Determination of Metallization Degree of Pre-reduced Chromite with Image and Rietveld Analysis

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Abstract
Metallization degree of pre-reduced chromite samples was determined using two methods. The chromite samples were reduced in the solid state by the use of methane-hydrogen gas mixtures. First method was image analysis of micrographs obtained by scanning electron microscope where heavier metallic phases appear as bright white areas which are relatively easy to segment using a thresholding algorithm. The second technique was Rietveld analysis of X-ray powder diffraction pattern which fits a calculated profile onto a measured X-ray diffraction pattern to gain information about phase quantities. Rietveld refinement and phase composition analysis was performed with PANalytical’s X’Pert HighScore Plus program from the XRPD (X-ray powder diffraction) data. The results from both techniques were in good agreement. Metallization degrees for the investigated samples ranged from 15 to 65 percent depending on the extent of reduction which was a function of time, reduction temperature and methane content of the gas mixture. These results are promising and show that either image analysis or X-ray Rietveld analysis can be used as a relatively fast method to determine the degree of metallization of pre-reduced samples in comparison to the slow and tedious chemical analysis.

Keywords: ferrous chromite, metallization, image analysis, Rietveld analysis

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INTRODUCTION
The pre-reduction of ferrous chromite brings advantages in the ferrochromium production when most chemical work can be accomplished at lower temperatures than smelting and only the final phase separation is then carried out at typical smelting temperatures [Kleynhans et al. 2017]. The reaction paths of chromite with methane are complicated due to the formation of carbides at relatively low temperatures [Anacleto et al. 2018; DeCampos & Eric 2006]. This conveys difficulties in the analysis of reduction products and thus reduction or metallization kinetics accompanied with carbide formation, cracking of the excess methane and soot formation on the surfaces of the reacting samples [Canaguier & Kolbeinsen 2018].

In this work, the aim is to determine how much metal, whether pure or carbide, was yielded by the methane prereduction experiments performed on Kemi Chromite ore of Finland. Two different analysis methods are utilized: image analysis of scanning electron microscope (SEM) micrographs and Rietveld analysis of X-ray powder diffraction (XRPD) patterns.

Image analysis (SEM)
Often the first step in image analysis is to divide an image into meaningful regions which could be used for further analysis. One of the simplest segmentation methods is thresholding which transforms an image \( f(x, y) \) to a binary image \( g(x, y) \) by choosing a threshold value \( T \).

\[
g(x, y) = \begin{cases} 
1, & \text{if } f(x, y) \geq T \\
0, & \text{otherwise}
\end{cases}
\]

(1)

Each pixel \( (x, y) \) is separated into one of two classes: object and background points. If the pixel intensity value is greater than the threshold \( T \), it is an object point, otherwise it is a background point [Gonzales, Woods & Eddins, 2004]. In Fig. 1 a SEM electron micrograph of reduced chromite was thresholded using two different values. The intensity histogram of the original image shows the number of pixels at each intensity value. The intensity values go from 0 (black) to 255 (white). Using a thresholding value of 30 effectively selects all but the darkest pixels. In order to select only the brightest pixels, a higher thresholding value, such as 240, needs to be selected.

A suitable threshold value can be chosen by evaluating the image by eye while changing the threshold value. However, this manual way quickly becomes tedious when processing large amounts of images and the attained results might not be reproducible. For this reason, thresholding is usually automated via an algorithm. There are several types of thresholding methods [Sezgin, Sankur & Survey, 2004] but in this work the focus was on the histogram shape-based methods which analyze, for example, the peaks and valleys of the histogram. Two algorithms discussed next were both global thresholding techniques which means the thresholding value was held constant throughout the image.
**Figure 1.** Thresholding the original electron image with a value of 30 basically includes all but the darkest pixels, while a thresholding value of 240 only selects the brightest pixels as object points and marks the rest as background points.

