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Disciplines

Engineering | Science and Technology Studies

Publication Details

Bjareborn, O., Arif, T., Monaghan, B. & Bumby, C. W. (2020). Fate of titanium in alkaline electro-reduction of sintered titanomagnetite. *Materials Research Express*, 7 (10).

PAPER • OPEN ACCESS

Fate of titanium in alkaline electro-reduction of sintered titanomagnetite

To cite this article: Oscar Bjareborn *et al* 2020 *Mater. Res. Express* **7** 106508

View the [article online](#) for updates and enhancements.

239th ECS Meeting

with the 18th International Meeting on Chemical Sensors (IMCS)

ABSTRACT DEADLINE: DECEMBER 4, 2020

May 30-June 3, 2021

SUBMIT NOW →

Materials Research Express

PAPER

OPEN ACCESS

RECEIVED

7 September 2020

REVISED

24 September 2020

ACCEPTED FOR PUBLICATION

30 September 2020

PUBLISHED

9 October 2020

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Fate of titanium in alkaline electro-reduction of sintered titanomagnetite

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E-mail: chris.bumby@vuw.ac.nz**Keywords:** titanomagnetite electrolysis, alkaline electrolyte, iron electrowinning, ceramic cathode

Abstract

Direct electrochemical reduction of iron ore in concentrated NaOH electrolyte has been proposed as a potential route to substantially reducing the global steel industry's CO₂ emissions. Here, we report the solid-state electro-reduction of sintered pellets formed from titanomagnetite ironsand. This commercial iron ore contains ~4 wt.% Ti which is directly incorporated within the magnetite lattice. At 110 °C, these pellets are electrochemically reduced and exhibit a well-defined reaction front which moves into the pellet as the reaction progresses. The electro-reduction process selectively produces iron metal, whilst the Ti content is not reduced. Instead, Ti becomes enriched in segregated oxide inclusions, which are subsequently transformed to a sodium iron titanate phase through taking up Na⁺ from the electrolyte. These inclusions adopt an elongated shape and appear to exhibit locally preferential alignment. This suggests that they may nucleate from the microscopic titanohematite lamellae which naturally occur within the original ironsand particles. The expulsion of contaminant Ti-oxides from the final reduced metal matrix has implications for the potential to development of an industrial electrochemical iron-making process utilising titanomagnetite ore.

1. Introduction

Iron and steelmaking is responsible for 7% of total global CO₂ emissions [1], making it the second largest industrial source. As a result, there is growing global interest in alternative iron-making processes with reduced emissions [2, 3]. One such approach is the electrowinning of iron [4–7] using electricity generated from renewable sources. Pyro-electrolysis of iron oxides can be achieved at high temperatures in molten electrolytes [8–10]. Alternatively, electroreduction can also take place in alkaline hydroxide solutions at temperatures below 150 °C [11–14]. This latter approach is attractive, as the lower process temperature enables a wider choice of anode materials. Whilst alkaline electrowinning approaches have been studied for magnetite (Fe₃O₄) and hematite (Fe₂O₃) feedstocks, there is little known about the applicability of this process to other forms of iron ore. One such ore is Titanomagnetite (TTM) which is commercially utilised as a steel-making feedstock in several countries [15, 16]. TTM has the stoichiometric form, Fe_{3–x}Ti_xO₄, and adopts the same crystal structure as magnetite. However, ironmaking from TTM ore is not entirely straightforward, as the titanium content prevents it from being processed via the traditional blast furnace route. This is due to the formation of a high viscosity Ti-containing slags which blocks the furnace [17]. Instead, TTM is commercially smelted using CO₂-intensive direct reduction methods [18], which employ coal as both the thermal fuel and chemical reductant. In principle, an alternative electro-reduction process could simultaneously decrease CO₂ emissions and also circumvent the problems of processing Ti-bearing slags.

