealing NiO Nanoparticles

3. RESULT AND DISCUSSION

3. 1. Structural Analysis

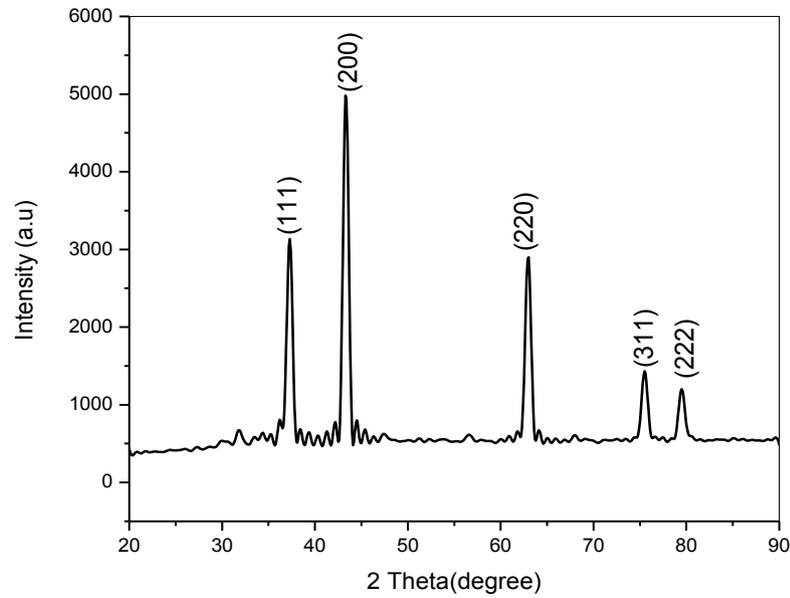


Figure 3. XRD Pattern of NiO nanoparticles annealed at 500 °C

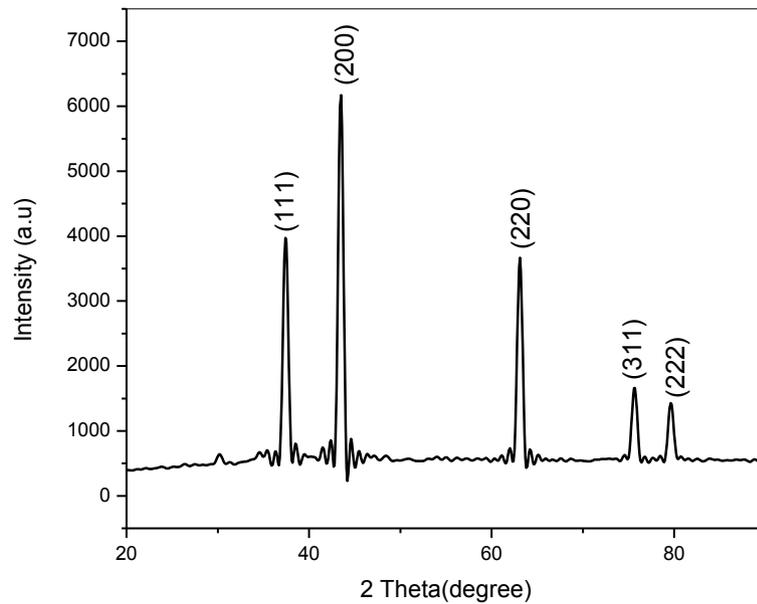


Figure 4. XRD Pattern of NiO nanoparticles annealed at 700 °C

X-Ray Diffraction pattern of Nickel Oxide nanoparticles was obtained with $\lambda = 1.54\text{\AA}$ and with theta ranging from 20-90 degree is shown in Fig. 3. The predominant peaks lies in (200), (111) and (220) plane which indicates the good crystallinity of the sample. The average grain size of NiO nanoparticles obtained in this process is 14.08 nm. The nickel oxide sample shows the cubic structure with $a = 4.17\text{\AA}$ which is in agreement with JCPDS file 47-1049 [25]. Figure 4 shows the XRD pattern of NiO nanoparticles annealed at 700 °C. The predominant peaks lies in the plane (200), (111) and (220) with high intensity. The average grain size of the particle is found to be 15.86 nm. The grain size of NiO nanoparticles annealed at 700 °C is slightly larger than the NiO nanoparticles annealed at 500 °C. From this we can say that, the grain size increases with increase in temperature. This behavior was expected because the heating facilitates the diffusion and agglomeration of the particles [26-28]. The diffraction peaks becomes more intense with higher temperature because, at higher temperatures the crystallite size will be large. This can be attributed to the thermally promoted