



Ferronickel particle formation during the carbothermic reduction of a limonitic laterite ore



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ABSTRACT

Attempts to economically extract nickel from the nickeliferous laterite ores face a number of challenges, including a limited ability to beneficiate the ore prior to processing. One method to address this issue would be to reduce the ore at moderate temperatures to form a ferronickel alloy, which could then be selectively recovered to form a nickel concentrate. The current work explores the effects of varying coal, elemental sulphur and pyrite additions and temperatures in the range of 1000–1200 °C on the formation of ferronickel alloy particles in a limonitic ore. It was observed that a small sulphur addition ($\leq 4\%$) yielded a significant increase in the average size of the ferronickel particles formed during reduction. A decrease in the average nickel grade of the ferronickel particles, resulting from the increased metallization of iron, was also observed in the presence of sulphur. The ferronickel particle morphology and distribution of sulphur in the reduced ore are consistent with a liquid-phase growth mechanism driven by the Fe–FeS eutectic at 988 °C.

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1. Introduction

Increasing attention is being paid to nickeliferous laterite ores as an alternative to nickel sulphide ores as a feed-stock for nickel metal production. These oxidic laterite ores account for up to 72% of global nickel resources and, unlike the nickel sulphide deposits which typically occur at depth, laterite ore deposits are located near the surface, allowing them to be more readily accessed (Dalvi et al., 2004). However, the processing of nickel laterite ores poses several challenges. The nickel content of these ores is in solid solution with hydrated iron oxides and silicate minerals, limiting the application of physical beneficiation methods to the removal of only the coarse, low-nickel high-silica fraction of the ore (Quast et al., 2015). Combined with the typically low nickel grades in these laterite ores (0.5–2%), large volumes of ore must be processed.

Current methods of processing laterite ores typically require either acid leaching, followed by electrowinning and further refining, or direct smelting of the majority of the mined ore. Both approaches are capital intensive and typically have higher net operating costs than similar nickel sulphide operations (Mitchell, 2015). Further, it has been demonstrated that the processing of nickel laterites is significantly less energy efficient than the processing of nickel sulphides (Domínguez et al., 2013) and it has

been estimated that processing of nickel laterites may have a significantly more negative environmental impact than the processing of sulphides (Mudd, 2010). To address these issues, it would be highly desirable to beneficiate the ore prior to processing.

One proposed method of concentrating the nickel content of these laterite ores is the selective reduction of the contained metal oxides at moderate temperatures to form a ferronickel alloy, which may then be separated from the gangue by magnetic separation, leaching, or other methods. The objective of such a strategy is to form a high nickel grade ferronickel suitable for further processing using existing infrastructure, and a reduction in the ore volume for subsequent processing to achieve a net savings on processing costs. The effectiveness of this strategy will largely depend on the separation efficiency of the process to recover the ferronickel from the reduced ore. This separation efficiency will in turn be a function of the nickel grade in the ferronickel, extent of nickel metallization (recovery of nickel to the alloy phase), and the size of the ferronickel particles formed. As in conventional mineral processing applications, it would be expected that the larger the particle size of the material of interest, the more easily it will be separated and recovered. Coarser particles may be more readily liberated from the surrounding gangue, and exhibit a higher separation efficiency in a variety of beneficiation methods, such as density separation or wet magnetic separation. This has been demonstrated in practice for the reduction roasting and magnetic separation of a laterite ore (Li et al., 2012).

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1.1. Prior experimental work

Several previous studies have explored this selective reduction methodology, with magnetic separation employed to recover the ferronickel produced (Elliott et al., 2015b; Jiang et al., 2013; Li et al., 2012, 2011; Liu et al., 2014; Zhu et al., 2012). The most promising results (i.e. the highest nickel grades and recoveries in the magnetic concentrate) correlate with the use of reduction conditions, which would produce the largest ferronickel particles, although none of these experimental works reported on the morphology of the ferronickel phase (Elliott et al., 2015a). Increased reduction temperatures and durations have been shown to result in larger ferronickel particles (Elliott et al., 2015b; Zhu et al., 2012). The addition of sulphur and fluxing agents, such as sodium sulphide, have also resulted in an increase in the size of ferronickel particles formed (Harris, 2012). The highest nickel concentrate grade (9.87%) reported in the literature for this methodology employed a reduction temperatures of 1200 °C, a reduction duration of 50 min, and a sodium sulphate addition of 10 wt% (Jiang et al., 2013).

While operating at high reduction temperatures for prolonged periods may promote larger ferronickel particles, it also directly counteracts the objective of implementing a selective reduction and concentration step (achieving a net energy and cost reduction). It has also been noted that operating at temperatures above 1000 °C will lead to sintering of the products, hindering subsequent recovery of the ferronickel (Tang et al., 2014). Thus, the role of sulphur and fluxing agents in the production of a high grade nickel concentrate at lower temperatures is of particular interest.

