Magnetic, chemical and x-ray studies of magnetites from Tamil Nadu, India

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Abstract. Natural intensity, susceptibility, and Koenigsberger ratio were determined and studies of Rayleigh loops, and high field hysteresis, and variation of susceptibility with temperature from -196°C to Curie temperature were made on a number of magnetite-quartzite and pyroxene granulite samples from Tamil Nadu. FeO , Fe_2O_3 , and TiO_2 proportions were estimated and cell dimensions were determined. From the magnetic studies it is inferred that in general the samples contain predominantly multidomain grains. In a few cases single-domain particles are detected, while in a few other samples a mixture of superparamagnetic particles and single domain states could be inferred. The relative remanence ratio is found to increase with coercive force. The ferromagnetic mineral in magnetite-quartzites is pure magnetite with a little alteration to hematite while in pyroxene granulites it is a titaniferous magnetite with a small percentage of TiO_2 . It is probable that the cell dimensions are dependent on oxidation in magnetites, and on the content of TiO_2 in titaniferous magnetites.

Keywords. Magnetic properties; magnetite; titaniferous magnetite; oxidation; cell dimension; x-ray studies; chemical studies.

1. Introduction

In Tamil Nadu there are several occurrences of iron formations which are commonly referred to as magnetite-quartzites. These occur in the precambrian high grade granulite terrain of peninsular India. Important occurrences are in Kanjamalai, Godumalai, Nainaramalai, Thirthamalai and Kavuthimalai-Vedippanimalai areas. The associated rocks are generally various types of granulites and gneisses, dunites, peridotites, pegmatites and dolerite dykes. Pyroxene granulites are the most intimately associated rocks with the magnetite-quartzites. A study of mineralogy and chemical composition of the magnetites and the magnetic properties of the magnetite-quartzites and pyroxene granulites is presented here, to find the nature of the magnetic minerals, magnetic granulometry and the dependence of the magnetic properties. Although similar studies have been made mostly on igneous rock bodies (Radhakrishnamurthy *et al* 1978; Deutsch

and Patzold 1976; Davis and Evans 1976) and recently on charnockites (Ramaswamy and Ramamurthy 1980) such studies are lacking on the magnetite-quartzites.

2. Experimental methods

A collection of 141 samples of magnetite-quartzites and 25 samples of the associated pyroxene-granulites was made. The magnetic properties of these samples were studied to determine the magnetic granulometry. Proportions of FeO, Fe_2O_3 , and TiO_2 , and cell dimensions of the separated magnetic concentrates were determined.

Intensity of natural remanent magnetization (J_n) and susceptibility (k) in a field of 0.5 Oe were determined on all the samples, and the Koenigsberger ratios (Q_n) were calculated. Rayleigh loops in a field of 10 Oe and high-field hysteresis in a field of 2400 Oe were studied for all the samples. Representative samples of 16 magnetite-quartzites and 8 pyroxene granulites were studied for variation of susceptibility (k), in a field of 0.5 Oe, with temperature (T) from -196°C to Curie temperature. Relative susceptibility (k_{-196}/k_{25}) values were determined. Similarly the relative remanence ratios (J_r/J_m) were also calculated.

Ferromagnetic concentrates from magnetite-quartzites and pyroxene granulites for chemical and x-ray analyses were obtained by crushing the samples to a fine powder in a gun metal mortar and the ferromagnetic fraction was separated by hand magnet and further purification and separation of silicates were carried out using Bromoform, Clerci solution, and acetone.

Total iron as Fe_2O_3 and TiO_2 were determined calorimetrically, and FeO was determined by permanganometric method. These three constituents were determined in magnetic concentrates from selected samples of 38 magnetite-quartzites and 12 pyroxene granulites. Fourteen magnetic concentrates from magnetite-quartzites and five concentrates from pyroxene granulites were subjected to x-ray studies and their cell dimensions were determined. These studies were carried out at the Indian Institute of Technology, Madras, and the Indian Institute of Science, Bangalore.

3. Magnetic properties

3.1. J_n , K , Q_n and Rayleigh loops

The ranges of J_n , K and Q_n are given in table 1 and their distribution is shown in the figure 1a, b. There is general similarity of the histograms for all the three properties both for the magnetite-quartzites and pyroxene granulites. Such relationship indicates that the magnetic properties are dependent on the amount of ferromagnetic constituents.

