

Table 1. Fractionation of MK ilmenite.

Ilmenite fraction	Current (A)	Mag. Susc. ($\times 10^{-6}$)	Content (wt. %)	Sp. gravity
MK1	0.15	230	15.4	4.214
MK2	0.20	129	22.6	4.335
MK3	0.25	82	30.5	4.568
MK4	0.30	58	15.4	4.418
MK5	0.35	42	4.0	4.395
MK6	0.40	32	8.0	4.224
MK7	>0.40	<32	4.0	4.147

ter (1959). It is seen that at a separation current of 0.25 A, corresponding to a χ of 82×10^{-6} cgs units, a maximum of 30.5 wt. % of the bulk ilmenite, with a sp. gravity of 4.568, can be separated (Table 1). In comparison with synthetic ilmenite ($\chi = 96 \times 10^{-6}$ cgs units, sp. gravity = 4.7), the MK3 fraction is thus the closest in magnetic susceptibility and specific gravity to ideal ilmenite. About 40 wt. % of the bulk ilmenite has higher values of χ than that of pure synthetic ilmenite (MK1 plus MK2).

The chemical analysis of the seven fractions is given in Table 2. In MK ilmenite, iron oxide varies between 44 and 32 wt. % and TiO_2 between 55 and 65 wt. %.

Table 2. Results of the chemical analysis.

Ilmenite fraction	Composition (%)					
	FeO	Fe_2O_3	$\text{FeO} + \text{Fe}_2\text{O}_3$	TiO_2	Mn	Cr
MK1	20.96	21.88	42.84	56.02	0.195	0.079
MK2	26.50	17.30	43.80	54.26	0.188	0.071
MK3	32.20	11.70	43.90	55.70	0.123	0.104
MK4	26.21	15.62	41.83	56.84	0.124	0.106
MK5	15.35	25.91	41.26	57.84	0.139	0.144
MK6	7.97	29.77	37.74	60.66	0.130	0.168
MK7	3.36	28.90	32.26	65.06	0.106	0.232

With increasing alteration as indicated by decreasing χ , the Mn content decreases while Cr content increases. The increase in Cr is presumably due to relative enrichment following the selective leaching of other constituents. The first four magnetic fractions, having an average Fe-oxide content of 43 wt. % and TiO_2 of 55.7 wt. %, are relatively unaltered ilmenite. In these fractions, the mean value of the $\text{FeO}/\text{Fe}_2\text{O}_3$ ratio is 1.73. In the fifth fraction, compared to the fourth one, the weight percentage of ilmenite sharply decreases from 15.4 to 4 and $\text{FeO}/\text{Fe}_2\text{O}_3$ ratio decreases from 1.68 to 0.59. The last three fractions, containing an average iron oxide content of 37 wt. % and TiO_2 of 61 wt. %, belong to the highly-altered variety of ilmenite with a mean $\text{FeO}/\text{Fe}_2\text{O}_3$ of 0.36.

Based on the chemical analysis, a cut-off grade can be fixed for unaltered and significantly-altered varieties of ilmenite at a χ of around 58×10^{-6} cgs units. This has been confirmed by XRD and TGA studies. XRD patterns (Figure 1) illustrate that sharp and prominent peaks of ilmenite persist only in the first four fractions whereas the remaining three fractions are pseudorutile-rich. A similar observation has been reported for Capel ilmenite (Wort and Jones 1980). TGA data (Figure 2) show that the weight gain above 600°C occurs only for the first four fractions, implying the presence of substantial amounts of Fe^{2+} . Weight loss due to bound