crystallite growth [29]. Table 1 gives the XRD parameters of NiO nanoparticles annealed at two different temperatures. From the table, the NiO nanoparticles shows cubic structure with lattice parameters value $a=b=c=4.17\text{\AA}$.

Table 1. XRD parameters of NiO nanoparticles

Sample	hkl plane	2 Theta(deg)	Theta(deg)	Grain size(nm)	Lattice constant (a=b=c)
NiO nanoparticles annealed at 500 °C	111	37.24	18.62	13.22	4.177
	200	43.29	21.64	13.88	4.176
	220	62.90	31.45	15.15	4.175
	311	75.43	37.71	15.38	4.173
	222	79.42	39.71	15.99	4.176
NiO nanoparticles annealed at 700 °C	111	37.40	18.70	14.92	4.160
	200	43.44	21.72	15.59	4.162
	220	63.03	31.51	17.06	4.167
	311	75.58	37.79	17.96	4.169
	222	79.57	39.78	19.41	4.169

3. 2. Surface Morphological Analysis

The surface morphological features of synthesized nanoparticles were studied by Scanning Electron Microscope. Fig. 5 shows the SEM image of NiO nanoparticles with magnifications of 10,000, 20,000, and 55,000. The instrumental parameters, accelerating voltage, spot size, and magnification and working distances are indicated on SEM image. The appearances of some particles are in spherical shape. We can observe that the particles are highly agglomerated and they are essentially cluster of nanoparticles. The observation of some larger nanoparticles may be attributed to the fact that NiO nanoparticles have the tendency to agglomerate due to their high surface energy and high surface tension of the ultrafine nanoparticles [30].