A study of all samples for Rayleigh loops indicated that they generally show either a loop or line. All gradations in the thickness of the loop are found from a maximum as shown in figure 2c to that of a line indicated in figure 2a. According to the criteria given by Radhakrishnamurty and Likhite (1970) the samples which show a loop may contain superparamagnetic (SP) particles while

Figure 1. (a) Distribution of J_n (A), Q_n (B), and K (C) in magnetite-quartzites. (b) Distribution of J_n (A), Q_n (B), and K (C) in pyroxene granulites.

Table 1. Ranges of J_n , K and Q_n .

	$J_n \times 10^{-3}$ emu/g		$K \times 10^{-3}$ emu/g		Q_n	
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum
Magnetite-quartzites	0.13	561.3	0.122	73.1	0.05	100.8
Pyroxene-granulites	0.01	11.5	0.013	26.4	0.033	11.3

Ranges of J_n , K , and Q_n .

those which show a line may have multidomain (MD), single domain (SD), or cation deficient (CD) particles. In the case of two samples K_0 and V_{15} a small constriction of the loop is observed (figure 2—b, K_0). Radhakrishnamurty and Sastry (1970) and Deutsch and Patzold (1976) have attributed such constrictions to strong interaction in dense SP particle clusters. The relative susceptibility values are given in table 2 and range between 0.28 to 0.92 in the case of magnetite-quartzites and 0.39 to 1.13 for pyroxene granulites. The high values obtained for the granulite samples may suggest the presence of very fine particles which may be SP even at -196°C .

3.2. k - T curves

From a study of variation of k with T it is clear that all the samples are similar and indicate Curie temperatures ranging between 535°C and 580°C . A representative k - T curve is shown in figure 3. The range of Curie temperatures obtained generally indicate the presence of almost pure magnetite (Tanaka and Kono 1974). Though the Curie temperature decreases with increasing content of TiO_2 , the samples of the present study contain only negligible amounts of TiO_2 and therefore it is probable that TiO_2 content has no influence on the Curie temperatures of the samples. The presence of strains, imperfections and impurities in the magnetites may generally lower their Curie temperature (Tarling 1971).

The k - T curves of the magnetite-quartzites are similar to those obtained by Radhakrishnamurty (1975), Radhakrishnamurty and Deutsch (1974), and Radhakrishnamurty *et al* (1978, 1979) for basalts containing MD grains. The characteristic sharp k -peak as shown in figure 3, occurring in the temperature range of -150°C to -146°C though similar to that of basalts (-165°C to -150°C) is slightly higher which could be due to impurities in the samples. In the samples studied for k - T curves the characteristics of maghaemite were not noticed.

3.3. High field hysteresis

The hysteresis loops in a field of 2400 Oe shown in D , E and F of figure 2 indicate that there are different types of loops. The J_r/J_m values for magnetite-

Figure 2. a, b, and c. Rayleigh loops at room temperature in a field of 10 O_e.
a—almost line, b—constricted loop, c—loop.

Scale : Horizontal axis-1 small division = 0.66 O_e.

Vertical axis-1 small division = 17×10^{-3} emu.

d, e and f. High field hysteresis loops at room temperature in a field of 2400 O_e.

Scale : Horizontal axis-1 small division = 57 O_e.

Vertical axis-1 small division

= Figure d. 1.11125 emu

= Figures e and f. 2.2225 emu.

Table 2. Magnetic properties of magnetite-quartzites and chemical composition and cell dimensions of the magnetites.

