

The non-oxidative dissolution of nickel concentrate in aqueous acidic solutions

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SYNOPSIS

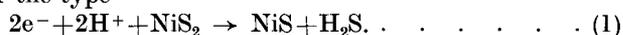
The kinetics of the reactions involved in the non-oxidative dissolution of nickel concentrate in hydrochloric acid were studied by electrochemical and powder-leaching methods. It was found that the rate of non-oxidative dissolution increases with a cathodic shift in the mixed potential, which can be achieved by an increase in the nickel-to-sulphur stoichiometric ratio at the reaction surface. An unactivated concentrate with a sulphur content of 36 per cent exhibited a mixed potential of +0,3V (SCE) and could not be leached satisfactorily in 3 M hydrochloric acid at 60°C. When activated material (18 per cent sulphur) was leached, it had a mixed potential of -0,3V, and high iron and nickel extraction rates. Results reported by other workers on the non-oxidative dissolution of sulphides are explained in terms of the mixed potentials at which dissolution occurs.

SAMEVATTING

Die kinetika van die reaksies betrokke by die nie-oksidatiewe oplossing van nikkelkonsentraat in soutsuur is met electrochemiese en poeierloog-metodes bestudeer. Daar is gevind dat die tempo van nie-oksidatiewe oplossing toeneem met 'n katodiese verskuiwing in die gemengde potensiaal wat bewerkstellig kan word deur 'n verhoging van die stoïgiometriese verhouding van die nikkel tot die swavel by die reaksieoppervlak. 'n Ongeaktiveerde konsentraat met 'n swavelinhoud van 36 persent het 'n gemengde potensiaal van +0,3V (VKE) gehad en kon nie bevredigend by 60°C in 3M soutsuur geloog word nie. Toe geaktiveerde materiaal (18 persent swavel) geloog is, het dit 'n gemengde potensiaal van -0,3V en hoë yster- en nikkelekstraksie-tempo's gehad. Die resultate wat aangegee word deur ander persone wat aan die nie-oksidatiewe oplossing van sulfiede werk, word in terme van die gemengde potensiale waarby die oplossing plaasvind verklaar.

Introduction

The kinetic reactions involved in the non-oxidative dissolution of synthetic nickel sulphides in hydrochloric acid have already been reported¹, and the dissolution of nickel matte is to be described in a future paper. These reaction rates were found to depend on the nickel-to-sulphur stoichiometric ratio of the reacting sulphide, which was interdependent with the potential assumed by, or applied to, the sulphide. Under oxidizing conditions (potential greater than -0,1 V), a metal-deficient sulphide surface was formed on all the nickel sulphides investigated, and non-oxidative dissolution proceeded slowly. At cathodic potentials (less than -0,4 V), sulphides of metal-rich bulk stoichiometry reacted less rapidly than at slightly more anodic potentials, because of the formation of a metal-rich surface. However, metal-deficient sulphides (e.g., nickel sulphide) dissolved readily at more cathodic potentials owing to reductions of the type



The reaction rates of these materials increased with cathodic shift in potential until the potential was -0,5 V, which is the limiting potential for the study described here. Thus, the sulphides can be divided into two groups. The first group consists of those sulphides for which the maximum non-oxidative dissolution rate is anodic of the natural mixed potential that they assumed on dissolution. These materials dissolve more rapidly under slightly oxidizing conditions, and are characterized by metal-rich stoichiometry. Nickel matte, which contains free nickel metal, falls into this category. The second group, which includes pyrite and nickel sulphide, consists of those sulphides that react more rapidly as the potential is shifted cathodically.

The non-oxidative dissolution of a nickel concentrate has been studied by Dyson² and Van Weert *et al.*³. The latter workers reported that, at 60°C, 90 per cent of the iron and 10 to 40 per cent of the nickel was extracted from a pyrrhotite concentrate in periods of around 3 hours. When the concentrate was leached in dilute hydrochloric acid, there was an incubation period, during which no hydrogen sulphide was observed. This incubation period was correlated with the potential of the leaching medium, which shifted from +300 to 0mV, at which point the evolution of hydrogen sulphide began. The cathodic shift in potential was attributed to the reduction of the iron(III) that had been formed by the dissolution of the magnetite present in the concentrate. Dyson² obtained poor nickel extractions when he reacted untreated nickel concentrate with hydrochloric acid. However, when the concentrate was pretreated with a reducing gas at around 750°C for the removal of excess sulphur, the concentrate was readily leached. The comparative non-reactivity of the non-activated concentrate was attributed to the formation of an inhibiting sulphur layer round the unreacted particles. This explanation was also used by Van Weert *et al.*³ and Ingraham *et al.*⁴ to explain similar results in which the dissolution of metal sulphide in acid was very slow. An alternative explanation for this behaviour is proposed later in this paper.

The aim of the work described here was the investigation of the non-oxidative dissolution of a nickel concentrate so that the kinetics and mechanism of metal extraction could be determined. The treatment of the concentrate by a matte-leaching process similar to that used by Falconbridge⁵ was also considered.

