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Report for Trans-Tasman Resources Limited

Results of Iron-sand Characterisation: "Where is the iron?"

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Results of Iron-sand characterisation: "Where is the iron?"



Executive Summary

This document reports collated data obtained from the study of a set of 40 drill holes within Trans Tasman Resources (TTR) Limited's mining permit area. Data on ironsand chemistry and morphology has been obtained following the methodology described in a previous report - "*Progress report on Ironsand Technology Research Aim 1.1 : Understanding Feedstock Variability*" (July 2016). For each hole site, a 'synthetic ROM' was produced by recombining individual 1 m drill core "segments" down to a specified mining depth. These mining depths were set by criteria previously agreed with TTR. The methodology used to determine each depth is summarised in Section 2 of this report. From each synthetic ROM sample we have then produced a magnetic concentrate using dry magnetic separation, which has been configured to simulate TTR's expected LIMS1 process. No grinding has been undertaken for any of the work presented here, so all data relates solely to raw unground ironsand particles.

A summary of the key findings in this document is as follows:

- 1. The required mining depth varies significantly across the mining site. In 19 of the 40 holes surveyed the mining depth was found to be equal to at least the full depth of the sampled drill hole (up to 11 metres in some cases). However the optimum mining depth for the remaining 21 holes was determined to be above the bottom of the drill hole, and in 2 holes the total mining depth equated to only a single 1 m drill-core horizon.
- 2. A key evaluation metric is the mass of Fe which passes through to the magnetic concentrate (LIMS1). This can be expressed as a percentage of the total ROM mass, calculated from the product of the Fe grade of the magnetic concentrate, $Fe_{XRFConc}$ and the mass ratio of the magnetic concentrate and the mass of the ROM samples. This value is shown in Figure 4.1a, which is also reproduced below.



Magnetically recovered iron (LIMS1) expressed as a percentage of the total mass of the ROM material for the 40 selected holes studied in this work (See Figure 4.1a on page 25).

(a) We find that the percentage of magnetically recovered Fe varies significantly across the 40 holes studied, with an absolute range from 2.3 wt. % to 13.3 wt. %. Across the full sample set

measured we find a mean value of 5.2 wt. % with a standard deviation of 2.6 wt. %.

- (b) We also find that the magnetic reject stream (i.e., tailings from LIMS1) consistently contains an Fe mass equating 3-4% of the total ROM mass. This is consistent with previous Davis tube measurements performed by TTR which also indicated that 3-4% of the ROM comprised non-magnetic Fe. This means that drill holes with less than ~7% Fe in the ROM can contain a similar fraction of magnetic and non-magnetic Fe. In such cases, the magnetic concentrate stream and reject stream often contain similar fractions of the total ROM Fe (as measured by XRF). (Refer to Section 4.1 on page 24 and Figure 4.1b)
- 3. For the 40 holes studied, the Fe grade of the magnetic concentrate (LIMS1) had an average of $27.5 \text{ wt. }\% \pm 7.4 \text{ wt. }\%$ standard deviation, and an absolute range from 11.0 wt. % to 40.9 wt. %.



Iron content in the magnetically concentrated sand for the 40 selected holes as measured by XRF (See Figure 3.7b on page 19).

- 4. A consequence of 2 and 3 above is that the relative mass of the magnetic concentrate (LIMS 1) is not well correlated with the Fe content of the ROM.
- 5. Laser diffraction measurements were used to determine particle size distributions (PSD) in the ROM, magnetic concentrate (LIMS1) and magnetic reject (LIMS1 reject) streams.
 - (a) PSD of the ROM exhibited some variability between holes. Additionally, 2 out of the 40 holes exhibited >10 vol. % of particles $< 50 \,\mu$ m in diameter.



Particle size distribution of the run of mine samples for the 40 selected holes as measured by laser diffraction (See Figure 3.2a on page 14).

- (b) The PSD of the magnetic concentrate (LIMS1) was found to closely mirror the PSD of the parent ROM sample.
- 6. In the magnetic concentrate (LIMS1), fine particles were found to have greater Fe content than coarser particles.



XRF Iron content for the individual sieve cuts in the magnetically concentrated sand for the 40 selected holes (See Figure 3.7c on page 19).

- 7. TTR have considered using particle size classification to separate a fraction of the magnetic concentrate prior to grinding. From the sample set studied, we calculate that 8 out of 40 holes would deliver an unmilled sieve cut fraction containing > 55 wt. % Fe, using particle size thresholds of either $< 125 \,\mu\text{m}$ or $< 150 \,\mu\text{m}$.
- 8. XRF chemical analysis shows that:
 - (a) Fe:Ti:V ratios are extremely consistent across all holes and magnetic concentration sieve cuts.
 - (b) Ca is negatively correlated with Fe content. That is, as Fe grade increases the Ca content decreases.
 - (c) The trend in P content is more complicated. In general, an increase in the Fe content in the LIMS1 concentrate corresponds to an increase in P content. At Fe grades > 50 wt. % we observe the P content to be > 0.2 wt. %. However, the ratio of P:Fe decreases with increasing Fe content. This suggests that beneficiation to Fe grades of ≥ 60 % (e.g. by grinding + LIMS2) may enable the P content to be reduced below 0.18 wt. % in the final LIMS2 concentrate.

