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Alteration and Exsolution Characteristics of Ilmenites of Moheskhali Island, Chittagong, Bangladesh

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Abstract

Mineralogical analysis of Moheskhali beach sands revealed the presence of high amount of ilmenite. About 50% of these ilmenites are unaltered which are characterized by the presence of exsolution of ilmenite with hematite, ilmenite with magnetite, and ilmenite with rutile. Others (50%) are unexsolved where 30-50% grains partially or fully altered. These altered and unaltered phases of ilmenite are confirmed by X-Ray diffraction study. Ilmenite-hematite exsolution comprising 70-80% of the total exsolution. The widely banded exsolved phases were formed by continuous exsolution mechanism while the second generation thinner bands were formed discontinuously. Seriate texture dominates (about 75%) over emulsion, granular, quadrangular, sub graphic, veined and special types. Optical study suggests that the alteration of ilmenite is seen to proceed along grain boundaries and/or fractures resulting in an amorphous to microcrystalline mass resembling leucoxene. The chemical composition of the alteration products of ilmenite frequently fluctuates within definite ranges (pseudoilmenite and pseudorutile ranges) of a nonstoichiometric composition and thus deviates from their ideal composition.

Key words: Ilmenite, Rutile, Opaque, Exlosution, Alteration.

Introduction

Ilmenite, the major source of titanium metal receives much attention among basic researchers due to its ubiquitous presence in rocks and sediment which are widely used as pigment, welding electrodes, coating and lining for blast furnace hearth. Alteration phenomenon, intergrowths, ferrous-ferric ratio and trace element distribution form prime concerns of Geochemists and Mineralogists with reference to this mineral. TiO_2 is enriched by all normal weathering process over all rocks, and the amount of enrichment is proportional to the intensity of weathering. Several papers have been published on the alteration of ilmenite. Most of the previous workers (Grey and Reid 1975, Wort and Jones 1981, Choudhury *et al.* 1989, Gupta *et al.* 1988, Hugo and Cornell 1991, Suresh Babu *et al.* 1994, Rao *et al.* 2002 and 2005) are agreed that the mineral ilmenite undergoes oxidation and leaching whereby iron is removed from the structure resulting in a relative enrichment in TiO_2. Most alteration leads to thermodynamically unstable, incomplete, irreversible phases that are extremely complex in elemental composition (Rao *et al.* 2005). Consequently, the physical and chemical characteristics such as bulk density, porosity, grain density, hardness, magnetic susceptibility and other properties are affected.

Moheskhali Island is 17 miles long and 6 miles wide. The main part of the island is low hills composed of sandstone and shales of Mio-Pliocene age. The heavy mineral deposits are exposed on the western side slope of the hill range some 3-4 miles inland from the present shore line (Fig. 1) and run more or less parallel to the foothills. The beach sands of Moheskhali Island belong to fine sand class, well sorted to very well sorted and negatively skewed (Mitra and Ahmed, 1990). The heavy minerals of the study area vary from 10 to 20%. Most of the heavy minerals lie within the size fraction of 1.5ϕ to 3.5ϕ and the percentage increase from 1.5ϕ to 3.5ϕ size fraction. The non-opaque heavy minerals are hornblende, garnet, epidote, actinolite-tremolite, sillimanite, sphene, zircon, monazite, etc. The opaque minerals content varies between 15 and 30% of the heavies. These are ilmenite, magnetite, titanomagnetite, hematite, iron-hydroxides, rutile and pyrite

Ilmenite, the main source of titanium of Bangladesh, is the major opaque mineral in the Moheskhali Island. It varies from 15 to 30% (Mitra and Ahmed, 1990). Both unaltered and altered ilmenites are recognized in the study area and 50% of the unaltered ilmenite is exsolved (Ahmed *et al.* 1992). In the present study, ilmenites of Moheskhali Island (Fig. 1) are studied for understanding their exsolution pattern and alteration.

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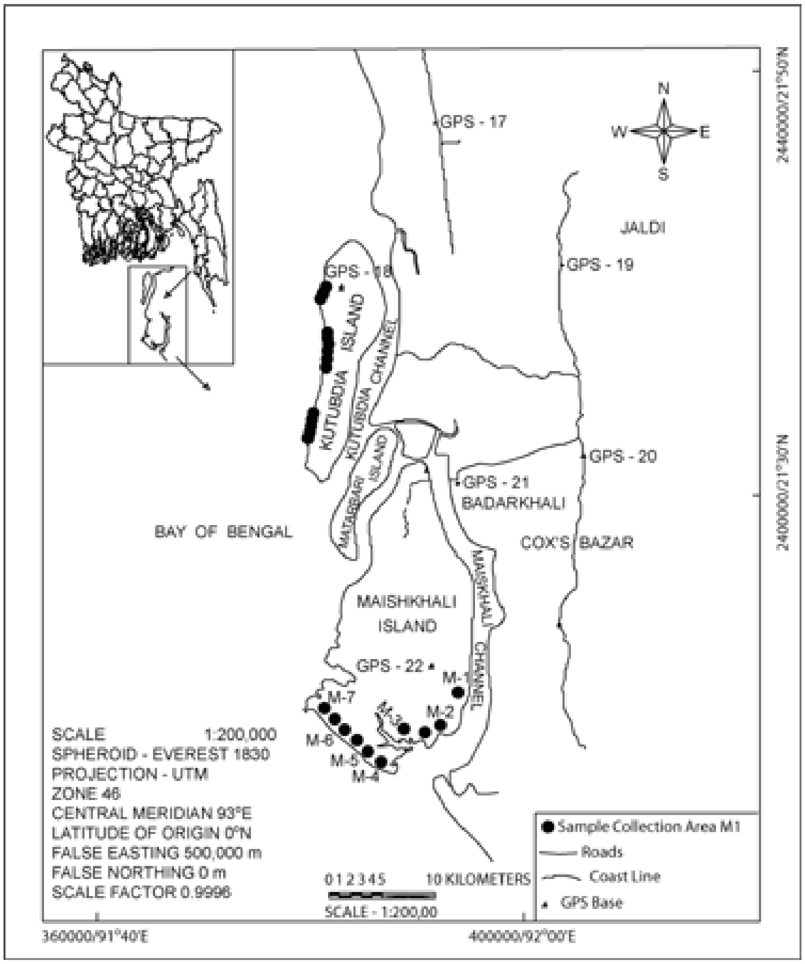


Fig. 1. Index map of Moheshkhali Island showing sample locations.

