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Solid–solid interactions in the NiO/Fe_2O_3 system with and without LiO_2 doping

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Abstract

The effects of calcination temperature, molar ratio and Li_2O doping on the interaction between ferric and nickel oxides were investigated using TG, DTA, dDTA and XRD techniques. The results obtained revealed that NiO retarded the crystallization of ferric oxide into the alpha phase and interacted with it to yield a well crystallized $NiFe_2O_4$ (trevorite) at temperatures starting from 700°C. However, the solid–solid interaction that gives rise to nickel ferrite was found to be affected by the molar ratio of NiO and Fe_2O_3 present. The complete transformation of the reacted oxides into the ferrite phase required prolonged heating at temperatures above 1100°C.

Lithium oxide doping at 700 and 800°C modified the formation of nickel ferrite; the presence of 0.75 or 1.5 mol% of Li_2O depressed the ferrite formation, which was, however, enhanced in the presence of 3 mol% of Li_2O. Heating of the mixed solids doped with 1.5 mol% Li_2O at 800°C led to the formation of β-$LiFeO_2$, which was converted into α- and/or β-$LiFe_5O_8$ by increasing the amount of Li_2O to 3 mol%.

The effect of Li_2O in modifying the solid–solid interactions between NiO and Fe_2O_3 and the formation of β-$LiFe_5O_8$ is discussed in the light of the dissolution of a portion of Li_2O and an increase in the mobility of Ni^{2+} ions in the nickel oxide lattice.

Keywords: Calcination; Crystallinity; dDTA; Doping; Ferrite; Mixed oxide; SSE

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1. Introduction

The coexistence of basic nickel carbonate and hydrated ferric oxide may affect their thermal decomposition and also affect the solid–solid interactions between the produced NiO and Fe_2O_3 solids [1]. Nickel ferrite can be obtained by heating ferric oxide with nickel carbonate or nickel oxide at temperatures starting from 800°C [2–4]. The solid–solid interactions between these oxides may be influenced by the prehistory of the parent solids, their ratio, and also by the addition of small amounts of lithium carbonate or lithium oxide [1]. It has been reported that lithium oxide doping promotes the formation of nickel ferrite [5]. The promoting effect of the Li_2O treatment has been attributed to an effective increase in the mobility of the reacting cations through the whole mass of each solid and through the newly formed ferrite film covering the surface of the grains of each oxide [5]. The ferrites are classified according to their structure and properties into two types. The first type has a cubic spinel structure (e.g. Mg, Co, Ni, Cu and Zn), whereas the second type exhibits different structures: e.g. the ferrites of the alkaline earth elements [6–8]. The spinel-type ferrites are commonly utilized in line electronic devices owing to their remarkable magnetic and semiconducting properties [2–4].

The present work reports a study on the thermal behaviour of pure and Li_2O-doped mixtures of NiO and Fe_2O_3 using TG, DTA and dDTA techniques. The solid–solid interactions between nickel oxide and ferric oxide were characterized by the X-ray diffraction technique.

2. Materials and experimental techniques

2.1. Materials

Hydrated ferric oxide was precipitated from ferric sulphate (BDH) solution using a dilute NH_4OH solution (0.2 N) at 70°C and pH 8. The gel obtained was washed with bidistilled water until free from ammonium and sulphate ions, then dried at 100°C to constant weight.

Pure Fe_2O_3 samples were obtained by thermal decomposition of the prepared hydrated ferric oxide in air at various temperatures between 300 and 800°C.

Pure NiO specimens were obtained by heating basic nickel carbonate in air at temperatures between 300 and 800°C. The basic carbonate used was of analytical grade and supplied by Prolabo Company.

Three specimens of ferric/nickel mixed oxides having different compositions were prepared by mechanical mixing of finely powdered basic $NiCO_3$ with hydrated solid ferric oxide. The mixed oxides were obtained by firing the mixed solids in air at 300–800°C for 4 h.

Samples of the doped mixed solids were prepared by treating a given mass of the prepared mixed solids with solutions containing different proportions of lithium nitrate. The extents of doping expressed in mol% of Li_2O were 0.75, 1.5 and 3. The pure and doped solids obtained were dried at 100°C, then ignited in air at 700 and 800°C.

2.2. Techniques

The DTA and TG of different samples were carried out using a Netzsch–Gerätebau GmbH thermal analysis apparatus (Bestell-Nr 348-742C). The rate of heating was 10°C min^{-1}. A 75 mg sample of the solid specimen was used in each case and the heating process was conducted in the presence of a current of dry air flowing at 50 ml min^{-1}.

