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TECTONICALLY DEFORMED ILMENITE IN TITANIFEROUS IRON ORES OF THE MAMBULA COMPLEX, ZULULAND, SOUTH AFRICA

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ABSTRACT

Ilmenite from the Mambula Complex, South Africa, shows tectonically induced lamellar twinning parallel to $\{10\bar{1}1\}$ and two distinct size-ranges of magnetite platelets that formed by simultaneous reduction and exsolution. The platelets of the earlier set are coarser, more sparsely distributed, and nucleated heterogeneously at energetically favorable sites along twin planes and at the intersection of twin planes. The platelets of the second set are much thinner and oriented parallel to $\{0001\}$; they were formed by nucleation and growth in areas away from the larger platelets of magnetite. Geothermometry based on coexisting ilmenite–magnetite pairs places an upper temperature limit on the episode of deformational twinning between 500 and 600°C at $\log_{10}f(O_2)$ between −17.5 and −18.5. A lower temperature limit of 480°C at $\log_{10}f(O_2)$ = −24.6, placed on the twinning episode, reflects the temperature at which ionic re-equilibration between coexisting ilmenite and magnetite effectively ceased. The dominant mode of ilmenite deformation at low strain-rates and elevated temperature is mechanical twinning on $\{10\bar{1}1\}$.

Keywords: Mambula Complex, South Africa, ilmenite, deformation, twinning, magnetite, exsolution.

SOMMAIRE

L'ilménite du complexe de Mambula (Afrique du Sud) illustre le développement, dû à la tectonique, de lamelles maclées parallèles à $\{10\bar{1}1\}$ et de magnétite en plaquettes de deux tailles distinctes formée par réduction et exsolution simultanées. Les plaquettes précoces, plus grossières et plus éparses, sont le produit d'une nucléation hétérogène dans les sites énergétiquement favorables dans les plans de macles et à leurs intersections. Les plaquettes de deuxième génération, beaucoup plus fines, orientées parallèlement à $\{0001\}$, se sont formées par nucléation et croissance dans les régions éloignées des premières plaquettes. Les couples magnétite–ilménite permettent d'assigner des limites supérieures de température (de 500 à 600°C) et de fugacité d'oxygène [$\log_{10}f(O_2)$ entre −17.5 et −18.5] à l'épisode de déformation qui a produit les macles. Une limite inférieure de 480°C et $\log_{10}f(O_2)$ = −24.6 pour le même épisode correspond à la température à laquelle prit fin la ré-équilibration ionique entre ilménite et magnétite. L'ilménite, à faibles taux de déformation et à haute température, produit surtout des macles mécaniques sur $\{10\bar{1}1\}$.

(Traduit par la Rédaction)

Mots-clés: complexe de Mambula (Afrique du Sud), ilménite, déformation, maclage, magnétite, exsolution.

INTRODUCTION

Ilmenite from various geological environments may show the development of lamellar twinning (*inter alia* Edwards 1938, Vaasjoki 1948, Ramdohr 1969). In particular, tectonically deformed crystals of terrestial ilmenite characteristically show the development of twinning parallel to $\{10\bar{1}1\}$ (Chakravarty 1961, Ramdohr 1969). Shock-induced twinning in ilmenite, in contrast, develops parallel to both $\{10\bar{1}1\}$ and $\{0001\}$ and is a common feature of ilmenite from lunar breccias (Minkin & Chao 1971, Sclar 1971, Smith & Steele 1976). The development of shock-induced twinning in ilmenite at low temperatures has been demonstrated experimentally (Minkin & Chao 1971, Sclar *et al.* 1973), but little information is currently available on tectonically deformed terrestrial ilmenite. The aim of this paper is to describe some samples of tectonically deformed ilmenite from the Mambula intrusive complex, South Africa, that display well-developed twinning and related features of magnetite exsolution.