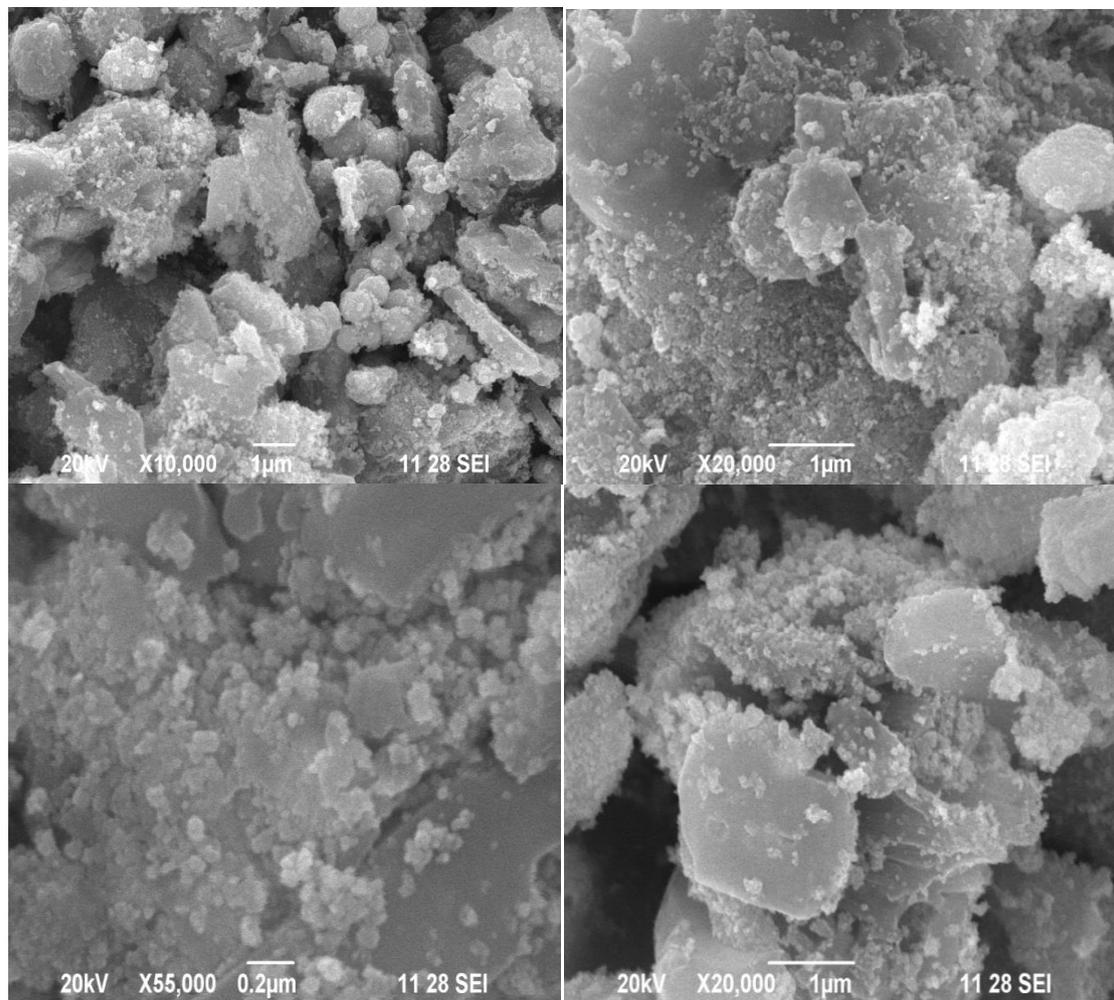
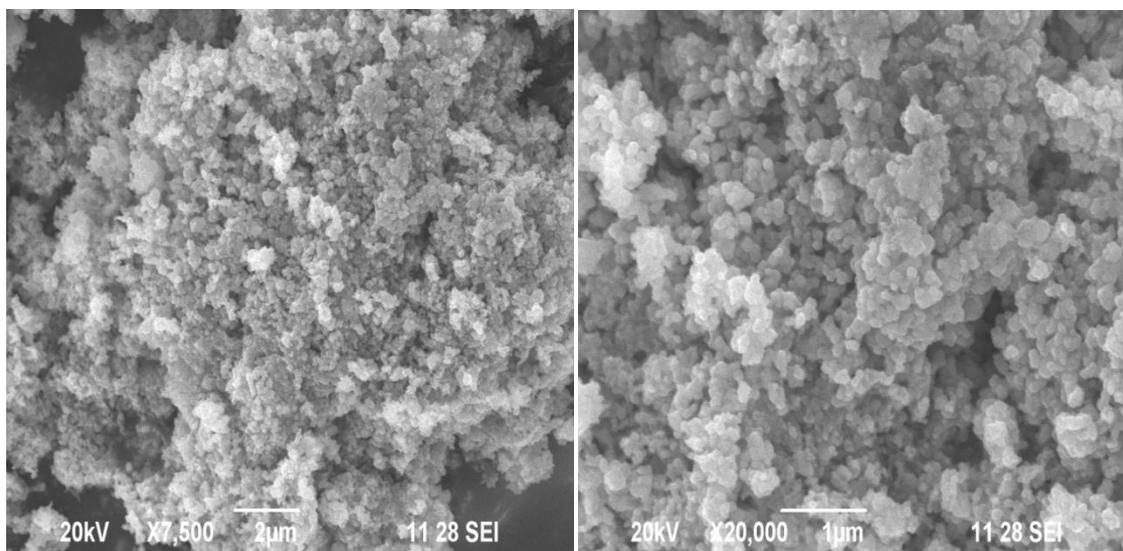


