

INVESTIGATION ON THE GARNIERITE AND LIMONITE MIXED LATERITE ORE FOR NICKEL RECOVERY

S.-W. Zhou ^{a,b}, C. Lu ^{a,b}, B. Zhang ^{a,b}, B. Li ^{a,b}, Y.-G. Wei ^{a,b,*}

^a Kunming University of Science and Technology, Engineering Research Center of Metallurgical Energy Conservation and Emission Reduction, Ministry of Education, Kunming, China

^b Kunming University of Science and Technology, National local Joint Engineering Research Center of Energy Saving and Environmental Protection Technology in Metallurgy and Chemical Engineering Industry, Kunming, China

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Abstract

Laterite, as an important resource of nickel, has become the focus of development and utilization. This study adopted the method of mixing ore (garnierite and limonite) to increase the recovery rate of nickel in garnierite ore. The phase transformation of the two ores was investigated during the heating process; dehydroxylation and recrystallization were observed and the iron oxides phase was finally transformed into the iron-containing spinel. By changing the proportion of the two ores, increasing the iron content in the sample was beneficial to the reduction of nickel. Then, Fe_2O_3 , Fe_3O_4 , and Fe were used as the iron source to study the mechanism of the iron-bearing minerals in promoting the reduction of nickel. The results indicated that hematite and/or magnetite would react with amorphous silicate minerals to generate magnesioferrite and enstatite, thereby avoiding additional forsterite generation; moreover, the trevorite phase would be formed, which strengthens the aggregation of nickel and iron.

Keywords: Garnierite ore; Limonite ore; Reduction; Phase transformation

1. Introduction

Nickel resources mainly involve two types of ore, including nickel sulphide ore and laterite ore. As nickel sulphide ore resources were mined continuously, its reserves decreased sharply. Plentiful laterite ores have attracted interest recently, and treatment processes have led to greater efforts to improve the recovery of nickel from laterite ore [1]. Laterite nickel ore, also known as nickel oxide ore, is formed by nickel, iron, and magnesium silicate olivine bedrock through long-term weathering and enrichment [2]. The top lateritic layer is commonly of a high laterisation degree of the limonite type, the lower portion is a garnierite type with lower laterisation degree, and the intermediate transition zone is a saprolitic type [3].

The laterite nickel ore is rich in reserves, but the complex ore phase and structure make it difficult for laterite to efficiently enrich nickel. Thus, many scholars studied the mechanism of laterite nickel ore dehydration and reduction of iron and nickel oxide under isothermal and non-isothermal conditions [4-8]. The phase transformation of laterite minerals (saprolite

and limonite) reduced at temperatures up to 800 °C and the phase reversibility with cooling were investigated, [9] indicating that dehydroxylation could release nickel which was embedded in main gangue minerals. An Australian garnieritic-type ore demonstrated that chlorite is converted into forsterite and enstatite at 700-800 °C with recrystallization between 800 and 850 °C [10]. However, the temperature of talc transformed to forsterite and enstatite reached 1000 °C. The microstructure and phase characterisations of laterite ore were investigated and the results show the nickel is mainly associated with goethite and serpentine particles, which altered factors that affect nickel recovery from a single phase (goethite or serpentine) [11, 12]. Nickel is difficult to be extracted from olivine, and nickel recovery is better for single-phase olivine with an Mg/Fe ratio < 8. Kawahara et al. [13] studied the reducibility of several types of laterite nickel ore and reported that the reducibility of nickel in ore increased with increasing iron concentration; poor reducibility of nickel in low-iron and/or high-magnesia laterites ore is attributed to magnesium replacement by nickel in the silicate lattice.

Aiming at the characteristic of difficult reduction

Corresponding author: weiygcp@aliyun.com

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and enrichment of the dispersive nickel phase in the ore, the low-nickel and low-iron garnierite was taken as the research object in the present study. The method of mixing ore (limonite-type laterite ore) was adopted to increase the iron content in the raw material for promoting the reduction of nickel. Based on the understanding of the phase transformation of the two ores during the high-temperature roasting process, a detailed investigation on the reduction roasting-magnetic separation of the mixed ore was carried out, and the interaction between the iron phase and nickel in the ore was clarified. The obtained results can clarify the mechanism of nickel phase reduction and enrichment in mixed ore.