Methodology

Representative beach samples were collected from the Moheskhali Island. After washing and drying 100 g of each sample is sieved through a Ro-tap sieve shaker. The heavy minerals were studied from two size fractions viz 1.5-2.5ϕ and 2.5-3.0ϕ from each sample. The heavy minerals of the fraction were separated using isodynamic magnetic separator, ilmenite and non-magnetic fractions being removed by maintaining the separator with a 25^{O} side slope, a current of 0.3 amp. and a tilt of 15^{O} in moderate vibration. For optical studies the grains were mounted in a solidified resin (araldite) and polished. Opaque minerals were studied under ore microscope and non-opaques under polarizing microscope. About 200 grains were counted in each section under a microscope. Bulk chemical analyses were done using atomic absorption spectrophotometer (Perkin Elmer Type-2380). The X-Ray analysis was carried out using Philips X-Ray dilfractometer. For mineral chemistry, ilmenites were analyzed with the help of JEOL-JXA-9600 EPMA super-probe model, with an accelerating voltage of 15 KV with a beam current of 0.01nA and a diameter of 10µm.

Results and Discussion

Optical microscopy

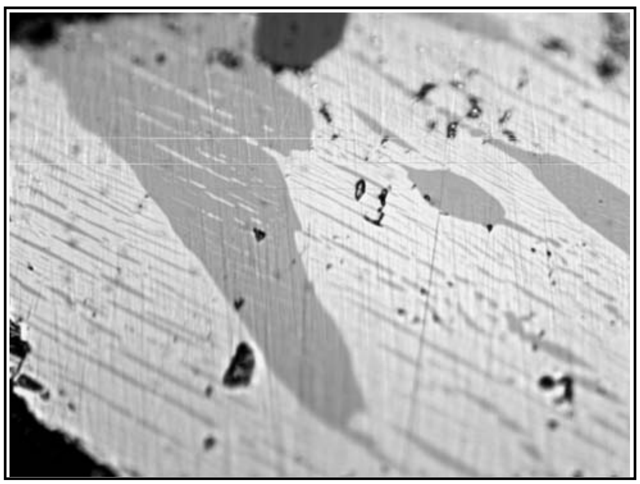
Exsolution

Unaltered ilmenite is brown to pinkish brown in color in contrast to the grayish white, minutely granular, porous appearance of the altered grains. The shape of ilmenite grains ranges from elongated lamellae, prismatic, irregular, subrounded to euhedral. The reflectivity is measured on an average 18.2. About 50% of the unaltered ilmenite shows a wide variety of exsolution features. Five major types of exsolution in ilmenite have been recognized in Moheskhali beach sand. These are ilmenite-hematite, magnetite-ilmenite, hematite -rutile, ilmenite-hematite-rutile and ilmenite-rutile.

Among these the most common (~ 80%) exsolution products in the investigated area are the exsolution of ferrianilmenite and titanohematite both simultaneously acting as the host and the guest in the same grain. In most cases, the lamellae of the guest are distributed bimodally (sometimes poly-modally) with modes of their thickness clustering around 2µ and 8µ (Fig. 2a, 2b, 2c, 3c, 4a, 4b). The distribution of minimum lamellae widths in ilmenite-hematite coexisting phases from the Moheshkhali Island is shown in Table I. These detrital ilmenites with preserved intergrowth are more commonly derived from igneous rather than metamorphic source rocks (Riezebos, 1979). The early generation coarser lamellae were formed at high temperature (600-530^{O}C) by a fast diffusion process and the finer ones formed at later stages of crystallization by slow diffusion process in the temperature range of 500-430^{O}C (Ahmed *et al.* 1992). Rarely some grains show equal amounts of ilmenite and hematite (Fig. 3c) indicating higher temperature of crystallization by a fast diffusion process (Ramdohr, 1980).

In some hematite (or ilmenite), smaller ilmenite (or hematite) exsolution bodies in parallel rows between the rows of large exsolution bodies of ilmenite (or hematite) are formed (Fig. 2d, 3a). Ilmenites with fine grained exsolution of hematite are also common where the depletion zone or zones of non-exsolution are concentrated along the grain boundaries (Fig. 3b). Sometimes the lamellae in the host shows micro-boudin-like feature (Fig. 3d) indicating its generation in stressed condition.