**Otsu’s Method**
Idea behind the Otsu’s method [Otsu, 1979] is to find the threshold that minimizes the within-class variance. This method assumes that the histogram is bimodal (has two maxima) and the valley between these two peaks is deep and sharp. Also, illumination should be uniform so only the object appearance differences cause the brightness behavior [Kittler & Illingworth, 1985]. The gray-level histogram \( h(i) \) of the examined image was first normalized and treated as a probability distribution.

\[
h(i) = \frac{n_i}{N_p}, \quad h(i) \geq 0, \quad \sum_{i=1}^{L} h(i) = 1
\]

where \( N_p \) is the number of pixels in the image, \( n_i \) is the amount of pixels at intensity level \( i \) and \( L \) is the total number of possible intensity levels in the image (256 for an 8-bit gray-level image). Now, pixels in the image were divided into two classes, \( C_0 \) and \( C_1 \) (background and objects, respectively) by a threshold value \( T \). So, \( C_0 \) denotes pixels with intensity levels \([0, \cdots, T-1]\) and \( C_1 \) refers to pixels with levels \([T, \cdots, L-1]\). The occurrence probabilities for these classes were calculated through;
Then the mean intensity and class variance values were evaluated statistically and from these through iteration of all the possible threshold levels $T$ (from 0 to 255 for an 8-bit image), the one that yields the lowest class variance value was chosen as the threshold.

**Huang’s Method**

Huang’s method [Huang & Wang, 1995] utilizes fuzzy logic with the selection of an optimal threshold. In contrast to Boolean logic where the variable values can be either 0 or 1, fuzzy logic allows variables to be any real number between 0 and 1. This is beneficial when selecting a threshold because class probability densities often overlap due to ambiguous data. Fuzzy logic deals with fuzzy sets whose elements have degrees of membership described with the aid of a membership function. In effect, this means that one element can be part of multiple sets. In case of selecting a threshold, a fuzzy set (an image) was considered as an array of fuzzy singletons (image pixels), each having a membership value associated with the intensity level of the pixel. Image $I$ could then be represented as

$$I = \{f(x,y), \mu_I(f(x,y))\},$$

where $\mu_I$ is the membership function. In this method the normalized histogram $h(i)$ which groups all pixels $f(x,y)$ having the same intensity/gray levels $i$, was utilized to calculate the membership function together with mean intensity values/levels with statistical procedures which is rather complex and beyond the scope of this paper and hence will not be illustrated here. The calculation procedure eventually provided the chosen threshold value.

**Rietveld analysis**

In order to get information about the crystal or chemical structure of a sample, it was necessary to build a mathematical diffraction pattern and compare it to the measured one. Integrated intensity $I_{hkl}$ for each $hkl$ (representing the crystallographic plane indices) reflection could be calculated with Eq. (6):

$$I_{hkl} = (F_{hkl})^2 \times LP_{hkl} \times P_{hkl} \times A \times E_{hkl}$$

where $F_{hkl}$ is the structure factor, $LP_{hkl}$ is the Lorentz-polarization factor, $A$ is the absorption factor, $P_{hkl}$ is the preferred orientation factor and $E_{hkl}$ is the extinction factor [Cullity, 1978]. The explanation and mathematical description of all the above mentioned factors is again complex and beyond the scope of this paper and can be found elsewhere [Leino, 2017]. The integrated intensity equation gives a single intensity value for a single $2\theta$ angle. This value is used to represent the area under a real X-ray diffraction peak. The Rietveld method distributes this single value across a range of $2\theta$ angles creating a peak profile similar to the observed one. The aim is to minimize the difference between the
calculated and observed peak profiles by using the method of least-squares. A simple and widely used way of fitting the background is to use a polynomial function in $2\theta$. Usually, Rietveld programs use a 5<sup>th</sup> degree polynomial and refinement of only three of the five coefficients is enough to achieve a good background fit. All of the five coefficients are needed only for more complex backgrounds. Also, for very complex backgrounds, specific background formulas are available [Taylor & Hinczak, 2006]. In order to set the calculated profile of each phase onto the same scale as the measured one, a scale factor is employed. Shape of the measured profile peak is dependent on various aspects such as the characteristics of the incident beam, experimental arrangement, diffractometer and sample size and shape [Delhez, 1992; Suortti, 1992]. Various peak shape functions are used to approximate these effects. In his original paper, Rietveld [Rietveld, 1969] used a Gaussian peak shape profile.