2. Experimental

2.1. Materials

Two types of low-grade nickel laterite ore used in the study were supplied by the Yunnan province of China. The chemical compositions of the two laterite ores are listed in Table 1.

The garnierite contains low Ni (0.82 %) and Fe (9.67 %), and high SiO₂ (37.49 %) and MgO (31.49 %). The Ni and Fe contents in the limonite reach 1.17 and 35.71 %, respectively. The anthracite was used as a reductant, and the proximate analysis results are listed in Table 2, which shows the fixed carbon is 76.43 %.

2.2. Reduction roasting

Laterite ores were dried at 60 °C for 24 h, and then ground using an XZM-100 laboratory vibratory mill to 95 % passing 0.25 mm. The dried laterite ore was first mixed with the reductant, and then the sample was placed in a horizontal tube furnace and heated to the required temperature for a given duration under nitrogen atmosphere which was introduced at a flow rate of 30 mL/min. After the reduction, the roasted sample was cooled in the tube furnace to prevent re-oxidation. The cooled roasted ore was wet-ground in the above-mentioned vibratory mill at a pulp density of 50 % solids, and then the slurry was subjected to magnetic separation (DTCXG-ZN50 Magnetic Tube, at a magnetic-field intensity of 200 mT) to obtain the ferronickel concentrate.

2.3. Equipment

The X-ray diffraction (XRD) was adopted to analyse the phase of the laterite ores and roasted samples, which were obtained using a Japan Science

D/max-R diffractometer with Cu Ka radiation ($\lambda=1.5406 \text{ \AA}$), operating at 40 kV and 30 mA. Thermogravimetric (TG) and differential scanning calorimetry (DSC) was performed on a NETZSCH STA 449F3 unit under a nitrogen atmosphere, and the samples were heated from 20 to 1200 °C at a constant rate of 10 °C/min. The Scanning Electron Microscopy coupled with Energy Dispersive Spectrometer (SEM-EDS, HITACHI-S3400N) was used to analyse the microstructure of the roasted sample.

3. Results and Discussion

3.1. Nickel distribution

The distribution form of nickel in the ore determines the difficulty degree of its extraction. Low grade and complex embedding form are the fundamental reasons for the difficult treatment of laterite nickel ore. The distribution ratio of nickel in Ni-bearing minerals is listed in Table 3, the analysis methods was described in a previous report [14]. Nickel is mainly associated with silicates and oxides in two types of ores and accounted for 95.13 % of the garnierite and 96.75 % of the limonite. For limonite, approximately 69.35 % of Ni element is distributed in silicates, and this ratio reaches 84.15 % in garnierite ore. Compared with oxides, nickel is more difficult to reduce in silicates [12, 15].

3.2. Phase transformation of laterite ore during roasting

3.2.1. Phase transformation of garnierite ore

The TG/DSC curves of the raw garnierite ore are shown in Fig. 1, which indicated a weakly endothermic peak below 120 °C, corresponding to the removal of adsorbed water from the sample. An obvious endothermic peak located at 610 °C is attributed to dehydroxylation of serpentine, with a weight loss of 9.5 %. With the increasing of temperature, serpentine undergoes recrystallisation and transforms into forsterite and enstatite with a strong exothermic peak at approximately 820 °C.

To study the phase transformation of the nickel laterite ore during the roasting process, the ore samples were roasted for 60 minutes at the setting

Table 2. Proximate analysis of anthracite (mass%)

Properties	Fixed carbon	Volatile	Ash	Moisture
Content	76.43	7.78	15.29	1.02

Table 1. Chemical analysis of the laterite ore sample (mass%)

Sample	TFe	Ni	Co	MgO	SiO ₂	Al ₂ O ₃	CaO
Garnierite	09.67	0.82	0.033	31.49	37.37	1.89	0.033
Limonite	35.71	1.17	0.047	4.54	16.75	11.17	0.053



temperature. The XRD patterns of garnierite samples roasted at various temperatures are shown in Fig. 2.