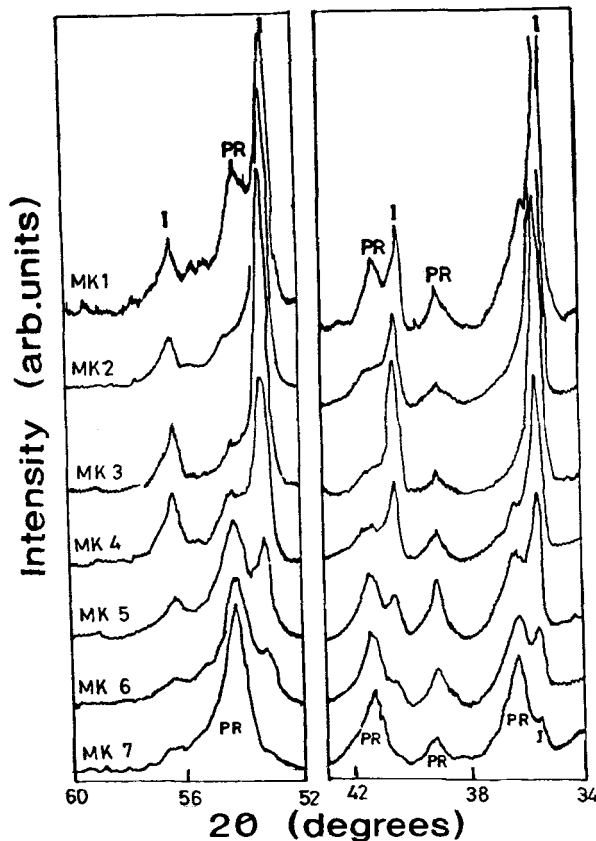


Figure 1. Powder XRD patterns of MK fractions showing ilmenite-rutile conversion. I—ilmenite; PR—Pseudorutile.

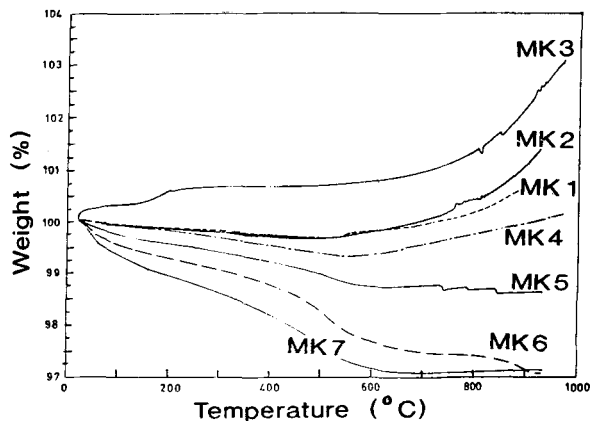


Figure 2. TGA curves of seven MK fractions.