GEOLOGICAL SETTING

The small (25 km^2) gabbroic Mambula Complex is located at the confluence of the Tugela and Mambula rivers in Zululand, South Africa (28°58′S, 31°03′E). It contains several titaniferous magnetite- and ilmenite-rich layers that commonly consist of more than 70% by volume of opaque oxides (Du Toit 1918, Reynolds 1979). These rocks have been metamorphosed under conditions approaching the almandine-amphibolite facies during the 900–1000 Ma Natal–Namaqua regional metamorphic event (Cain 1975), which resulted in the partial recrystallization of the opaque oxides in the Fe–Ti ores (Reynolds 1979). These ores exhibit overall granoblastic polygonal textures. Reactivated oxidation of external granules and exsolution of ilmenite from the Ti-magnetite have also resulted in the development of abundant coarse granular crystals of ilmenite that range between 0.7 and 1.5 mm in size. This ilmenite is generally clear of intergrowths and shows the development of sparsely distributed twins that are attributed to annealing. In contrast, ilmenite in magnetite-rich layers from the southern margin of the complex shows the development of abundant

TABLE 1. COMPOSITIONS OF ILMENITE AND MAGNETITE FROM THE MAMBULA COMPLEX

	1	2	3	4	5	MAGNETITE	MB6
SiO_2	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	2.44
TiO_2	48.30	49.27	50.37	51.04	51.87	0.27	13.82
FeO	40.51	41.28	41.58	42.07	40.11	30.99	29.86
MgO	1.10	1.14	1.45	1.50	3.17	0.10	2.20
MnO	0.96	0.98	1.11	1.15	0.87	0.03	0.33
Fe_2O_3*	7.96	6.55	4.78	3.27	4.67	67.87	43.11
Al_2O_3	0.18	0.14	0.19	0.09	0.13	0.40	6.85
Cr_2O_3	0.03	0.04	0.03	0.02	N.D.	0.13	0.05
V_2O_3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.69
TOTAL	99.04	99.40	99.51	99.14	100.82	99.79	99.35
CATIONS PER FORMULA UNIT (ilmenite 2, magnetite 3)							
Ti	0.9224	0.9364	0.9531	0.9681	0.9559	0.0079	
Fe^{2+}	0.8603	0.8725	0.8751	0.8873	0.8221	1.0012	
Mg	0.0415	0.0430	0.0544	0.0564	0.1157	0.0058	
Mn	0.0206	0.0209	0.0236	0.0245	0.0181	0.0009	
Fe^{3+}	0.1522	0.1246	0.0906	0.0622	0.0863	1.9732	
Al	0.0027	0.0021	0.0029	0.0014	0.0019	0.0090	
Cr	0.0003	0.0005	0.0003	0.0002	-	0.0020	
RECALCULATED MOLE FRACTIONS (Stormer 1983)							
	X'_{ilm}	X'_{ilm}	X'_{ilm}	X'_{ilm}	X'_{ilm}	X'_{usp}	
	0.921	0.936	0.953	0.968	0.954	0.008	

*Fe_2O_3 calculated according to the method of Stormer (1983).

5: Average composition of undeformed ilmenite crystals from 16 samples of Mambula titaniferous iron ore (Reynolds 1979).

MB6: Bulk chemical analysis of Fe-Ti oxide ore containing twinned ilmenite crystals. Analyst = National Institute for Metallurgy (now Mintek), South Africa. (High Fe_2O_3 value reflects partial oxidation caused by surface weathering).

N.D. Not determined.

twin-lamellae that exhibit bending and dislocation features. Numerous platelets of exsolved magnetite are also present. The associated magnetite is virtually Ti-free (Table 1) and has recrystallized to form fine-grained granoblastic polygonal aggregates with the ilmenite. The magnetite contains relict exsolution bodies of Al-rich transparent spinel, but modified ilmenite lamellae are only sparingly present. The coexisting silicates have been altered to fine-grained aggregates of amphibole and chlorite, whereas a distinct tectonically induced foliation is developed in the enclosing metagabbro.