Pure ilmenite solid solution exsolves by continuous exsolution mechanism but Fe_2O_3 -rich ilmenite solid-solution



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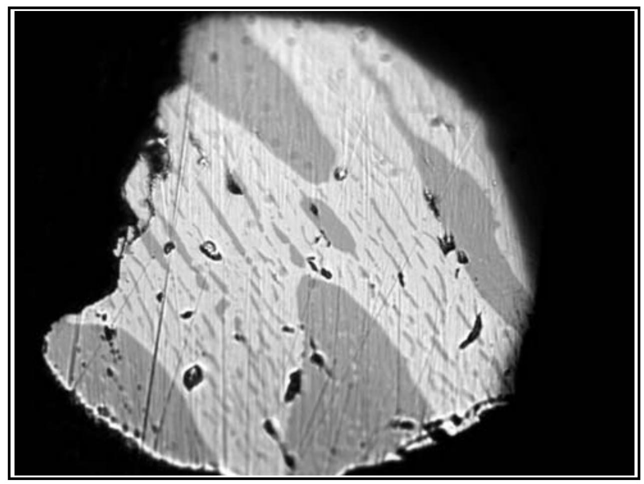

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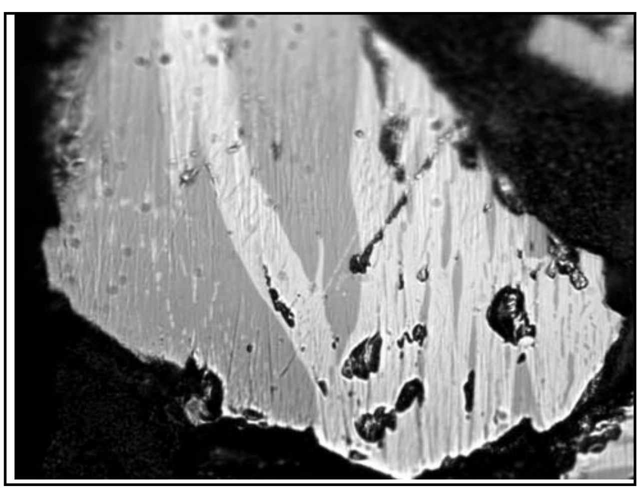

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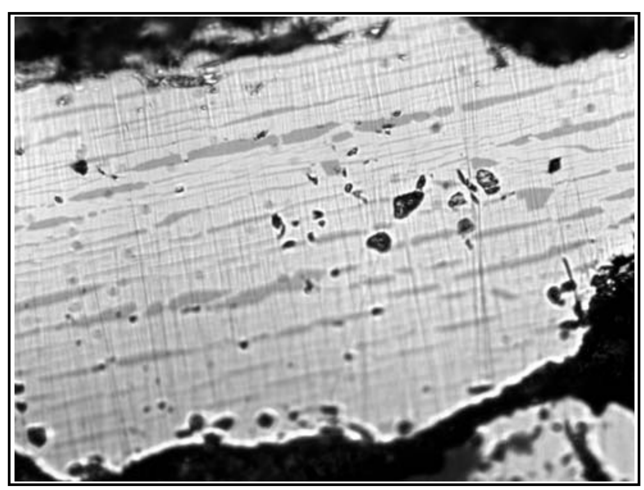


2(d)

Fig. 2. Seriate texture of Ilmenite-Hematite a, b, c) Two generations exsolution lamellae. d) Finer curved lenses of Ilmenite in hematite which are formed by stress conditions.

Table I: Distribution of minimum lamellae widths in ilmenite-hematite coexisting phases from study area (in percent)

Sample No	No exsolution	<1μ m	1-6μ m	6-12μ m	12-20μ m	>20μ m
M-1	57.84	3.30	21.00	12.52	3.84	1.50
M-2	38.00	7.08	30.08	15.72	6.60	2.52
M-3	43.15	8.29	26.44	15.28	5.10	1.74
M-4	51.73	5.60	24.20	13.00	5.16	2.03

exsolves discontinuously (Kretchsmar and McNutt, 1971, Haggerty, 1976). Exsolution textures in the two phase ilmenite-hematite were formed either by continuous or by discontinuous mechanism and sometimes by both. In Fig. 2a, 2b and 2c, the first generation thicker lamellae are formed by continuous mechanism while the second generation thinner lamellae are formed discontinuously. In hemo-ilmenite the exsolved Hem_{ss} solute is a relatively evenly distributed single generation constituent. Hematite-rich solid solutions are restricted to acid intrusive and anorthosite suites; where Hemss are seen to exsolve coexisting with ilmenite-rich solid solution (Haggerty, 1976).

ilmenite/hematite ratio in exsolved ilmenite is shown in Table II where the average ratio of ilmenite/hematite in exsolved ilmenite is 0.97. The average percentage of pure ilmenite (exsolved + unexsolved) and pure hematite (exsolved) are found to be 73.74% and 26.26% respectively.

X-ray diffraction analyses

Using the intensity ratios of peaks (104) of ilmenite and hematite, the percentage of hematite and ilmenite in bulk samples are determined with reference to the calibration curve of Kretchmar and McNutt 1971, as shown in Fig. 6. X-ray powder diffraction data (Table III) of four ilmenite samples of different grain sizes show a general increase of cell parameters with decreasing weight percentage of hematite.