The Rietveld refinement method makes use of refinement parameters which are divided into two groups: global and phase-dependent. Global parameters include the machine zero offset, specimen displacement and pattern background etc. and are, for the most part, stable. Phase-dependent parameters include phase spike widths, unit scale dimensions and so on. Some of these phase-dependent parameters are stable but most are unstable. An effective refinement strategy involves a step-by-step parameter turn on sequence where only the stable parameters are refined first and additional parameters are turned on for the following cycles [Young, 1992]. This kind of systematic approach aims to prevent situations where a premature refinement of non-linear and very unstable parameters leads to erratic behavior or failure of the whole refinement. When parameters are turned on one by one, it is also easier to spot a troublemaking parameter if the refinement is not going well.

In this work Rietveld refinement and phase composition analysis was performed with available PANalytical’s X’Pert HighScore Plus program from the XRPD data which utilizes all the principles mentioned above.

**RESEARCH METHODS**

Image analysis was performed on the SEM micrographs taken from Leikola’s [Leikola, 2015] samples in addition to those of the newly prepared and reacted samples. Each new sample was prepared from approximately five grams of chromite concentrate powder (from Kemi, Finland) which was reduced for 60 minutes at temperatures and CH<sub>4</sub>-H<sub>2</sub> atmospheres shown in Table 1. Additionally, three E series of experiments (E04, E10 and E16) were also performed at 1350ºC with 30% methane in the gas mixture and a duration of 75, 90, and 120 minutes. To decrease the particle size and ensure a uniform size distribution, each sample was ground with the Retsch XRD-Mill McCrone for 10 minutes. Size distribution of each ground sample was examined with Malvern Instruments Mastersizer 2000. After the XRPD measurements, the powder samples were cast into epoxy and wet ground with silicon carbide papers and polished with 3 and 1 μm monocrystalline diamonds. In order to increase the conductivity and get better image quality in SEM, the polished samples were carbon coated with a Leica EM SCD050 cool sputtering device.
The Rietveld analysis on E series samples (see Table 2) were rather erratic due to extensive carbon precipitation and hence were abandoned.

The gas components used were argon (Ar 99.999 %), methane (CH$_4$ 99.5 %) and hydrogen (H$_2$ 99.99 %) from Aga-Linde and total gas flow was kept at 800 mL/min during the experiments. A closed electrical, horizontal tube furnace was utilized to conduct the experiments. SEM micrographs were taken with a LEO model 1450 VP (Carl Zeiss, Germany) using Oxford Instruments INCA software at x1500 magnification. A total of approximately 25 images were taken for each sample.

The SEM micrographs had four distinctive phases in addition to the black epoxy. Leikola’s [Leikola, 2015] study confirmed this, where her samples, examined using energy dispersive X-ray spectroscopy (EDS), showed the phases to be silicate (very dark gray), two kinds of chromite (dark-partially reduced and light gray-unreduced) and metal/metal carbide (white). These phases are marked in Figure 2. MATLAB (R2016a) with the Image Processing Toolbox (version 9.4) was used to perform the image analysis.

**Table 1.** Experimental temperatures and atmospheres employed for each sample.

| Sample | T [°C] | CH$_4$ [vol%] | CH$_4$ [ml/min] | H$_2$ [vol%] | H$_2$ [ml/min] |
|--------|--------|----------------|----------------|---------------|----------------|
| A04    | 1100   | 10             | 80             | 90            | 720            |
| A10    | 1100   | 20             | 160            | 80            | 640            |
| A16    | 1100   | 30             | 240            | 70            | 560            |
| B04    | 1200   | 10             | 80             | 90            | 720            |
| B10    | 1200   | 20             | 160            | 80            | 640            |
| B16    | 1200   | 30             | 240            | 70            | 560            |
| C04    | 1300   | 10             | 80             | 90            | 720            |
| C10    | 1300   | 20             | 160            | 80            | 640            |
| C16    | 1300   | 30             | 240            | 70            | 560            |
| D04    | 1350   | 10             | 80             | 90            | 720            |
| D10    | 1350   | 20             | 160            | 80            | 640            |
| D16    | 1350   | 30             | 240            | 70            | 560            |