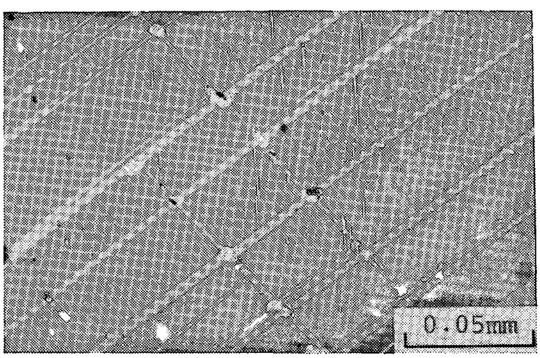

FIG. 1. Narrow twin-lamellae (lighter) in ilmenite showing the development of partly martitized magnetite platelets at the intersection of twin lamellae. Incident light, oil immersion.

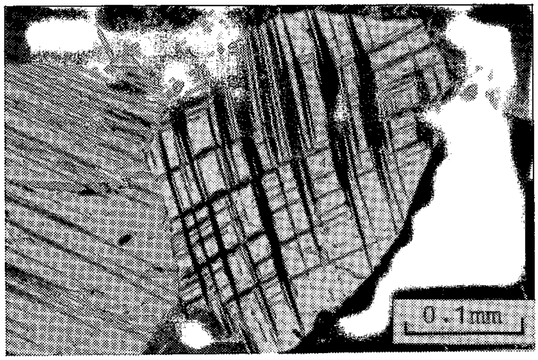

FIG. 2. Well-developed deformation-induced twin lamellae in ilmenite. Incident light, oil immersion, partially crossed polars.

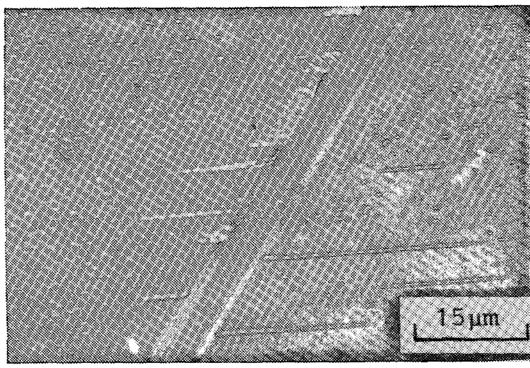

FIG. 3. Development of magnetite platelets (white, partly martitized) along twin planes in ilmenite. Note the absence of finer exsolution bodies in the immediate vicinity of the larger bodies, and their abundant development at a greater distance. Incident light, oil immersion.

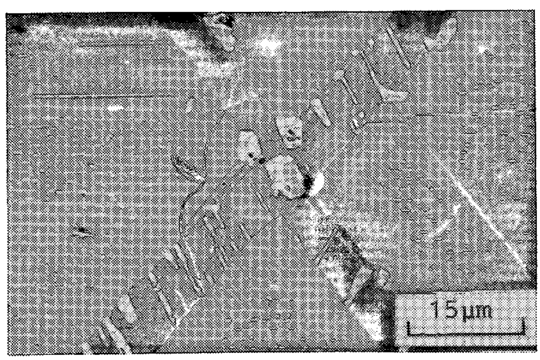

FIG. 4. Two intersecting twin-lamellae in ilmenite (darker) with associated larger magnetite platelets (white, partly martitized) separated from areas of fine magnetite exsolution by narrow, intergrowth-free zones.