Specimen No.	$J_n \times 10^{-3}$ emu/g	(k) suscepti- bility $\times 10^{-3}$ emu/g	Q_n (J_n/J_d)	Relative suscepti- bility k_{100}/k_{25}	Coercive force (H_c) Oe	J_r/J_m	Curie tempera- ture (T_c)	FeO	Fe ₂ O ₃	TiO ₂	Cell dimension Å
K ₀	304.6	32.19	18.92	0.28	56	0.077	..	25.90	70.90	0.04	8.3892
K ₉	13.1	43.82	0.59	..	..	..	..	24.80	72.53	0.03	8.3831
K ₁₀	3.47	5.2	1.33	0.47	111	0.166	..	25.81	72.80	0.034	8.3956
K ₂₆	18.02	0.86	41.90	0.66	444	0.500	..	29.90	67.83	0.01	..
K ₃₀	118.6	38.7	6.13	0.36	56	0.026	545	..	..	..	..
K ₃₁	561.3	4.2	267.28	0.49	111	0.100	..	27.13	71.37	0.02	8.3958
N ₂	2.14	49.36	0.08	..	..	..	..	24.95	72.80	0.05	8.3875
N ₆	1.17	0.41	5.57	0.92	333	0.333	580	27.10	70.65	0.019	..
N ₁₆	497.4	19.92	43.94	..	..	..	..	28.50	69.82	0.01	8.3930
N ₁₉	404.3	3.49	231.69	0.63	111	0.100	..	27.90	69.84	0.024	..
N ₂₃	65.01	4.73	27.48	0.46	22	0.008	..	29.61	68.42	0.03	..
N ₂₆	119.00	35.32	6.74	0.35	28	0.008	..	24.92	73.33	0.01	8.3815
T ₃	175.2	8.03	43.63	..	..	..	..	24.93	72.21	0.019	8.3852
T ₄	147.3	12.14	21.99	0.39	333	0.107	..	25.12	71.90	0.02	..
T ₇	369.6	9.14	80.87	0.43	167	0.083	..	25.53	72.71	0.033	8.3785
T ₁₁	506.8	9.66	104.92	0.33	111	0.094	565	26.72	70.52	0.08	8.3980
V ₁₅	74.66	33.93	4.40	0.53	160	0.120	535	25.90	71.73	0.032	8.3945
V ₁₈	47.80	0.97	98.56	0.85	512	0.499	..	28.90	69.20	0.018	8.3948
V ₂₇	587.00	11.73	100.08	0.40	240	0.250	..	25.32	72.54	0.022	8.3945
V ₃₆	0.33	1.16	0.58	0.82	480	0.375	..	26.67	71.85	0.021	8.3991
*V ₄₁	0.362	0.22	3.29	0.39	..	..	..	27.50	68.65	2.80	8.3936
*V ₄₃	0.359	0.061	11.39	1.13	..	..	..	29.10	67.50	1.40	..
*V ₆₃	0.072	0.059	2.44	1.00	..	..	..	28.40	65.30	1.34	..
*V ₆₄	0.989	1.05	1.88	0.57	..	..	..	28.60	64.10	5.50	8.4340
*T ₆₄	2.283	17.84	0.255	..	..	..	..	23.90	73.60	1.06	8.3904
*T ₆₆	2.46	17.83	0.276	..	..	..	..	24.71	72.54	1.72	8.404
*T ₆₈	6.421	26.36	0.487	..	..	..	..	22.84	74.70	1.24	8.3928

* Magnetic properties of pyroxene granulites and chemical composition and cell dimensions of titaniferous magnetites.

Figure 3. k - t curve from -196°C to Curie temperature.

quartzites (table 2) range between 0.008 and 0.50. The high values of J_r/J_m are generally associated with high H_s . The J_r/J_m ratio is independent of anisotropy (Dunlop, 1972). According to Nagata (1961) there will be a general increase in J_r/J_m with H_s in igneous rocks. Such relationship is found in the magnetite-quartzites which is shown in figure 4. The theoretical value of 0.5 for J_r/J_m is expected for SD particles distributed in a nonmagnetic matrix (Dunlop 1972; Dunlop *et al* 1973; Deutsch and Patzold 1976). The increase in H_s and saturation remanence reflects the increase in J_r/J_m ratio as the fraction of single-domain regions increase (Kono 1977). From table 2 it can be seen that the samples K_{23} , and V_{18} have a value of 0.5 for J_r/J_m and these samples also exhibit the highest H_s which probably indicates the presence of SD particles. A similar interpretation has been given by Day *et al* (1976). These samples give Q_n values of 41.9 and 98.56 respectively. According to Radhakrishnamurty and Deutsch (1974) the value of Q_n ranging between 10 and 100 indicates the presence of SD particles in basalts. Therefore the referred samples of magnetite-quartzites may contain SD particles. Radhakrishnamurty *et al* (1977) suggest the presence of MD grains if the J_r/J_m is less than 0.2. In most cases of magnetite-quartzites the J_r/J_m is less than 0.2 and it is probable that they contain mostly MD grains. In other cases where the J_r/J_m value is above 0.2 but below 0.5 there may be a mixture of MD and SD grains. Similar interpretations have been given by Davis and Evans (1976).