The main minerals, which are evident from the XRD patterns of the garnierite raw ore, are lizardite $[(Mg,Fe)_3Si_2O_5(OH)_4]$, quartz (SiO_2), and maghemite ($\gamma\text{-Fe}_2O_3$). At 300 and 500 °C, part of the maghemite was transformed, generating a new phase hematite. At a roasting temperature up to 610 °C, the peak for lizardite completely disappeared, the peak intensity of hematite ($\alpha\text{-Fe}_2O_3$) increased gradually, without the appearance of the silicate mineral phase. In the combined thermogravimetric curves, serpentine underwent dehydroxylation and the crystal structure was broken. The serpentine was converted into amorphous silicate minerals; therefore, there is no

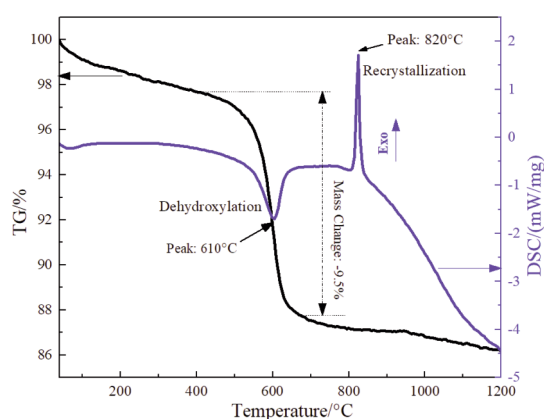
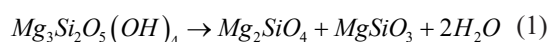


Figure 1. TG/DSC curves of the raw garnierite ore

peak of silicate minerals in the XRD patterns. At 700, 800, and 900 °C, the structure of silicate minerals transformed from non-crystalline to crystalline, and the new phases forsterite and enstatite were found. TG curve (Fig. 1) depicts a strong exothermic peak located at 820 °C, so the transformation from non-crystalline silicate minerals to forsterite (Mg_2SiO_4) and enstatite ($MgSiO_3$) is an exothermic process in accordance with Reaction (1) [16, 17].



As temperature increased to 1000 °C, hematite diffraction peak intensity was significantly reduced, and the new spinel phases were formed: trevorite ($NiFe_2O_4$) and magnesioferrite ($MgFe_2O_4$). Further increasing the temperature above 1200 °C, the diffraction peaks for hematite completely disappeared, and the trevorite peak intensity had an increase as a result.

3.2.2. Phase transformation of limonite ore

Similar tests were conducted for limonite ore, the TG/DSC curves of the raw limonite ore are presented in Fig. 3. Three main thermal effects were observed. The first endothermic peak at approximately 100 °C corresponded to the elimination of the absorbed moisture. The second endothermic peaks as shown at approximately 280 °C represented the dehydroxylation of goethite to form hematite, with a

Table 3. Distribution ration of nickel at different minerals in the laterite ore (mass%)

Sample	Existential Phases	Oxides	Silicates	Sulfides	Sulfates	Total
Garnierite	Grade	0.09	0.69	0.03	0.01	0.82
	Distribution ratio	10.98	84.15	3.65	1.22	100
Limonite	Grade	0.32	0.81	0.022	0.016	1.17
	Distribution ratio	27.40	69.35	1.88	1.37	100

