Chemical and petrographic characterization of ilmenite and magnetite in oxide-rich cumulates of the Sokndal Region, Rogaland, Norway

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Igneous ilmenite and magnetite in some oxide-rich cumulates of the Sokndal region show chemical features and microtextures that reflect the progress of magmatic evolution and subsolidus re-equilibration. These features also relate to the patterns of aeromagnetic anomalies in the region. The most primitive oxides studied occur in the Tellnes ilmenite norite. These are followed by oxides in the Bjerkreim-Sokndal layered intrusion, first in ilmenite norites and finally in magnetite-rich norites. The trend of magmatic oxide crystallization in Bjerkreim-Sokndal, as previously determined by Wilson et al. (1996), was from hematite-rich ilmenite with minor end-member magnetite to hematite-poor ilmenite with titanomagnetite, a trend indicating progressive diminution of Fe_2O_3. This trend was accompanied by decreasing MgO and Cr_2O_3, and by gradually increasing V_2O_3 to the point where magnetite became a primary magmatic precipitate: beyond this V_2O_3 also decreased. In primitive samples the ilmenite has abundant hematite exsolution lamellae, whereas the magnetite has end-member composition and is free or nearly free of lamellae. Highly evolved samples contain near end-member ilmenite without exsolution, and titanomagnetite with fine exsolution of ulvöspinel subsequently oxidized to ilmenite and/or oxidation-exsolution lamellae of ilmenite. MgO is most strongly concentrated in ilmenite; Cr_2O_3 and V_2O_3 are strongly concentrated in hematite exsolution lamellae, but even more strongly in coexisting magnetite. The most chemically pure ilmenite, with the exception of MgO, occurs in small abundance in the most evolved titanomagnetite-rich cumulates.

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Introduction

In 1995, NGU completed a detailed aeromagnetic survey of the Sokndal region as part of a broad investigation into ilmenite resources (Rønning 1995). The resulting aeromagnetic map showed a dramatic pattern of aeromagnetic highs and lows. McEnroe et al. (1996) demonstrated that the magnetic lows are produced by rocks with a high ratio of natural remanent magnetization (NRM) to susceptibility (X), commonly referred to as the Koenigsberger ratio (Q value) where $Q = NRM/X \cdot F$ (F = present local magnetic field strength). Rocks with high Q values include ilmenite-rich norites and anorthosites with very fine-grained oxides, and the anomalies are dominated by strong remanent negative inclination. Conversely, the extreme magnetic highs are produced by rocks with a very low ratio of natural remanent magnetization to susceptibility. Rocks with these low Q values are magnetite-rich, including norites, mangerites and quartz mangerites, and give rise to induced magnetic anomalies. During the study of the rock-magnetic properties and paleomagnetism of these rocks, a series of detailed electron-probe analyses was performed on the oxide minerals, with special attention to analytical precision for major elements and the economically significant trace elements Cr and V. These are summarized here and will be explored in greater detail elsewhere. A detailed study is also in progress to understand the extremely strong and stable remanent magnetization in the ilmenite-rich rocks. The stable remanence and high Q values found in hematite-ilmenite rocks could have consequences for the interpretation of long- and short-wavelength magnetic anomalies.

Groups of samples

Mineral analyses are presented from five parts of the region (Fig. 1). The first group of samples is from the Tellnes ilmenite norite orebody in a magnetic low dominated by the reversed remanent magnetization of the Åna-Sira anorthosite. The remaining four groups are from the Bjerkreim-Sokndal layered intrusion. Of these four, two groups are from more primitive ilmenite-bearing norites that form magnetic lows in the lower part of the intrusion as exposed at Heskestad and Mydland, though well above the lowest levels exposed in the Bjerkreim lobe. The remaining two groups are from the more evolved magnetite-rich norites that form the magnetic highs in the upper part of the layered intrusion at Mydland and at Bakka.

General oxide compositions and magma evolution

The derivation and evolution of the magma from which the Tellnes ilmenite norite was formed has been much debated and is still unsettled (Krause et al. 1985, Wilmart et al. 1989,

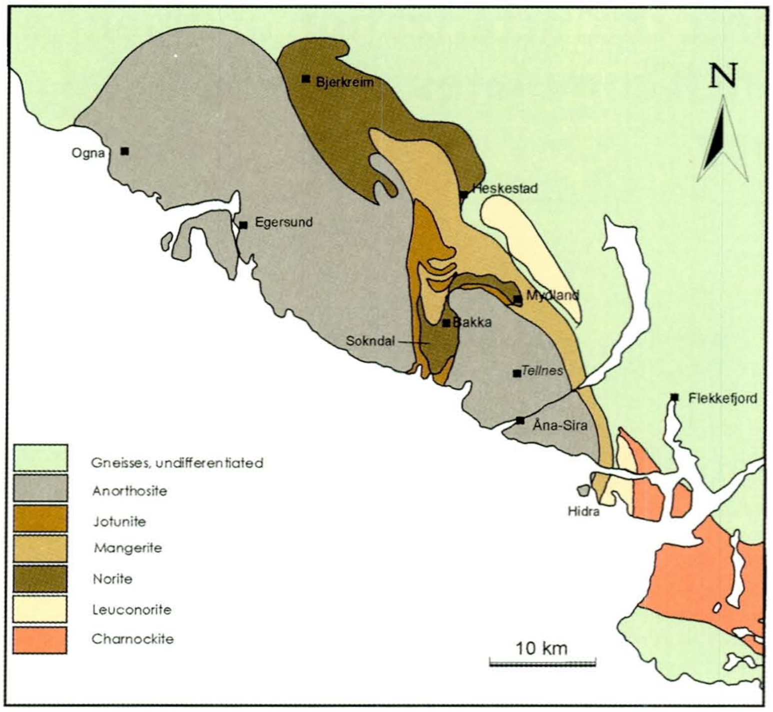

Fig. 1. Simplified geological map from NGU report 96.059 (modified from Duchesne & Michot 1987), showing the sampling localities at Tellnes Mine, Heskestad, Mydland and Bakka.