Nomenclature and abbreviations

| A_{FeSEM} | Area of the iron as a percentage of the total area of the particles as imaged in the SEM | | |
|--|--|--|--|
| Dx_y | The particle size at which $y \%$ of the particles are below this size. | | |
| $Fe_{XRFConc}$ Iron concentration in the magnetically concentrated sand as measured by XRF (wt. %) | | | |
| Fe_{XRF_i} | Iron concentration in the magnetically concentrated s and from sieve cut i as measured by XRF (wt. %) | | |
| Fe_{XRFROM} Iron concentration in the ROM sand as measured by XRF (wt. %) | | | |
| Fe_{Mag} | Percentage of iron collected by the dry magnetic separation process (wt. $\%$) | | |
| LIMS 1 | Low intensity magnetic separation pass 1 | | |
| LMF_i | The locked mass fraction in sieve cut i | | |
| m_i | Mass of sieve cut i | | |
| $m_{MagConc}$ | Mass of the sand concentrated by magnetic separation (g) | | |
| $m_{Mag Reject}$ Mass of the sand rejected by magnetic separation (g) | | | |
| ROM | Run of mine | | |
| SEM | Scanning electron microscope | | |
| TLMF | Total locked mass fraction $(\%)$ | | |
| UTM | Universal Transverse Mercator | | |
| ρ_{TM} | Density of the titanomagnetite phase | | |
| $ ho_g$ | Density of the gangue | | |

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Background

This report describes ongoing work carried out on Research aim 1.1 of the "Ironsand technology" project being undertaken in collaboration between the Robinson research institute (VUW) and Callaghan Innovation. We have undertaken a detailed study of the chemical composition and micro-morphology of sub-sea ironsand obtained from Trans Tasman Resources (TTR) Limited's mining permit area – including both run-of-mine and magnetic concentrate (LIMS1 equivalent) samples. The goals of this aim were to:

- Characterise the variability of the ROM and LIMS1 fractions across TTR's mining permit area.
- Characterise the distribution of iron-bearing inclusions within individual sand particles.
- Describe the variability in iron distribution (versus particle size) that occurs across the mining permit area.

Material for this work was sourced from holes drilled in the permit area by TTR. The methods used are intended to simulate process streams that occur in the early stages of TTR's proposed beneficiation process, up to the production of a LIMS1 concentrate. Consequently, the results, discussions and conclusions on magnetic separation within this report should be interpreted only in relation to TTR's LIMS1 process. No grinding has been undertaken in the work reported in this document.

It is important to note that the irons and samples were stored in polyweave bags with a coarse weave after collection off the Taranaki Bight. Consequently, some of the very fine s and may have been lost through the weave and we do not know how much leaked out prior to analysis. This may have implications for some of the reported measurements of the $<50 \ \mu m$ size fraction in the ROM sample.

Method

Ironsand samples have been collected, processed and analysed by various techniques to gain an understanding of the variability of the sand in the permit area. The outline below includes some repetition of information given in the previous report. This is done for clarity and ease of reference.

2.1 Sample preparation

All samples were obtained from TTR's sample storage warehouse in Porirua. TTR have drilled > 150 sample holes across their entire mining permit area, but characterisation of all of these holes would exceed the budget for this work. Instead, and in agreement with TTR, it was decided to reduce the number of holes for this study to 40 holes. TTR has collected sand in 1 m depth segments (or 'horizons') from holes over a systematic grid of approximately 1 kilometre spacing (60 holes), and from additional sites between these grid points. The 40 holes chosen for study here comprise 30 holes from the grid points and 10 holes chosen from the in-between sites. The off-grid samples were chosen to extend the range of iron contents examined, or in a few cases to replace nearby grid samples that were not readily available in sufficient quantity. This led to a set of holes spread across the permit area in a new grid with 1.4 km spacing (plus extras) and with a broad range of initial Fe grades. The location of each hole studied in this work is shown in Figure 2.1.

For each of the selected holes, a synthetic ROM was produced by combining horizons collected between the sea bed and a specified mining depth. The expected mining depth for each hole was defined according to the following criteria:

- Condition of the sand Gravel, coarse sand, fine sand, silt etc.
- Iron content from x-ray fluorescence (XRF).
- The depth of the deposit, to an absolute limit of 11 m set by Mining Permit conditions.

The protocol followed was to calculate the cumulative magnetic Fe fraction of ROM over the full depth of the hole to its bottom, or to 11 m (whichever occurred first), using previous measurements obtained by the Davis Tube method (following TTR's standard conditions). If this fraction was less than 3.5 % by mass, the bottom-most horizons were sequentially discarded until the cumulative magnetic Fe fraction was greater than or equal to 3.5 mass percent. If the new bottom horizon thus established did not itself have magnetic fraction greater than or equal to 3.5 %, then it too was discarded and the next-higher horizon subjected to the same test. Finally, the expected cumulative fraction of Fe in the Davis Tube concentrate from the hole to the selected mining depth was calculated; if it did not equal or exceed 55 % by mass, then the bottom-most horizons were sequentially discarded until the criterion was satisfied.

Representative synthetic samples of the expected ROM from each hole were obtained by recombining the required drill horizons above the determined mining depth. It was determined that the sand from each drill horizon should be combined on an equal volume basis using the dry bulk density. This was calculated using the Fe_2O_3 percentage measured by XRF for each horizon and an empirical formula (equation 2.1), both supplied by TTR.

$$Dry \, bulk \, density = \frac{Fe_2 O_3 \, wt. \,\% \times 0.6994 \times 81.191}{51.064} \tag{2.1}$$



Figure 2.1: Location of the holes drilled in the permit area. Holes selected for this work are labelled and indicated with green circles. The other holes collected in the permit area by TTR are shown in red.