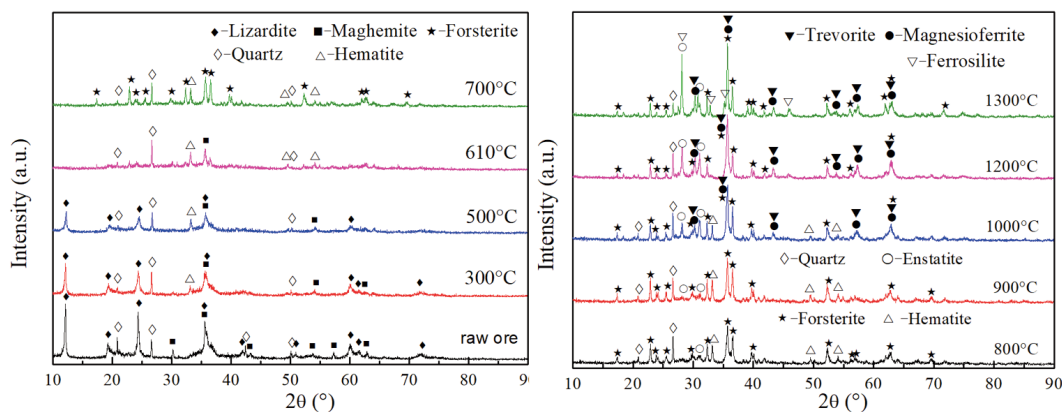
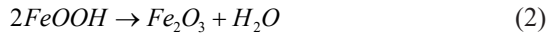


Figure 2. XRD patterns of the garnierite ore after roasting at different temperatures



weight loss of 4.7 %, in accordance with Reaction (2) [16, 18].



This decomposition temperature of goethite was low compared to the report, in which the decomposition temperature was 310 °C [19] or 330 °C [20]; this phenomenon may be attributed to the poor crystalline structure of the goethite in the ore. At approximately 850 °C, the third exothermic peak mainly represented the sintering of the particles, which generated $\alpha\text{-Fe}_2\text{O}_3$ particles with an irregular shape [21]. This peak indicated a recrystallisation phenomenon with no associated weight loss.

Fig. 4 shows that the main minerals of limonite raw ore are goethite (FeOOH) and maghemite. After roasting at 200 °C, the diffraction peak was basically identical to the raw ore, indicating that the ore phase did not change at this temperature. At a roasting temperature up to 300 °C, the goethite peak completely disappeared, and the hematite peak appeared simultaneously. This is consistent with the results of thermogravimetric analysis; namely, goethite is transformed to hematite at 280 °C. As the

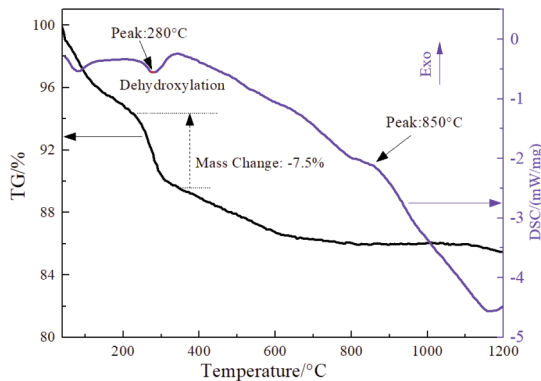


Figure 3. TG/DSC curves of the raw limonite ore

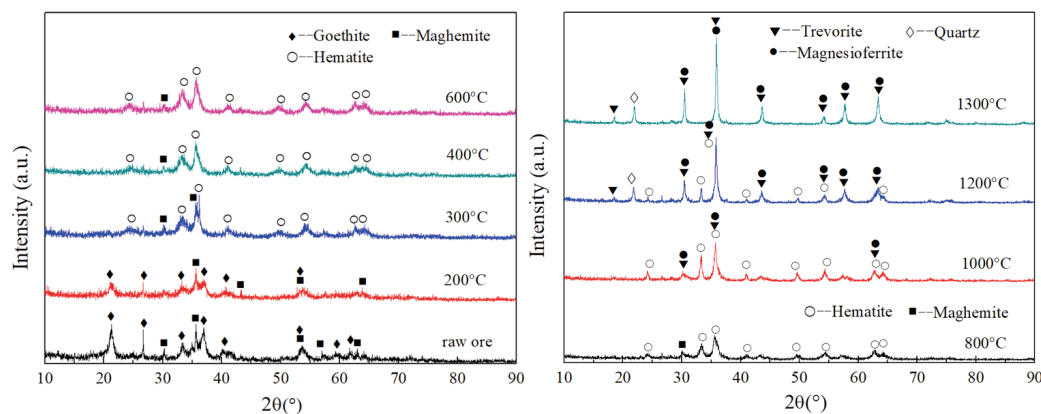


Figure 4. XRD patterns of the limonite ore after roasting at different temperatures. goethite (FeOOH); trevorite (NiFe_2O_4), magnesioferrite (MgFe_2O_4), hematite ($\alpha\text{-Fe}_2\text{O}_3$), maghemite ($\gamma\text{-Fe}_2\text{O}_3$), quartz (SiO_2)