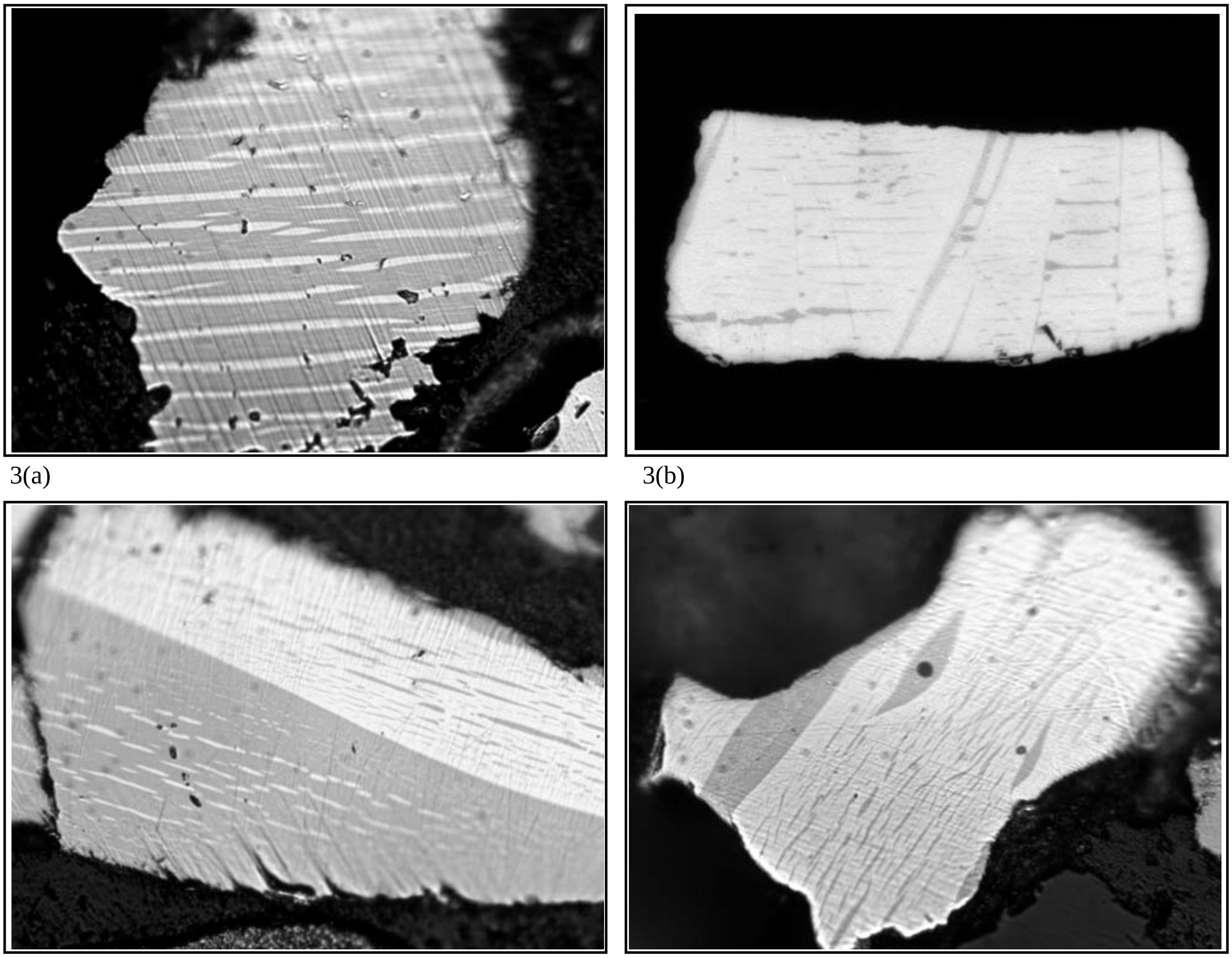
Table II. Optical modal analysis of exsolved ilmenites from Study area

Sample no.	Percentage of exsolved ilmenite	Percentage of different types of exsolution textures. The values in the parenthesis are average ilmenite/hematite ratio.							Total ilmenite (exsolved + unexsolved) %	Total hematite (exsolved) %
		Seriate	Emulsion	Granular	Sub-graphic	Quadrangular	Veined	Others		
M-2 (3.0-4.5ϕ)	42.16	33.68 (0.41)	4.22 (0.47)	0.69 (0.96)	0.42 (0.47)	0.42 (1.22)	0.63 (0.43)	2.10 (0.79)	70.80	29.20
M-2 (1.5-3.0ϕ)	62.00	53.94 (1.24)	4.34 (0.61)	1.55 (0.64)	-	0.93 (0.54)	0.62 (0.67)	0.62 (0.69)	70.93	29.07
M-4 (3.0-4.5ϕ)	56.85	44.41 (0.70)	6.89 (3.34)	1.22 (1.50)	1.14 (1.22)	0.58 (1.56)	-	2.61 (1.13)	69.83	30.17
M-4 (1.5-3.0ϕ)	48.27	38.49 (0.92)	4.88 (1.86)	1.49 (1.38)	1.20 (0.59)	-	0.24 (0.75)	1.97 (0.45)	75.37	24.63
Av.% present	51.74	43.11	4.40	1.25	0.65	0.41	0.25	1.67	73.74	26.26
Av./ilm./hem ratio		0.90	1.85	0.94	0.71	0.81	0.58	0.89		

The exsolved ilmenite of Moheskhali exhibits different types of well developed textural patterns namely seriate (Fig. 2 and 3), emulsion (Fig. 4a and 4b), granular (Fig. 4c and 4d), quadrangular, sub graphic (Fig. 5a), veined and special type (Fig. 5b, 5c and 5d). Seriate type is predominant (about 80%) where the coarse ilmenite (or hematite) exsolution bodies contain rows of small bodies of hematite (or ilmenite) of various sizes. Modal analysis of exsolved ilmenites from the study area is shown in Table II. These exsolution bodies have the same general orientation parallel to the (0001) direction of the ilmenite and hematite host. In the study area, the average percentage of different textures with their

Bulk chemical analysis

By atomic absorption Ti, Fe, Mn, Mg and Cr contents in ilmenites were determined and the results are shown in Table IV. Moheskhali ilmenites contain 39.76 to 42.85% TiO_2 and 23.70 to 28.04% Fe_2O_3. The ratio of ilmenite ($FeO.\ TiO_2$) to hematite (Fe_2O_3) varies from 2.46 to 3.16. The coarse fraction (1.5-3.0ϕ) is found to contain more TiO_2% than the finer fraction (3.0-4.5ϕ). The percentage of Mn, Mg and Cr in ilmenite is insignificant.



3(a) 3(b) 3(c) 3(d)

Fig. 3. Seriate texture of Ilmenite-Hematite a) Parallel rows of exsolution lamellae of Hematite in Ilmenite. b) Multi generation exsolved phases of Ilmenite in Hematite. c) Equal amount of Hematite and Ilmenite as a decomposition product of a high temperature solutions. d) Two generations Ilmenite exsolution in Hematite host formed by stressed conditions.