Duchesne 1999). On the other hand, the magmatic derivation and evolution of the Bjerkreim-Sokndal layered intrusion has been thoroughly documented (Wilson et al. 1996). Nevertheless, the stages of relative magmatic evolution of all the samples can be roughly worked out using such ratios as MgO/ (MgO + FeO) in the silicates, and the relative contents of MgO, V_2O_3, and Cr_2O_3 in the oxides. These show that the oxide minerals of the Tellnes ilmenite norite are more primitive than those sampled by us in the layered intrusion. Thus, the Tellnes deposit cannot be correlated with late stages in evolution of the Bjerkreim-Sokndal intrusion, nor with the internal mangerite dike system in the Åna-Sira Anorthosite, as suggested by Krause et al. (1985). Detailed geochemistry on the Tellnes ilmenite norite and the associated monzonorite to quartz mangerite dike (Wilmart et al. 1989), indicates they cannot be co-magmatic and suggests the ilmenite norite could be derived from crystallization of the enclosing anorthosite or possibly the most primitive parts of the Bjerkreim-Sokndal intrusion not covered in our sampling. More recent precise U-Pb geochronology (Schärer et al. 1996) indicates that the Tellnes ilmenite norite is about 10 m. y. younger than the other intrusions in the region, so that there are no exposed rocks available for study representing its source magma. Further extensive consideration of the origin of the oxide-rich deposits was given by Duchesne (1999).

Based on the broader study of the Bjerkreim-Sokndal layered intrusion by Wilson et al. (1996), the Heskestad, Mydland and Bakka locations of this study fall either in or slightly below their MCU-IV cyclic unit or in the upper transition from norite to mangerite and quartz mangerite. The magmatic evolution trend of the oxides of the layered intrusion (Wilson et al. 1996) involves early precipitation of hematite-rich ilmenite together with minor Ti-poor magnetite. These are followed by a gradual decrease in hematite content of ilmenite coupled with increasing Ti content of magnetite. The end result is an appearance of progressive reduction in the oxides. This was probably not produced by a change of external environment, but by extraction of cumulates, driving the magma toward more reducing compositions, even in this example where early ilmenite dominated over magnetite. This trend is not to be confused with apparent oxidation or reduction effects within individual oxide grains, that were mainly the product of localized subsolidus exchange reactions.

The oxide compositions and petrographic observations of this study illustrate very well the conclusions of Wilson et al. (1996). The overall chemical results are illustrated by four tie lines in Fig. 2, meant to show our best estimate of the equilibrium tie lines of the oxides in the triangle FeO (+MgO) - Fe_2O_3- TiO_2 under high-temperature conditions.[1] These values were obtained qualitatively from typical analyses of oxide grains, as much as possible including minor exsolution lamellae, and will be followed up by more precise estimates based on modal analysis of oxide grains. Duchesne (1999), by contrast, made bulk XRF analyses of high-purity oxide mineral separates which should give a good measure of the bulk composition of host plus lamellae. His ilmenite and magnetite compositions (his Table 4) from the Tellnes deposit compare favorably with ours, although the ilmenite actually plots closer to our TE-41 from Heskestad than to our TE-3, the most Cr-rich of our three samples from the Tellnes deposit.

The most oxygen-rich tie line (TE-3) in Fig. 2 is for the

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[1] A maddening and easily overlooked numerical feature of Fig. 2 is that the distance of an analysis point from the ilmenite toward the hematite end-member is an indirect and non-linear function of hematite content. For example, a grain of composition hematite $_{50}$, $Fe^{2+}_{0.5}\,Ti^{4+}_{0.5}Fe^{3+}_{1}O_3$, plots at 0.33 mol. % Fe_2O_3. The ilmenite TE-3, plotting at .077 mol. % Fe_2O_3, actually contains 14.5% of the hematite end-member. This problem is eliminated by plotting on a less traditional cation basis.