Experimental Method

Technique

Electrodes were constructed from both untreated nickel concentrate and activated nickel concentrate as

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follows. The concentrate was intimately mixed with Teflon powder, and the mixture was compressed at a pressure of 300 MPa. The pellet was sealed in a Teflon holder, and was used with the rotating-disc assembly described previously¹. The electrode of unactivated concentrate had a resistance of 16 k Ω , which made the measurement of current-potential curves unpractical. However, the electrode of activated concentrate, which had a low resistance, was satisfactory for the study of normal current-potential behaviour. Mineral electrodes were constructed from core-drilled samples of bulk pentlandite, chalcopyrite, pyrite, and nickeliferous pyrrhotite. The experimental techniques were the same as those described previously¹.

Leaching was carried out in the glass apparatus described previously.¹ The potentials of the resulting slurry were measured at a platinum-plate electrode against a saturated-calomel electrode, and are quoted as such.

Materials

The flotation concentrate used in this study was produced from a low-grade South African nickel-copper ore. Chemical and mineralogical analyses of the material are given in Table I. The concentrate was activated in 10 g batches at 850°C in a silica tube with hydrogen as the reducing gas. A loss in mass of 10 to 15 per cent was recorded. The amount of sulphur remaining in the activated concentrate was dependent on the original sulphur content of the nickel concentrate and on the length of time for which the material was held at 850°C. Analyses of the batch of activated concentrate that was used in the electrodes are shown in Table I. Other batches of concentrate were activated for different periods to produce concentrates of different sulphur contents.

The samples used as electrodes were analysed by X-ray-diffraction and electron-microprobe analysis. The chalcopyrite and pyrite were relatively pure (more than 95 per cent), and the pyrrhotite had a pentlandite content of 10 per cent. The pyrrhotite fraction showed a metal-to-sulphur stoichiometric ratio of $M_{0,88}S$. The major impurity in the pentlandite was nickeliferous pyrrhotite, at a concentration of 20 per cent.

TABLE I
ANALYSIS OF CONCENTRATES

Element	Amount in concentrate %	Amount in activated concentrate %
Ni	7,5	7,9
Cu	11,6	12,2
Fe	25,8	27,5
S	26,9	22,9
Ni + Cu + Fe (M)	$M_{0,91}S$	$M_{1,15}S$

The principal sulphide minerals present in the materials were as follows, in decreasing order of abundance: Concentrate: pyrite, pyrrhotite, chalcopyrite and pentlandite. Activated concentrate: troilite, bornite, pentlandite, chalcocite and pyrite.

TABLE II

EFFECT OF NICKEL METAL AND HYDROGEN SULPHIDE ON THE RATE OF DISSOLUTION OF THE ELECTRODES

Contact with nickel metal	$10^1 k^*_{Ni}$ mol.cm ⁻² .s ⁻¹	$10^1 k^*_{Fe}$ mol.cm ⁻² .s ⁻¹	E_{OC} V(SCE)
Activated concentrate	61(9)	270(73)	-0,30(-0,01)
Untreated concentrate	290(6)	41(11)	-0,29(0,05)
$10^{-3} M H_2S$ in solution			
Activated concentrate	12(32)	50(210)	-0,02(-0,12)
Untreated concentrate	19(11)	83(73)	0,05(0,05)

Conditions 3 M HCl, 60°C, 1 500 r/min

Figures in parentheses indicate dissolution rates in the absence of nickel metal or hydrogen sulphides.

Results and Discussion

Bulk Electrode Studies

Because the electrode made from unactivated nickel concentrate had a resistance of 16 k Ω , the effect of potential on the dissolution rate of the concentrate could not be studied. The activated concentrate showed an increase in the rate of nickel and iron dissolution with cathodic shift in potential (Fig. 1). This behaviour is similar to that found for metal-deficient iron and nickel sulphides¹. Thus, the activated concentrate belongs in the second of the two groups described in the introduction to this paper. Since the unactivated nickel concentrate is even more metal-deficient than the activated material, it is reasonable to assume that it will also react more rapidly at increasingly cathodic potentials. Thus, any alteration to the non-oxidative dissolution system that shifts the mixed potential of the reaction to a more cathodic value will result in a more rapid dissolution rate. That Van Weert *et al.*³ should have correlated potential with the evolution of hydrogen sulphide can be explained in terms of this hypothesis. While magnetite is present on the mineral surface or iron(III) is present in solution, the mixed potential at the sulphide surface will be sufficiently oxidizing to inhibit non-oxidative dissolution, but, as the magnetite and iron(III) are removed from the system, the potential will shift cathodically and hydrogen sulphide will start evolving rapidly.

Open-circuit potentials and reaction rates for the dissolution of activated and non-activated concentrate are given in Table II.