Figure 2.3: Map showing hole depth used for each hole considered within TTR mining permit area

Samples from TTR's "Chem" and "Log" separates were riffled at the TTR warehouse to representatively obtain the required weight for each horizon using a box riffler and a rotary micro-riffler. These horizon samples were combined for each hole as above then returned to the Gracefield Innovation Quarter for further processing.

The procedure for preparing the combined horizons for each hole for measurement was as follows:

- 1. Pass the hole sample through a 2 mm sieve to remove large pieces of shell etc., noting the mass removed.
- 2. Riffle the scalped sample to provide run of mine (ROM) samples for both particle size and XRF measurements.
- 3. Magnetically separate the ROM sample to divide it into
 - (a) a magnetically concentrated sample "Concentrate". (This is a simulant of the expected LIMS1 stream for each given hole.)
 - (b) a rejected material sample "Magnetic rejects"
- 4. Riffle the magnetic rejects to produce a representative sub-sample for particle size measurement.
- 5. Riffle the concentrate to provide representative sub-samples for both particle size and XRF measurements.
- 6. Pass all three particle size measurement sub-samples (ROM, concentrate and magnetic rejects) through a 1 mm sieve, noting the mass.¹
- Sieve the remaining concentrate sample and weigh the sieve fractions. Sieve sizes used were 1000 μm, 710 μm, 500 μm, 355 μm, 300 μm, 250 μm, 212 μm, 180 μm, 150 μm, 125 μm, 106 μm and 63 μm.
- 8. Recombine appropriate sieve fractions to provide concentrate sample cuts of
 - (a) $< 125\,\mu{
 m m}$
 - (b) $125 \,\mu m 150 \,\mu m$
 - (c) $150 \,\mu m 180 \,\mu m$
 - (d) $180\,\mu m 250\,\mu m$
 - (e) $250 \,\mu m 355 \,\mu m$
 - (f) $355 \,\mu m 2000 \,\mu m$
- 9. Riffle each sample cut to divide it into representative sub-samples for electron microscope imaging and XRF analysis. When there was insufficient material for both techniques, XRF was given preference.

2.2 Particle size measurement

Particle size measurement was carried out using two techniques. One technique (sieving) is based on weight fractions while the other (laser diffraction) is based on volume fractions.

2.2.1 Sieving – Weight measurement

As described in section 2.1 the concentrate from magnetic separation was passed through 12 different sieves ranging in aperture size from 63 µm up to 1 mm, resulting in 13 size fraction 'cuts' of the sample. Each cut was weighed and then plotted on a cumulative fraction versus size curve to allow determination of the Dx_{10} , Dx_{50} and Dx_{90} values. Dx_y values denote the size (diameter) are those for which y % of the sample mass occurs as smaller particles.

 $^{^{-1}}$ It was found that samples that had not been sieved to remove particles > 1 mm in size would jam in the tubing of the particle size measurement equipment.

2.2.2 Laser diffraction – Volume measurement

Particle size measurements were made using a Malvern Mastersizer 3000. Samples for measurement were collected from the run of mine (ROM) sand, magnetic-concentrate sand and the magnetic-reject sand. Each of these samples was approximately 10 g in weight.

A particular range of obscuration level is required for making reliable particle size measurements so not all of the riffled sample was needed. There was usually a delay between riffling and measuring the particle size and the samples were moved around in and between the laboratories. Therefore, the samples were gently rotated to mix the sand in order to counter any effects of segregation that may have occurred. Each sample was then spooned into the Mastersizer 3000 for measurement until the obscuration level was within the required range. The particle size was measured in three replicates with a three minute delay between measurements. At the end of each set of measurements the three results were automatically averaged, and both a graphical report and a data text file were generated.

2.3 Magnetic separation

Using optimised operating parameters developed earlier in the project the ROM samples were passed through the dry magnetic separator. At the end of the run the magnetic-concentrate sand and the magnetic-reject sand were both weighed and recorded.

2.4 X-ray fluorescence

XRF measurements were undertaken by SpectraChem Analytical (CRL Energy Limited). Close communication was maintained between SpectraChem Analytical, RRI and CI to ensure that samples were handled by all parties in such a way as to produce accurate and representative results.

2.5 Electron microscopy and image analysis

In most cases, enough ironsand sample was obtained to divide the sample into two samples per sieve fraction outlined in step 8 of Section 2.1. To obtain a representative sample in each a riffler was used to make this division. Epo-tek 301 resin mixed with a fine carbon powder at a concentration of 4 % was used to embed samples. The carbon powder addition was carried out to separate particles to enable the image analysis software to correctly identify individual particles from scanning electron microscope (SEM) images. The carbon powder also increased the conductivity of the sample enough to allow SEM imaging without need of carbon coating the samples, thereby reducing sample preparation time. Embedded samples were ground, polished and then imaged by SEM using the backscatter detector to differentiate between iron bearing and non-iron bearing phases in the grains. The collected images were then analysed using an in-house automated imaging program to extract and plot iron content, liberation level and iron grain size distributions.

The image analysis software uses a stereological correction to adjust for biases associated with extrapolating 3D liberation data from 2D particle sections. Additionally, the software performs a statistical analysis to ensure accuracy of the results. Standard deviations are sample standard deviations estimating population parameters and are therefore calculated using N-1 rather than N in the denominator of the defining equation. The software also allows for individual particles to contain multiple iron grains and the total iron fraction is given by

$$\frac{Number of \ iron \ pixels}{Number of \ particle \ pixels} \times 100$$
(2.2)

This approach counts all particles in the image, including those intercepting the edge of the image. Total iron grain sizes and total particle sizes are averages of the average values from each image and do not include stereological correction. Finally, averaged numerical values are produced and presented in an output report.