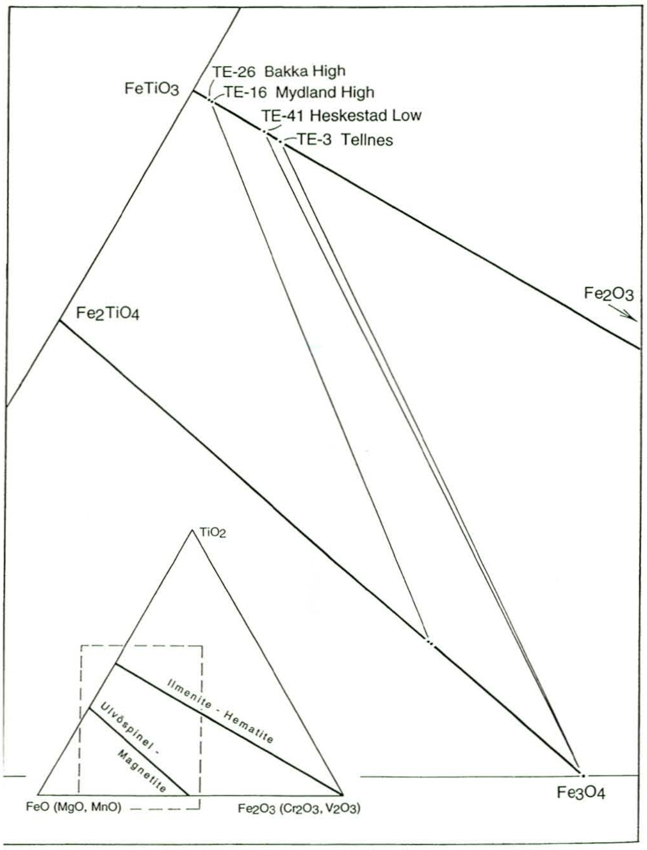

Fig. 2. Part of the system FeO-TiO_2-Fe_2O_3 in mole % (with subordinate oxides included) showing approximate equilibrium tie lines between bulk ilmenite and magnetite for four samples from the Sokndal region. Inset shows the location of the detailed diagram in the whole system. Various forms of evidence (see text) indicate that these tie lines show a progression in magmatic crystallization from most primitive (TE-3) to most evolved (TE-26), in which the oxides decreased in their oxygen content. The positions of the tie lines explain much about the subsolidus behavior of the oxides in different samples. Words 'high' and 'low' refer to field locations, the Bakka and Mydland magnetic highs, and the Heskestad and Tellnes magnetic lows.

Tellnes deposit. Another relatively primitive oxygen-rich tie line (TE-41) is for the ilmenite-rich norite of the layered intrusion in the magnetic low at Heskestad. More evolved, oxygen-poor tie lines represent the more evolved magnetite-rich norites in the magnetic highs at Mydland (TE-16) and at Bakka (TE-26).

Explanation of subsolidus equilibration

Based on the tie lines in Fig. 2, it is easy to explain the nature of subsolidus evolution at each of the locations. At Tellnes, and in the Heskestad magnetic low, the ilmenite is extensively exsolved with hematite lamellae, whereas the magnetite, where present, is a nearly Ti-free end-member composition with little or no exsolution. By contrast, in samples from the magnetic highs at Mydland and Bakka, the ilmenite is Ti-rich with relatively little hematite-exsolution, whereas the magnetite is relatively Ti-rich with oxidation-exsolution lamellae of ilmenite and/or exsolution of ulvöspinel subsequently oxidized to ilmenite. The explanations based on Fig. 2 are supported in detail, both by petrographic observations and by electron-probe analyses.

Detailed oxide petrography

Reflected- and transmitted-light microscopy were performed to understand better the magnetic phases and correlative magnetic properties, to establish if the magnetic carriers were in equilibrium with the mineral assemblage, and to determine the degree of alteration. In all samples the oxides are well equilibrated showing coarse anhedral grains. Discrete ilmenite grains are usually >1mm. Some oxides are rimmed by pyroxene, and many are in clusters with orthopyroxene and/or clinopyroxene. The samples described below correspond to the four tie lies shown in Fig. 2.

The ilmenites from the Tellnes deposit described in detail below (Fig. 3) are in sample TE-3 considered to be most primitive, based on our microprobe analyses. All ilmenites are ferrian ilmenite with multiple generations of titanohematite-hematite exsolution lamellae parallel to (0001). Second- and third- generation titanohematite lamellae are observed down to the micron scale. Based on measured magnetic properties (McEnroe et al. 1996, McEnroe 1997), it is proposed that these lamellae continue down to the nanometer scale, as has been shown for the ilmenite-hematite series in samples from the granulite region of southwestern Sweden (McEnroe 1996, Harrison et al. 1998). Pleonaste plates, as precipitates, are common in the ilmenite. Around the pleonaste plates there are diffusion haloes, where ilmenite is free of hematite exsolution (Fig. 3a). These are where the ilmenite was depleted in Fe^{3+} and hence not available for later hematite exsolution. These areas yielded the ilmenite compositions closest to $FeTiO_3$ because they are depleted in hematite exsolution. Small sulfide 'inclusions' or precipitates are present in numerous ilmenites. Coexisting magnetite grains have minor pleonaste exsolution. Where magnetite is adjacent to hemo-ilmenite, a magnetite-pleonaste-ilmenite reaction symplectite commonly has formed (Fig. 3b). Very fine-grained ilmenite with hematite exsolution, and magnetite with ilmenite oxy-exsolution and rutile plates are present in clinopyroxene grains. This magnetite-ilmenite exsolution occurs in plates and blades parallel to (010) in clinopyroxene (Morse 1970, Fleet et al. 1980). These abundant very fine-grained oxides in clinopyroxene have a strong effect on the magnetic properties and aeromagnetic signature of the Tellnes intrusion. As is to be expected from their different mode of occurence, these oxides commonly have compositions distinct from the discrete oxide grains. The chemical summary figures in this paper (Figs. 5 & 6) are limited to compositions in the discrete oxide grains.