Any advantages in the pre-concentration of the laterite ore resulting from a sulphur addition will be partially counteracted by the increased sulphur content of the alloy. One of the primary applications of nickel is in the production of stainless steel, where trace concentrations of sulphur will negatively impact the mechanical properties of the final product. Therefore, it is desirable to use the minimum sulphur addition to achieve the maximum improvement in the nickel grade and recovery in the ferronickel.

1.2. Effect of sulphur on ferronickel particle formation

The mechanism by which the addition of sulphur affects the formation of ferronickel particles during the reduction roasting of a laterite ore has been thoroughly described by Rao et al. (2016). During a reduction roast of a limonitic laterite in the presence of sulphur, three major phases will be formed: metal oxides, metal sulphides, and a ferronickel alloy. Given the high iron grade (>50%) and low nickel grade (<2%) in limonite ores, the behaviour of these phases can be approximated as an Fe–O–S system. It is the interaction of the FeS and metallic Fe formed during the reduction roast in particular that is the driving force for the enhanced growth of ferronickel particles in the presence of sulphur. The binary Fe–S system exhibits an Fe–FeS eutectic at 988 °C, allowing for the formation of a liquid phase at a relatively low temperature. Provided the reduction temperature is higher than this eutectic, an iron saturated melt will exist at the interface between the ferronickel alloy particles and the surrounding metal sulphides. The presence of this phase causes liquid phase sintering of particles through capillary action between neighbouring particles, facilitating the agglomeration of the smaller particles. Rao et al. (2016) also noted that in this liquid phase, Ni, Fe, Ni₃S₂ and FeS will be completely miscible, allowing for transport of both nickel and iron through the liquid phase by capillary action. As the mixture cools, iron and nickel metal will precipitate first, forming a solid ferronickel alloy phase, followed by the precipitation of the remaining metal sulphides.

The sulphidation of a limonitic nickel laterite in the absence of a reductant has previously been studied by Harris (2012), including a thermogravimetric (TGA) study, in which liquid formation was observed at 950 °C, providing evidence for the formation of a liquid Fe–Ni–O–S phase. Harris reported that the growth of mixed iron and nickel sulphide particles occurred via a liquid phase mechanism, with transport driven by strong capillary forces and the precipitation of submicron ferronickel particles from a sulphur deficient matte phase as the ore mixture cooled. It was also noted that higher temperatures and longer reaction times yielded larger sulphide particles.

In a similar TGA/DTA study conducted on the same material in the presence of a carbon addition the change in sample heating rate as a result of melting was obscured by the reduction of iron oxides between 800 and 1000 °C. However, subsequent microscopic analysis of the formed ferronickel particles showed particle morphology consistent with growth from a liquid phase, as well as increasing ferronickel particle size with increasing temperature (Elliott et al., 2015b). These observations are in agreement with the proposed mechanism by which the presence of sulphur enhances the growth of the ferronickel particles.

In the present work, the residual sulphur content of reduced ore samples was used to deduce the iron sulphide content of the ore. At the reduction temperatures considered here, sulphur will only be stable as either a metal sulphide or combined with organic hydrocarbons (Gryglewicz and Jasienko, 1992). Given the conditions employed, it is assumed that the amount of organic sulphur was negligible. Based on the mechanism outlined by Rao et al., it was expected that increased iron sulphide content will result in increased liquid formation therefore larger ferronickel particles.

The present work aimed to further investigate the mechanism behind the growth of the ferronickel particles during the carbothermic reduction of a limonitic laterite ore. Of particular interest were the effects of the amount and mode of sulphur addition to the ore on the size and nickel grade of the ferronickel particles formed. Both elemental sulphur and pyrite were investigated as methods of forming a stable sulphide phase in the ore, thereby generating the desired eutectic melting behaviour at the Fe–FeS interface.

2. Experimental

2.1. Raw materials

The ore used in this study was provided by the Ivory Coast operations of Xstrata. Table 1 provides the ore composition as determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analysis and X-ray Diffraction (XRD) analysis, which showed goethite as the only identifiable mineral present. The ore composition is given on a dry, calcined basis as all chemically bound water was removed prior to the reaction temperatures being reached (Elliott et al., 2015b).

Bituminous coal from Ward's Science was used as the reductant; the composition of the coal used is given in Table 2. Reagent grade elemental sulphur was from Fischer Scientific. Pyrite was ground from a mineral sample obtained from Ward's Science. Both the coal and the pyrite were ground to –200 mesh for use in briquetting.

2.2. Reduction tests

The as-received ore was dried in an oven at 100 °C for 24 h to remove free water, then ground to –100 mesh and stored in an airtight container at room temperature. The limonitic ore, bituminous coal and elemental sulphur or pyrite were mechanically mixed in

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