It has previously been shown that solid-state electro-reduction of iron oxides can be achieved in concentrated alkaline solutions, which are well suited to fostering the required anodic evolution of oxygen [19–21]. This approach has been used to electrochemically reduce both conductive iron oxide cathodes [22–24],

Figure 1. Schematic showing pellet preparation method by uniaxial compression of ironsand in a die.

and also agitated slurries of fine iron ore particles in suspension [5, 11, 19, 21, 25, 26]. However, the mechanism by which these reactions proceed remains a matter of some conjecture. Ionic Fe species are very poorly soluble in alkaline electrolytes, but some authors have nonetheless suggested that continuous dissolution and redeposition of Fe ions may occur at the cathode [12, 22, 23, 27]. In contrast, others have described a solid-state electro-deoxidation of the iron oxide, similar to the FFC Cambridge process [19–21, 28]. Evidence for this latter mechanism includes the observation of a well-defined reaction front at both microscopic [19, 20] and macroscopic scales [23].

TTM possesses the same crystal structure and Fe oxidation states as magnetite, so might be expected to follow a similar electro-reduction pathway. However, TTM is more thermodynamically stable than non-titaniferous magnetite [29–32], and the role and fate of Ti^{4+} during alkaline electroreduction has not previously been studied. This paper describes an experimental study into the feasibility of alkaline electroreduction of TTM ironsand, and the fate of titanium species during this process. Sintered TTM pellets have been employed as the cathode electrode in these experiments, and the electrochemical reactions taking place within these pellets have been studied to provide insights into the mechanism of electroreduction, and the fate of contaminants in the iron ore.

The TTM ironsand used in this work has been sourced from New Zealand, which has abundant deposits of chemically uniform ironsand, containing approximately 8% TiO_2 by mass [33–36]. Similar ironsands are also found in other large deposits in the South Pacific [35–37].

2. Experimental

New Zealand titanomagnetite ironsand concentrate was commercially sourced from Industrial Sands Ltd (Waikato North Head). Samples were washed in water to remove impurity fines, and then sieved to produce a particle size fraction of 125–150 μm which was used throughout this work. Ironsand pellets were prepared by uniaxial pressing at 35 MPa in a die with an inner diameter of 10 mm, using a hydraulic press. A schematic of this process is shown in figure 1. The pellets were then sintered at 1300 $^{\circ}\text{C}$ for 2 h in an argon atmosphere. Each pellet was mechanically thinned (i.e. polished) to a thickness of 1.5 mm, then washed in distilled water and ethanol, and dried in a flow of clean air.

Electrochemical reduction of these ironsand pellets was studied using the experimental configuration presented in figure 2. The working electrode comprised a stainless-steel rod (SS316L), to which the ironsand pellet was mounted using silver conductive paint. The steel rod was electrically insulated with polytetrafluoroethylene (PTFE) film tape and shrink tubing. This left only the top surface of the ironsand pellet exposed to the electrolyte, forming an electroactive surface area for the working electrode of 78.5 mm^2 . The anode was a platinised titanium mesh cylinder, which surrounded the working electrode in order to minimise electrolyte resistance. Three-electrode studies were carried out using a $\text{Hg}|\text{HgO}$ (20% KOH) reference electrode (+0.098 V versus NHE at 110 $^{\circ}\text{C}$). To avoid contamination, the reference electrode compartment was connected to the electrolyte via a sintered junction extension filled with 20% KOH. This three-electrode configuration was connected to a potentiostat (Metrohm Autolab) controlled by PC software (NOVA 2.1). Cyclic voltammograms (CV) were recorded at a scan rate of 20 mV s^{-1} , starting from the open circuit potential (E_{oc}), and following the scheme $E_{\text{oc}} \rightarrow -1.3 \text{ V} \rightarrow -0.3 \text{ V}$, unless indicated otherwise.