An oxide assemblage similar to that of the Tellnes ilmenite norite, though lower in overall abundance of oxides, is found in the area of a magnetic low over part of the Bjerkreim-Sokndal layered intrusion near Heskestad. Fig. 3 shows representative microphotographs of sample TE-41 from the Heskestad region. Ferrian ilmenite and minor magnetite are the dominant opaque phases. All ferrian ilmenites

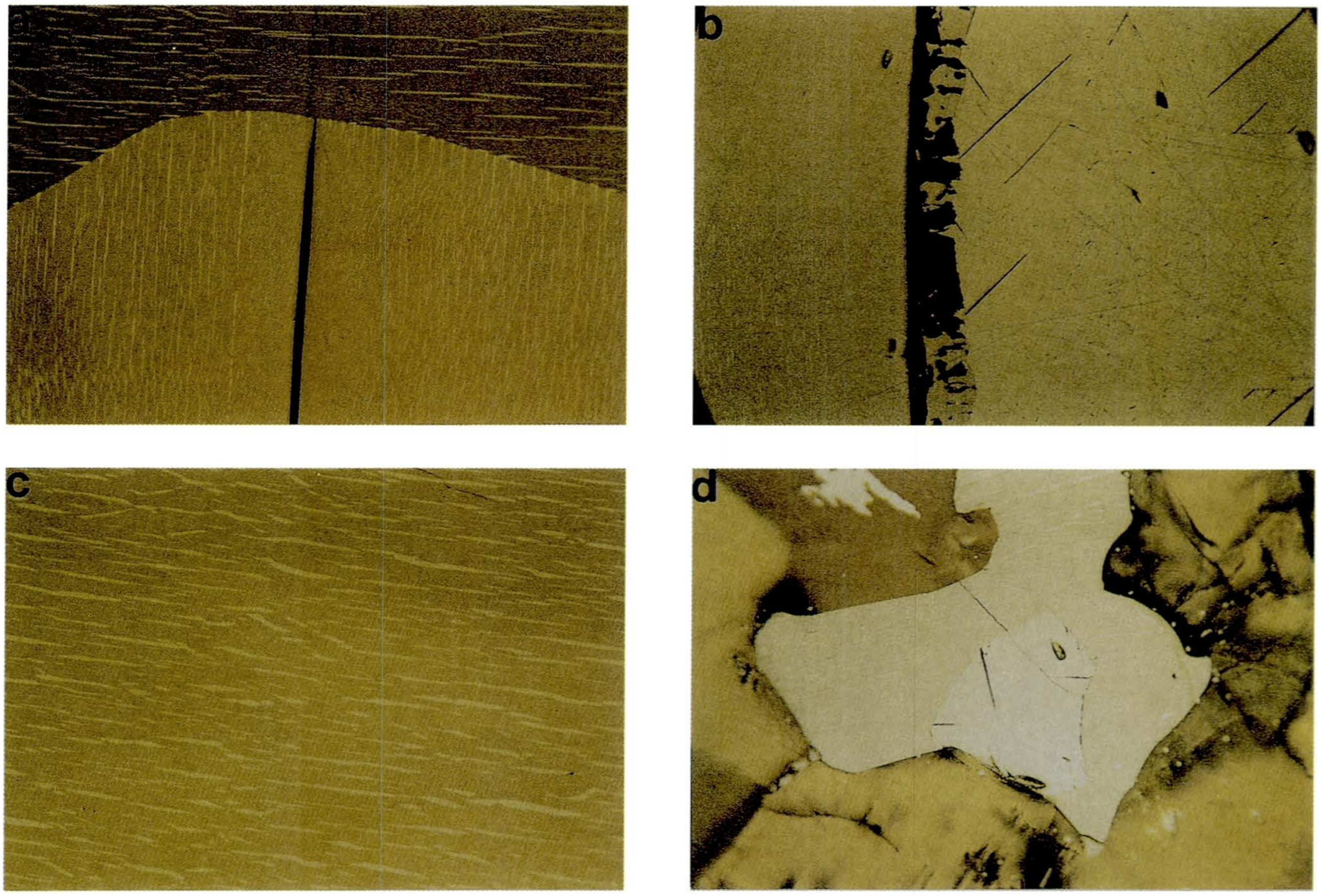

Fig. 3. Reflected-light photomicrographs: (a). Ilmenite with multiple generations of very fine {0001} hematite exsolution lamellae. Internal to the ilmenite is a spinel plate surrounded by an area free of hematite lamellae. Note concentration of hematite lamellae at twin plane (Tellnes mine). Width of field 60μ. Photographed with a blue filter. (b) Magnetite with minor {100} spinel lamellae but free of ilmenite lamellae. Adjacent ilmenite grain contains hematite lamellae and at the contact between magnetite and ilmenite a magnetite-ilmenite-spinel symplectite (Tellnes mine). Width of field 120μ. Photographed with a blue filter. (c) Ilmenite with multiple generations of fine hematite exsolution lamellae (Heskestad). Width of field 120μ. Photographed with a white filter. (d) Ilmenite with fine hematite exsolution lamellae enclosing a magnetite grain with minor spinel lamellae (Heskestad). Width of field 330μ. Photographed with a white filter.

contain multiple generations of very fine (0001) hematite exsolution lamellae (Fig 3c). Minor aluminous spinel plates, as precipitates are common in the ferrian ilmenite grains as are minor sulfide 'inclusions'. Magnetite has minor aluminous spinel exsolution (Fig 3d). A few magnetite grains have rare oxidation-exsolution lamellae of ilmenite. In all cases, spinel lamellae have nucleated on the ilmenite lamellae. When ilmenite was oxy-exsolved from magnetite it pushed the bulk composition towards spinel saturation and produced a second generation of spinel exsolution.