Tectonically Deformed Ilmenite

The ilmenite exhibits polygonal outlines, and the degree of twinning is variable, suggesting an orientation effect. The twin lamellae are normally 0.001 - 0.05 mm wide, and it is assumed that they are developed parallel to the rhombohedral planes of the ilmenite (Fig. 1) as determined by Chakravarty (1961), Ramdohr (1969), Minkin & Chao (1971) and Sclar *et al.* (1973). Both the width and spacing of the lamellae are highly variable within any individual crystal, and they commonly taper and pinch out before the margin of the crystal is reached (Fig. 2). This is a characteristic feature of pressure twins that develop in response to imposed stress (Stanton 1972). In the more extreme cases, the twin lamellae are themselves bent, and translation along the twin planes is evident. This mechanical twinning on $\{10\bar{1}1\}$ is believed to be the most important deformation mechanism in ilmenite at relatively low strain-rates and elevated temperatures (Minkin & Chao 1971, Sclar *et al.* 1973). The basal planes of the ilmenite crystals are clearly defined by the orientation of the magnetite platelets that are developed parallel to {0001} (Buddington & Lindsley 1964, Haggerty 1976). No twin lamellae are developed parallel to this direction, which supports the contention that tectonic deformation of ilmenite does not result in the development of mechanical twinning parallel to {0001} (Minkin & Chao 1971, Sclar *et al.* 1973).

Small magnetite platelets 1–2 μm thick and up to 10 μm long are generally present at the intersection of twin lamellae and at irregularly spaced intervals along the twin composition-planes (Figs. 1, 3). They appear to have nucleated heterogeneously and grown at loci of high dislocation-energy caused by the twinning (Champness & Lorimer 1976). Similar textures have been reported from annealed deformed alloys in which nuclei of the exsolved phase form in regions in which the local degree of deformation is highest (Cahn 1970). It is conceivable that the ilmenite solid-solution may have been slightly supersaturated at the ambient temperature at the time of deformation, and the sudden creation of energetically favorable sites of nucleation initiated exsolution. The growth of these magnetite domains effectively depleted the immediate surroundings in the magnetite component and led to the establishment of concentration gradients between the magnetite lamellae and the undepleted supersaturated areas further away. This behavior is analogous to the well-documented depletion zones that are commonly present around the large exsolution bodies of Al-rich spinel in aluminous Ti-magnetite crystals (*inter alia* Ramdohr 1969, Bowles 1977).

The formation of crystallographically oriented lamellae of magnetite in ilmenite is generally ascribed to the contemporaneous reduction and exsolution of an original ilmenite–hematite solid solution. This is because magnetite is virtually insoluble in ilmenite at geologically realistic temperatures (Buddington & Lindsley 1964, Haggerty 1976). This implies that the range of oxygen fugacities at the time of deformation and exsolution was sufficiently low to stabilize ilmenite + magnetite, rather than an ilmenite + hematite assemblage. The magnetite in the platelets now shows varying degrees of martitization owing to later processes of surface weathering.

A further set of very much finer micrometre-sized platelets of magnetite is developed parallel to {0001} of the ilmenite in areas between the wider-spaced twin lamellae (Figs. 3, 4). These platelets are notably absent from areas immediately adjacent to the twin planes and the larger platelets of magnetite reflecting depletion of the magnetite component in such zones. It appears that processes of long-range diffusion were ineffective in substantially decreasing the concentration gradients, so that supersaturation was increased in the undepleted areas once postmetamorphic cooling commenced. This resulted in nucleation and growth of the fine magnetite bodies in these areas once supersaturation had reached sufficiently high levels. The formation of magnetite, rather than hematite bodies, is again ascribed to contemporaneous reduction with exsolution, indicating that the range of oxygen fugacities remained low during this stage of postmetamorphic cooling.

A crystal of ilmenite showing the development of similar exsolution bodies of magnetite at the intersection of sparse twin-lamellae has been illustrated by Ramdohr (1969, Fig. 576a). Ramdohr noted that these magnetite bodies developed after strong deformational twinning, but did not offer any explanation. An analogous situation involving the presence of twin lamellae in an exsolved hemo-ilmenite grain has also been illustrated by Edwards (1938, Fig. 7). The abundant robust lamellae of hematite in this sample vary in width and orientation across twin planes; Edwards cited this as evidence to show that exsolution had occurred after the episode of twinning. This suggests that these hematite bodies also nucleated heterogeneously at loci of high dislocation-energy resulting from the development of twin planes. The preferential location of the magnetite platelets at the intersections of twin lamellae in the deformed ilmenite from the Mambula Complex clearly shows that their development followed the twinning event.