The results obtained under open-circuit conditions were dependent on the particular surface that was exposed, probably because of the non-homogeneity of the disc. However, when the same electrode, polished lightly between experiments, was used, a reproducibility of about 30 per cent could be obtained. The different values for the open-circuit dissolution rates of the concentrates reflect completely different electrode surfaces. However, as Table II shows, the rates of dissolution of both nickel and iron are faster, and the open-circuit potentials more cathodic, for the activated concentrate than for the unactivated material in the absence of added nickel metal or hydrogen sulphide. This can be

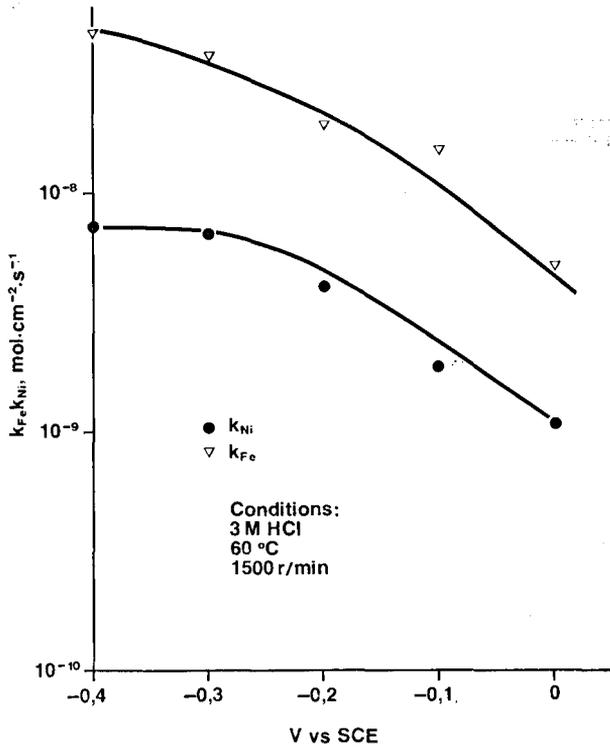


Fig. 1 Dependence of the non-oxidative dissolution rate of an activated concentrate on potential

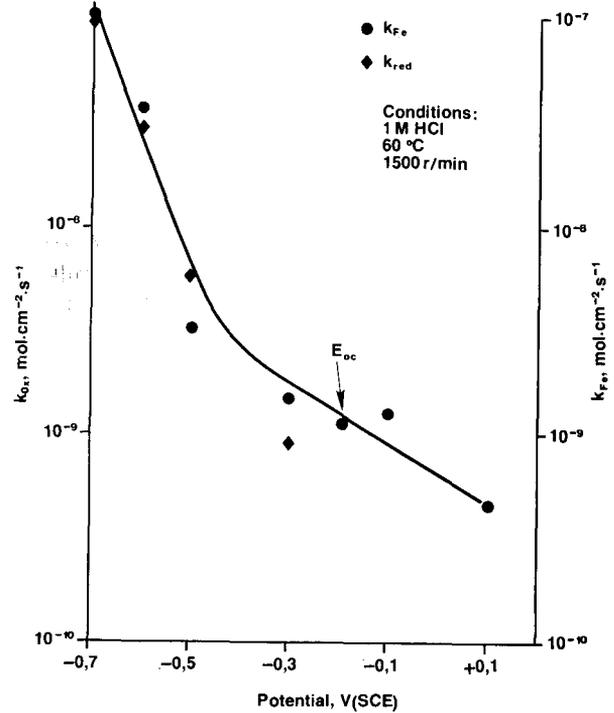


Fig. 3—The effect of potential on the rate of non-oxidative dissolution of pyrite

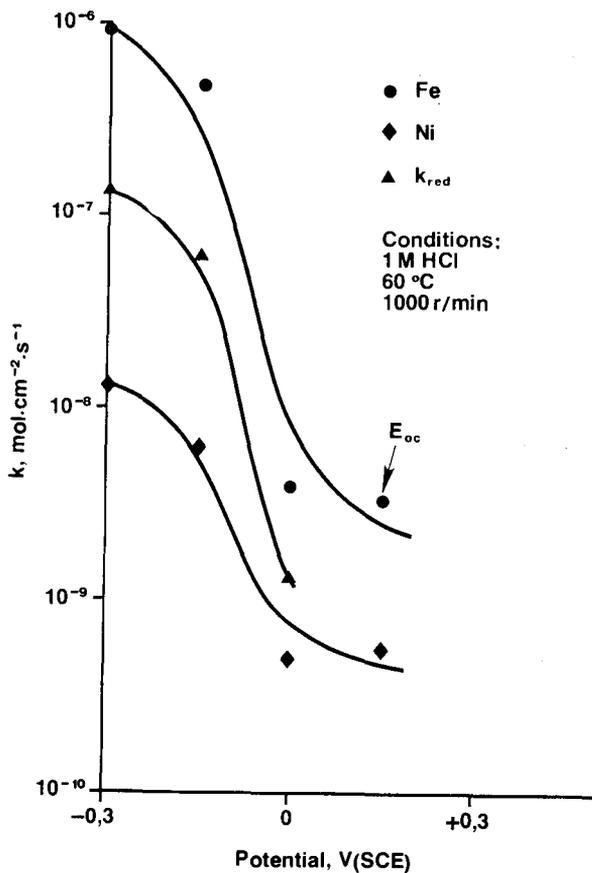


Fig. 2—The effect of potential on the rate of non-oxidative dissolution of pyrrhotite