The oxide mineral assemblage in the magnetite-rich norite from the area in the Bjerkreim-Sokndal layered intrusion we refer to as the Mydland magnetic high is very different from the samples described above. Fig. 4a and b shows the two dominant oxides, subordinate ilmenite and very abundant magnetite. Ilmenite is free of hematite exsolution though minor spinel exsolution is present. When ilmenite is adjacent to magnetite a minor ilmenite-spinel-magnetite symplectite is present. The magnetite grains (Fig. 4a) display a very fine cloth-like pattern of ilmenite replacing exsolved ulvöspinel on (100) and well developed trellis oxidation-exsolution lamellae of ilmenite on (111). All trellis lamellae of ilmenite have enclosed and marginal spinel. Multiple generations of spinel exsolution parallel to (100) are present throughout the magnetite grains. The first generation of spinel exsolution is surrounded by a rim of ilmenite, which in turn is surrounded by magnetite where neither ilmenite nor spinel lamellae are present. Second-generation spinel lamellae are also surrounded by ilmenite. Subsequent smaller spinel rods do not have ilmenite rims. Secondary alteration of titanomagnetite to titanomaghemite was observed in many grains.

Samples from the magnetite-rich cumulate norite of the aeromagnetic high over the Bjerkreim-Sokndal layered intrusion at Bakka contain subordinate homogeneous ilmenite and abundant magnetite with ilmenite oxidation-exsolution. Fig. 4c shows the ilmenite and magnetite in sample TE-26. Although abundant ilmenite is present in this sample, most of it exists as very fine oxy-exsolution lamellae in magnetite. Large, first-generation, aluminous spinel exsolution lamellae are surrounded by areas of nearly pure magnetite which in turn are surrounded by ilmenite oxidation-exsolution lamellae. Between these areas an oxidation-exsolution pattern is present which is similar to that discussed above for the Myd-

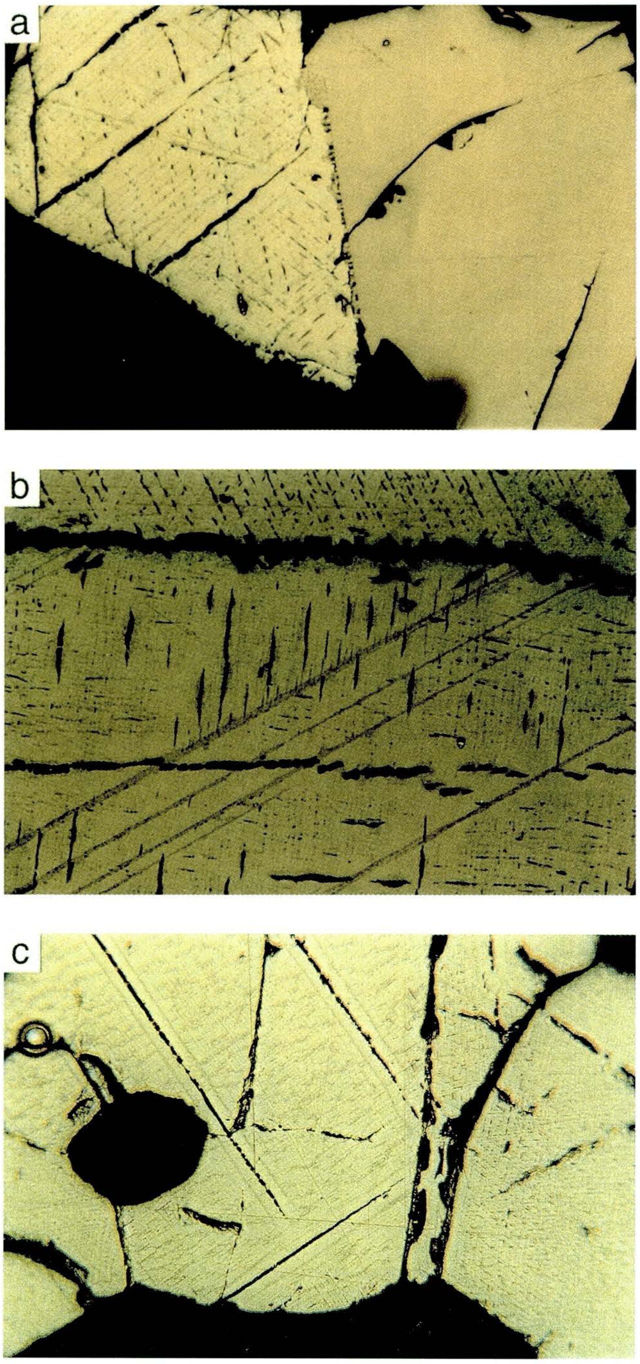

Fig. 4. (a) Mydland. Magnetite on left, with oxidation-exsolution lamellae of ilmenite and very fine spinel {100} exsolution. A minor magnetite-spinel-ilmenite symplectite at contact with ilmenite grains. Note lack of hematite exsolution in ilmenite (TE16). Width of field 200μ. Photographed with a blue filter. (b) Magnetite with {111} lamellae of ilmenite and {100} lamellae of spinel. Coarser spinel is rimmed by ilmenite (TE 16). Width of field 200μ. Photographed with a blue filter. (c) Bakka (TE26). Magnetite with very fine {100} cloth-like exsolution of ulvöspinel subsequently oxidized to ilmenite. Note the transitional pattern from a lit-par-lit Usp_{ss} to a lamellar Usp_{ss}. Also present are two generations of spinel along {100} planes. Width of field 200μ. Photographed with a white filter.

land sample. The very fine exsolution pattern is indicative of {100} ulvöspinel exsolution from magnetite. Subsequently, the ulvöspinel exsolution was oxidized to ilmenite. Locally within this region there are fine plates of spinel. Overall, the Bakka sample contains less spinel than that from Mydland.