Electro-reduction experiments were performed in a stirred alkaline electrolyte (50 wt% $\text{NaOH-H}_2\text{O}$), which was contained within a PTFE beaker heated in a heated silicone oil bath. Systematic CV experiments were performed at 50, 80 and 110 $^{\circ}\text{C}$ on ironsand pellets. After reduction, the ironsand pellet was removed, rinsed in distilled water and dried in a vacuum oven at 80 $^{\circ}\text{C}$. XRD analysis of the top surface of the reduced pellets was

Figure 2. Schematic diagram of the electrochemical cell used in this work.

performed to verify the presence of metallic iron. X-ray diffraction (XRD) spectra were recorded employing a Bruker D8 Advance diffractometer with Co-K α (Cobalt (Co): 6.9257 keV, $\lambda = 1.7902 \text{ \AA}$). The pellets were then set in an epoxy resin and a cross section was polished for SEM analysis. Morphology and microstructural features were characterized by SEM and EDS using a FEI Quanta 450 and a FEI Nano-SEM Nova 450 (both equipped with EDAX EDS detectors).

3. Results and discussion

3.1. Fabrication and characterization of ironsand pellets

NZ ironsand is a well-studied mineral which is known to consist mostly of homogeneous spheroidal particles of TTM [33, 38]. A small amount of titanohematite (TTH) is also present in the form of lamellae observed within a minority of particles [34, 39]. TTH is a solid solution within the hematite-ilmenite (Fe_2O_3 - FeTiO_3) series.

The naturally occurring TTH lamella contain substantially higher levels of Ti than the surrounding TTM matrix (see figure 3(c)). Elemental composition of the naturally occurring ironsand studied in this work was analysed by X-ray fluorescence and is given in table 1. With the exception of Fe, composition is given as equivalent wt.% of the common oxide. In addition to TTM, appreciable levels of Al, Si and Mg are also found to be present. SEM-EDS (not shown) indicates that whilst silicates are largely present as discrete gangue inclusions, both Al and Mg enter solid-solution within the TTM crystal lattice.

The porosity of the sintered pellets was measured using the Archimedes' principle, and indicated an open porosity of 15%–20%. SEM analysis of a cross-section of sintered pellet before reduction is presented in figure 4. This shows a well sintered pellet with multiple inter-particle bonds formed by 'necking' between neighbouring particles. Some variation in titanium content is observed across the pellet, in the form of fine lamellar features (assumed to be TTH), as well as a small number of individual grains with relatively high titanium content. Figure 5 shows XRD patterns obtained from both the raw ironsand powder, and from a pellet sintered under argon atmosphere for 24 h at 1300 °C. The sintered pellet shows a small increase of the amplitude of hematite peaks compared to the initial ironsand sample, which indicates some thermal transformation of TTM to titanohematite during the sintering process [40]. However, titanomagnetite remains the dominant phase present after sintering process.

3.2. Electrochemical behaviour of ironsand pellets in NaOH 50 wt% solution

Initial electrochemical experiments were performed to investigate the electrochemical behaviour of the ironsand in strong alkaline electrolyte. The temperature range studied in current manuscript is consistent with temperature range for conventional magnetite electrowinning in literature [8]. This temperature range is also

Figure 3. Back-scatter SEM images of a polished cross-section of NZ ironsand (125–150 μm). Darker grey grains in the image (a) are ‘gangue’ particles containing lower concentration of Fe. The image (b) shows a single TTM particle containing exsolved TTH lamellae. Titanium concentration within these lamellae is shown by inset EDS-map (c).

Table 1. XRF analysis of the TTM ironsand, obtained after initial roasting in air at 1000 $^{\circ}\text{C}$ for 1 h. LOI = mass loss after roasting, where negative value indicates sample gained mass due to oxygen take-up during roast step.