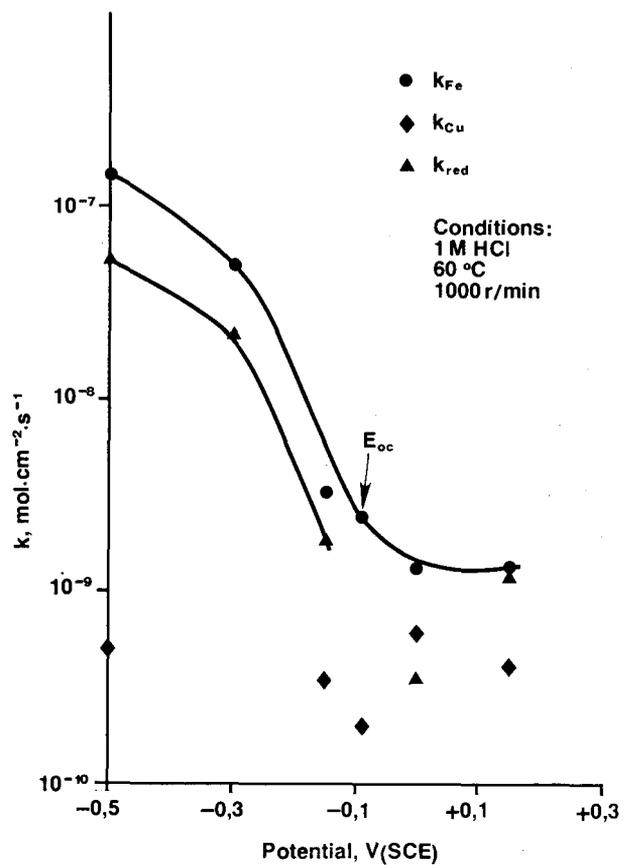
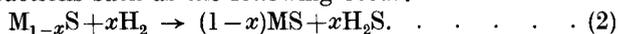
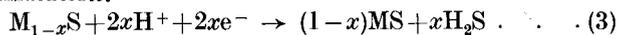


Fig. 4—The effect of potential on the rate of non-oxidative dissolution of chalcopyrite

attributed to the relative sulphur contents of the two materials. When the concentrate is activated at 850°C, reactions such as the following occur:

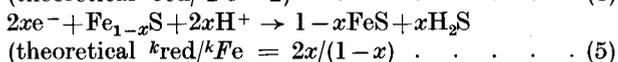
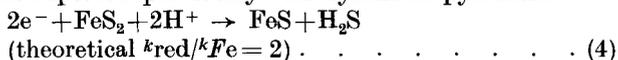


This can be considered to be the same as electrochemical reduction of the sulphide surface prior to non-oxidative dissolution:



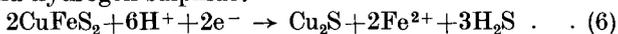
Such a reduction will occur at potentials cathodic to the natural mixed potential, and hence activation is the equivalent of shifting the reaction potential to a more cathodic value. As shown in Fig. 1, this will, in turn, result in a faster rate of non-oxidative dissolution of the concentrate.

Since an electrode of satisfactory conductivity could not be constructed from the concentrate, the potential dependence of the rate of dissolution of the individual minerals contained in the concentrate was investigated. Electrodes were prepared from mineral specimens of pyrite, pentlandite, nickeliferous pyrrhotite, and chalcopyrite. The effect of potential on the rate of non-oxidative dissolution and on the resultant current (on the assumption of a two-electron process) of each of these minerals is illustrated in Figs. 2 to 5. For each mineral, the rate of non-oxidative dissolution increased as the potential was shifted cathodic to the open-circuit potential. Thus, the rate-determining step in the dissolution of the minerals is the reduction of the mineral to remove an excess of sulphur present at the mineral surface. Once this has been achieved, the rate of non-oxidative dissolution is fast. For pyrite ($k_{red}/k_{Fe} \approx 2$) and pyrrhotite ($k_{red}/k_{Fe} \approx 0.3$), the ratio of reduction current to iron-dissolution rate indicates that these species are reduced to stoichiometric iron sulphide (troilite) prior to dissolution, as shown in equations (4) and (5). Troilite then dissolves rapidly to yield iron(II) and hydrogen sulphide. This phenomenon was reported previously⁶ for synthetic pyrrhotite.



As the activation of the concentrate by reaction with hydrogen at elevated temperatures converts pyrite and pyrrhotite to troilite, this is the equivalent of the application of a cathodic potential during the dissolution. Hence, for similar reasons to those illustrated electrochemically, the rate of leaching of the iron sulphides is enhanced by activation.