An X-ray investigation of the thermal products from the various solids was carried out using a Philips type PW 1390 diffractometer. Some of the patterns were run with iron-filtered cobalt radiation ($\lambda = 1.7889$ Å) at 30 kV and 10 mA with a scanning speed in 2θ of 2° min^{-1}, and the others with nickel-filtered copper radiation ($\lambda = 1.5405$ Å) at 36 kV and 16 mA with a scanning speed in 2θ of 2° min^{-1}.

3. Results and discussion

3.1. Thermal behaviour of free oxides

Fig. 1 shows the TG, DTA and dDTA curves of hydrated ferric oxide. Four endothermic peaks and one exothermic peak are observed in the DTA curve. The first two peaks are relatively weak, but the other peaks are strong and sharp, especially the third one. The maxima of these peaks are located at 130, 205, 280, 340 and 385°C, respectively. The endothermic peaks are associated with weight

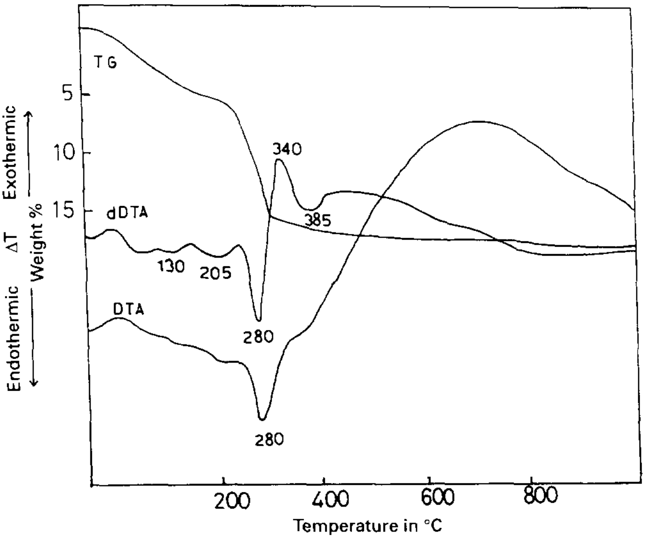

Fig. 1. TG, DTA and dDTA of hydrated ferric oxide.

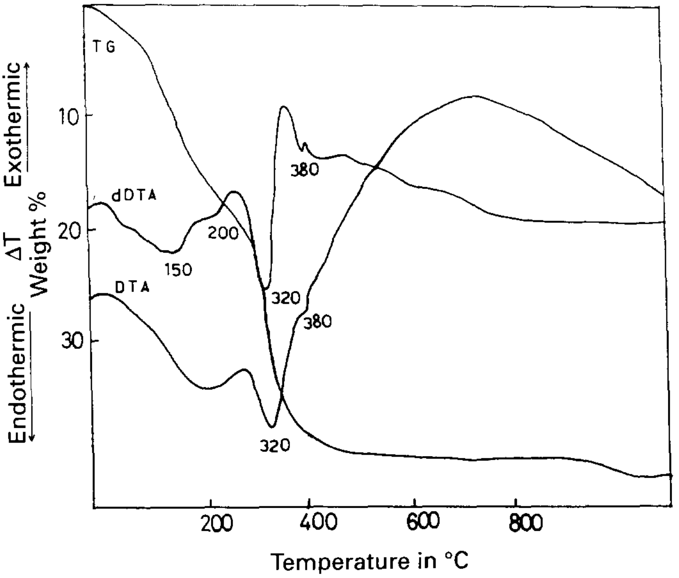

Fig. 2. TG, DTA and dDTA of basic nickel carbonate.

losses of 4%, 2.8%, 9.2% and 2.1%, respectively. The peak at 130°C indicates the removal of physisorbed water; the actual loss in weight of the investigated hydrated oxide amounts to 14.1%, which suggests the formula of $Fe_2O_3 \cdot 1.5H_2O$. The peaks at 205 and 280°C correspond to dehydroxylation of structural water from iron oxyhydroxide. The exothermic peak is attributed to the ready nucleation of the hydrated residue to α-Fe_2O_3 [9,10]. This speculation will be confirmed later in this work by XRD investigation. The endothermic peak at 385°C, with 2.1% loss in weight, did not appear in the DTA curve of an iron oxide gel aged for 5 months at pH 5 [11]. This peak may characterize the complete removal of the last traces of hydroxyl groups present in the interior of grains of the produced crystalline Fe_2O_3. The process of dehydroxylation of structural water from hydrated ferric oxide can be regarded as a surface reaction proceeding progressively from the outermost surface layers of the solid towards the interior of its particles. Similar behaviour has been found in the case of thermal dehydroxylation of nickel hydroxide, which starts at 250°C; the solid obtained at 350°C still contained a very small proportion of undecomposed hydroxide, found in the interior of the NiO grains [12].