	Fe ^a	TiO ₂	Al ₂ O ₃	MgO	SiO ₂	V ₂ O ₅	MnO ₂	Other	LOI
wt%	59.1	7.9	3.8	2.8	2.0	0.6	0.6	0.69	–3

^a Calculated from equiv. Fe₂O₃ value in fully oxidised sample.

suitable for electrolyte stability and furthermore avoids the water loss which can occur at higher temperatures. Figure 6 shows cyclic voltammetry (C-V) curves obtained from the ironsand pellet electrodes at three different temperatures, during cyclic scanning between -0.3 V and -1.3 V (relative to a Hg/HgO reference electrode). In each case the first 2 full cycles are shown. At 50 $^{\circ}\text{C}$, cathodic peaks C_1 and C_2 are observed during the first potential scan in the cathodic direction. Further negative polarization beyond C_2 gives rise to a rapidly increasing cathodic current B. At 80 $^{\circ}\text{C}$ and 110 $^{\circ}\text{C}$ the cathodic peak C_2 is present, but C_1 is only revealed as a masked shoulder. In addition, two anodic peaks were obtained at 50 and 80 $^{\circ}\text{C}$, A_1 ($\sim -0.5\text{ V}$) and A_2 ($\sim -0.9\text{ V}$), but only A_2 was observed at 110 $^{\circ}\text{C}$.

The C-V curves shown in figure 6 are very similar to those reported previously in conventional magnetite studies [8, 20, 21, 41]. We therefore attribute the peak C_1 to partial reduction of Fe(III) to Fe(II) species and peak C_2 to reduction of Fe(II) to metallic Fe. It is of note that distinct differences were observed in the nature of the C_2 peak during the first cycle on a virgin pellet, versus all subsequent cycles. During the first cycle, Fe metal is initially reduced from the TTM matrix. As the cycle returns through the anodic peaks (A_1 and A_2) this Fe metal is then electrochemically re-oxidised to form a surface iron hydroxide [42]. This newly formed hydroxide then participates in all subsequent cycles, masking any subsequent peaks arising from reduction of the TTM matrix. In addition to basic thermodynamic considerations [43], the electrical conductivity of magnetite and solid-state

Figure 4. SEM–BSD image (top left) and corresponding EDS elemental maps of oxygen (top right), Iron (bottom left) and titanium (bottom right) of the sintered pellet before reduction.

Figure 5. XRD-patterns for raw NZ ironsand (bottom) and sintered (1300 °C, 2 h under argon) pellet (top). The pellet sample was ground to a powder to ensure this data is representative of the full pellet volume.

Figure 6. C-V curves (scan rate of 20 mV s^{-1}) obtained from sintered ironsand (pellet) electrodes in NaOH 50 wt% solution at temperatures of: (a) 50°C ; (b) 80°C ; and (c) 110°C .

diffusion rates also both increase with temperature, such that a combination of these effects probably contribute to this strong current density enhancement with temperature [44–46].

3.3. Electro-reduced ironsand pellets

A pellet reduced at -1.0 A for 2 h was then removed from the cell and examined. Figures 7(a) and (b) show SEM images of the resulting electro-reduced surface. The particulate structure of the unreduced pellet has been replaced by a coalescent network of metallic globules. XRD analysis (figure 7(c)) shows that BCC α -iron is now the major phase present at this surface, and that the original titano-magnetite (spinel) phase is undetectable. Note that XRD peaks are sharp in this figure because this is high purity elemental iron in stark contrast with previous figure where the peaks are broad due to the presence of impurities in the inhomogeneous natural occurring mineral sand.

Figure 7. Surface of pellet after electro-reduction in 50 wt% NaOH, 110 °C, 2 h and -1.0 A cm^{-2} . (a) Low magnification SEM image. (b) High magnification SEM image of highlighted area. (c) XRD pattern obtained from pellet surface, green stars indicate peaks corresponding to α -iron.

Figure 8. Overlay image of element maps for Fe (orange), Ti (blue) and O (green) from EDS mapping of a cross-sectioned iron sand pellet reduced for 16 h at 1 A cm^{-2} .