4. Chemical studies

4.1. Chemical studies

The results of the chemical analysis are shown in table 2. The molecular percentages of the analyses are calculated and plotted in the $\text{FeO-Fe}_2\text{O}_3\text{-TiO}_2$ ternary diagram (figure 5A, after Akimoto and Katsura 1959). From the figure it can be seen that the ferromagnetic concentrates from magnetite-quartzites fall near the Fe_3O_4 point and on the line $\text{Fe}_3\text{O}_4\text{-Fe}_2\text{O}_3$. This indicates that the TiO_2 in them is very small and mineralogically they are pure magnetites with a small proportion of haematite. The poverty of TiO_2 in magnetites from metamorphic

Figure 4. Relationship between relative remanence ratio (J_r/J_m) and coercive force (H_c) in magnetite quartzites.

rocks, as compared to those from most igneous rocks, was reported by Buddington *et al* (1955). Ore microscopic studies also indicate the presence of pure magnetite with some alteration to haematite. In the magnetites, which are hereafter referred as such, ilmenite lamellae have not been noticed. The small proportion of TiO_2 present in the samples may not be in solid solution in magnetite. The ferromagnetic concentrates from pyroxene granulites fall near Fe_3O_4 point (figure 5A) but slightly higher than those of the concentrates from magnetite-quartzites, because of the presence of TiO_2 in higher proportions. Ore microscopic study of granulites indicated the presence of magnetite and ilmenite. The concentrates from granulites are referred to as titaniferous magnetites to distinguish them from the magnetites separated from magnetite-quartzites. The effect of differences in chemical composition of the samples is not reflected in magnetic properties since there is not much variation in the composition of magnetite or titaniferous magnetites.

The atomic ratio of $Fe/(Fe + Ti)$ and the atomic proportion of metals to oxygen calculated on the basis of 32 oxygen atoms $32 X (Fe + Ti)/O$ are represented in figure 5B (after Akimoto and Katsura 1959) by taking the former on the abscissa and the later in the ordinate. The atomic ratio of $Fe/Fe + Ti$ ranges from 0.9995 to 1.0000 for magnetites and from 0.9458 to 0.9907 for titaniferous magnetites. The value of $32 X (Fe + Ti)/O$ is a measure of the degree of oxidation which is in the range between 23.37 and 23.96 for magnetites and varies from 23.14 to 23.82 in titaniferous magnetites. As can be seen from figure 5b all points of

Figure 5. (a) FeO—Fe₂O₃—TiO₂ ternary diagram after Akimoto and Katsura, 1959. (○) Magnetite quartzites. (●) Pyroxene granulites. (b) Relation between 32 (Fe + Ti)/O and Fe/(Fe + Ti). Fe includes Fe²⁺ and Fe³⁺. After Akimoto and Katsura 1959.

The bottom figure is an enlarged view of the hatched portion of the top figure. (○) Magnetite (M) from magnetite quartzites. (●) Titaniferous magnetites (TM) from pyroxene granulites.

magnetites are distributed very close to EF line which suggests different degrees of oxidation from magnetite to haematite. All points of titaniferous magnetites fall above the line EBC indicating the absence of ulvospinel in solid solution and the presence of ilmenite.

From the weight percentages, normative minerals are recalculated. In the proportion of the normative minerals the percentage of haematite in the magnetites ranges from 1.36 to 18.64 and in titaniferous magnetites it varies between 3.63 and 26.43. The excess of haematite cannot be accounted by considering it to be in solid solution in magnetite. Greig *et al* (1935) and Edwards (1949) found that at 1075° C magnetite with 8% Fe₂O₃ in solid solution is in equilibrium with haematite containing less than 0.5% Fe₂O₄, and that at 1452° C the magnetite can contain 30% Fe₂O₃ which on cooling separates out as haematite. It is therefore unlikely that excess of Fe₂O₃ occurs as solid solution in magnetites. Basta (1959) suggests that oxidation of natural magnetite results in the production of γ -Fe₂O₃ (maghaemite) which is unstable at moderate to high temperatures and inverts to α -Fe₂O₃. For this reason he concludes that it is unlikely to occur in metamorphic rocks. Gjelsvik (1957) suggests that high Fe₂O₃ is certainly due to oxidation and confirms it by ore microscopic studies. According to Abdullah

and Atherton (1964) the Fe_2O_3 surplus appears commonly in magnetites from metamorphic rocks and is often due to late oxidation or martitization or even due to the development of maghaemite and not due to the presence of solid solution, since the temperatures of the formation of metamorphic magnetites do not reach the high temperatures where solid solution may exist. Since the area from which the samples are collected belongs to the high grade granulite facies of the regional metamorphism (in which the temperatures may have reached to about 800°C to 900°C which are much lower than the temperatures where solid solution can exist) it is suggested that the greater or lesser amounts of excess Fe_2O_3 is due to varying degrees of oxidation without maghaemitization. X-ray diffraction studies, magnetic properties and ore microscopic studies revealed the absence of maghaemite.