Alteration of Ilmenite

A conspicuous differential response to weathering (chemical action of sea water) has been noted among those with exsolution (hematite + ilmenite) and without (unexsolved). The later shows alteration upto 40% by volume with the development of leucoxene, pseudorutile etc. while the exsolved ones are almost free from such effects of alteration.In general intergrowth of ilmenite-hematite appears to survive weathering and stand longer transport compared to magnetite-hematite; whereas ilmenite-magnetite intergrowths are the least durable (Molinaroli and Basu, 1987). The degree of alteration of different ilmenite deposits in the world is quite varied; those of South Africa and Brazil being altered relatively very little but those of Australia, India and America show extensive alteration. Alteration phenomenon

Table III. Composition of ilmenite separates determined By X-ray diffraction.

Sample	I Ilm/1$_{(104)}$ $_{(104)Hem}$	wt% Hematite	Cell parameters (Å)
M-2 (3.0 - 4.5ϕ)	1.52	28.12	a = 5.077 c = 14.003
M-2 (1.5 - 3.0ϕ)	1.69	26.14	a = 5.080 c = 14.017
M-4 (3.0 - 4.5ϕ)	1.61	27.42	a = 5.080 c = 14.015
M-4 (1.5 - 3.0ϕ)	1.92	23.55	a = 5.082 c = 14.020

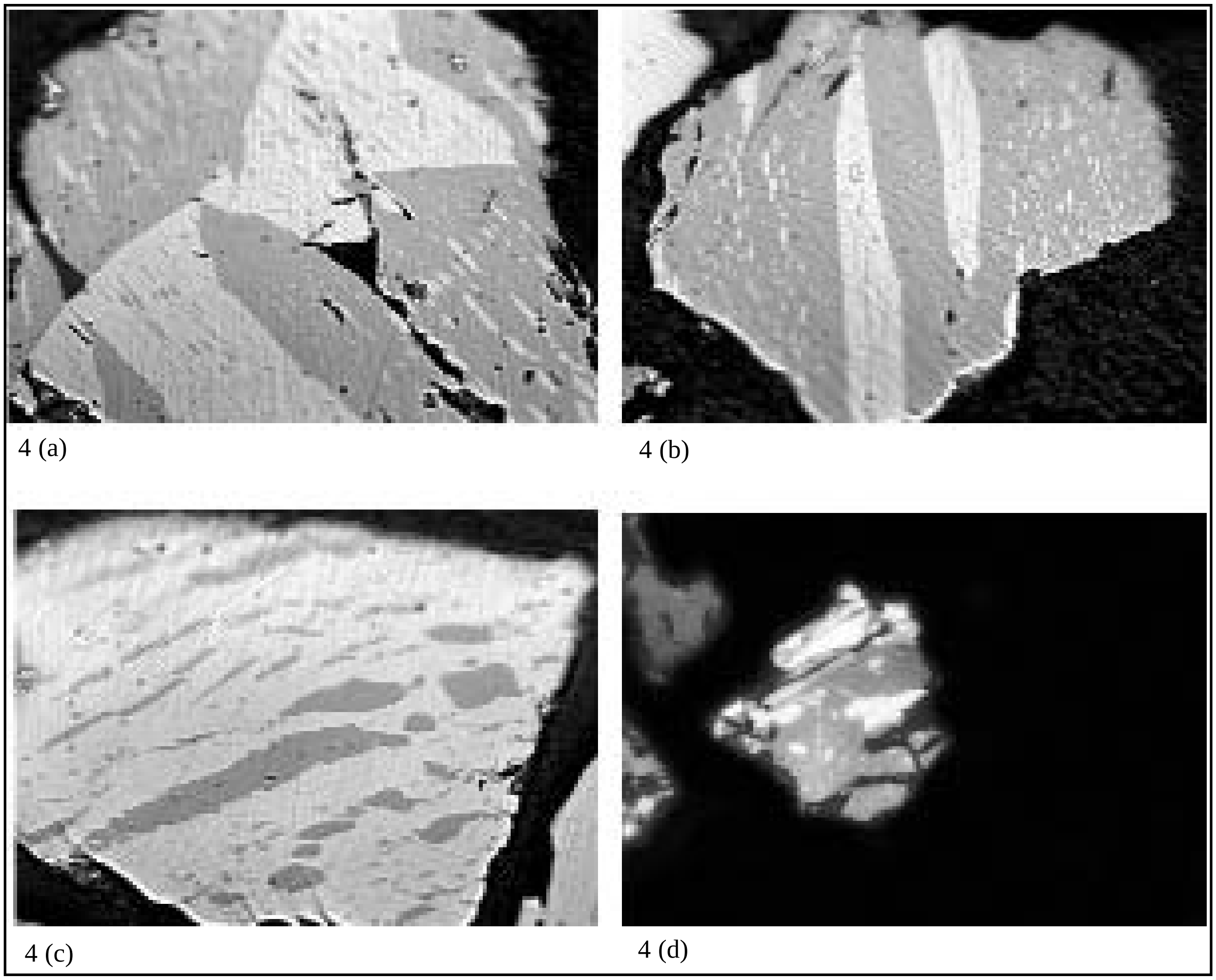


Fig. 4. a, b) Emulsion texture. c, d) Granular texture.

in the study area is less (10 to 20% of the total ilmenite) comparative to eastern and southern coast of India (Mitra and Ahmed, 1990). Alteration is seen only in unexsolved ilmenite, while the exsolved ones are almost free from such alteration. In the study area three stages of alteration is rec ognized viz, pseudoilmenite, pseudorutile and leucoxene. Though ilmenite alteration is neither uniform nor continuous; the alteration in the study area either may be continuously to pseudoilmenite-pseudorutile-leucoxene or directly to leucoxene or to pseudorutile. Table V shows the reflectiv