Detailed electron probe analyses

The electron probe analyses of ilmenite and of magnetite are presented in two composite figures (Figs. 5 & 6). Instrumental conditions for the analyses are presented in the Appendix. Analyses of spinel exsolutions, locally in magnetite and in ilmenite, that are the product of more complex primary substitutions and later subsolidus reactions, will be discussed elsewhere. A more mineralogically oriented study of the oxides would show these analyses in cation proportions that can be related more directly to mineral end members (McEnroe et al. in review). For this study, however, the results are presented in weight % oxides, a method of greater familiarity in the mineral industry.

Fig. 5 shows all analyses of ilmenite. When using the electron probe, Fe analyses are customarily reported in terms of weight % FeO. On this basis pure hematite (Fe_2O_3) is 89.99 wt. % FeO, whereas end-member $FeTiO_3$ ilmenite contains 47.35 % FeO. As can be seen in Fig. 5A, some of the ilmenites contain hematite in solid solution or as fine exsolution lamellae. Thus, as TiO_2 decreases, total Fe as FeO increases, trending toward the end-member value for hematite. Conversely, when total Fe is adequately proportioned to Fe_2O_3 and FeO, and a plot is made of FeO vs TiO_2 as in Fig. 5B, the analyses show increasing FeO with TiO_2 from the ideal hematite composition at the origin. Analyses having lower FeO than ideal ilmenite at 47.35% FeO are ilmenites with significant amounts of MgO substituting for FeO, particularly in Tellnes ilmenites. When MgO is plotted against TiO_2, as in Fig. 5C, it is obvious that MgO is concentrated in TiO_2-rich ilmenite end members and is less important in TiO_2-poorer compositions involving greater hematite component. The MgO content in Tellnes ilmenites is particularly prominent, reaching values above 4.5 wt. %, consistent with the apparently primitive nature of these rocks.

When the minor oxides V_2O_3 and Cr_2O_3 are plotted against TiO_2 as in Figs. 5D and 5E, there is an inverse correlation, indicating that these oxides tend to be concentrated in the hematite component or in hematite-rich exsolution lamellae. In these plots it emerges that Cr_2O_3 is concentrated in the most primitive Mg-rich samples from the Tellnes Mine which also are the most extensively exsolved, whereas V_2O_3 is concentrated particularly in the ilmenite norites from Heskestad and Mydland that crystallized from more evolved magmas than Tellnes, but before magnetite joined ilmenite as a primary magmatic precipitate. Ilmenites from the magnetite-ilmenite norites at Mydland and Bakka are all high in TiO_2, lacking significant hematite exsolution, and are also depleted in MgO, V_2O_3 and Cr_2O_3, due to their mature stage of magmatic evolution.

Fig. 6 shows all of the analyses of magnetite with all oxides plotted in weight percent. As in Fig. 5A, all Fe is plot-