4.1. X-ray studies

The cell dimensions determined are given in table 2, for magnetites ranging between $8.3785\text{ \AA}$ to $8.3991\text{ \AA}$. In the case of titaniferous magnetites the range is $8.3904\text{ \AA}$ to $8.4340\text{ \AA}$. The best known value of the cell dimension of pure natural magnetite, according to Basta (1957), is 8.3963 ± 0.0005 at 18°C . The cell dimensions determined show deviations from the standard value. The dependence of cell dimension of the magnetite on the proportion of TiO_2 and the oxidation state have been suggested by Basta (1957, 1959). Similar interpretations were given by Nagata (1961), and Stacey and Banerjee (1974). The TiO_2 contents in the magnetites are very low. There is no effect of the presence of TiO_2 on cell dimensions in magnetites whereas in the case of five samples of titaniferous magnetite there is a relationship of increasing cell dimension with increase in TiO_2 content (figure 6). Of all the samples determined V_{44} has the highest cell size of $8.4340\text{ \AA}$ which also has the highest content of TiO_2 5.2%. Ore microscopic examination has revealed the presence of magnetite with exsolution lamellae of ilmenite where as in the case of magnetite-quartzites the presence of ilmenite could not be noticed. Therefore the observed increase in cell sizes of the titaniferous magnetites from granulites is considered here as due to the presence of TiO_2 .

To test the dependence of the cell dimension on the oxidation in the case of magnetites the oxidation values [the atomic proportion of metals to oxygen calculated on the basis of 32 oxygen atoms $-32 \times (\text{Fe} + \text{Ti})/0$] are plotted against cell dimensions in a graph (figure 6) which shows a general decrease of cell dimension with increasing oxidation. Oxidation parameters (Z) of Ozima and Sakamoto (1971) were also calculated and they do not show any relationship with the cell dimensions.

Akimoto *et al* (1957), O'Reilly and Banerjee (1967), O'Reilly and Readman (1971), and Ozima and Sakamoto (1971) have conducted experiments on synthetic oxidized titanomagnetites with different mole fractions of Fe_2TiO_4 and studied the relationship existing between saturation magnetization, Curie temperature and cell dimensions. They have drawn the contours of equal Curie temperature, saturation magnetization, and cell dimensions in the $\text{FeO}-\text{Fe}_2\text{O}_3-\text{TiO}_2$ ternary diagram. The results obtained by them do not correlate with each other (Stacey and Banerjee 1974) and with the data on natural samples of the same composition, (Joshima 1973). The compositions of the samples cannot be plotted in either Ozima and Sakamoto, or O'Reilly and Readman diagrams since the compositional

Figure 6. (a) Relationship between $32(\text{Fe} + \text{Ti})/\text{O}$ and cell dimensions in magnetites separated from magnetite quartzites. (b) Relationship between TiO_2 per cent and cell dimension in titaniferous magnetites separated from pyroxene of granulites.

range of their synthetic samples are widely different from the samples studied. If the plots of the chemical analyses of the samples of the present discussion are made in Akimoto *et al* (1957) diagrams, they indicate Curie temperature of 580°C and cell dimension of $8.395\text{ \AA}$ for the magnetites. The Curie temperatures and cell dimensions obtained from the diagram do not correspond with the actual results obtained. This discrepancy may be due to the fact that Akimoto *et al* (1957) carried out the experiments on synthetic samples. Another possibility is that the separated ferromagnetic minerals are not perfectly in single phase, but contain two or more phases which cannot be detected by x-ray diffraction or chemical analyses (Joshima 1973).

5. Conclusions

The observed magnetic properties are considered as due to the presence of mainly MD grains with a combination of SD and SP particles except in a few cases where SD particles may be dominant, while in few other samples a mixture of SP and SD states could be inferred. It is found that the J_r/J_m ratio increases with increase in H_c . Magnetite from magnetite-quartzites is pure except for a little alteration to haematite. The excess of Fe_2O_3 in these is due to varying degrees of alteration but not due to the presence of Fe_2O_3 in solid solution. The cell dimensions are dependent on oxidation and TiO_2 content.

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