Fig. 2 shows the TG, DTA and dDTA curves of the nickel carbonate used at temperatures between room temperature and 1000°C. Three endothermic peaks are observed in the DTA curve. The first peak is strong and broad, extending between room temperature and 280°C, with its maximum at 180°C. The second peak is strong and sharp, having its maximum at 320°C, and the last peak is weak but sharp and located at 380°C. These peaks are associated with weight losses of 20.5%, 19% and 1.7%, respectively, i.e. the total loss in weight reaches 41.2%. This value

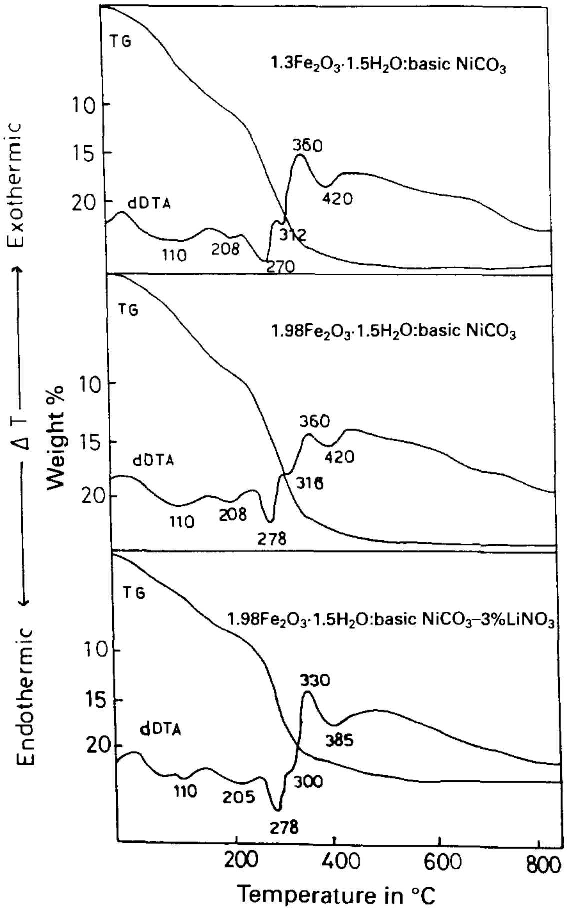

Fig. 3. TG, DTA and dDTA of different mixed solids.

permitted us to suggest the formula of the investigated basic nickel carbonate as $NiCO_3 \cdot 2Ni(OH)_2 \cdot 4.3H_2O$. The first peak corresponds to the removal of water of crystallization of the basic nickel carbonate according to

$$NiCO_3 \cdot 2Ni(OH)_2 \cdot 4.3H_2O \rightarrow NiCO_3 \cdot 2Ni(OH)_2 + 4.3H_2O$$

In fact, the loss in weight corresponding to this reaction is 20.3%, which is very close to that found experimentally. The peak at 320°C corresponds to simultaneous thermal decomposition of the $NiCO_3$ and $Ni(OH)_2$ of the anhydrous basic nickel carbonate. The endothermic peak at 380°C might indicate the complete thermal decomposition of the last traces of undecomposed nickel hydroxide in the interior of NiO particles [12].

It can be seen from Fig. 2 that the solid produced by the thermal decomposition of basic nickel carbonate remains stable at temperatures between 450 and 900°C, then loses ≈1% of its weight on heating at 950°C. This value indicates the departure of all excess oxygen present in non-stoichiometric NiO to produce a stoichiometric green nickel oxide solid.

3.2. Thermal behaviour of different mixed oxides

Fig. 3 shows the TG and dDTA curves of some mixed solids having the molar compositions of $1.3Fe_2O_3 \cdot 1.5H_2O$: basic $NiCO_3$, $1.98Fe_2O_3 \cdot 1.5H_2O$:basic $NiCO_3$, and $1.98Fe_2O_3 \cdot 1.5H_2O$:basic $NiCO_3$ pretreated with 3 mol% of $LiNO_3$. These samples are designated as Fe Ni-I, Fe Ni-II and Fe Ni-II-Li. The DTA curves of these solids exhibit five endothermic peaks and one exothermic peak. The exothermic peak indicating the crystallization of α-Fe_2O_3 was found at 360 and 330°C for the pure and the doped solids, respectively. The last endothermic peak, corresponding to the removal of the last traces of hydroxyl groups present in the interior of particles of the produced α-Fe_2O_3, was detected at 420 and 380°C for the pure and the doped mixed solids. The DTA curves of the different mixed solids investigated did not include any thermal effect at temperatures between 450 and 1000°C, and the weight of these solids did not undergo any change on heating at ≥450°C. The total weight losses accompanying the thermal treatment of various mixed solids were 27.6, 25 and 24.6% for Fe Ni-I, Fe Ni-II and Fe Ni-II-Li, respectively. The positions of endothermic peaks indicating dehydroxylation of structural water from iron oxyhydroxide and decomposition of anhydrous basic nickel carbonate were not much affected by heating mixtures of the two compounds.