temperature increased to 800 °C, the peak intensity of maghemite decreased, while the peak intensity of hematite had an increase. This finding indicates that the maghemite gradually converted into hematite, which is similar to the DSC curve (Fig. 4) through the appearance of the exothermic peak at 850 °C. As the temperature was increased to 1000 °C, the maghemite peak disappeared and the characteristic peaks of magnesioferrite (MgFe_2O_4) and trevorite (NiFe_2O_4) appeared. As the temperature was further increased, the hematite phase disappeared, and the magnesioferrite and trevorite peak strengthened.

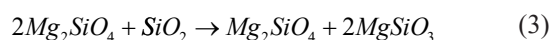
3.3. Effect of limonite content on garnierite ore reduction

The garnierite sample with various proportions of limonite ore (ranging from 0 to 80 %) was roasted at 1200 °C for 60 minutes. Coal dosages added in the sample were based on the C/O ratio; namely, the amount of coal addition in terms of the gram-atomic ratio of the fixed carbon in the coal added to the combined oxygen in nickel and iron oxides. Fig. 5 shows the magnetic separation results, indicating that as limonite ore increased to 60 %, the nickel recovery gradually improved from 30.52 % to 71.55 %; the corresponding iron recovery increased from 47.23 % to 81.72 %. Thereafter, the recoveries of nickel and iron remained stable. The experimental results demonstrated that the reduction rate of nickel in garnierite was promoted by the addition of limonite ore.

As analysed above, the main mineral phases of garnierite ore roasted at 1200 °C included forsterite, enstatite, magnesioferrite, trevorite, and quartz; limonite ore after roasting included hematite, trevorite, magnesioferrite, and quartz. Fig. 6 shows the XRD patterns of the mixture of the two ores after being roasted at 1200 °C.

The diffraction peak of forsterite in the roasted

sample reflected a weak intensity when adding 20 % of limonite. With increasing the limonite proportion, the forsterite peak gradually disappeared. This may be attributed to the formation of enstatite, as expressed in Reaction (3). However, the nickel element which was embedded in enstatite was easier to extract than nickel in the forsterite, [15] which could be a major reason for improving nickel recovery. In addition, the hematite peak was unobserved and the spinel peak intensity (maghemite and magnesioferrite) strengthened. The Ni-Fe alloy could be generated from the Fe-rich matrix [12]; thus, the formation of the iron-containing spinel may be beneficial to the reduction of nickel and improve the nickel recovery rate in the later process.



3.4. Reaction mechanism analysis

The iron in the limonite ore is mainly in the form of goethite and maghemite, and they would be converted into hematite as the temperature is

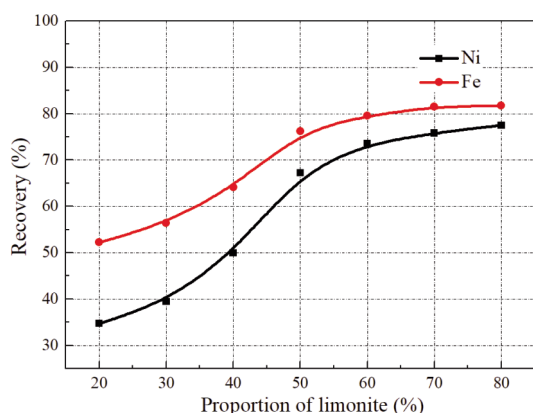


Figure 5. Effect of limonite ore proportion on the recovery of iron and nickel in mixed ore