Table IV. Bulk Chemical analysis of ilmenites

Sample No.	TiO_2 %	FeO %	Fe_2O_3 %	MnO %	MgO %	Cr_2O_3	$FeO.TiO_2$ %	$\frac{FeO.TiO_2}{Fe_2O_3}$
M-2 (3.0-4.5ϕ)	39.76	30.82	27.84	1.14	0.27	0.06	70.58	2.54
M-2 (1.5-3.0ϕ)	41.35	31.96	25.31	1.17	0.04	0.02	73.31	2.89
M-4 (3.0-4.5ϕ)	39.12	29.83	28.04	1.30	0.34	0.308	68.95	2.46
M-4 (1.5-3.0ϕ)	42.85	32.02	23.70	1.04	0.08	0.03	74.87	3.16

Table V. Reflectivity of ilmenite and its alteration products

Mineral	Reflectivity
Ilmenite	19.2
Pseudoilmenite	20.0
Pseudorutile	21.8
Leucoxene	23.4

ity of ilmenite and its alteration products. X-ray diffraction data and chemical composition of ilmenite and its alteration products are given in Table VI and Table VII respectively. The chemical composition of the alteration products of ilmenite frequently fluctuates within definite ranges of non-stoichiometric composition and thus deviates from their ideal composition (Table VII)

Pseudoilmenite, the first phase of alteration occurs due the incomplete alteration of ilmenite to pseudorutile. Here patchy intergrowths of both altered and unaltered ilmenite present (Fig. 7). Alteration is taking place from the periph ery, along irregular veins and cracks. It is grey in color and isotropic (Fig. 7). Pseudoilmenite can be replaced by

Table VI. X-ray diffraction data of ilmenite and its alteration products

Ilmenite		Phase-1 Pseudoilmenite M-1		Phase-1 Pseudoilmenite M-2		Phase-2 Pseudorutile M-1		Phase-2 Pseudorutile M-2		Phase-3 Leucoxene M-1		Phase-3 Leucoxene M-2	
dA^O	I/I_1	dA^O	I/I_1	dA^O	I/I_1	dA^O	I/I_1	dA^O	I/I_1	dA^O	I/I_1	dA^O	I/I_1
3.737	30	3.85	30	3.722	20	2.697	26(H)	2.690	24(H)	3.251	100	3.242	94
2.754	100	2.742	100	2.764	100	2.751	20(IL)	2.726	20(IL)	2.482	38	2.784	42
2.544	70	2.591	55	2.506	50	2.480	65	2.478	60	2.181	14	2.170	17
2.237	30	2.242	25	2..172	10(PR)	2.087	36	2.060	30	2.049	6	2.186	10
1.868	40	1.802	30	1.792	25	1.683	100	1.672	100(IL)	1.683	50	1.720	55
1.726	55	1.770	60	1.709	50	1.702	12 (IL)	1.705	10(IL)	1.619	18	1.624	20
1.635	9	1.645	6	1.660	8	1.476	20	1.470	20	1.479	4	1.468	5
1.506	30	1.509	20	1.495	25	1.441	22	1.304	10	1.449	10	1.442	8
1.469	35	1.412	30	1.412	20	1.362	11	1.436	25	1.357	13	1.361	11
1.342	13	1.294	10	1.352	7					1.254	9		
		a_0=5.011 $\pm$0.02 A^O c_0=14.340 $\pm$0.012 A^O		a_0=5.182 $\pm$0.016 A^O c_0=13.85 $\pm$0.027 A^O		a_0=2.863 $\pm$0.034 A^O c_0=4.590 $\pm$0.042 A^O		a_0=2.860 $\pm$0.016 A^O c_0=4.531 $\pm$0.028 A^O		a_0=4.599 $\pm$0.017 A^O c_0=2.498 $\pm$0.031 A^O			

Table VII. Chemical compositions of ilmenite and its alteration products

Sample No.	FeO	Fe_2O_3	TiO_2	MnO	MgO	Al_2O_3	Fe/Ti-molar ratio	Formula	Mineral
M-1	43.86	3.28	50.65	1.38	0.52	0.09	0.98	$(Fe^{2+}_{1.87} Mn_{0.06} Mg_{0.04} Fe^{3+}_{0.10}) Ti_{1.93} O_6$	Ilmenite
M-2	20.24	23.68	53.98	1.36	0.50	0.11	0.89	$(Fe^{2+}_{0.77} Mn_{0.05} Mg_{0.03} Fe^{3+}_{0.83}) Ti_{1.93} O_6$	Pseudoilmenite
M-3	22.45	21.29	54.10	1.29	0.48	0.08	0.96	$(Fe^{2+}_{0.88} Mn_{0.05} Mg_{0.04} Fe^{3+}_{0.77}) Ti_{1.93} O_6$	Pseudoilmenite
M-4	8.96	31.06	58.98	0.78	0.45	0.10	0.65	$(Fe^{2+}_{0.27} Mn_{0.03} Mg_{0.03} Fe^{3+}_{107}) Ti_{202} O_6$	Pseudorutile
M-5	4.02	34.12	59.94	0.82	0.40	0.14	0.62	$(Fe^{2+}_{0.13} Mn_{0.06} Mg_{0.03} Fe^{3+}_{0.02}) Ti_{1.17} O_6$	Pseudorutile
M-6	-	1.21	91.90	0.13	0.87	0.48	0.02	$(Mg_{0.03} Fe^{2+}_{0.01} Al_{0.02} Ti_{0.93} O_6$	Leucoxene
M-7	-	0.21	98.13	-	-	0.40	0.004	TiO_2	Leucoxene