Examples of the statistical data available from this approach are shown in Figure 2.4. Figures 2.4a and 2.4b plot the cumulative distribution of iron as a function of the liberation state and are examples of samples with mostly liberated and mostly locked iron, respectively. The histograms (Figures 2.4c and 2.4d) are another representation of the same data and categorise each particle within one of four liberation categories with respect to the equivalent iron grain diameter. Since the grains are seldom perfect circles



Figure 2.4: Examples of the statistical data generated from the image analysis.

in the images this is a diameter back-calculated from the measured area. The liberation categories chosen here are based on those used in a previous QEMSCAN report to TTR from Amdel-Perth. These categories are:

- Locked (<30% of the grain section area is iron ore)
- Low Middling (30-60% is iron ore)
- High Middling (60-90% is iron ore)
- Liberated (>90% of the grain section area is iron ore).

Liberated, high-middling, low-middling and locked particle and iron sizes are averages of the non-zero average values from each image. This is because at large sieve sizes the images do not always have a particle in every liberation category. These values include a stereological correction whereby particles with diameters less than the lower sieve cut-off are not counted. Particles at the edge of the image are excluded from this calculation.

The overall SEM image analysis method was found to yield reproducible and accurate results using sample sizes of approximately 8 g. Typically between 8,000 and 40,000 particles were counted – depending on particle size – from 64 images in order to estimate the

- total iron content
- iron liberation distribution
- size of iron grains.

Results

3.1 Particle size

Particle sizes were measured by sieving (reported as percentage of total mass) and by laser diffraction (reported as percentage of total volume). Cumulative particle size distributions from both techniques are given in Figure 3.1. The distributions given in Figure 3.1a are based on the mass fraction of the particles below the stated sieve size. For example, since the finest sieve used was 63 µm aperture the value for 63 µm in the figure is the weight fraction collected in the pan at the bottom of the sieve stack. The majority of particle size distributions for the holes are broadly consistent, although there is a range in particle sizes between the holes and a small number of outliers display significantly larger overall particle sizes (see Table 3.1).

Graphs showing the Dx_{10} , Dx_{50} and Dx_{90} particle sizes are shown in Figure 3.2 for the ROM, concentrate and magnetic rejects. There is some variability in the values from hole to hole. For comparison, the Dx_{10} , Dx_{50} and Dx_{90} sizes from sieving have been extracted from Figure 3.1a and are shown in Figure 3.3. In general the two histograms (laser diffraction and sieving) are broadly consistent, but there are some differences leading to some holes showing for laser measurement larger particle sizes than other holes while the order is reversed for sieving.

Plots of the percentage of particles under $50 \,\mu\text{m}$ are given in Figure 3.4. In general the amount of material less than $50 \,\mu\text{m}$ is quite small – mainly below 4% by volume. Of the 40 holes investigated there are only three holes, two holes and four holes with percentages greater than this for the ROM, concentrate and magnetic rejects, respectively.

Since the concentrate and tails are split from the ROM it would be expected that the sum of the percentage of grains below 50 µm from the concentrate and tails would approximate the percentage for the ROM. However, as can be seen in Figures 3.4 and 3.5 there are holes where the percentages for the concentrate and/or the tails are greater than that for the ROM – particularly noticeable in the STH009RC and STH010RC samples. This may be due to attrition of the sand in processing steps following the collection of the ROM PSD sample and prior to collecting the PSD samples for the concentrate and/or gangue break apart.

3.2 Magnetic separation

Between 7.8% and 40% of the ROM material was collected as magnetic concentrate through the dry magnetic separation process (Figure 3.6a). Viewing the recovery by location (Figure 3.6b) shows that the largest yields come from the north eastern region of the permit area and also from a band of holes a little to the east of the centre of the permit area.

Table 3.1: Ranges in the particle size distributions across the majority of holes (Outliers excluded).

| | \mathbf{Sieve} | Laser |
|------------------|--------------------|-----------------------|
| Dx ₁₀ | $88-140\mu m$ | $93-164\mu\mathrm{m}$ |
| Dx_{50} | $159-267\mu{ m m}$ | $192-324\mu m$ |
| Dx_{90} | $281-891\mu m$ | $339-840\mu m$ |



(a) By sieve weight for every hole surveyed.



Concentrate - Laser PSD results

(b) By laser diffraction (volume) for every hole surveyed.

Figure 3.1: Cumulative particle size distribution graphs for all holes within the permit area selected for this characterisation work.







(b) Magnetic concentrate.



(c) Rejects from magnetic separation.

Figure 3.2: Summary of the laser diffraction measurements of the particle size distributions. X-axes ordered alphanumerically.



Figure 3.3: Summary of the sieve measured particle sizes for the magnetic concentrate. Note: there was no sieving of the ROM or magnetic rejects and consequently no data available. X-axis ordered alphanumerically.

3.3 X-ray fluorescence

Analysis of the chemisty of the sand is split into iron content and minor element concentrations. Turning first to the iron content, all the holes show less than 19% iron (as Fe) in the ROM (Figure 3.7a). However, after magnetic separation the iron content of the concentrate is increased to more than double the ROM value for all of the holes studied (Figure 3.7b). There is quite a range in the concentrate iron content ranging from 11% to 41%, which is consistent with the variability seen in the magnetic recovery ratios. Comparing the iron content by sieve cut size (Figure 3.7c) it is clear that the iron content is higher as the particle size becomes smaller.