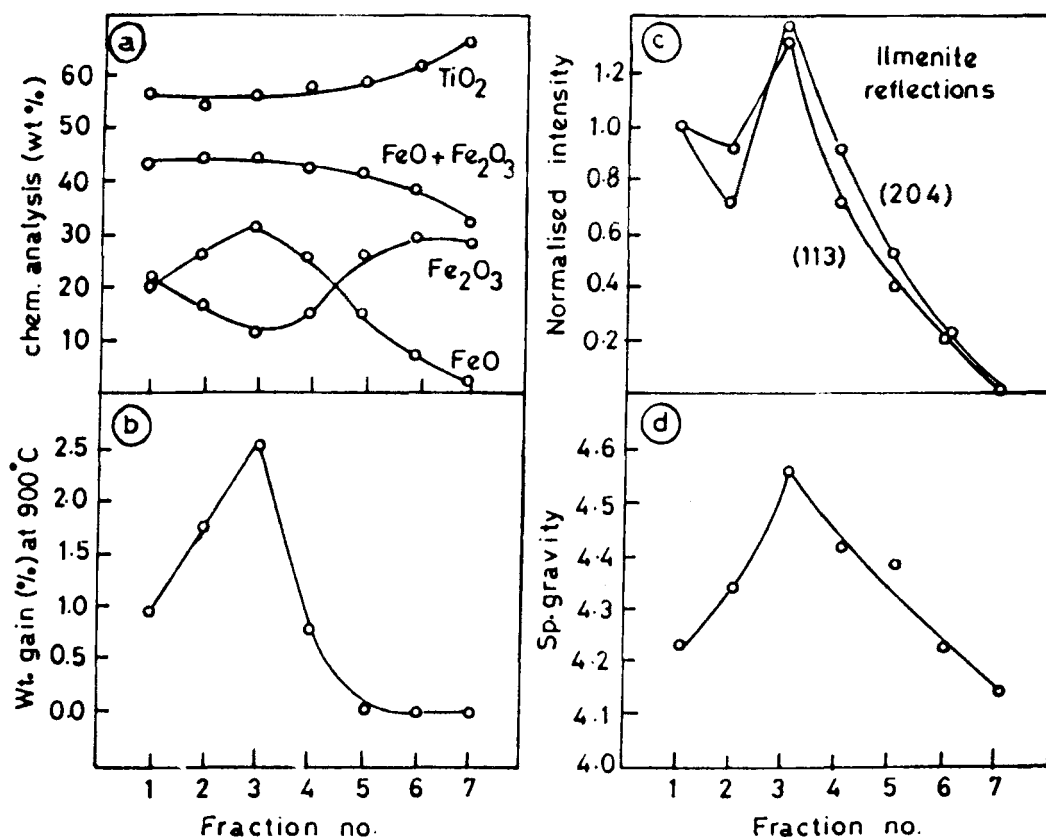


Figure 3. Synoptic diagram showing differential alteration.

water increases as the degree of alteration increases. The pseudorutile structure may actually contain hydroxyl ions (Frost *et al* 1983; Waychunas 1991). Thus the content of bound water might be correlated with the presence of the pseudorutile phase.

While the Australian Capel ilmenite fraction having χ of 81×10^{-6} cgs units contains 14.8 wt. % Fe₂O₃ (Wort and Jones 1980), the similar MK3 contains only 11.7 wt. %, but with a common TiO₂ of 55.7 wt. %. Maximum-altered Capel ilmenite contains 25 wt. % Fe₂O₃ against 29 wt. % in the similarly-altered MK7 fraction with 63.2 and 65 wt. % TiO₂ respectively.

Though ilmenite alteration is neither uniform nor continuous, the weathering mechanism is illustrated as a two-stage (Grey and Reid 1975) or a multi-stage process (Hugo and Cornell 1991). The present chemical and XRD data suggest that the initial step (ferrous-ferric iron transformation stage) is relatively advanced compared to the next Fe-removal stage. In other words the first stage is conformable to the widespread ferrous-ferric iron conversion in nature, a common low-temperature geochemical phenomenon, while the leaching step takes place only in favorable environments. However, oxidation to ferric state and peripheral removal of iron from the system can take place simultaneously. The incomplete alteration leads to the coexistence of

ilmenite, pseudorutile and rutile. Thus different fractions could be rich in one of these phases.

Though magnetic susceptibility is inversely related to the degree of alteration in ilmenite, it is reported that the highly magnetic fractions need not be those containing the least-altered ilmenite (Wort and Jones 1980). The present XRD, TGA, chemical analysis and sp. gravity studies (Figure 3) support this observation. Among the first three fractions the third one contains more FeO than the other two, total iron oxide being similar. Therefore ferrous-ferric iron conversion is less in the first two fractions. Neither Fe₂O₃ nor total Fe-oxides exceeds the equivalent amount of TiO₂ present in any of the ilmenite fractions. This points out that the Fe₂O₃ component cannot be due to ilmenite-hematite solid solution. Therefore, the ferric component can either form pseudorutile or hematite as a product of alteration. However, in the samples studied, pseudorutile was identified as the major product of alteration. Recently, our Mossbauer studies have confirmed that alteration in MK ilmenite stops at the pseudorutile stage (Das *et al* 1993).