The rate of iron dissolution from chalcopyrite is increased when a cathodic potential is applied to the electrode. Chalcopyrite is reduced to yield chalcocite and hydrogen sulphide:



Copper is not dissolved owing to its insolubility in the presence of hydrogen sulphide. The mixed potential recorded during the dissolution of the concentrate was not sufficiently cathodic for reaction (6) to occur. Activation of the concentrate at high temperature, however, converted the copper minerals to chalcocite and bornite, thus liberating the iron for subsequent dissolution.

The rate of nickel and iron dissolution from pentlan-

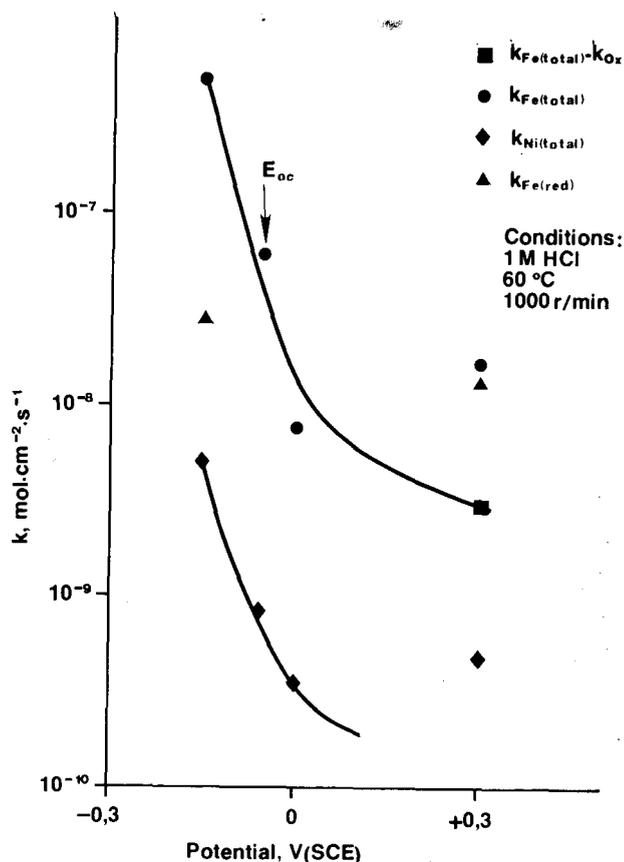


Fig. 5—The effect of potential on the rate of non-oxidative dissolution of pentlandite

dite increases with cathodic shift in potential. However, this may be partially due to dissolution of a pyrrhotite impurity in the pentlandite electrode. This increase in dissolution rate is observed in the potential region below that of the activated and non-activated concentrates. Thus, although activation of the concentrate does not change the chemical composition of the pentlandite, it may render it more liable to dissolution.

The open-circuit potential of minerals in 1M hydrochloric acid and the rates of dissolution of iron and nickel (or copper) are illustrated in Table III. The open-circuit potential of troilite is fairly cathodic, and dissolution is fast. The rate of iron dissolution from the pentlandite sample is also fast, but the rate of nickel dissolution is much slower. This probably reflects fast dissolution of the pyrrhotite impurity present in the pentlandite, particularly as a cathodic open-circuit potential was observed. The following reactivity order can be postulated from the results, but it must be emphasized that this order depends on the reaction potential and stoichiometry of the individual sulphide mineral:

troilite > pyrrhotite > pentlandite > chalcopyrite \approx pyrite.

Contact between the nickel metal immersed in the solution and the electrodes of untreated and activated concentrate resulted in a cathodic shift in the mixed potentials of both the untreated and the activated concentrates to about -0.3 V (Table II). The rate of nickel

TABLE III

DISSOLUTION OF MINERALS UNDER OPEN-CIRCUIT CONDITIONS

Mineral	Open circuit potential (E _{OC})	k _{Fe}	k _{Ni(Cu)}
NiFeS ₂	-0,06	6 × 10 ⁻⁸	8 × 10 ⁻¹⁰
CuFeS ₂	-0,10	2,5 × 10 ⁻⁹	2 × 10 ⁻¹⁰
FeS ₂	+0,05	1 × 10 ⁻⁹	—
Fe _{1-x} S	+0,15	3 × 10 ⁻⁹	5 × 10 ⁻¹⁰
FeS*	-0,1	≈ 2 × 10 ⁻⁷	—

*Calculated from results presented in references 6 and 7.

dissolution was increased at this potential, but this can be attributed at least partially to the simultaneous dissolution of the nickel metal. However, the rate of iron dissolution from the electrodes also increased, suggesting that the rates of non-oxidative dissolution of both the activated and the untreated concentrates are appreciably faster at the more-cathodic mixed potential. This observation is in agreement with the results obtained when the potential was varied by use of a potentiostat (Fig. 1).