Inspection of Figs. 1, 2 and 3 reveals that (i) NiO retards the crystallization of α-Fe_2O_3: the exothermic peak corresponding to this process was shifted from 340 to 360°C in the presence of nickel oxide; (ii) NiO retards also the departure of the last traces of OH groups present in the interior of α-Fe_2O_3: the endothermic peak relating to this change was shifted from 385 to 420°C; (iii) lithium oxide doping enhances the process of crystallization of ferric oxide into α-Fe_2O_3.

3.3. XRD investigation of the thermal products of pure and mixed oxide solids

XRD diffractograms of hydrated ferric oxide preheated in air at various temperatures between 250 and 1000°C, not given here, reveal that the hydrated ferric oxide used, having the formula $Fe_2O_3 \cdot 1.5H_2O$, loses its water of constitution on heating at 250°C, forming anhydrous α-ferric oxide with moderate crystallinity. The endothermic peak located at 280°C in the DTA curve of the hydrated ferric oxide (see Fig. 1) indicates the removal of water of constitution, yielding anhydrous Fe_2O_3 of moderate crystallinity. It can also be observed from the XRD measurements that an increase in calcination temperature from 250 to 900°C resulted in a progressive improvement in the degree of crystallinity of the α-ferric oxide produced. The solids obtained at 900 and 1000°C exhibited an excellent degree of crystallinity.

The colour of ferric oxide heated at 250-700°C was reddish brown, turning to dark brown at 800°C and black at 900 or 1000°C. The reddish and dark brown colours are characteristic for ferric oxide (haematite, α-Fe_2O_3), whereas the black colour might indicate ferric oxide in magnetite form (Fe_3O_4). The fact that the diffraction lines typical of the Fe_3O_4 phase were not detected in the XRD patterns of the solids heated at 900 and 1000°C might indicate the amorphous nature of this or its phase presence in minute amounts covering the surface of the dominant α-ferric oxide. The presence of magnetite in a ferric oxide sample preheated in air at 1000°C has been confirmed by using a strong hand magnet, which attracted a small portion of the ferric oxide solid. It can thus be concluded that the complete transformation of Fe_2O_3 into well crystallized Fe_3O_4 requires prolonged heating at temperatures above 1000°C or in the presence of a reducing atmosphere; the heating of ferric oxide supported on alumina at a temperature as high as 1100°C was not sufficient to effect even the partial transformation of α-Fe_2O_3 into Fe_3O_4 [13].

Preliminary experiments showed that the thermal product of basic nickel carbonate calcined in air at temperatures between 300 and 900°C consists of a well crystallized NiO phase. However, the colour of the solid obtained changes from black through grey to light grey and green on increasing the heating temperature in the range 300–1000°C. The observed loss in weight of NiO being heated at 950°C ($\approx 1\%$, see Fig. 2) corresponds to the formation of a stoichiometric nickel oxide.

The green colour indicates a stoichiometric NiO phase, whereas the other colours are indicative of a non-stoichiometric solid that contains a slight excess of oxygen with respect to the amount present in stoichiometric NiO. This excess oxygen is accommodated in the oxide lattice as cationic vacancies, with subsequent transformation of some of the Ni^{2+} into Ni^{3+} ions [14,15], which could be created by interaction between NiO and oxygen [16–18].

The XRD patterns of the mixed solid having the formula $Fe_2O_3 \cdot 1.5H_2O$:0.05 basic $NiCO_3$ preheated in air at 600 and 800°C, not given here, indicate that the mixed solid heated at 600°C consists of well crystallized α-Fe_2O_3, with some diffraction lines corresponding to a free NiO phase. Increasing the heating temperature to 800°C caused the appearance of some diffraction lines of a well crystallized $NiFe_2O_4$ phase. The differentiation between free NiO, Fe_2O_3 and $NiFe_2O_4$ phases was not an easy task owing to the presence of common diffraction lines in their XRD patterns. However, this problem has been solved by adopting the so-called key lines proposed by one of the authors [1]. The absence of any diffraction lines indicative of $NiFe_2O_4$ in the mixed solid preheated at 600°C showed the absence of any solid–solid interaction between NiO and Fe_2O_3.

Indeed, the presence of unreacted NiO in this solid sample might suggest the absence of solid solution of NiO in Fe_2O_3. This conclusion is not unexpected, simply because the Ni^{2+} ion is bigger than the Fe^{3+} ion, 0.78 and 0.64 Å, respectively [14].