The removal of oxygen from the metal surface layer is also clearly evident in elemental maps of a cross-sectioned electro-reduced pellet. Figure 8 shows an element overlay image for oxygen, iron and titanium obtained via EDS-mapping. The top part in this image represents the electrolyte-pellet interface, whilst the bottom of the image was closest to the electrode during electroreduction. A metallic iron surface layer (orange) can clearly be seen from which oxygen has been almost entirely depleted. Exceptions to this are a few particles containing very high levels of Ti, and a few small elongated lamella (green), which are discussed further in the next section.

Figure 8 also shows that the original particulate structure of the pellet has been largely destroyed in the metallic region and replaced by a porous dendritic interconnected network. This increase in porosity is due to the change in density from the initial TTM phase ($4800\text{--}5300 \text{ kg m}^{-3}$) [33] to iron metal (7200 kg m^{-3}). Progressing deeper into the pellet, a clear reaction front interface can be observed separating the electro-reduced

Figure 9. SEM–BSE images of the cross section of ironsand pellets. Effect of current density and time on the depth of reaction front.

Table 2. Reduction conditions, and the depth of the reaction front for samples 1 to 7. All experiments were all performed in 50 wt% NaOH at 110 °C.

#	Time (h)	Current (A)	Electrode Current density A cm ⁻²	Total charge delivered C cm ⁻²	Reaction front depth (μm)
1	2	−0.5	1.00	7157	220
2	2	−1.0	1.30	9171	370
3	2	−1.8	2.30	16 509	340
4	4	−0.3	0.40	5503	110
5	16	−0.8	1.00	58 700	440
6	17	−1.0	1.30	77 961	470
7	1.8	−1.8	2.30	53 408	390

metallic region (orange) from the oxygen rich TTM region (green). This lower region appears to be unaffected by the electroreduction process, with the original particle morphology still clearly apparent.

In order to investigate the progression of this reaction front in more detail, a series of ironsand pellets were reduced for different durations at various constant current densities, under the conditions given in table 2.

Figure 9 shows back-scatter (BSE) SEM images of cross-sections of a subset of these samples, each reduced for 2 h at increasing current levels. In each case the reaction front formed between the metallic iron layer and the unreacted inner TTM zone can again be clearly seen. Overall, the depth of this reaction front is observed to increase with increasing total charge delivered to the electrodes. However, once the front reaches a depth of approx. 350 μm, further progress is significantly slowed, and applying further charge has little effect on the remaining unreduced iron oxides in the pellet.

This stagnation behaviour may be explained by the thin planar pellet electrode geometry used for these experiments. As the electroreduction of the pellet progresses, the outer metallic iron layer forms at all surfaces, and including the outer edges of the 1.5 mm thick pellet. Eventually the iron layer at each edge extends far enough to meet with the backside electrode. Evidence for this ‘edge shorting effect’ can be seen in the top and bottom images in figure 9, which both show the left-hand edges of the respective pellets. In each case, the metallic iron layer is observed to continue down the left-hand edge of the pellets towards the backside electrode. Once the metallic iron layer meets the back electrode it will ‘short-circuit’ the unreduced region remaining in the centre of the pellet (as the unreduced oxide has a much lower electrical conductivity). From this point onwards, charge is then transferred directly from the back electrode into the iron-electrolyte interface, leading to the preferential formation of hydrogen at this interface [4], rather than reduction of iron at the interior of the pellet.

3.4. Morphology and phase development

In general, lower current densities are observed to produce a denser metallic network within the electro-reduced layer, whilst higher currents produce a more porous dendritic structure. At all current densities it is notable that small oxide inclusions remain within the electro-reduced layer which are not converted to iron metal. These residual oxides are found to be enriched in titanium content. This is because the initial unreduced ironsand contains approx. 4 wt. % Ti, which cannot be reduced to titanium metal in an NaOH solution. As such Ti-oxides must be exsolved during the electro-reduction process, either into the electrolyte solution or as phase-segregated solids.