Figure 5. SEM images of NiO nanoparticles annealed at 500 °C



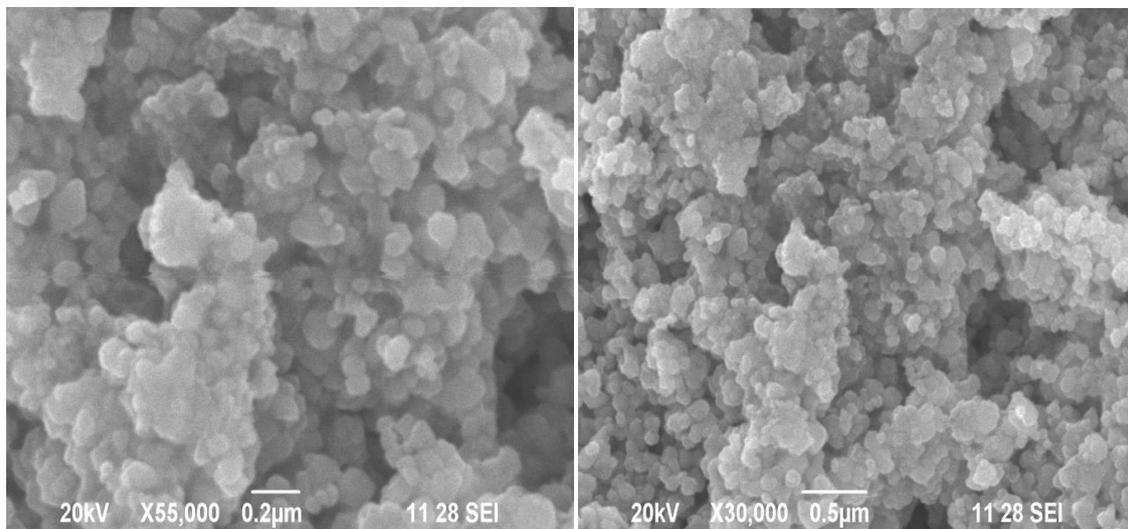


Figure 6. SEM images of NiO nanoparticles annealed at 700 °C

Figure 6 shows the SEM micrographs annealed at 700 °C with magnifications in the range of 7,500,20,000,55,000,30,000. Some of the particles are in spherical shape. The particles are uniformly distributed and attached to each other to form agglomerated particles. The aggregate size increases on increasing the annealing temperature from 500 to 700 °C, this agglomeration resulted in an increase of the crystallite size of the particles [31].

3. 3. Thermal Analysis

Many compounds are not stable and decompose into some other compounds on heating. To check the thermal stability the prepared sample is characterized with the help of the Thermo gravimetric analysis. Figure 7 (a) shows the TGA curve of synthesized NiO annealed at 700 °C which was carried out from 40 to 730 °C. The weight loss occurs in two stages: (i) 80-90 °C with 0.5% weight loss due to dehydration [32] (ii) 100-120 °C with 0.3% weight loss due to evaporation of water.

In the DTA curve, a sharp endothermic peak is observed at 89 °C. This might be attributed to the thermal dehydration of the precursor and the evaporation of physically adsorbed impurities. When the hydroxyl group is removed from Ni(OH)₂ the NiO crystal phase is expected to be formed (Fig. 7(b)).