Fig. 4 shows the X-ray diffractograms of basic $NiCO_3$:0.2($Fe_2O_3 \cdot 1.5H_2O$) subjected to thermal treatment in air at 600 and 800°C. All the diffraction lines of well crystallized NiO were detected in the XRD patterns of mixed solid specimens calcined at 600 and 800°C. It can also be seen from Fig. 4 that all diffraction lines of α-Fe_2O_3 were observed in diffractograms of the solid samples calcined at 600 and

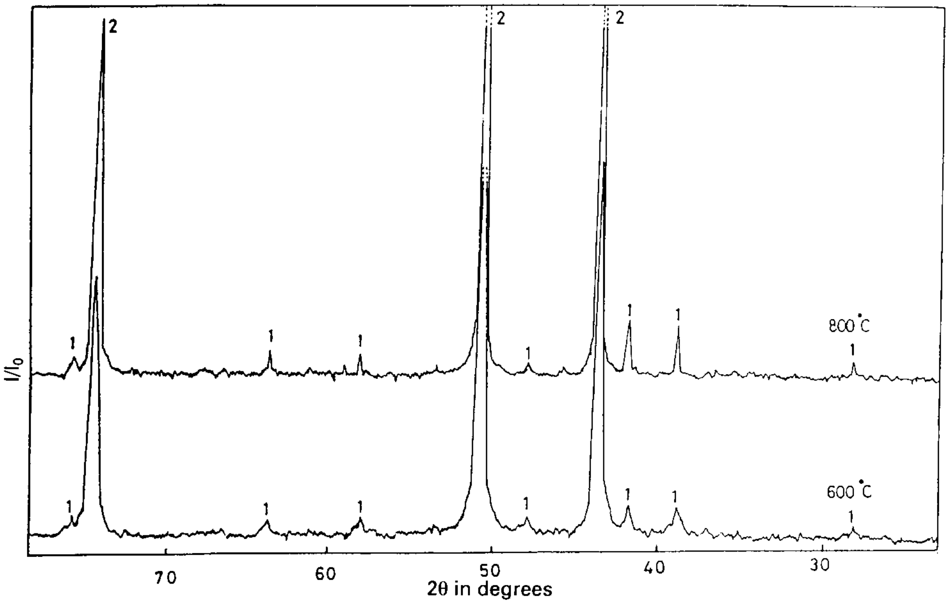

Fig. 4. X-ray diffractograms of basic $NiCO_3$:0.2($Fe_2O_3 \cdot 1.5H_2O$) calcined in air at 600 and 800°C; 1, alpha-Fe_2O_3; 2, NiO.

800°C. However, the degree of crystallinity of this phase was much increased by raising the calcination temperature from 600 to 800°C. Indeed, the α-Fe_2O_3 produced at 600°C exhibited a relatively small degree of crystallinity. It has been shown that pure hydrated ferric oxide is transformed into well crystallized α-Fe_2O_3 by heating in air at temperatures starting from 500°C. These findings indicate that the presence of nickel oxide with ferric oxide retards the crystallization process of the α-Fe_2O_3 phase. These results are in good agreement with those of the thermal behaviour of the mixed solid (see Figs. 1 and 3). In fact, the exothermic peak relating to the crystallization of α-Fe_2O_3 moved from 340°C in the absence of nickel oxide to 360°C in its presence. The retardation effect of NiO on the crystallization process of α-Fe_2O_3 may result from some kind of dispersion of NiO crystallites through the whole mass of ferric oxide grains, hindering their crystallization.

A comparison between the XRD patterns of Fe_2O_3:0.05 NiO and NiO:0.2Fe_2O_3 solids preheated at 800°C indicates the formation of well crystallized $NiFe_2O_4$ at 800°C in the case of Fe_2O_3 treated with 5 mol% of NiO, whereas no ferrite phase has formed at 800°C in the case of NiO treated with 20 mol% of Fe_2O_3. These results clearly indicate that nickel oxide can dissolve a certain portion of Fe_2O_3, thus modifying its electronic structure, via substitution of some lattice Ni^{2+} ions by Fe^{3+} ions with subsequent removal of excess oxygen. The dissolution process can take place according to two different mechanisms, depending on the prehistory of the solid sample and the calcination conditions. The incorporation process is accompanied by the disappearance of some Ni^{3+} present in non-stoichiometric NiO