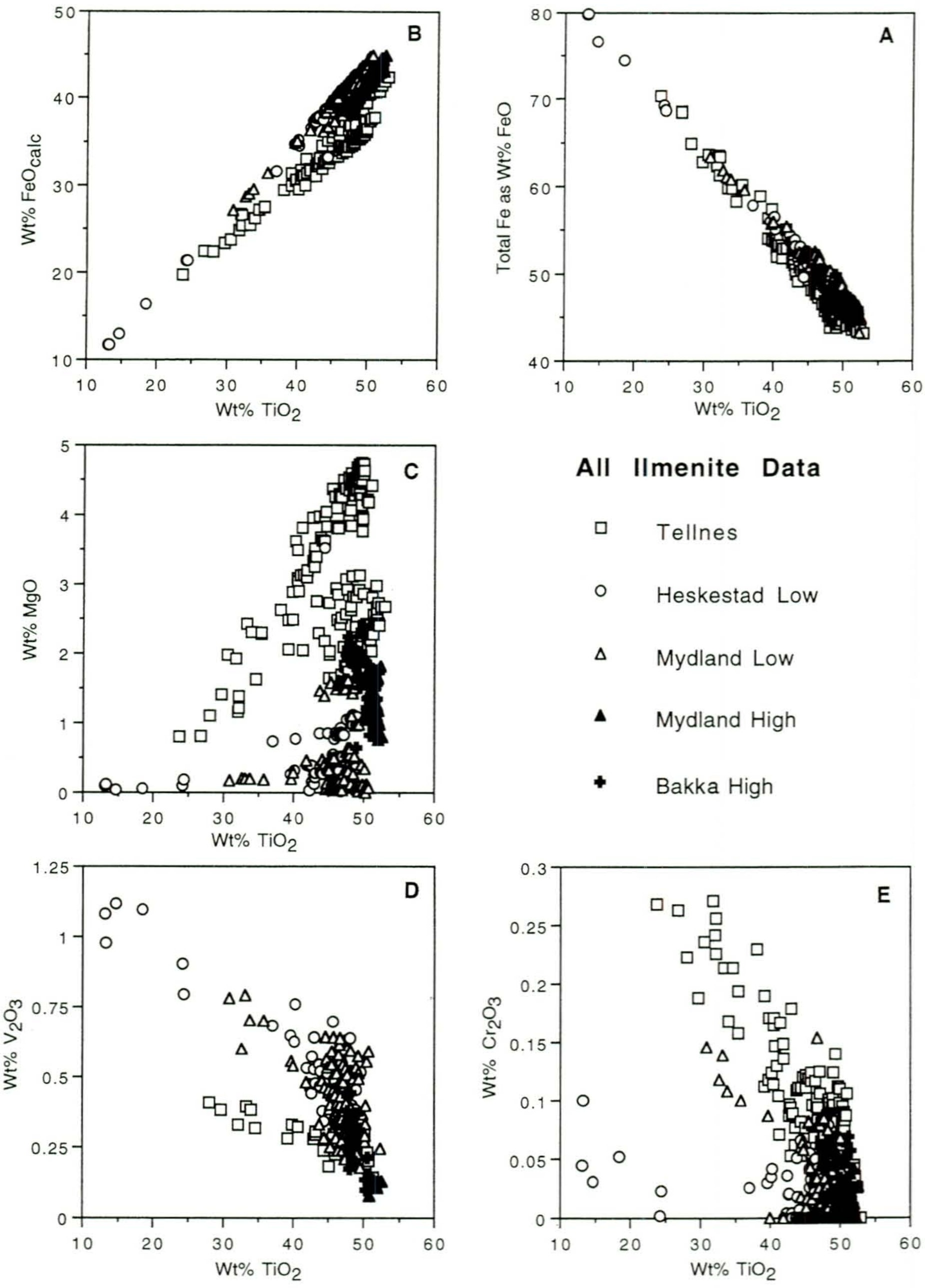

Fig. 5. Electron probe analyses of ilmenite from oxide-rich cumulates in five areas in the Sokndal region, plotted in terms of weight % of other oxides against weight % TiO_2. The areas are listed in order from magmatically most primitive (Tellnes) to most evolved (Bakka). Diagrams in counterclockwise order are: A) Total Fe as wt.% FeO; B) wt. % FeO (calculated); C) wt. % MgO; D) wt. % V_2O_3; and E) wt. % Cr_2O_3. In 5A, pure hematite lies at 0% TiO_2 and 89% FeO.

ted as FeO in Fig. 6A. In this case, note that magnetites from magnetic lows at Tellnes, Heskestad and Mydland plot at end-member magnetite with 93.09 wt. % FeO. Only magnetites from the magnetic highs at Mydland and Bakka contain substantial TiO_2, and hence also less total Fe. In Fig. 6B, the same analyses are plotted in terms of calculated weight % FeO and TiO_2. End-member magnetites plot at 31.03 % FeO, with greater FeO only in more ulvöspinel-rich compositions which at 18.6 wt. % TiO_2 corresponds to about 52% ulvöspinel end member. Fig. 6C shows MgO plotted against TiO_2. MgO is notably lower in magnetites than in many ilmenites (compare Fig. 5C) and seems to be highest in some near end-member magnetites than in titaniferous magnetites from the same samples from the Mydland and Bakka aeromagnetic high areas.

In magnetites, V_2O_3 is highest in Ti-poor magnetites (Fig. 6D), particularly in samples from the ilmenite norites from the Heskestad and Mydland magnetic lows. In these norites, ilmenite was the dominant primary precipitate oxide mineral. Magnetite had either just joined ilmenite or was precipitated from intercumulus liquid. Once magnetite became the dominant primary oxide precipitate, the magma was rapidly depleted in vanadium and late Ti-rich magnetites are very low in V_2O_3. Cr_2O_3 is only significant in Ti-poor magnetites (Fig. 6E), and then only in magnetites in the primitive rocks of the Tellnes body.

Summary

The analytical results of the present study are summarized as follows. Ilmenite from the primitive Tellnes ilmenite norite contains up to 4.8 wt.% MgO substituting for FeO. Areas rich in hematite exsolution lamellae are also enriched in V_2O_3 (up