First, Otsu’s method [Otsu, 1979] was used to calculate three threshold levels for the SEM micrograph to separate the phases. This caused the last threshold level to include some brighter gray areas in addition to totally white ones. Thus, this last threshold section was thresholded again. This additional thresholding was done with the Otsu’s method [Otsu, 1979] but for some images this approach was too generous, i.e. it still caused some light gray areas to remain. Huang’s method [Huang & Wang, 1995] was found to be more suitable for the images in question and was thus used to further segment the brightest phases.
Figure 3 shows the original SEM micrograph and an image where the brightest threshold level was colored in green and placed on top of the original image for comparison. As one can see, the thresholding method finds the brightest metal phase with good accuracy. Next, the original image was separated into binary sub images according to the calculated threshold levels. This way, each phase had its own sub image where a white pixel indicated the phase is present and a black pixel that it is absent. To exclude the black epoxy from the calculations, area fraction $A_f$ was calculated for each phase by dividing the area of the phase with area of all phases. This area percentage was then converted to a mass percentage $M_p$ by multiplying with the density of the phase in question: The brightest phase was found, in the Rietveld analysis, to consist mostly of chromium carbide so a density of 7.8 g/cm$^3$ was used for it [ref]. A density of 4.8 g/cm$^3$ was used for the two chromite phases and a density of 2.0 g/cm$^3$ for the darkest phase consisting of silicates.

![Figure 2.](image)

**Figure 2.** Different phases found in the sample; the bright white phases are metal or metal carbides, the darker and lighter gray are unreduced chromite and the very dark gray includes silicates.

All density values were given by PANalytical’s X’Pert HighScore Plus program (version 4.0). Finally, the mass percentages were normalized. Even though the mass percentages were calculated for all of the phases, only the most interesting metal phase was analyzed and discussed.

XRPD measurements were conducted with PANalytical’s X’pert3 Powder X-ray diffraction system using Cu K$\alpha$ radiation. Samples were back-loaded into a
spinning sample holder. Qualitative analyses were conducted with the PANalytical’s X’Pert HighScore Plus program (version 4.0). The program rated different minerals based on how well they fitted on the XRD pattern. The likely minerals contained by the sample were chosen with the help of the score given by the program and prior knowledge of the sample and reduction process. The quantitative Rietveld analyses were also performed with the same software, using refinement steps suggested by Taylor [Taylor & Hinczak, 2006] and Young [Young, 1992].

RESULTS AND DISCUSSION

The results from the image and Rietveld analyses are presented and discussed here. It is evident from experiments that the metal content of the partially reduced samples increases with increasing reduction temperature. The CH₄ content or reduction time does not seem to play a major role at the lower reduction temperatures. At higher temperatures, the methane content plays a much more significant role especially when the reduction time is short. Image analysis were also performed in this study on
Figure 3. Original SEM micrograph and an image, where the brightest threshold level is calculated with the algorithm, colored green and placed on top of the original image.

on Leikola’s [Leikola, 2015] available samples which were already mounted and polished. Table 2 compares the metal content results between the image
analysis performed on Leikola’s samples and samples from this work for experiments performed under similar conditions. Leikola’s samples have higher metal content across the board but the difference is not significant. The difference may be due to the less amount of Leikola’s samples (1g compared to 5 g samples of this work) used in the experiments. The percent metallization values determined by chemical methods on Leikola’s samples are also in good agreement with the values reported in Table 2. The standard deviations as a percentage of all the samples recorded in Table 2 were in the 0.22% to 1.66% range.

**Table 2.** Image analysis and Rietveld analysis results for the metal content (% metallization) of reduced samples from Leikola’s work [Leikola, 2015] and this work. Results from E series of experiments are only from this work conducted at 1350°C with 30% methane for 75 (E04), 90 (E10) and 120 (E16) minutes.