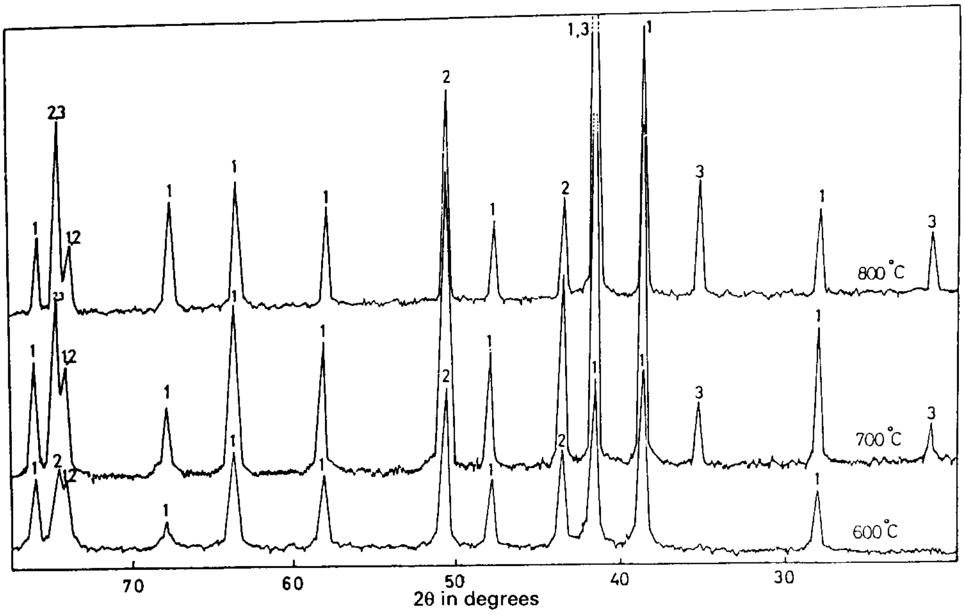

Fig. 5. X-ray diffractograms of basic $NiCO_3$:1.98($Fe_2O_3 \cdot 1.5H_2O$) calcined in air at 600, 700 and 800°C; 1, alpha-Fe_2O_3; 2, NiO; 3, $NiFe_2O_4$.

and the creation of cationic vacancies in the case of stoichiometric NiO. These two mechanisms can be simplified by the use of Kröger's [19] propositions in the following manner

$$Fe_2O_3 + 2Ni^{3+} \rightarrow 2Fe^{3+}(Ni^{2+}) + 0.5O_2(g) \tag{1}$$

$$Fe_2O_3 + NiO \rightarrow 2Fe^{3+}(Ni^{2+}) + V(Ni^{2+}) \tag{2}$$

where $Fe^{3+}(Ni^{2+})$ is a trivalent iron ion located in the position of a host cation (Ni^{2+}) of the NiO lattice, Ni^{3+} represents a positive hole localized on a Ni^{2+} ion and is present in non-stoichiometric and solid nickel oxide, and $V(Ni^{2+})$ represents an uncharged cationic vacancy. The presence of these vacancies may enhance the sintering process of NiO [20].

The dissolution process of Fe^{3+} in NiO according to the first mechanism, which requires the presence of Ni^{3+}, predominates at moderate temperatures (below 600°C). The second mechanism governs the doping process at relatively high temperatures, above 600°C [20]. The formation of a nickel ferrite phase by heating nickel/ferric oxides at 800°C suggests a limited solubility of Fe^{3+} in the NiO lattice.

Fig. 5 depicts the X-ray diffractograms of basic nickel carbonate mixed with 1.98 mol of hydrated ferric oxide and preheated in air at 600, 700 and 800°C. The diffraction lines of well crystallized α-Fe_2O_3 and NiO only are detected in the pattern of the mixed solid preheated at 600°C. Increasing the calcination temperature above this limit (700 or 800°C) brought about the appearance of the diffraction