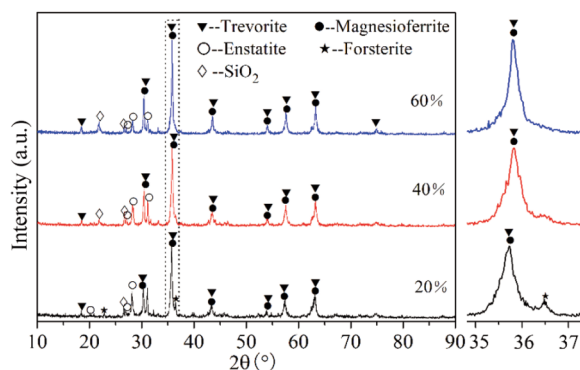


Figure 6. XRD patterns of mixed ore after roasting at 1200 °C for 60 minutes with different proportion of limonite ore proportion. Quartz (SiO_2), trevorite ($NiFe_2O_4$), magnesioferrite ($MgFe_2O_4$), enstatite ($MgSiO_3$), forsterite (Mg_2SiO_4)

increased. In order to investigate the effect of iron phase in mixed ore on nickel reduction, Fe_2O_3 , Fe_3O_4 , and Fe were used as iron source and added to the garnierite ore, respectively. The added quantities of Fe_2O_3 , Fe_3O_4 , and Fe are based on the Fe/Ni ratio, which was calculated from the composition of mixed ore (garnierite mixed with limonite in various proportions). Fig. 7 shows the results of nickel reduction rate in the sample (reduction at 1200 °C for 60 minutes) as a function of Fe/Ni ratio. With the addition of Fe_2O_3 , the metallization rate of nickel exhibited a gradual improvement from 75.0 to 83.0 % with increasing Fe/Ni ratio from 16.7 to 24.6. With the addition of Fe_3O_4 , as the Fe/Ni ratio increased from 16.7 to 24.6, the metallization rate of nickel improved from 74.3 to 76.5 % and stabilized thereafter. Fe_2O_3 and Fe_3O_4 reflect varying degrees of promoting the nickel oxide reduction in the sample. However, the metallization rate of nickel is stabilized at approximately 71 %, with the addition of metallic Fe, indicating that metallic iron did not obviously influence the nickel reduction.

The XRD patterns of the mixture (garnierite ore and Fe_2O_3) with various Fe/Ni ratios after roasting at 1200 °C are shown in Fig. 8. Without adding Fe_2O_3 , the pattern (Fig. 8a) exhibited a high intensity of forsterite diffraction peak. As Fe_2O_3 dosage increased, the forsterite diffraction peak intensity was significantly reduced, and the intensity of the enstatite and spinel (trevorite and magnesioferrite) diffraction peak increased accordingly. The phase for the Fe_2O_3 did not appear in the XRD analysis results.

Combining the XRD analysis results in Fig. 2, for the garnierite roasted at different temperatures, the peak intensity of hematite was significantly reduced and finally disappeared, and the intensity of enstatite and spinel (trevorite and magnesioferrite) diffraction peaks accordingly appeared when roasting temperature was above 900 °C, illustrating

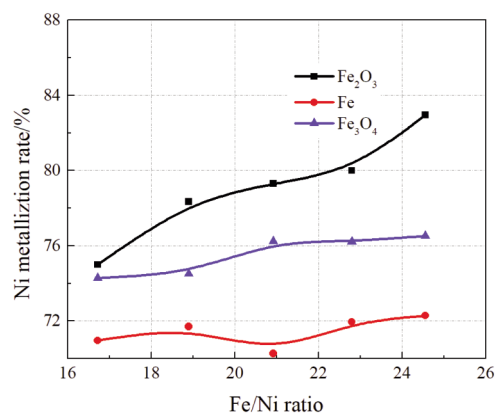
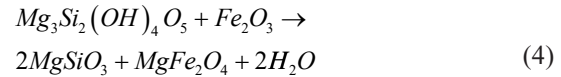