The addition of hydrogen sulphide to the reactant solution resulted in an anodic shift in mixed potential, with a corresponding decrease in the rate of metal dissolution for the activated concentrate (Table II). No significant effect on the rate of dissolution of metal from the treated concentrate was observed. It is unlikely that the addition of 10⁻³M hydrogen sulphide presents any thermodynamic limitation on the rate of dissolution of nickel or iron at this low pH value, but it may alter the nature of the surface of the reactant sulphide, thus changing the mixed potential and dissolution rate.

Leaching of Particulate Concentrates

When the untreated concentrate was leached in 3 M hydrochloric acid at 60°C, extractions of 12 per cent nickel and 7 per cent iron were recorded after 5 minutes. However, after 6 hours these figures had risen to only 17 per cent and 9,5 per cent respectively. Thus, as reported by Dyson², the non-oxidative extraction of nickel from untreated concentrates is not feasible under normal conditions. When the activated material described in Table I was leached for 6 hours under identical conditions, extractions of 70 per cent nickel and 91 per

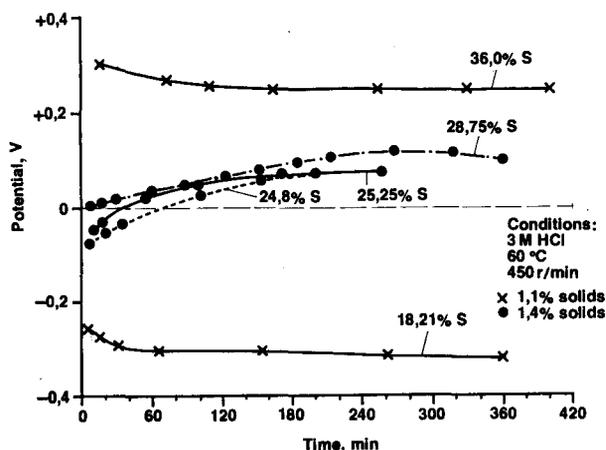


Fig. 6—The effect of sulphur in the concentrate on potential

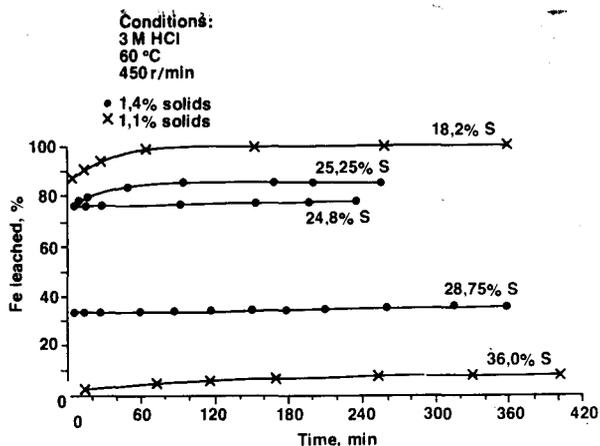


Fig. 7—The effect of sulphur in the concentrate on the extraction of iron

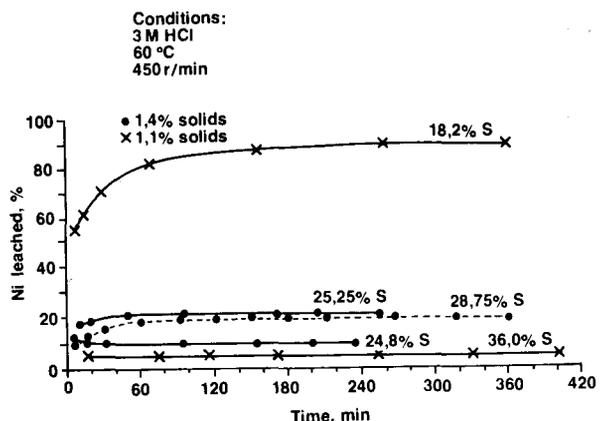
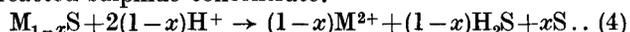


Fig. 8—The effect of sulphur in the concentrate on the extraction of nickel

cent iron were recorded. The slow leaching rates of untreated concentrates that are high in sulphur has been attributed by Dyson² and others^{3,4} to the formation of an inhibiting layer of sulphur round the partially reacted sulphide concentrate:



However, a calculation of the volume of sulphur that can be produced from the stoichiometric metal deficiency of a concentrate shows that it occupies less than 20 per cent of the total volume of the dissolved concentrate. Since the temperature at which leaching occurs is below the melting point of sulphur, a complete inhibiting layer of sulphur cannot be formed around the reaction surface. Instead, a porous layer is present that inhibits the reaction rate only by a reduction in the surface area available for reaction. This does not explain the very poor reactivity of the untreated material observed by Dyson² and also in this study. Instead, the differences in leaching rates of the activated and untreated concentrates are probably due to the difference in mixed potential at which they react (i.e., differences in metal-to-sulphur stoichiometric ratio at the reaction interface). This is illustrated in Figs. 6 to 8, which show that there is a direct correlation between the extraction rate of both nickel and iron and the potential assumed by