* Analyses exclusive of the trace elements Cr^{3+}, V^{3+}, Ni etc. present in the samples

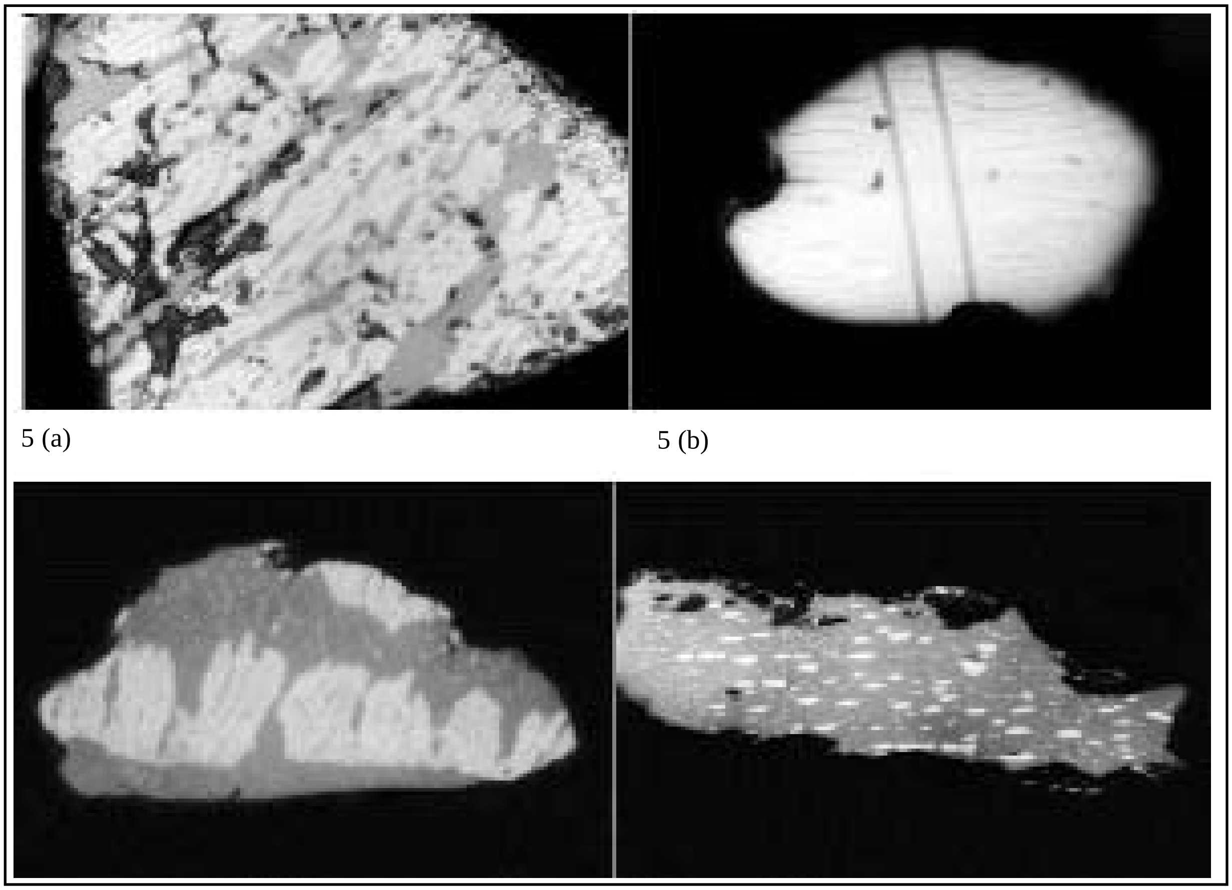


Fig. 5. a) Sub-graphic texture. b) Blitz texture of Hematite-Ilmenite-Rutile. c) Special type - forest fire texture. d) Special type - Deer skin texture.

pseudorutile (Fig. 7) or by leucoxene (Fig. 7). Pseudoilmenite is characterized by the simultaneous presence of bivalent and trivalent iron in its lattice. Its chemical composition may vary within a definite stoichiometric range (Table VII). The decrease of Fe content at this alteration study is accompanied an increase of Ti content and hence the molar ratio of Fe/Ti for ilmenite (1.02) decreases to about 0.84 for pseudoilmenite. Both optical and chemical properties of pseudoilmenite are distinctly different from those of ilmenite and pseudorutile, through the X-ray analyses correspond to those of ilmenite (Table VI). The X-ray patterns are diffused.

Pseudorutile is very fine grained, slightly grayer and isotropic nature. The reflectance of pseudorutile is higher than that of pseudoilmenite. The Fe_2O_3-content at this stage distinctly predominates over FeO-content (Table VII) and the Fe/Ti molar ratio decreases from 0.84 for pseudoilmenite to 0.64

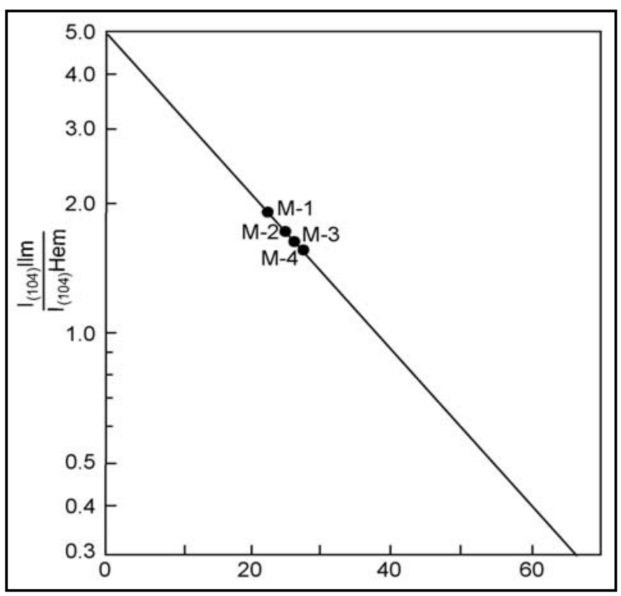


Fig. 6. X-ray deteriminative calibration curve representing ilmenite (104) peak intensity/hematite (104) peak intensity plotted on a logarithmic scale (5%, uncertainty).