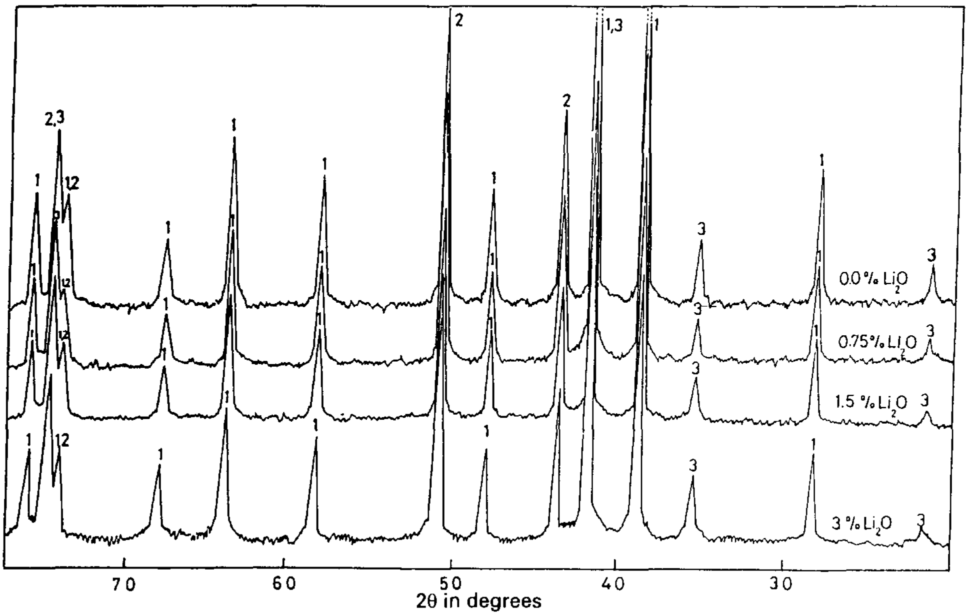

Fig. 6. X-ray diffractograms of basic $NiCO_3$:198($Fe_2O_3 \cdot 1.5H_2O$) doped with Li_2O and calcined in air at 700°C; 1, alpha-Fe_2O_3; 2, NiO; 3, $NiFe_2O_4$.

lines of the nickel ferrite phase accompanying the diffraction lines of unreacted NiO and Fe_2O_3.

These results indicate that: (i) no solid–solid interaction between NiO and Fe_2O_3 takes place at 600°C; (ii) NiO interacts with Fe_2O_3, in the solid state, at 700°C to produce a $NiFe_2O_4$ (trevorite) phase; (iii) the crystallinity of the nickel ferrite phase increases on increasing the heating temperature from 700 to 800°C; (iv) the complete transformation of NiO and Fe_2O_3 to $NiFe_2O_4$ (trevorite) requires prolonged heating of the mixed oxide solid at elevated temperatures [1].

The fact that no thermal peak relating to the formation of a nickel ferrite phase has been detected in the DTA curves (Fig. 3) of the various mixed solids heated up to 1000°C indicates that the solid–solid interaction process between NiO and Fe_2O_3 takes place at a rate too small to be detected by normal DTA techniques. It has been reported by one of the authors [1] that nickel oxide interacts with Fe_2O_3 to produce nickel ferrite at temperatures starting from 800°C, instead of the 700°C in the present work. The ferric oxide employed by these authors was a sintered sample of α-ferric oxide supplied by the Fluka company. These results clearly indicate the role of the prehistory of the ferric oxide in nickel ferrite formation.

Figs. 6 and 7 show the X-ray diffractograms of the mixed solids doped with 0.75, 1.5 and 3 mol% of Li_2O and heated at 700 and 800°C. The diffraction lines of well crystallized α-Fe_2O_3, NiO and $NiFe_2O_4$ were detected in the patterns of the pure mixed solids preheated in air at 700 and 800°C. The presence of Li_2O as dopant

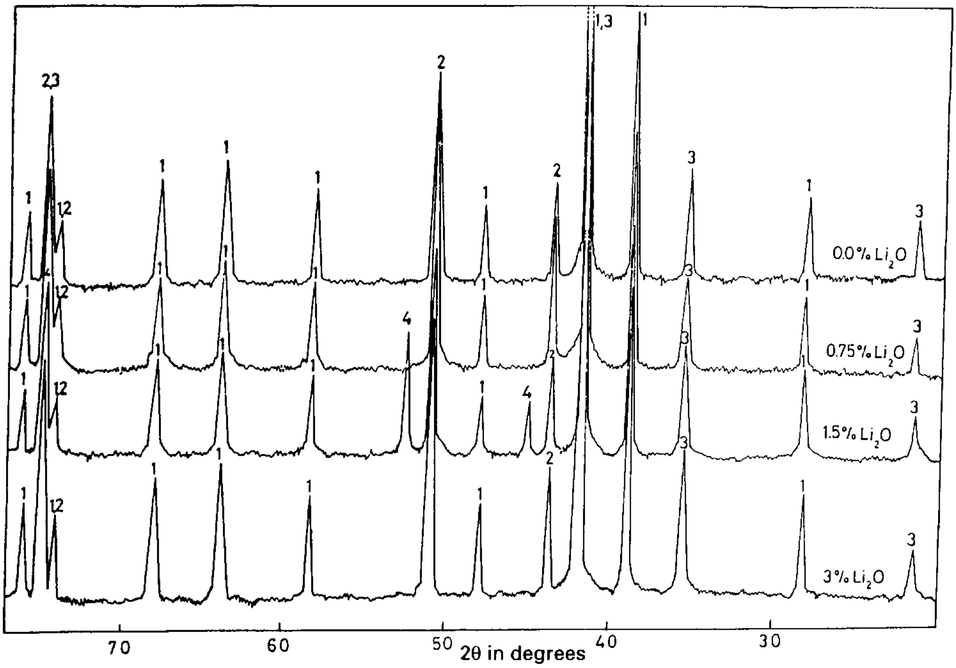

Fig. 7. X-ray diffractograms of basic $NiCO_3$:1.98($Fe_2O_3 \cdot 1.5H_2O$) doped with Li_2O and calcined in air at 800°C; 1, alpha-Fe_2O_3; 2, NiO; 3, $NiFe_2O_4$; 4, β-$LiFeO_2$.