For the minor constituents, the titanium (Figure 3.8a), vanadium (Figure 3.8b) and phosphorus (Figure 3.8c) concentrations are increased in the magnetic concentrate compared to the ROM, while the calcium concentration is decreased (Figure 3.8d). With decreasing particle size the titanium, vanadium and phosphorus content increases, but the calcium content decreases. The calcium content is at least two orders of magnitude greater than that of vanadium and phosphorus.

3.4 Image analysis of scanning electron micrographs

It is important to note that the scanning electron microscope (SEM) imaging was carried out only on sieved fractions of the magnetically concentrated sand and not on the ROM material. In agreement with the XRF data, the analysis of the SEM images shows the area of the particles made up of iron increases as the particle size decreases (Figure 3.9). Between 150 µm and 180 µm there is a large range of iron fractions in the particles, extending from locked to liberated levels. Above 180 µm iron becomes progressively more locked with the iron fraction below 37 % in all cases.

Greater detail can be seen in Figure 3.10. Here the iron is split into the four categories of locked, low middling, high middling and liberated as defined in section 2.5. At the lower particle sizes the majority of the sand is predominantly liberated iron – or more accurately titanomagnetite. With increasing particle size the iron becomes more bound with gangue. A noteable exception is sample X327 where we observe an unusually high percentage of liberated grains for particle sizes $250 \,\mu\text{m}$ and larger (Figures 3.10e and 3.10f).



Figure 3.4: Percentage of the particles under $50 \,\mu\text{m}$ plotted against their location in the permit area and histograms showing the scatter – From laser measurement. Histogram X-axes ordered alphanumerically.



Figure 3.5: Histogram showing the magnitude of the discrepencies in the laser PSD measurements between the ROM, concentrate and rejects for the fraction of particles under 50 µm. The difference is given by ROM - (Concentrate + Rejects). Yellow indicates there was more ROM than concentrate + rejects while red indicates the opposite. Ideally the difference should be zero. X-axis ordered alphanumerically.



(a) Histogram showing the variability in the magnetic fraction across the holes. X-axis ordered alphanumerically.



(b) Magnetic fraction collected (wt.%) with respect to the location of each hole in the permit area.Figure 3.6: Magnetic material collected through dry magnetic separation for each hole surveyed.



(a) Run of mine. Mean: 8.6 wt. %. Standard deviation: 3.2 wt. %.



(b) Magnetically concentrated. Mean: 27.5 wt.%. Standard deviation: $11.0\,{\rm wt.\%}.$



(c) Iron concentration for the magnetically concentrated sand for each of the sieve cuts.

Figure 3.7: Concentration of iron measured by XRF for each hole surveyed. X-axes indexed alphanumerically.



Figure 3.8: Concentrations of minor elements measured by XRF on the ROM, magnetic concentrate and on each of the sieve cuts of the magnetically concentrated sand. X-axes ordered alphanumerically.



Figure 3.9: The area of all the particles was measured from each image for each sieve cut size and for each hole surveyed. Iron area is the percentage of the area in the particles found to be iron. X-axis ordered alphanumerically.





X402-

X404-X510-

X515-X516-X517-X517-X520x523x526x532x532x534-

X537 X544 X5551 X5552 X5554 X557 X559

X561 X574

X521

0.

S305

STH009RC -STH010RC -X300 -

X313B X316-X320-

X321-X325-X328-X328-X330-X333-X333-X339-X339-

X309-X310-

×303-

Figure 3.10: Influence of the grain size on the partitioning of the iron in the gangue. Percentage of particles lying inside of the four defined classes of iron liberation, shown independently for each sieved fraction. Note there was insufficient sample available to analyse the two missing holes by this method. X-axes ordered alphanumerically.







(f)

Discussion

4.1 Iron Recovery

Iron recovery, Fe_{Mag} , is defined here as the iron content of the magnetically concentrated sample, reported as a mass percentage of the ROM sample. Mathematically this is defined as

$$Fe_{Mag} = Fe_{XRFConc} \times \frac{m_{MagConc}}{m_{MagConc} + m_{MagReject}}$$
(4.1)

where $Fe_{XRFConc}$ is the weight percent iron of the magnetic concentrate measured by XRF and the masses of the magnetic concentrate and rejects are given by $m_{MagConc}$ and $m_{MagReject}$, respectively. This is shown for each hole in Figure 4.1a and reveals that, depending on the hole, between 2.3% and 13.3% of the ROM is magnetically recoverable iron.

The iron content of the ROM was also measured. Subtracting Fe_{Mag} from this value yields the amount of iron in the ROM which is not recovered by the magnetic separation process and therefore lost. From Figure 4.1b it can be seen that in many cases the amount of lost iron is almost as much as the iron recovered, and in a few cases more iron is lost than is recovered. Further work is being carried out to confirm this high iron loss result. This lost iron is expected to occur either as non-magnetic oxides (ilmenite or haematite) or very small magnetite grains encapsulated in large gangue particles.

4.1.1 Magnetic separation and x-ray fluorescence

In general, there is an increase in the mass of the sand recovered by magnetic separation with increasing iron content in the ROM (Figure 4.2a). Consequently, this has an effect on the concentration of the iron after magnetic separation and likewise the spread in the iron content increases with increasing mass percent of the material recovered (Figure 4.2b).