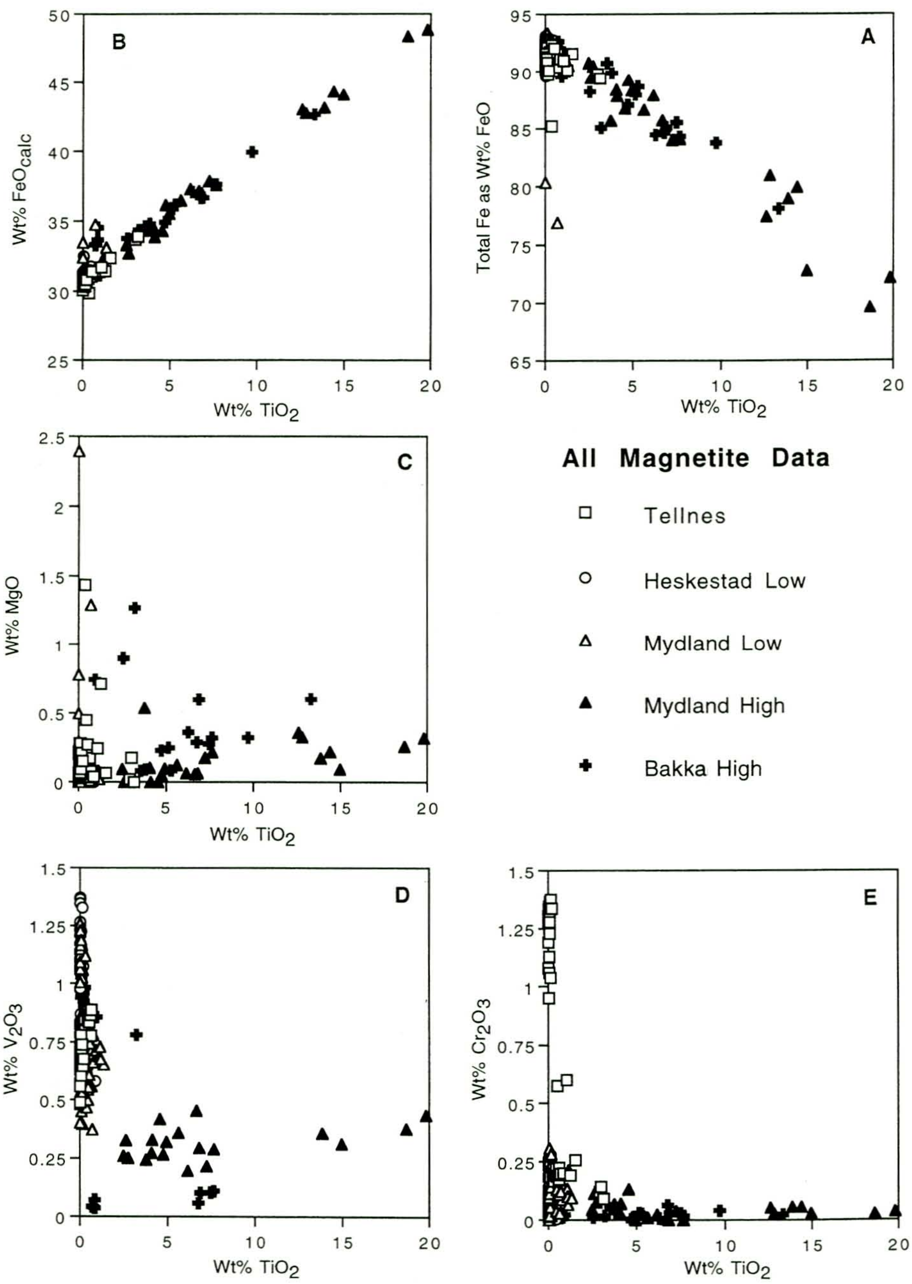

Fig. 6. Electron probe analyses of magnetite from oxide-rich cumulates in five areas in the Sokndal region, plotted in terms of weight % of other oxides against weight % TiO_2. The areas are listed in order from magmatically most primitive (Tellnes) to most evolved (Bakka). Diagrams in counterclockwise order are: A) Total Fe as wt.% FeO; B) wt. % FeO (calculated); C) wt. % MgO; D) wt. % V_2O_3; and E) wt. % Cr_2O_3.

to 0.4 wt.%) and Cr_2O_3 (up to 0.27 wt.%). Coexisting Ti-free magnetite contains about 0.4 wt.% MgO, 0.75 wt.% V_2O_3, and 1.25 wt.% Cr_2O_3. Ilmenite from the ilmenite-rich norite samples at Mydland and Heskestad in the Bjerkreim-Sokndal layered intrusion contain up to 1.6 and 1.0 wt.% MgO, respectively. Areas rich in hematite exsolution lamellae are enriched in V_2O_3 (up to 0.75 and 1.15 wt.%) and Cr_2O_3 (up to 0.15 and 0.1 wt.%). Lower MgO and Cr_2O_3, and higher V_2O_3 in the Heskestad sample as compared to the Tellnes and Mydland samples is indicative of the progress of magmatic crystallization with V_2O_3 enrichment in the liquid before the onset of primary precipitation of magnetite. Ti-free magnetites in these samples from the layered intrusion contain up to 0.75 wt.% MgO, up to 1.4 wt.% V_2O_3, and up to 0.3 wt.% Cr_2O_3.

Hematite-poor ilmenite from magnetite-rich norite samples at Mydland and Bakka in the layered intrusion contains up to 2.3 and 2.0 wt.% MgO, about 0.1 and 0.2 wt.% V_2O_3, and 0.07 and 0.07 wt.% Cr_2O_3, respectively. Analyses of magnetites in these magnetite-rich norites show variable TiO_2 consistent with the observed exsolution of ulvöspinel and/or oxidation-exsolution of ilmenite. Magnetites from Mydland and Bakka show slightly increasing MgO with increasing TiO_2 up to a maximum of 0.4 and 0.6 wt.% MgO, decreasing V_2O_3 with increasing TiO_2 from a maximum of 0.8 and 1.0 wt.% V_2O_3, and no obvious relationship between Cr_2O_3 and TiO_2 with a maximum of 0.15 wt.% Cr_2O_3. The magmatically evolved nature of these magnetite norite cumulates is suggested by the low values in magnetite of MgO and Cr_2O_3, and also of V_2O_3, indicating crystallization well after magnetite became a primary magmatic precipitate. Contrary to the consistent trends for magnetite, ilmenites in the magnetite-rich norites at Mydland and Bakka show higher MgO than the ilmenites in the ilmenite-rich norites at Mydland and Heskestad. This trend might reflect subsolidus reequilibration of these small amounts of ilmenite.

Future prospecting for workable and chemically suitable ilmenite resources will focus on mineral concentrations, on aeromagnetic signatures, and also on the relationships between magmatic evolution, major- and trace-element chemistry, and subsolidus re-equilibration, as outlined here.

Appendix

Analytical method

Microprobe analyses were made at the University of Massachusetts with a Cameca SX50 electron microprobe set at an accelerating potential of 15 keV, a sample current of 15 nA, and a typical beam diameter of 1 μm. Counting times of 20 or 40 seconds per element were used. Corrections for differential matrix effects were done using the Cameca online PAP correction routine. Analytical precision is estimated at ± 0.1 weight percent for oxide components present at the 1 weight percent level. Analytical precision on typical values of 0.5 weight percent V_2O_3 and Cr_2O_3 is estimated to be ± 0.06 weight percent at the 95% confidence level. In addition, there is believed to be a systematic overestimate of V_2O_3 in ilmenite of about +0.1 weight percent caused by Ti Kβ -V Kα interference.

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