brought about some changes in the degree of crystallinity of $NiFe_2O_4$ (trevorite) produced at 700°C; the presence of 0.75 mol% of Li_2O decreased the crystallinity of $NiFe_2O_4$ formed at 700 and 800°C. Increasing the amount of Li_2O to 3.0 mol% effected an increase in the crystallinity of $NiFe_2O_4$ (trevorite) to a value near that observed for the pure mixed solid heated at 700°C. Increasing the precalcination temperature of the doped solid to 800°C resulted in (i) the appearance of new diffraction lines different from those relating to NiO, Fe_2O_3 and $NiFe_2O_4$; (ii) some changes in the degree of crystallinity of the produced $NiFe_2O_4$; (iii) new diffraction lines which appeared in the case of the mixed solid sample doped with 1.5 mol% of Li_2O but disappeared on increasing the Li_2O content to 3 mol%. The new diffraction lines are located at *d* spacings of 2.04, 2.36 and 1.48 Å, and are characteristic of a β-$LiFeO_2$ phase [21].

The presence of 0.75 mol% of Li_2O decreases slightly the crystallinity of the $NiFe_2O_4$ (trevorite) phase produced at 800°C. The crystallinity of this phase increases on raising the amount of Li_2O to 3 mol%. The formation of β-$LiFeO_2$ takes place according to

$$Fe_2O_3 + Li_2O \xrightarrow{800^\circ C} 2LiFeO_2(\beta\text{-})$$

The formation of the lithium ferrite phase and the modification in the crystallinity of $NiFe_2O_4$ produced at 800°C might indicate that a portion of the Li_2O has dissolved in the NiO lattice and the other portion has interacted with Fe_2O_3 to yield lithium ferrite. It is well known that NiO can dissolve up to 16 mol% of Li_2O via substitution of some of the host Ni^{2+} ions with subsequent formation of Ni^{3+} according to

$$Li_2O + 0.5O_2(g) \rightarrow 2Li(Ni^{2+}) + 2Ni^{3+}$$

where $Li(Ni^{2+})$ is a monovalent ion located in the position of a host (Ni^{2+}) ion of the NiO lattice. The incorporation of lithium ions according to this mechanism leads to an increase in the amount of excess oxygen with subsequent transformation of Ni^{2+} to Ni^{3+} [20].

The formation of $NiFe_2O_4$ takes place according to

$$NiO + Fe_2O_3 \xrightarrow{700,\ 800^\circ C} NiFe_2O_4$$

This solid-solid interaction requires the presence of nickel ions in the divalent state, and the transformation of some of the Ni^{2+} into Ni^{3+} by Li_2O doping is expected to decrease the number of divalent Ni^{2+} contributing to $NiFe_2O_4$ formation. Consequently, the presence of Li_2O might impede nickel ferrite formation. This speculation is experimentally verified in the case of the 0.75 mol% Li_2O-doped mixed solid. However, the increase in the amount of Li_2O to 3 mol% resulted in an increase in the crystallinity of the $NiFe_2O_4$ produced at 800°C (see Fig. 7), indicating an enhancement of $NiFe_2O_4$ formation. This anomaly could be resolved by assuming an induced increase in the mobility of Ni^{2+} ions in the presence of heavy doping (3 mol% Li_2O). Similar results have been reported by one of the authors [1].

The disappearance of the diffraction lines of β-$LiFeO_2$ in the case of the mixed solid doped with 3 mol% of Li_2O and preheated at 800°C might indicate its transformation into another lithium iron oxide phase [5] having characteristic diffraction lines common to NiO, Fe_2O_3 and $NiFe_2O_4$; these phases are α- and/or β-$LiFe_5O_8$ [22]. It seems that the increase in Li_2O content to 3 mol% results in enhanced dissolution of Li^+ ions in the NiO lattice, leaving a minor portion of free Li_2O that can interact with free iron oxide; these conditions favour the formation of $LiFe_5O_8$ which takes place according to the reaction $Li_2O + 5Fe_2O_3 \rightarrow 2LiFe_5O_8$.

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