Figure 10. (a) SEM–image of cross section of iron sand pellet (sample 5) after reduction at -1.8 A cm^{-2} in 50 wt% aq. NaOH at 110°C for 16 h. (b) Image of reduced iron and titanium-rich inclusions occurring near the pellet surface (c) Magnified image of inclusion and surrounding metal from image b, showing locations of EDS spot analyses. (d) Image of titanium rich inclusions and surrounding iron from deeper within pellet, near the reaction front. (e) Magnified image of inclusion and surrounding metal from image d, showing locations of EDS spot analyses.

Figure 10 shows SEM images of residual oxide inclusions found within the electro-reduced layer, after extended electroreduction at -1.8 A cm^{-2} . Magnified regions are shown both from near the pellet surface (figures 10(b) and (c)), and near the reaction front boundary (figures 10(d) and (e)). Table 3 and figure 11 show accompanying EDS analyses of these images. Spot analysis confirms that Ti levels in the metallic iron is low, with detected levels of $\sim 1\%$ Ti possibly being due to nanoscale oxide inclusions. As this is insufficient to account for all of the titanium originally present, we surmise that some Ti migration has occurred into the unreduced material during the reaction. This has stabilised these residual oxides to further electrochemical reduction. The oxide inclusions are observed throughout the electro-reduced layer, but there are clear differences between the oxide inclusions observed at the surface and those deeper inside the pellet.

Oxide inclusions observed deep within the pellet comprise two distinct oxide phases. These can be distinguished in figure 10(e) as regions of either darker or lighter grey contrast. The lighter phase may be Ti-enriched TTM, as the measured Ti/Fe ratios lie within the titanium-saturation limit ($x = 1$) of the $\text{Fe}_{3-x}\text{Ti}_x\text{O}_4$ spinel [see points 3 and 4 in table 3(ii)]. By contrast, the Ti/Fe ratio in the darker phase is much higher [points 5 and 6 in table 3(ii)]. The sodium level in the darker phase is also substantially higher than the background level. These observations are consistent with the formation of a sodium iron titanate phase, such as $\text{Na}_x\text{Fe}_x\text{Ti}_{2-x}\text{O}_4$ [47, 48]. This is forming from the outer edges of the inclusion, through taking up additional Na from the NaOH electrolyte.

Figure 11. EDS elemental maps of oxygen (green), sodium (brown), titanium (blue) and iron (red) for oxide inclusions shown in figure 10(d).

Table 3. Quantitative EDS point analysis of lamellae and surrounding metal in the electrochemically reduced iron sand pellet.

Spot #	O	Na	Mg	Ti	Fe	Ti/Fe	Na/Fe
(i) Points from figure 10(c), Top layer composition (atomic %)							
1	46.1	3.2	0.7	10.4	39.4	0.26	0.08
2	40.8	9.1	1.6	10.6	37.6	0.28	0.24
3	51.9	2.9	1.5	7.7	35.0	0.22	0.08
4	10.3	0	0	1.9	87.7	0.02	—
5	46.2	13.0	1.6	11.1	27.9	0.40	0.47
(ii) Points from figure 10(e), Interphase layer composition (atomic %)							
1	6.9	4.6	0	1.1	87.2	0.01	0.05
2	7.7	5.0	0	1.5	85.7	0.01	0.06
3	53.0	4.3	1.6	9.2	30.8	0.30	0.14
4	53.5	2.1	1.7	8.1	33.4	0.24	0.06
5	60.2	8.9	2.6	17.7	10.1	1.75	0.88
6	61.0	7.8	2.4	16.7	11.3	1.47	0.69