the slurry. Concentrates that are low in sulphur yield a slurry that exhibits a cathodic potential around $-0,3V$ and shows complete iron and nickel dissolution. Conversely, concentrates that are high in sulphur yield an anodic potential, and little iron or nickel is extracted. As a general rule, the concentrate should be activated for a time sufficient to yield a product containing less than 20 per cent sulphur and having a reaction potential approaching $-0,3V$. It should be noted at this stage that the temperature of activation that was found to be most successful for this concentrate ($850^{\circ}C$) is higher than that suggested by Dyson² ($750^{\circ}C$). Even prolonged treatment (24 hours) of the 36 per cent sulphur concentrate at $750^{\circ}C$ did not yield a concentrate that could readily be leached to completion. This probably reflects the greater percentage of sulphur present in the starting material.

The bubbling of hydrogen sulphide through the solution during the leaching of an activated concentrate had little effect on the rate of extraction of nickel and iron (Fig. 9). This shows that there is no thermodynamic limitation on the rate at which nickel and iron dissolve. There is no necessity, therefore, for hydrogen sulphide to be removed from the reaction vessel for a good extraction to be obtained.

The addition of elemental sulphur prior to the initiation of the leaching reaction caused the mixed potential to shift anodically from $-300mV$ to $+50mV$ during the reaction. This caused a virtual cessation in the dissolution of nickel and iron after about $1\frac{1}{2}$ hours, as the potential reached around $0 mV$, although the reaction had proceeded at an acceptable rate before

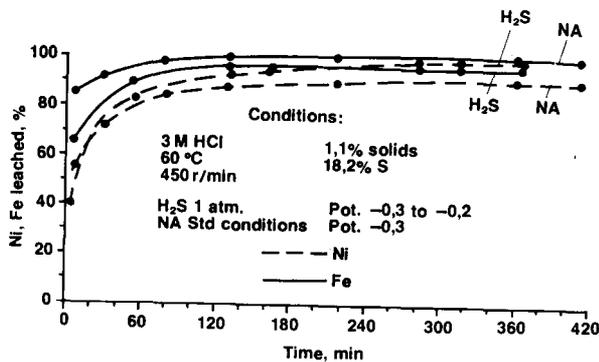


Fig. 9—The effect of hydrogen sulphide on the rate of dissolution of the nickel concentrate

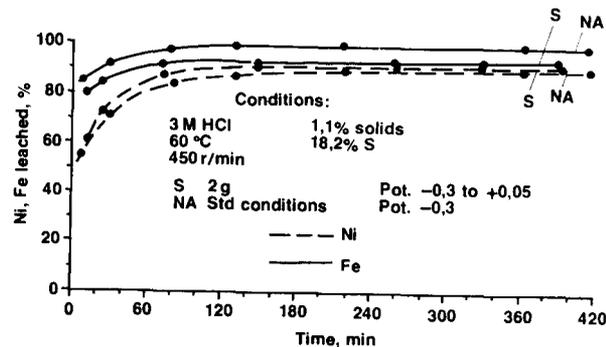


Fig. 10—The effect of sulphur on the extraction of nickel and iron

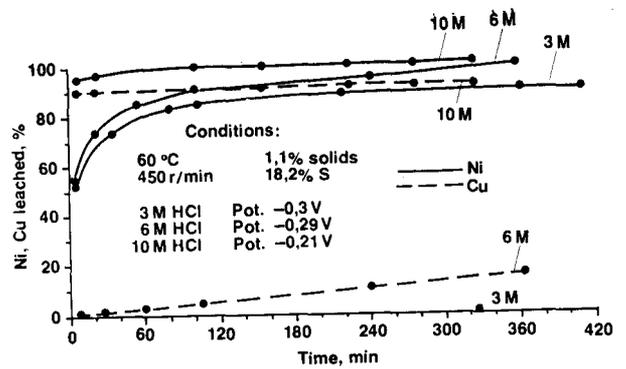


Fig. 11—The effect of acid concentration on the extraction of nickel and copper

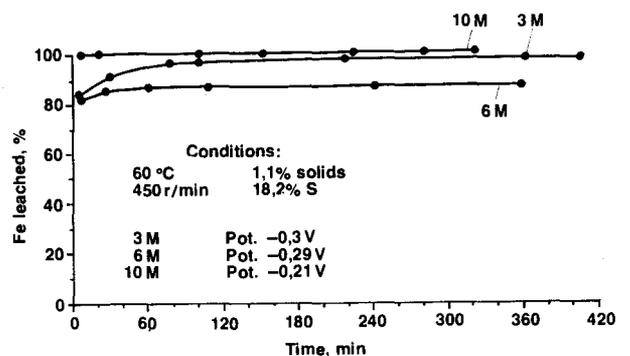


Fig. 12—The effect of acid concentration on the extraction of iron

this (Fig. 10). The same behaviour was observed when sulphur was added during the leaching of a nickel matte. The addition of sulphur prior to leaching should have no effect on the reaction rate if the addition is controlled by the diffusion of the reactants or products through a sulphur layer formed *in situ*, as was suggested previously². This again confirms that the relationship between mixed potential and dissolution rates is the determining factor in the faster nickel and iron extraction achieved from the activated concentrate.