To explain this a total locked mass fraction (TLMF) was calculated from the locked mass fraction (LMF_i) – obtained from the SEM image analysis – and mass (m_i) for each of the six sieve fraction cuts for each hole (equation 4.2).

$$TLMF = \frac{\sum LMF_i m_i}{\sum m_i} \tag{4.2}$$

This is shown for each hole in Figure 4.3a. Figure 4.3b shows the locked fractions with respect to the iron content of the magnetic concentrate. There is a clear and understandable relationship between these two attributes because an increase in the locked iron percentage inherently means there will be more gangue and consequently the iron content will be lower. The integrated locked fraction was applied to Figure 4.2b and clearly shows a separation based on this relationship (Figure 4.4). Although the magnetic recovery increases, the iron concentration varies increasingly strongly and is dependent on the amount of gangue locking the iron.

This leads to a wide degree of variability in the liberation state of particles obtained from different holes by magnetic separation. Particles of iron for a hole may be encased in more or less gangue leading to differing levels of locked iron, respectively. This means that the magnetic mass fraction of unground ROM material determined by dry magnetic separation is not a good measure of the amount of iron that is retrievable from a hole.



(a) Magnetic iron. Mean: 5.18 wt.%. Standard deviation: 2.62 wt.%.



(b) Comparison showing the iron collected and the iron rejected by magnetic separation. The iron content measured in the ROM is also included as total Fe.

Figure 4.1: Iron content of the run of mine samples. See text on the preceding page for an explanation of how the magnetic iron was calculated. X-axes ordered alphanumerically.



(a) Magnetics collected from ROM samples with respect to the iron content of the ROM.



(b) Iron content in the magnetically concentrated samples.

Figure 4.2: Relationship between the weight fraction of the sand collected by magnetic separation and iron content measured by x-ray fluorescence.



(a) Integrated fraction of the iron that is locked for each hole. This is calculated based on the locked fraction shown in Figure 3.10 and the weight for each cut. See text for more detail. X-axis ordered alphanumerically.



(b) Comparison between the integrated locked fraction from Figure 4.3a with the concentrate iron content measured by XRF.

Figure 4.3: Integrated locked fractions and their correlation with the iron content measured in the concentrate.



Figure 4.4: Integrated locked fraction applied to Figure 4.2b. The value of the integrated locked fraction is indicated by the colour map. XRF measurements were made for the samples with no colour on the bottom right, but there was insufficient material available to also carry out the SEM analysis from which the locked fraction of the iron was calculated.

4.1.2 Integrated iron content

The iron content was integrated (IIC) with respect to the mass of each sieve fraction cut using equations 4.3 and 4.4. Subscripts I and D are used to denote whether the calculation is based on increasing or decreasing particle size, respectively.

$$IIC_{I}(i) = \frac{\sum_{n=1}^{i} Fe_{XRF_{n}}m_{n}}{\sum_{n=1}^{i} m_{n}}$$
(4.3)

$$IIC_{D}(i) = \frac{\sum_{n=i}^{6} Fe_{XRF_{n}}m_{n}}{\sum_{n=i}^{6} m_{n}}$$
(4.4)

where Fe_{XRF_n} and m_n are the XRF measured iron concentration and mass for sieve cut n, respectively. This calculation was made for each incremental sieve cut i and the results are presented in Figures 4.5a and 4.5b.

Equation 4.3 and the resulting Figure 4.5a can inform decisions on the size threshold below which the sand may be sold without further beneficiation. We assume the criterion for selecting a sieve cut to be excused grinding is for Fe_{XRF} to be 55% or greater. This must have the prerequisite condition that the level of phosphorus be lower than 0.18 wt % and will be considered in section 4.2. There are few holes that meet this criterion, even at the finest particle sizes (Figure 4.5a).

Equation 4.4 and the resulting Figure 4.5b may be used to decide if there is a particle size above which it is not economic to process. We have assumed that Iron content below 3.5 wt. % is likely to be unsuitable for further processing. From Figure 4.5b we observe that all the sand collected across the whole particle size distribution would be worth processing.

4.2 Relationships of minor elements with iron

In addition to iron in the magnetic concentrate, other elements are also present. Minor elements are important because they can impact down-stream processes, either as a problem due to unintended alloying effects, or as a potentially beneficial additional product stream.

Vanadium is present in the iron sand and has high value. It could become another revenue stream if it is present in high enough concentrations and can be extracted economically. Throughout all of the



(a) Integration with increasing grain size according to equation 4.3. This indicates a % Fe grade for a sieve cut of the specified size from the LIMS 1 concentrate. May be useful in determining if the sand can be sold "as is" for a given particle size.



(b) Integration with decreasing grain size according to equation 4.4. May be useful to determine if there is a particle size above which it is uneconomic to process.

Figure 4.5: Integrated iron content for each sieve cut. X-axes ordered alphanumerically.



Figure 4.6: Relationship between XRF Fe (wt. %) and selected minor elements in the magnetically concentrated iron sand.



Figure 4.7: Comparison of hole iron content derived from x-ray fluorescence and from scanning electron micrographs.

sand samples surveyed, the concentration of vanadium is approximately 200 times lower than iron, and scales almost linearly with the iron concentration (Figures 4.6c and 4.6d). Titanium is also present and like vanadium its concentration also scales almost linearly with iron concentration. Similarly to iron, the concentration of both vanadium and titanium is highest for the smallest particle sizes.

The linear relationships in Figures 4.6b and 4.6d allow for a determination of the stoichiometry of the titanomagnetite phase. Fitting a linear relationship to Figures 4.6a and 4.6c yields slopes of 0.088 and 0.006 for the titanium and vanadium, respectively. Thus we calculate the titanomagnetite to be in the form $Fe_{2.74}Ti_{0.24}V_{0.02}O_4$.