| Sample | Leikola samples (image analysis) wt% metal | This work (image analysis) wt% metal | This work (Rietveld analysis) wt% metal |
|--------|-------------------------------------------|-----------------------------------|----------------------------------------|
| A04    | 22.91                                     | 20.14                             | 19.27                                  |
| A10    | 20.93                                     | 19.21                             | 20.34                                  |
| A16    | 21.41                                     | 18.17                             | 17.86                                  |
| B04    | 32.90                                     | 31.96                             | 32.11                                  |
| B10    | 35.79                                     | 34.92                             | 36.04                                  |
| B16    | 28.91                                     | 27.76                             | 29.91                                  |
| C04    | 47.30                                     | 46.91                             | 45.63                                  |
| C10    | 46.52                                     | 45.86                             | 43.92                                  |
| C16    | 48.28                                     | 47.89                             | 46.11                                  |
| D04    | 51.75                                     | 50.81                             | 48.24                                  |
| D10    | 58.17                                     | 58.01                             | 57.51                                  |
| D16    | 18.65*                                    | 66.09                             | 65.43                                  |
| E04    | -                                         | 72.61                             | **                                     |
| E10    | -                                         | 76.86                             | **                                     |
| E16    | -                                         | 83.62                             | **                                     |

Figure 4 shows an example XRD pattern of sample B16 where the major peaks are labelled. As can be seen, most of the peaks belong to the three major phases: chromite, chromium carbide and spinel. The carbon peak around $2\theta = 26^\circ$ included at least two different carbon phases which were identified to be graphite 2H and graphite 3R. The range $43^\circ - 46^\circ$ also includes other phases, such as iron and iron carbide, buried under the chromite and chromium carbide peaks. Due to severe overlapping of the phase peaks, especially at around $43^\circ - 46^\circ$, some phases were likely missed during the qualitative analysis. Thus, some form of additional and more accurate analysis would have been beneficial to identify these minor phases which would have increased the accuracy of the Rietveld refinement.

The results from Rietveld analysis for the samples of this work are also presented in Table 2. Figure 5 illustrates the results of Rietveld procedure in a column graph. It must be noted that the carbon, mineral, chromite and carbide/metal content graphs combine the corresponding phases of all the
graphite, existing non-reducible oxide phases like magnesium-aluminium spinels, as yet unreduced chromite phase and all the metallic phases including all the carbides respectively. Carbon content was around 25–35 weight percent in the A and B sets where the amount of methane does not seem to affect the carbon deposition. In the higher temperature experiments (C and D sets) the carbon content was highly affected by the experiment atmosphere but the amount of carbon remained more or less the same as in the A and B sets or even lower, with the exception of sample C16 which had a much higher carbon level, probably due to the high level of CH$_4$ (30 vol%) in the gas. It is also possible that i) the carbon may deposit on the walls of the furnace tube or the outer surface of the crucible at the higher temperatures or ii) reduction process consumed more carbon as the temperature rose and thus the carbon content was lower. However, the carbon content of the E-series samples (where the reduction periods were longer and gas phase contained 30 % CH$_4$ at the highest temperature of 1350 °C ) were very high, and well above 60 % and

![Figure 4. XRD pattern of the sample B16 where the major peaks are labeled.](image)

suppressed the peaks of the other constituents in such a way that reliable and consistent results could not be obtained. Chromite content decreases as the reduction temperature increased as expected, with the exception of samples D04 and D10, which were also not in accord with the increasing carbide/metal contents. This may be due to peak overlaps. Mineral content was not affected by the experiment parameters and remained more or less the same across all samples. The carbide/metal content followed the same trend with the image analysis results for this work’s samples.
For calculation purposes, when necessary (i.e. when the total analysis value deviated from 100 %) the percentage of carbon, chromite, mineral and carbide/metal content values directly from the Rietveld analysis were first normalized to 100 %. This procedure slightly changed the recorded percentages of the reported phases. Then the normalized carbon percentage was subtracted from 100 % and the total of the values of the three remaining phases were again normalized to 100 %. The subtraction of carbon values was obviously due to the fact that carbon had precipitated directly from the gas phase and is not a constituent of the reduced chromite. Thus, the total content of three phases; chromite, mineral and carbide/metal added up to 100 %. The new normalized values for the metal phase now represent the fraction of metallization of the reduced chromite sample. Percent metal values in Table 2 for Rietveld analysis were calculated by this procedure. A comparison of the percent metal values in Table 2 from the image analysis and from the Rietveld analysis clearly shows that the agreement between the two sets of results is excellent.