Hexagonal *a* and *c* lattice parameters of ilmenite and the volume of the unit cell are illustrated in Figure 4. This has been done by the careful identification of the reflections of ilmenite and pseudorutile and assigning

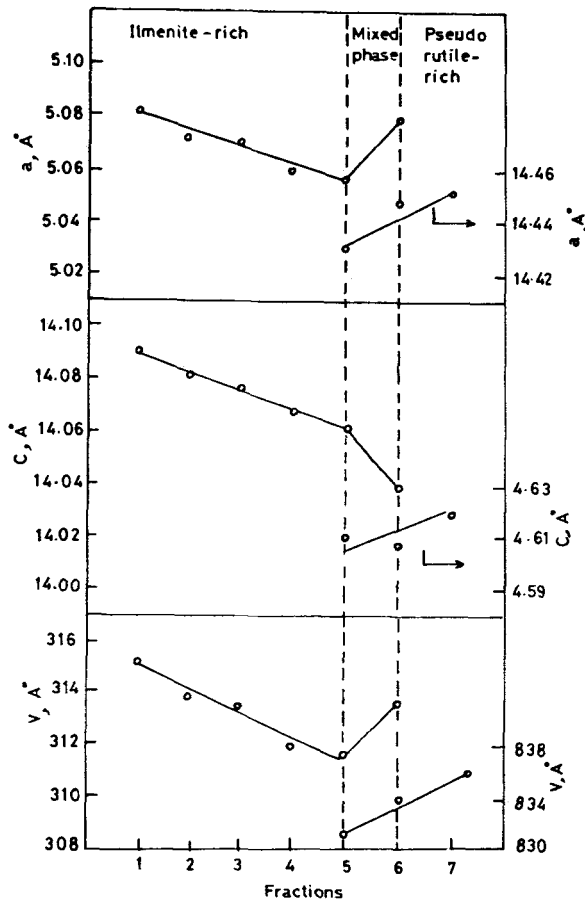


Figure 4. Variation of the lattice parameters and cell volume.

the (hkl) values based on the reported standard reflections of ilmenite (JCPDS file No.3-781). In the case of $\text{Fe}_2\text{Ti}_3\text{O}_9$ (pseudorutile), different XRD data are reported with slight variations in d-values and intensity ratios (Overholt *et al* 1950; Teufer and Temple 1966; Grey and Reid 1975). XRD values quoted in the latest reference were taken into consideration for the analysis of the pseudorutile phase.

Both the a and c lattice parameters of ilmenite were found to progressively decrease resulting in a reduction in the unit cell volume. According to the ionic radius concept, this decrease is anticipated if there is a Fe^{2+} - Fe^{3+} conversion as Fe^{3+} is smaller compared to Fe^{2+} . This conversion of ferrous to ferric iron while retaining the hexagonal structure can be achieved either by assuming excess oxygen in the lattice (FeTiO_{3+x}) or by ejection of some Ti or Fe by diffusion or by the reduction of some of Ti^{4+} to Ti^{3+} . The latter is very unlikely under natural conditions. Retaining the oxygen content during the ferrous-ferric iron transformation is possible by the simultaneous release of Fe^{3+} ions (Subrahmanyam *et al* 1982). In the 6th fraction,

where both ilmenite and pseudorutile co-exist, the ilmenite a parameter shows an increase and the c parameter a decrease. This has resulted in an increase in the unit cell volume (Figure 4), showing that the ilmenite structure expands, thus facilitating the Fe-leaching from the ilmenite but only to form pseudorutile.

CONCLUSIONS

Intense alteration with loss of much Fe has affected less than 20% of the material and apparently weathering has not been able to convert a significant quantity of ilmenite to rutile. The 3rd magnetic fraction with an approximate magnetic susceptibility of 82×10^{-6} cgs units is considered as the relatively-pure ilmenite of the deposit. XRD analysis shows that the hexagonal ilmenite structure contracts in the initial stages of alteration followed by an expansion before transforming to pseudorutile. It is clear that identification of a relatively-homogeneous raw material or an effectively-fractionated bulk ore is important for a given process and for a specific end product. Such an approach would lead not only to optimum utilization of ilmenite resources but also would facilitate the production of different grades of synthetic rutile.

ACKNOWLEDGMENTS

Ilmenite samples have been provided by the factories of Indian Rare Earths Limited. We acknowledge our colleagues Mr. S. Velusamy and Mr. S. Sasibhooshanan for their cordial help. Comments made by the anonymous referee were beneficial.

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(Received 14 July 1993; accepted 21 June 1994; Ms. 2401R)