Phosphorus is also present in the sand, and can be detrimental to the mechanical properties of steel because - although it increases strength and hardness - it also causes a reduction in ductility and impact toughness. There is an increase in the level of phosphorus with increasing iron content in the sand (Figure 4.6e). However, the rate of increase in phosphorus concentration is lower than the rate of increase of iron so the ratio of phosphorus to iron (P/Fe) decreases with increasing iron concentration (Figure 4.6f). Since the P/Fe ratio is lowest in the finest particles, this suggests that the total phosphorus level in the coarser particles may be reduced through grinding to smaller particle sizes than are found in the unmilled concentrate.

The calcium present is contributed by the gangue material. So, it is natural to see a decrease in the calcium content with decreasing grain size and increasing iron content (Figure 4.6g). For the sieve cuts above $250 \,\mu\text{m}$ the calcium concentration is usually between $50 \,\%$ and $100 \,\%$ that of iron and occasionally higher. Similarly to phosphorus grinding of the iron sand is expected to reduce the calcium concentration.

4.3 Electron microscopy and x-ray fluorescence

In this work we have used two different techniques to measure the Fe % in the ROM. These are by XRF chemical analysis, and by analysis of images collected from the SEM. There is a clear relationship between the two techniques (Figure 4.7), but it is not linear. This arises because the SEM image analysis relies on an area average while XRF uses a mass average. Due to the differences in density between the

titanomagnetite and the gangue the relationship is hyperbolic and can be expressed as

$$Fe_{XRFConc} = \frac{A_{FeSEM} \times \rho_{TM}}{A_{FeSEM} \times \rho_{TM} + (1 - A_{FeSEM}) \times \rho_g}$$
(4.5)

where ρ_{TM} and ρ_g are the densities of the iron and gangue, respectively, and A_{FeSEM} is the area fraction of the particles made up of iron (or more specifically iron oxide predominantly in the form of titanomagnetite). There is a good correlation between the two techniques with little deviation of the measured data from the fitted curve. Since XRF is a recognised method for elemental analysis, this provides a clear calibration basis for the SEM measurements. Additionally, the SEM technique gives further information on the condition of the iron (i.e., locked, low middling, high middling, liberated) that the XRF technique does not provide.

Summary

Research aim 1.1 – Understanding the feedstock variability – is now complete. Main findings from this work are listed below.

- Depending on the drill hole selected, between 2.3% and 13.3% of the ROM is magnetically recoverable iron.
- In many cases there is as much iron lost in the magnetic rejects as there is collected by magnetic separation. The form of this iron is still to be investigated, but it is clearly not highly ferromagnetic.
- Comparing the $Fe_{XRFConc}$ with the magnetic mass fraction obtained in this work by dry separation shows that magnetic mass fraction is not a good measure of the total iron recoverable for a hole.
- Only 20% of the holes have a sieve cut which may be defined that would pass a 55 wt. % iron threshold for selling the ore as mined.
- Using 3.5 wt. % iron as the minimum cut-off below which it is not economic to process, we find that the whole particle size distribution is worth processing for each of the synthetic ROM samples analysed. This seems to indicate that the mining depth criteria employed to generate the synthetic ROM samples are appropriate and do not result in the removal of excess low grade ore.
- Phosphorus concentrations increase with increasing iron content, but the ratio of phosphorus to iron concentration decreases. This implies that grinding prior to magnetic processing should further reduce the phosphorus in the separated product, potentially enabling commercial threshold levels to be met.
- Titanium and vanadium scale in the same proportions with respect to iron throughout all of the holes studied, thus indicating that the titanomagnetite stoichiometry is uniform throughout the permit area. It is found to be in the form Fe_{2.74}Ti_{0.24}V_{0.02}O₄.

A separate report will be provided to TTR containing detailed measurment data obtained from each survey hole. A further report will also be provided, discussing the use of saturation magnetisation measurements as an alternative approach to determining the magnetic iron within ironsand samples obtained from TTR's permit area.
CallaghanInnovation

Report for Trans-Tasman Resources Limited

Single Page Summaries for Collected Iron-sand Holes

Prepared by: Nigel Ross, James Storey, Vlatko Materić

Date: 5 July 2017

Single page sheets have been compiled summarising the results of the characterisation work for each of the holes selected from the holes collected from the permit area by Trans-Tasman Resources Limited (TTR) – See Figure 1 on next page. These summary sheets have been completed based on the understanding Callaghan Innovation (CI) and the Robinson Research Institute (RRI) have on the requirements TTR make for classifying the sand. As such they are not a final version and feedback is encouraged from TTR on how these summaries may be tailored to better provide information they need.

At present, information is missing from the summaries on the range in particle sizes to use for grinding. Although a lot of data has been generated there are no criteria available to CI and RRI on which to make informed judgements based on our SEM liberation data. These criteria need to be developed in consultation with TTR and are expected to become clearer during the grinding studies.

There are only 8 holes for which sieve cuts meet the 55 wt. % iron content criterion provided by TTR. Depending on the hole selected this would yield between 15 and 93 kg iron per tonne of ROM processed. However, the phosphorus contents are at levels where acceptance is becoming marginal.

| | Range | Average | Standard deviation |
|-------------------------|---------------------------------|---------------------------|-----------------------------|
| Total Iron in ROM | 49 – 184 kg tonne ⁻¹ | 86 kg tonne ⁻¹ | 32.4 kg tonne ⁻¹ |
| Magnetic Iron in ROM | 23 – 134 kg tonne ⁻¹ | 52 kg tonne ⁻¹ | 26.2 kg tonne ⁻¹ |
| Fe in concentrate (XRF) | 11.0 – 40.9 wt. % | 27.5 wt. % | 7.36 wt. % |
| P in concentrate (XRF) | 0.13 – 0.20 wt. % | 0.17 wt. % | 0.01 wt. % |

Below is a table summarising important results across all 40 holes selected from the permit area.