![Graphs showing carbon, chromite, mineral and carbide/metal contents](image)

**Figure 5.** Carbon, chromite, spinel and metal contents acquired from the Rietveld analysis.

The chemical analysis was made on samples A10, B10, C10 and D10 from Table 2 yielding the following metallization degrees:
A10: 20.1 %, B10: 36.0 %, C10: 45.9 %, and D10: 58.0 %.

These numbers represent total metallization in terms of Fe and Cr metallization and defined as following:

\[ M_i (\text{metallization of species } i) = \frac{(\% \text{ of metallized } i \text{ in the sample by chemical analysis})}{(\text{total } \% \text{ of } i \text{ in the sample from ore composition})}. \]

Obviously these metallization degree percentages are slightly different than the total metal % values from image analysis and Rietveld analysis as they determine the metal phase percentage over the whole sample, but the agreement is excellent proving the fact that both of these techniques can give very good representative results if done properly.

**SUMMARY AND CONCLUSIONS**

The aim of this work was to determine the metal content of reduced chromite concentrate samples using image analysis and Rietveld analysis. The samples produced by Leikola [Leikola, 2015] were used as a basis for image analysis but since no sample powder was left for the XRDP measurements, new experiments were conducted with the same parameters used in Leikola’s work. The four reduction temperatures used were 1100°C (A-set), 1200°C (B-set), 1300°C (C-set) and 1350°C (D and E-sets) and the three methane contents were 10, 20 and 30 volume percent with hydrogen gas being the balance. The reduction period for the A-, B-, C- and D-sets was chosen as 60 minutes. The E-set of tests were conducted at 1350°C using 30% CH₄ in the gas phase for reduction periods of 75, 90 and 120 minutes.

The image analyses were conducted from SEM micrographs using a thresholding algorithm written in MATLAB. A total of four phases were separated from the micrographs and for each phase, a normalized weight percentage was calculated. Only the metallized phase consisting of metal alloys and carbides was of interests in this work for pre-reduction of reduced chromite. For all the samples, the metal content was found to steadily increase with the higher reduction temperature, increasing reduction period and increasing CH₄ content in the gas phase. Results for the samples produced in this work were slightly lower across the board then Leikola’s samples. The likely causes for the differing metal content were i) smaller particle size of this work’s samples which created a more uniform phase distribution and prevented formation large metal particles that has the tendency of dominating the analysis and ii) a larger sample size used in this work which might have slowed the reduction process. Overall, image analysis worked well for the samples in question. The bright metal phase was easy to segment from other gray phases. Also, hundreds of SEM micrographs were quickly analyzed with the algorithm. However, since the bright area of the SEM micrograph included all the heavier metallic phases, they were always analyzed as one bundled phase without an option for a more broken down examination.
Rietveld refinement and the phase composition analysis was performed with PANalytical’s X’Pert HighScore Plus program from the XRPD data. Metal content results from the Rietveld analysis were quite close to those obtained from image analysis. However, a more accurate knowledge of the mineral composition would have likely increased the quality of the results gained from the Rietveld analysis. Another factor was the low number of parameters refined for the minor phases in order to keep the refinement stable. Altogether, the Rietveld refinement is an effective quantitative analysis method but it requires experience from the user. One has to precisely know the chemical/mineral composition of the studied sample, have the experience to get good XRPD measurements and have a deep knowledge of the utilized mathematics to determine whether the obtained results are feasible. Also, a high number of substances and impurities in this kind of mineralogical samples complicate the procedure and make it more time consuming.

Metallization degree results for these laboratory scale experiments look promising. Improvements for subsequent image analyses would include automation of the SEM imaging process to increase the amount of analyzed images. This way the whole sample could be imaged, reducing the chance that one highly reduced or non-reduced area dominating the analysis. If the Rietveld method is used, the chemical and mineralogical compositions of the samples need to be known accurately. Also, the analyses should be performed by an experienced operator.

We would consider image analysis to be somewhat more reliable than Rietveld method as long as significant number of representative electron images are scanned, due to the fact that it is a more direct method looking to the metallized and unmetallized regions on the samples, all of which are already visible.

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