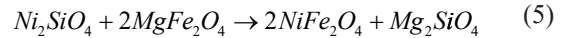
Figure 7. Effect of different Fe/Ni ratio on the nickel metallization rate

that hematite reacts with magnesium-bearing minerals and/or nickel-bearing minerals to generate trevorite and magnesioferrite. Silicate minerals are the nickel and magnesium host phases. From the viewpoint of thermodynamics, it is difficult to generate the spinel phase by the reaction of hematite with forsterite and/or enstatite. The formation of magnesioferrite is mainly due to the reaction between hematite and amorphous silicate minerals produced by serpentine dehydroxylation. This process can be expressed by Reaction (4), which avoids the generation of olivine and embeds nickel in enstatite. The newly generated magnesioferrite reacts with nickel silicate to form trevorite, in accordance with Reaction (5), and nickel in trevorite is more easy to reduce. Furthermore, nickel oxide would react with hematite to form trevorite, as in Reaction (6). Figure 7 shows that Fe_3O_4 does influence the nickel metallization rate, but the promotional role of Fe_2O_3 is stronger under the same Fe/Ni ratio conditions. The addition of Fe_3O_4 does

not prevent the generation of olivine, but instead generates magnesioferrite, in accordance with Reaction (7) and (8).



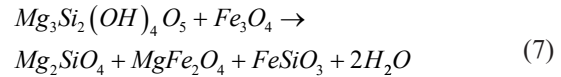
$$\Delta G^\theta = 153.667 - 0.287TkJ/mol$$



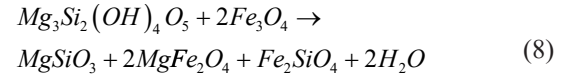
$$\Delta G^\theta = -47.366 - 0.032TkJ/mol$$



$$\Delta G^\theta = -17.596 - 0.0025TkJ/mol$$



$$\Delta G^\theta = 211.029 - 0.271TkJ/mol$$



$$\Delta G^\theta = 197.063 - 0.246TkJ/mol$$

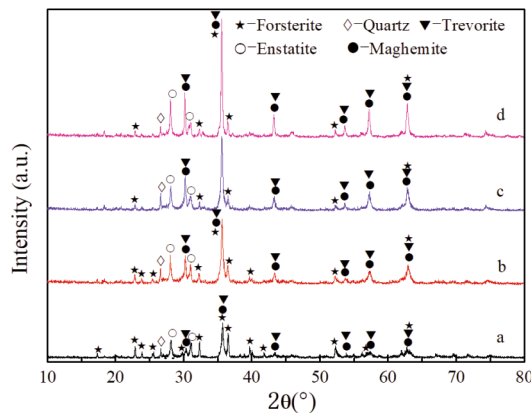


Figure 8. XRD patterns of the mixture (garnierite ore and Fe_2O_3) with different Fe/Ni ratio after roasting at $1200^\circ C$ for 60 minutes. a: Fe/Ni=11.79; b: Fe/Ni=16.72; c: Fe/Ni=20.92; d: Fe/Ni=24.56; quartz (SiO_2), trevorite ($NiFe_2O_4$), magnesioferrite ($MgFe_2O_4$), enstatite ($MgSiO_3$), forsterite (Mg_2SiO_4)

Figure 9 presents SEM images and EDS spectra of the reduction roasted mixture (garnierite ore and Fe_2O_3) at $1200^\circ C$ for 60 minutes with Fe/Ni=24.6. The SEM-EDS results clearly clarified the metallic iron in the sample (point 1), which is the reduced product of added Fe_2O_3 and attached with ferronickel particles (point 2). The reaction product of trevorite is a continuous series of metallic solid solutions.[22] Therefore, it is demonstrated that nickel-bearing minerals are transformed to $NiFe_2O_4$ before reduction, and the formation of trevorite strengthens the aggregation of nickel and iron.

Based on the above analyses, the reduction of nickel in the garnierite promoted by hematite [Fe_2O_3] can be described as follow: Fe_2O_3 first reacts with amorphous silicate minerals which are produced by serpentine dehydroxylation, to avoid the generation of olivine; then, the newly generated magnesioferrite reacts with nickel silicate (nickel-containing enstatite) to produce trevorite that is easier to reduce.