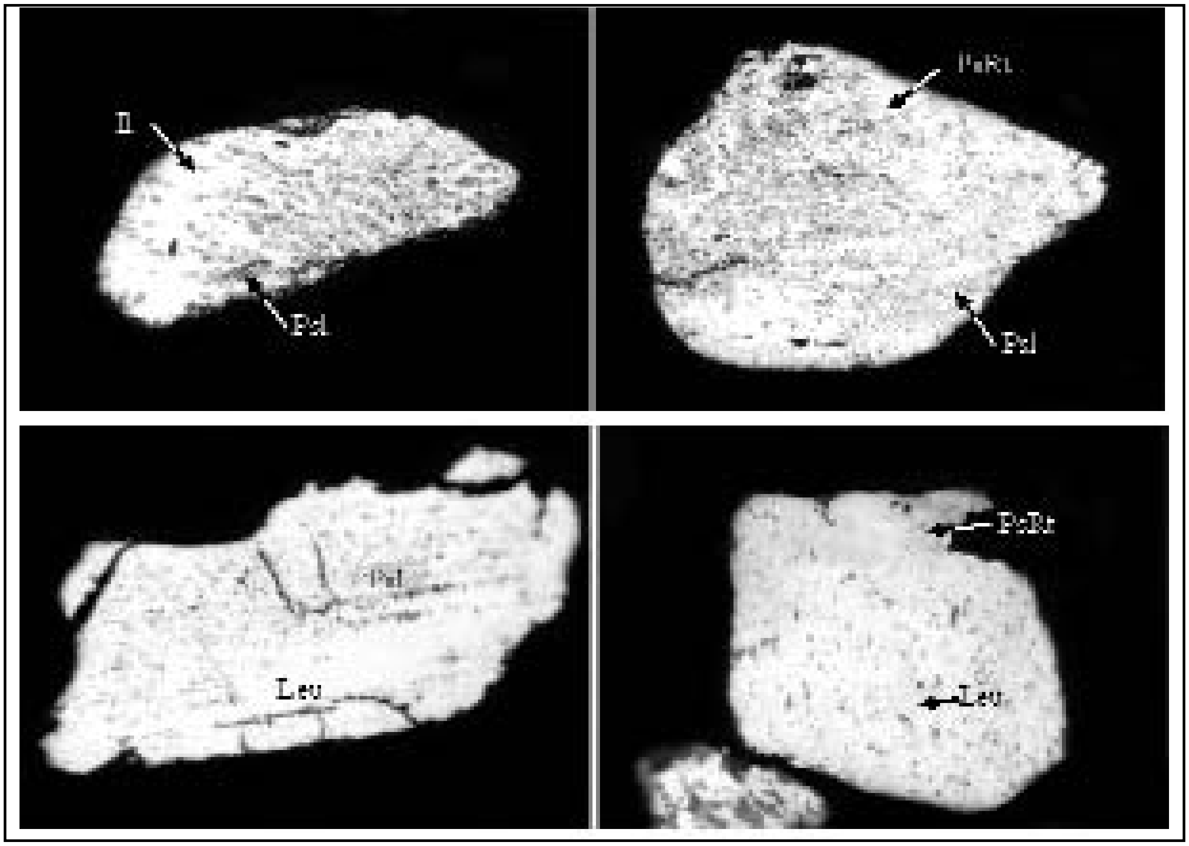


Fig. 7. Alteration of ilmenite a) Ilmenite (Il) is replaced by pseudoilmenite (psl) b) Pseudorutile (Psrt) and pseudoilmenite replaces ilmenite c) Pseudoilmenite is directly altered to leucoxere (Leu) d) In contrast to pseudorutile leucoxene shows yellowsih-white internal reflection

for pseudorutile. With continued removal of iron from the grain, pseudorutile converts to leucoxene either from the core of the grain or along the grain boundaries (Fig. 7). The areas formed of leucoxene are readily distinguished from pseudorutile or pseudoilmenite by their bright internal reflection under cross-nicols. In reflected light leucoxene is cream-colored. Cell volume, when computed from the data in Table III for altered ilmenite, clearly decreases from ilmenite to leucoxene leading to the shrinkage cracks in pseudorutile and leucoxene.

Conclusion

The major opaque mineral in Moheskhali beach is ilmenite which consists of 10 to 35% of the total heavies. About 50% of the unaltered ilmenites exsolves hematites in different textural patterns and due to this reason iron content in the ilmenite concentrate is likely to be more in the study area. About 70% of the exsolution texture is seriate texture where hematite lamellae in ilmenite and ilmenite lamellae in hematite are exsolved from $FeTiO_3$-Fe_2O_3 solid solution

From the modal analysis, chemical analysis and x-ray study, the percentage of ilmenite (exsolved + unexsolved) and hematite (exsolved) are observed to be 73.74% and 26.26% respectively. The widths of lamellae in detrital ilmenites are suggestive of igneous provenance. The widely banded exsolved phases were formed by continuous exsolution mechanism at higher temperature while the second generation thinner bands were formed discontinuously at lower temperature. The unexsolved ilmenites in the study area show alteration along grain boundaries and fractures. This alteration leads to the formation of leucoxene. Chemical analysis indicates enrichment of TiO_2, MgO and Al_2O_3 with loss of iron and MnO. It is also noted that the alteration of ilmenite is not uniform and the extent of alteration varies from grain to grain.

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