The following pages are:

- A summary of the single page sheets for the run of mine and simulated LIMS1 (Concentrate) samples.
- The single page sheets for each hole. Explanatory notes may be found on the reverse side of each sheet to allow each one to be used independently of the rest or this report.



Figure 1 Location of the sample holes drilled by TTR and those selected for this characterisation work (Green circles).

S305

Summary











Definitions

Total Iron - Total iron in the ROM (= Fe_{XRF ROM} × 10) Mag. Iron - Total iron in magnetics from the ROM (= Fe_{XRF Concentrate} × %_{magnetics} × 10) Mag. Phos - Total phosphorus in magnetics from the ROM (= P_{XRF Concentrate} × %_{magnetics} × 10) Proposed sieve size - For the concentrate based on 55 wt. % Fe cut-off (See "Sieving cut size selection" graph) Sieved Fe grade - Integrated iron content for the proposed sieve size Sieved P grade - Integrated phosphorus content for the proposed sieve size Fe recoverable - Weight of iron recoverable by sieving the concentrate Magnetics - Weight percent of magnetically separated sand with respect to the pre-separation weight

Scanning electron microscope image analysis

A particle can contain multiple iron grains.

Standard deviations are sample standard deviations. i.e.,

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

Iron Fraction

Iron fraction values are averages over the number of images n (=64 typically).

The total iron fraction is given by (# iron pixels/# particle pixels)*100 and includes edge particles.

The iron fractions for liberated, high-middling, low-middling and locked particles exclude edge particles.

Iron and Particle sizes

Diameter values are effective circular diameters calculated from

 $d = 2 \sqrt{\frac{A}{\pi}}$, where A is the area.

The total iron size and total particle size are averages of the average values from each image. They include <u>no</u> stereological correction.

STH009RC

Summary











Definitions

Total Iron - Total iron in the ROM (= Fe_{XRF ROM} × 10)

Mag. Iron - Total iron in magnetics from the ROM (= $Fe_{XRF Concentrate} \times \%_{magnetics} \times 10$)

Mag. Phos - Total phosphorus in magnetics from the ROM (= $P_{XRF Concentrate} \times \%_{magnetics} \times 10$)

Proposed sieve size - For the concentrate based on 55 wt. % Fe cut-off (See "Sieving cut size selection" graph)

Sieved Fe grade - Integrated iron content for the proposed sieve size

 $\ensuremath{\textit{Sieved}}\ensuremath{\,P}\xspace$ grade - Integrated phosphorus content for the proposed sieve size

Fe recoverable - Weight of iron recoverable by sieving the concentrate

Magnetics - Weight percent of magnetically separated sand with respect to the pre-separation weight

Scanning electron microscope image analysis

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Iron and Particle sizes

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STH010RC











Definitions

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Mag. Iron - Total iron in magnetics from the ROM (= $Fe_{XRF Concentrate} \times \%_{magnetics} \times 10$)

Mag. Phos - Total phosphorus in magnetics from the ROM (= $P_{XRF Concentrate} \times \%_{magnetics} \times 10$)

Proposed sieve size - For the concentrate based on 55 wt. % Fe cut-off (See "Sieving cut size selection" graph)

Sieved Fe grade - Integrated iron content for the proposed sieve size

 $\ensuremath{\textit{Sieved}}\ensuremath{\,P}\xspace$ grade - Integrated phosphorus content for the proposed sieve size

Fe recoverable - Weight of iron recoverable by sieving the concentrate

Magnetics - Weight percent of magnetically separated sand with respect to the pre-separation weight

Scanning electron microscope image analysis

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Scanning electron microscope image analysis

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Fe recoverable - Weight of iron recoverable by sieving the concentrate

Magnetics - Weight percent of magnetically separated sand with respect to the pre-separation weight

Scanning electron microscope image analysis

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Sieved Fe grade - Integrated iron content for the proposed sieve size

 $\ensuremath{\textit{Sieved}}\ensuremath{\,P}\xspace$ grade - Integrated phosphorus content for the proposed sieve size

Fe recoverable - Weight of iron recoverable by sieving the concentrate

Magnetics - Weight percent of magnetically separated sand with respect to the pre-separation weight

Scanning electron microscope image analysis

A particle can contain multiple iron grains.

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Iron Fraction

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Iron and Particle sizes

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X313B

Summary











Definitions

Total Iron - Total iron in the ROM (= Fe_{XRF ROM} × 10)

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Proposed sieve size - For the concentrate based on 55 wt. % Fe cut-off (See "Sieving cut size selection" graph)

Sieved Fe grade - Integrated iron content for the proposed sieve size

 $\ensuremath{\textit{Sieved}}\ensuremath{\,P}\xspace$ grade - Integrated phosphorus content for the proposed sieve size

Fe recoverable - Weight of iron recoverable by sieving the concentrate

Magnetics - Weight percent of magnetically separated sand with respect to the pre-separation weight

Scanning electron microscope image analysis

A particle can contain multiple iron grains.

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Scanning electron microscope image analysis

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Scanning electron microscope image analysis

A particle can contain multiple iron grains.

Standard deviations are sample standard deviations. i.e.,

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Iron fraction values are averages over the number of images n (=64 typically).

The total iron fraction is given by (# iron pixels/# particle pixels)*100 and includes edge particles.

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