

# Preparation of nickel-ferrites from complex compounds with acetylacetonato-ligands in different atmosphere conditions

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Ultrafine nickel-ferrite materials were prepared from complex compounds with acetylacetonato-ligands, according to a previously developed method. The influence of different atmospheres (air, oxygen and nitrogen) applied during thermal decomposition of precursors was studied. Thermal decomposition was carried out with the heating rate of 10°C/min and the cooling rate of 20°C/min. Characterization of the final products was performed by X-ray and TEM analysis. Reviewing the products, we concluded that the optimal temperatures for the preparation of pure ultrafine ferrite materials were 400°C for air atmosphere, and 500°C for O<sub>2</sub> and N<sub>2</sub> atmospheres. All samples prepared at lower temperatures show the presence of the nickel-phase, while all samples prepared at higher temperatures show the presence of the hematite-phase.

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## 1. Introduction

In recent years, nickel ferrites have been widely used in many fields of technics, especially in microelectronics. Like many other fine-particle ferrites, they received a considerable attention due to their application in high-density magnetic recording media, as suspension materials in ferromagnetic liquids, and as catalyst. The interest in these materials is permanently increasing, because of their usability under extreme conditions. They possess unique chemical and magnetic properties. These properties depend strongly on preparation conditions. Many interesting methods for the preparation and characterization of nickel-ferrites have recently been investigated, such as the pyrolysis method [1], the coprecipitation method [2-4], the combined method coprecipitation/mechanical milling [5], the sol-gel process [6], the combustion method [7] and many others. One of the most interesting methods is the synthesis from complex compounds with acetylacetonato-ligands by thermal decomposition [8,9]. Using this process, ultrafine nickel-ferrites with particle size less than 20 nm can be obtained. That was the reason why we investigated this preparation routine. We wanted to optimize preparation conditions by using different atmospheres (nitrogen, oxygen and air) during thermal decomposition of complex precursors.

## 2. Experimental

The complex compounds [Ni(AA)<sub>2</sub>] and [Fe(AA)<sub>3</sub>], where AA denotes acetylacetonato (2,4-pentadione), were synthesized according to the previously described method. Substances were mixed in stoichiometric ratios, homogenized and thermally decomposed, as in one of our earlier works [9]. Thermal decomposition was carried out at the heating rate of 10 °C/min. The cooling rate was 20 °C/min. The products were characterized using X-ray powder diffraction and transmission electron microscopy (TEM).

## 3. Results and discussion

Results of X-ray examinations are shown in Fig. 1 for samples produced at 400°C (c), 500 °C (b) and 600 °C (a) in N<sub>2</sub> atmosphere, in Fig. 2 for samples produced at 300 °C (c), 400 °C (b) and 500 °C (a) in O<sub>2</sub> atmosphere, in Fig. 3 for samples produced at 300°C (c), 400°C (b) and 500 °C (a) in air atmosphere (all samples so far have been kept at the final temperature for 1 min.) and in Fig. 4 for samples produced at 300 °C (c), 400 °C (b) and 500 °C (a) in air atmosphere (kept at the final temperature for 1 hour).

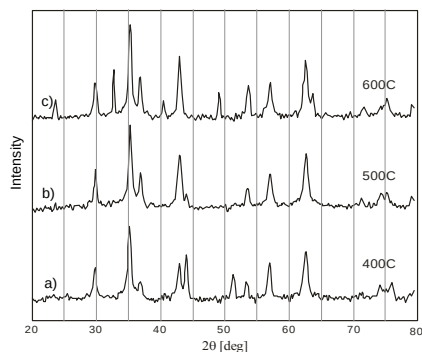


Fig.1 Nitrogen (1 min).

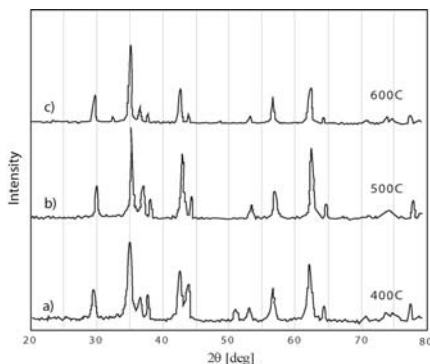


Fig. 2. Oxygen (1 min).

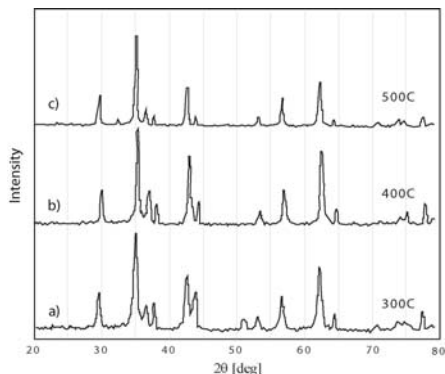


Fig. 3. Air (1 min).

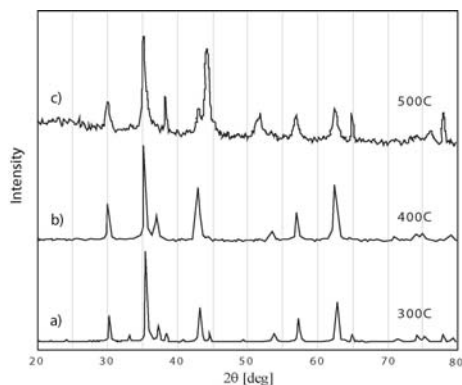


Fig. 4. Air (1 hour).

All diffractograms show well defined peaks. The peaks were analyzed and compared with ASTM cards [10]. Most of them are spinel reflections which belong to  $\text{NiFe}_2\text{O}_4$ . Other reflections, assigned to samples marked (c), show the presence of nickel-phase at  $2\theta$  angles of  $40.45^\circ$ ,  $51.85^\circ$ ,  $76.35^\circ$ . Samples marked (b) have spinel reflections only. Samples marked (a) show the presence of hematite-phase at  $2\theta$  angles of  $33.18^\circ$ ,  $64.02^\circ$ .

Calculated cell parameters for all samples are given in the Table 1.

Table 1. Calculated cell parameters for samples obtained at different atmosphere conditions [ASTM  $a_0 = 8.3390$ ].

| Temperature (°C) | Nitrogen | Oxygen | Air (1 min) | Air (1 hour) |
|------------------|----------|--------|-------------|--------------|
| 300              |          |        | 8.3382      | 8.3788       |
| 400              | 8.3496   | 8.3619 | 8.3380      | 8.3374       |
| 500              | 8.3438   | 8.3451 | 8.3337      | 8.3359       |
| 600              | 8.3271   | 8.3342 |             |              |

The results obtained by electron microscopy show that all samples are ultrafine, with particle sizes in accordance with previously described results [8].

#### 4. Conclusions

It was shown that the preparation of ultrafine nickel-ferrite is possible in different atmosphere conditions (nitrogen, oxygen and air), at relatively low temperatures ( $400^\circ\text{C} - 500^\circ\text{C}$ ). All investigated systems show the same effect, formerly described for nitrogen atmosphere conditions, with the nickel phase present at lower and hematite phase at higher temperatures. It was found that the optimum temperatures for the formation of ultrafine nickel-ferrite materials using this method are  $400^\circ\text{C}$  for samples made in air atmosphere, and  $500^\circ\text{C}$  for samples made in nitrogen and oxygen atmospheres.

#### Acknowledgements

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