The oxide inclusions nearer the surface have experienced a longer period of local reduction, as the reaction front moved pass these features at an earlier time. Unlike the deeper-occurring oxides, these ‘older’ inclusions now show a pronounced outer shell which separates the interior oxide from the surrounding iron metal (figure 10(c)). This shell-layer shows elevated levels of both titanium and sodium [see points 2 and 5 in table 3(i)]

Figure 12. Back-scatter SEM image of cross section of iron sand pellet (sample 5) after reduction at -1.8 A cm^{-2} in 50 wt% aq. NaOH at 110°C for 16 h. SEM-BS image of partially reduced iron sand particle with exsolved TTH lamella. Bottom images show EDS maps of this particle for: Ti (blue), Fe (red) and O (green).

and has probably formed from the Na-Ti-Fe-O phase discussed above. However, the Ti/Fe ratio is substantially lower in this shell material, suggesting that titanate species may have leached into the electrolyte over time [49]. The presence of void-like features between the shell and the enclosed interior oxide provide further evidence that some of this material has been leached.

A common feature of all of the observed oxide inclusions is that they adopt an elongated shape, with some preferential local alignment. This morphology can be understood by examining the partially reduced iron sand particles found nearest to the reaction front. Figure 12 shows one such particle, where the particle surface has been largely converted to metallic iron (white), but the inner region remains in the oxide phase (light grey). This ‘shrinking core’ behaviour occurs because the electrochemical reduction takes place at the electrolyte surface, which is yet to propagate to the inner core of the particle.

EDS maps (Figures 12(b)–(d)) show that the unreduced region of the particle contains multiple Ti-enriched lamellae. These are the naturally occurring TTH lamella found in the unreduced iron sand. At the surface, the reaction appears to be selective; converting the TTM matrix to iron metal but leaving some of the Ti-enriched lamella behind. Close examination of both figures 10(a) and 12(a) reveal a substantial number of similar fine lamella relics are present in the lower portion of each image. It appears that these lamellae form a nucleus for further growth of residual oxides, thickening over time through the accretion surrounding exsolved material. As a result, the residual oxides retain an elongated shape, and remain approximately aligned with the crystal twinning-planes of the parent iron sand particle.

4. Conclusion

We have demonstrated the electrochemical reduction of sintered titano-magnetite ironsand cathodes in concentrated NaOH electrolyte to produce α -iron. Reduction starts at the surface and proceeds inwards, forming a porous metallic layer with a well-defined reaction front. The increased porosity is a result of the volume reduction which accompanies the removal of oxygen from the parent oxide.

The electro-reduction process is selective for iron, with unreduced titanium-bearing oxides being exsolved as residual solid inclusions in the metallic layer. These inclusions appear to nucleate from TTH lamellae within the original ironsand, which thicken as the reaction progresses. EDS analysis shows that at least two distinct phases are formed within the oxide inclusions, a Fe-Ti-O oxide likely corresponding to Ti-enriched TTM, and a sodium iron titanate phase formed through uptake of Na^+ from the electrolyte solution. After extended reaction times, a sodium titanate outer layer is observed around each oxide inclusion.

These laboratory-scale results demonstrate that solid-state electro-reduction of titanomagnetite ironsand is feasible, and a potential route for the low- CO_2 production of iron from this naturally occurring ore. However, the sintered cathode approach explored here exhibits very low faradaic efficiencies and is not easily scalable, so industrial application would require an alternative approach. The slurry reduction method [5, 11, 19, 25] offers more promise in this regard and will be the subject of future work. In that case, the expulsion of solid titanium-bearing oxides from the metallic iron may prove particularly advantageous, as this could result in these metallurgically-troublesome inclusions being rejected from the final electrodeposited metal product.

Acknowledgments

This work was supported by funding from NZ MBIE Endeavour fund, Grant No. RTVU1404. The authors wish to thank Sarah Spencer for assistance with SEM microscopy and Martin Ryan for assistance with XRD measurements undertaken during this work.

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