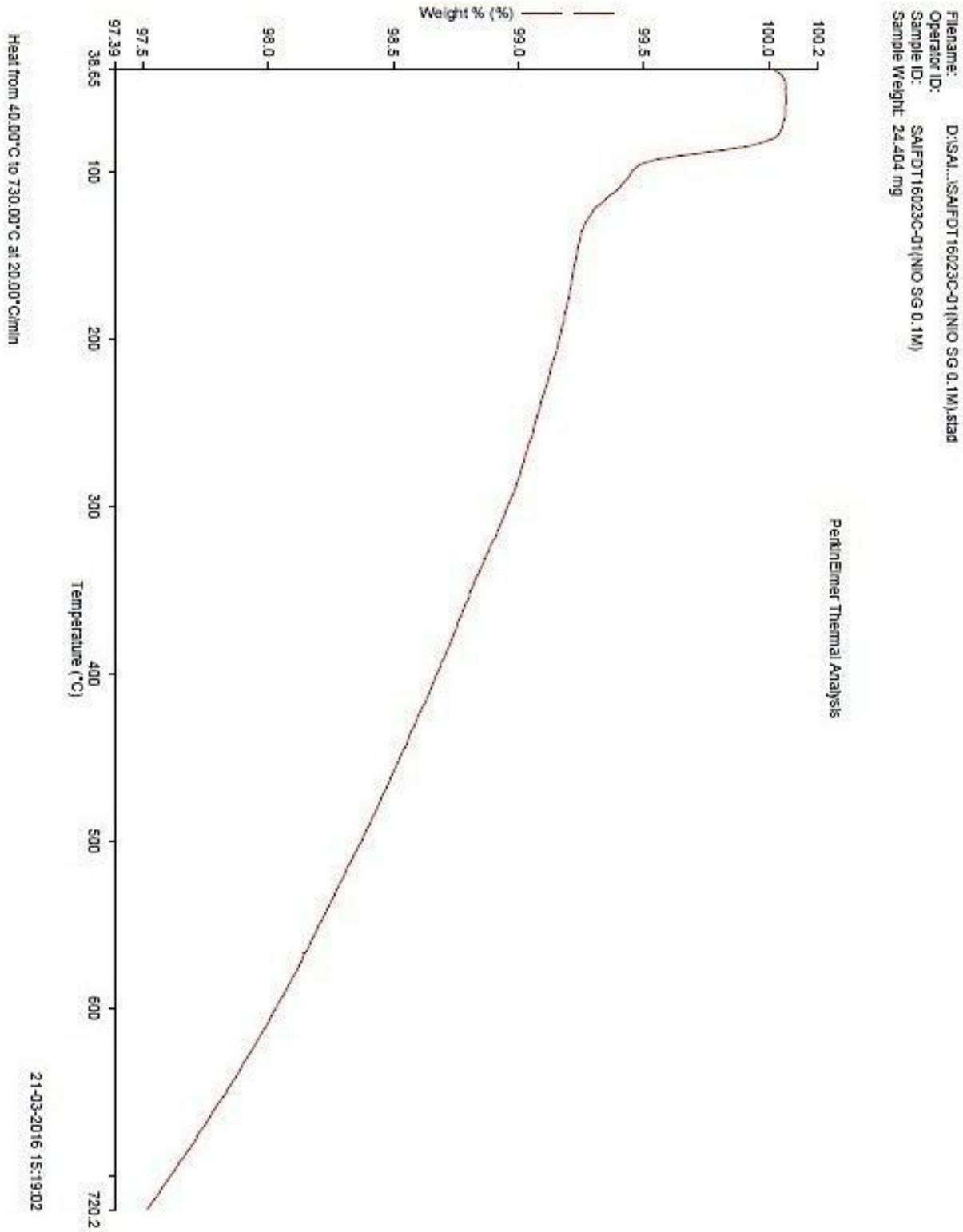


Figure 7(a). TGA curve of NiO nanoparticles annealed at 700 °C

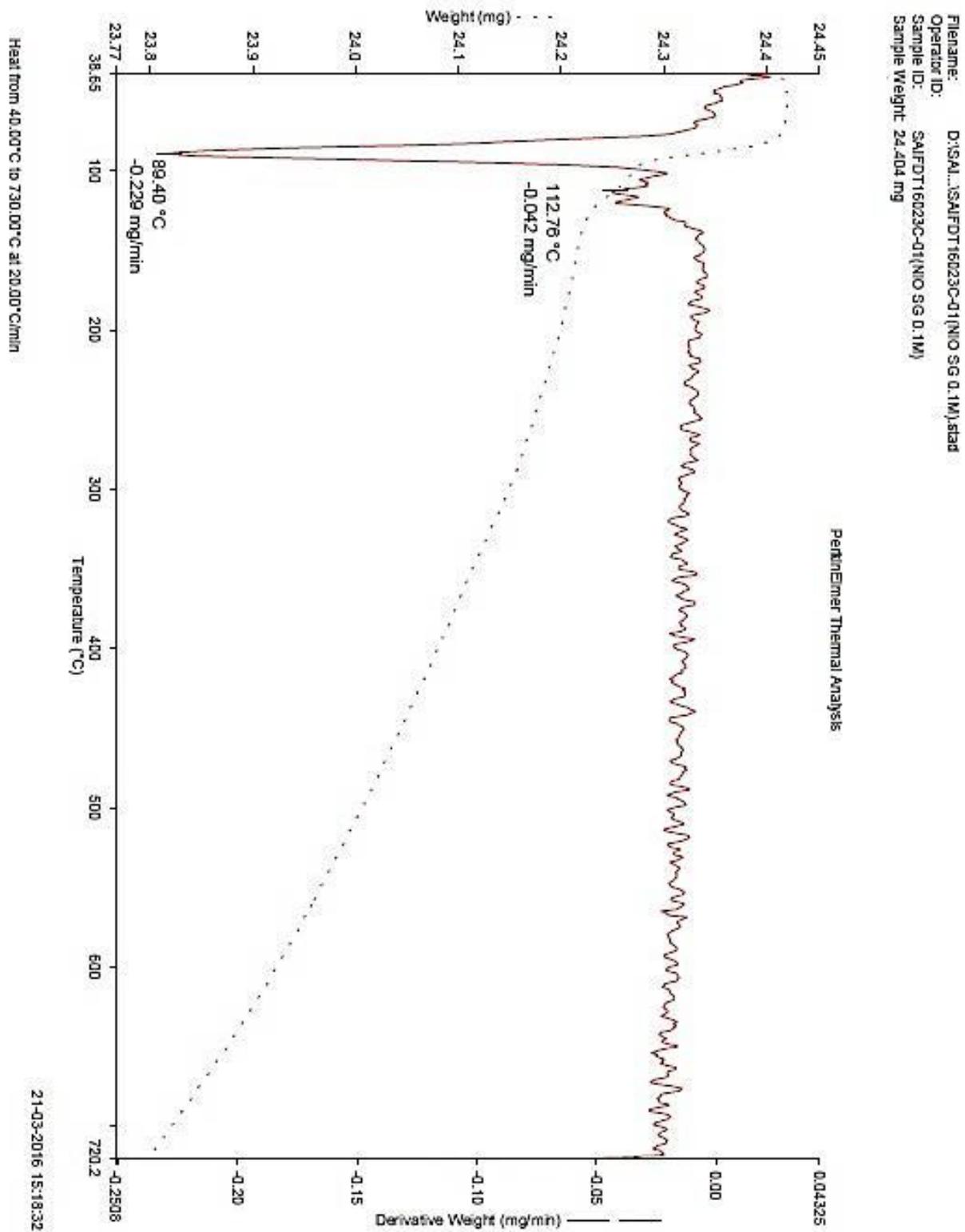


Figure 7(b). DTA curve of NiO nanoparticles annealed at 700 °C

4. CONCLUSION

The Nickel Oxide nanoparticles have been prepared by sol gel method and the prepared Nickel Oxide nano particles were annealed at two different temperatures 500 °C and 700 °C. XRD results revealed that the grain size of samples increases with increasing annealing temperature. SEM results showed that the particles are highly agglomerated and they are essentially a cluster of nano particles. The thermal stability of the sample was examined by TGA analysis which showed NiO nanoparticles formed between 130-720 °C. The prepared Nickel oxide can be used for ceramic structures and lithium ion micro batteries.

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