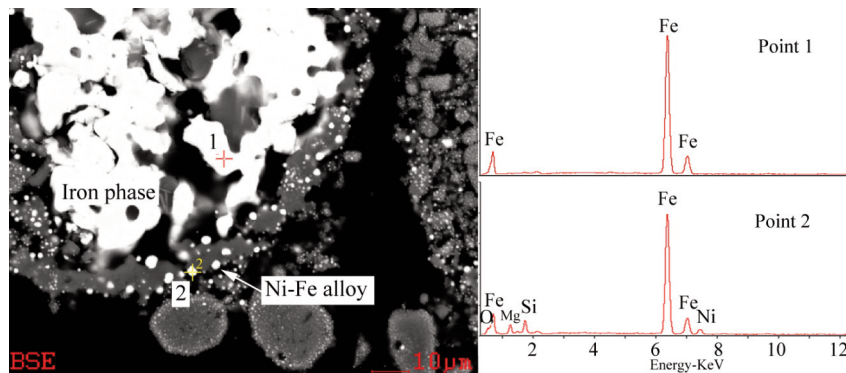


Figure 9. SEM/EDS analyses of reduction roasted mixture (garnierite ore and Fe_2O_3) at $1200^\circ C$ for 60 minutes with Fe/Ni=24.6

4. Conclusions

(1) With increasing the temperature, lizardite within garnierite undergoes dehydroxylation and recrystallization at 610 and 820 °C, respectively. Limonite mainly consists of goethite and maghemite phase. The dehydroxylation of goethite to form hematite occurs at approximately 280 °C; while maghemite is gradually converted into hematite at roasting temperature below 800 °C. Hematite would begin to transform to iron-containing spinel above 1000 °C.

(2) The addition of limonite is beneficial to the reduction nickel within garnierite. As limonite ore increases from 20 to 60 %, nickel recovery gradually improves from 30.5 to 71.6 %.

(3) Hematite [Fe₂O₃] reacts with amorphous silicate minerals produced by serpentine dehydroxylation to avoid the generation of olivine, embedding nickel in enstatite. The newly generated magnesioferrite also reacts with nickel silicate to produce a more easily reduced trevorite, which strengthens the aggregation of nickel and iron.

Acknowledgements

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ISTRAŽIVANJE GARNIJERITSKO-LIMONITSKIH LATERITSKIH RUDA ZA ISKORIŠĆENJE NIKLA

S.-W. Zhou ^{a,b}, C. Lu ^{a,b}, B. Zhang ^{a,b}, B. Li ^{a,b}, Y.-G. Wei ^{a,b,*}

^a Univerzitet nauke i tehnologije Kunming, Inženjerski istraživački centar za uštedu energije i smanjenje emisije, Ministarstvo obrazovanja, Kunming, Kina

^b Univerzitet nauke i tehnologije Kunming, Zdrženi nacionalni i lokalni istraživački centar za uštedu energije i tehnologiju zaštite životne sredine u metalurškoj i hemijskoj industriji, Kunming, Kina

Apstrakt

Laterit, kao važan resurs nikla, u fokusu je razvoja i primene. U ovom istraživanju prihvaćen je metod mešanja rude (garnijerita i limonita) da bi se povećalo iskorišćenje nikla iz rude garnijerita. Ispitivana je fazna transformacija dve rude tokom procesa zagrevanja; primećene su dehidroksilacija i rekristalizacija, i faza oksida železa je konačno transformisana u spinel sa sadržajem železa. Menjanjem odnosa dve rude dolazilo je do povećanja sadržaja železa u uzorku, što je bilo korisno za redukciju nikla. Zatim, Fe_2O_3 , Fe_3O_4 , i Fe su korišćeni kao izvori železa da bi se proučio mehanizam minerala bogatih železom koji utiču na redukciju nikla. Rezultati su pokazali da hematit i/ili magnetit reaguju sa amorfnim silikatnim mineralima i generišu magnezioferit i enstatit, i na taj način se izbegava stvaranje dodatnog forsterita; štaviše, formira se faza trevorita, što ojačava agregaciju nikla i železa.

Ključne reči: Ruda garnijerita; Ruda limonita; Redukcija; Fazna transformacija