The effect of acid concentration on the rate of leaching of nickel, iron, and copper is shown in Figs. 11 and 12. The rates of dissolution of nickel and iron are only slightly faster at the higher concentrations of acid, but the dissolution of copper increases markedly, from virtually nil in 3M hydrochloric acid to 90 per cent in 10 M hydrochloric acid. This was attributed to thermodynamic limitations on the dissolution of copper in dilute acid. These limitations are at least partially removed with acids at the higher strengths. It is interesting to note that, if the concentration of hydrochloric acid is adjusted, copper can either be leached from the activated concentrate or remain undissolved, whereas there is only a small change in the percentage dissolution of nickel and iron.

Although it was not a major objective of this study, consideration was given to the further treatment of the leach liquor by a method similar to that used in the Falconbridge matte-leaching process⁵. It was found that, after the residue had been filtered from the non-oxidative

leach of nickel concentrate, the pregnant liquor could be oxidized and the iron solvent extracted as iron(III) chloride. Owing to the high concentration of iron in the concentrate, a large quantity of iron(III) chloride would have to be extracted and, for the concentrate to be treated economically, the iron(III) chloride would have to be reconverted to hydrochloric acid and iron oxide. Alternatively, the iron could be precipitated as goethite or hematite by neutralization at elevated temperatures. The pregnant solution remaining after the removal of the iron could be recycled to the initial leach, and the metal concentrations could so be built up until the copper, cobalt, and nickel could be recovered by conventional methods.

Conclusions

The rate of non-oxidative dissolution of nickel concentrate depends on the potential at which the reaction takes place. This is, in turn, dependent on the metal-to-sulphur stoichiometric ratio at the reaction interface. For the non-oxidative dissolution rate to be maximized, the composition of the reactant interface should be altered in such a way as to yield the optimum mixed potential, which is a value cathodic of $-0.4V$. This alteration of reaction potential can be achieved by the addition of a reducing reagent, by the application of a direct current, or by chemical alteration of the stoichiometry of the material to be leached. The activation of the concentrate by hydrogen at high temperature is an example of the last method.

The activated nickel concentrate can be treated at a reasonable rate by a non-oxidative dissolution process in hydrochloric acid. A complete separation of nickel and iron from copper can be obtained in this way. An untreated nickel concentrate high in sulphur cannot be processed satisfactorily by non-oxidative dissolution in hydrochloric acid.

Acknowledgements

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International Mining, Britain

Mining engineers are well aware that the mineral resources of the world, particularly those that contribute to energy requirements, are vital to world economy, and that it is their duty to develop the technology that can more and more efficiently make those resources available for use. Mining operations and the development of mining equipment are long-lead processes; and it is with complete confidence in the future that preparations are going ahead for the International Mining Exhibition '82, which is to be held at the National Exhibition Centre, Birmingham, from 6th to 11th September, 1982, to coincide with the conference mentioned below.

The Association of British Mining Equipment Companies (ABMEC), who are sponsoring the exhibition, have determined the theme as 'Mining and Mineral Processing'; and it is intended that the scope of exhibits should cover open-pit methods as well as deep mining, exploitation of stratified minerals as well as ore-bodies, and all stages of the process from initial exploration and location of the 'green field' to preparation of the product for the market. Restoration of the land to other uses, especially after open-cast working, will feature in the show.

Further information is obtainable from Mr R. Buckley, ABMEC Secretaries, Peat, Marwick, Mitchell & Co., P.O. Box 121, Sheffield S13 9JQ. Telephone: 0742-21071.

The 1982 International Conference on Mining Transport and Handling Systems to be held in Birmingham,

England, will be conducted in four languages - German, French, Spanish, and English - with simultaneous translation service for the major presentations and discussion. The addition of Spanish to the conference language line-up for the first time reflects the increasing world-wide importance of mining matters, and emphasizes the expanding mineral-resource industries in Central and South America.

Scheduled to take place from September 7th to 9th, 1982, at the National Exhibition and Conference Centre in Birmingham alongside a world exhibition of mining equipment (see above), the Conference will review current practice and future developments in transport and handling. It will concern itself with mineral, materials, and personnel transport in both underground and surface-mining operations, and papers will cover the full range of mineral mining activity, although giving emphasis to coal.

Contributions of up to 5000 words are being invited, and prospective speakers are asked to send a 200-word synopsis of their suggested papers by the end of February 1981. Complete manuscripts of the papers that are accepted will be required by January 1982.

Further details can be obtained from NCB 82, National Coal Board, The Lodge, Doncaster DN1 2DX, England. Telephone (0302) 66611 ext. 3611, telex: 82161 CBHOB G.