Mineral Chemistry

Representative compositions of ilmenite and coexisting granular magnetite are presented in Table 1. The analyses were carried out on a Cambridge

Microscan V electron microprobe using wavelength-dispersion techniques and utilizing well-analyzed mineral standards. Raw data were reduced using the Bence–Albee routine. The Fe^{2+}/Fe^{3+} distribution and mole fractions of hematite and ulvöspinel were calculated by the method of Stormer (1983).

The analyses show that the ilmenite is close to its theoretical end-member composition; it contains only minor Mg and Mn. Both these elements are enriched in the ilmenite relative to the coexisting magnetite; this reflects their preferential partitioning into the rhombohedral phase during cooling and subsequent subsolidus re-equilibration (*inter alia* Vincent & Phillips 1954, Neumann 1974, Bowles 1977, Himmelberg & Ford 1977). The calculated Fe_2O_3 contents are variable, and reflect the degree to which exsolution and depletion have taken place. Composition 1 shows the highest Fe_2O_3 content and represents an area of fine magnetite platelets (now oxidized to martite) that cannot effectively be avoided by the electron beam. This bulk composition is slightly more Fe_2O_3-rich than homogeneous ilmenite crystals from slowly cooled igneous rocks, that generally contain less than 6% Fe_2O_3. This is considered to be close to the original pre-exsolution composition of the ilmenite–hematite solid solution in areas away from the larger magnetite platelets. Composition 4, in contrast, shows the lowest Fe_2O_3 content and represents a Fe^{3+}-depleted area immediately adjacent to a large magnetite platelet. Compositions 2 and 3 are representative of ilmenite that is free of exsolved phases, in areas intermediate between 1 and 4.

The coexisting granular magnetite is also close to its theoretical end-member composition and contains only very minor amounts of Mg and Mn. The original titaniferous character of this magnetite is indicated by the coexistence of abundant granular ilmenite, the presence of transparent Al-rich exsolution bodies and the survival of minor lamellae of relict ilmenite, and is also suggested by a comparison with opaque oxide-rich samples from less-deformed areas of the complex. The low residual TiO_2 content of this magnetite, therefore, indicates that extensive unmixing of the original magnetite–ulvöspinel solid solution has occurred. Elevated temperatures during regional metamorphism in the almandine-amphibolite facies (Cain 1975) and subsequent cooling after the thermal metamorphic peak reactivated exsolution and contemporaneous processes of oxidation and exsolution that effectively purged the magnetite of its Ti-bearing component. The Mg and Mn contents of the magnetite would also have been further reduced *via* subsolidus re-equilibration with the coexisting ilmenite during this period. The Al and Cr contents of the magnetite are higher than those of the ilmenite and reflect the preferential partitioning of these elements into the cubic phase relative to the coexisting rhombohedral phase. MB6 represents a bulk chemical composition of a silicate-poor portion of the Fe–Ti oxide ore that hosts the deformed ilmenite grains. This sample contains approximately 8% by volume of chlorite as the major impurity, and provides an indication of the composition of the original Ti-magnetite, assuming that the original oxide assemblage consisted largely of Ti-magnetite with only minor granular ilmenite (Reynolds 1979).

Temperature Estimates for Deformational Twinning

The revised ilmenite – Ti-magnetite geothermometer of Spencer & Lindsley (1981) cannot be applied directly to the deformed Fe^{3+}-poor ilmenite and coexisting Ti-poor magnetite because the intersection of calculated mole fractions of hematite and ulvöspinel fall outside the calibrated compositional range. Extrapolation of the relevant composition lines into this region indicates that final re-equilibration occurred at approximately 480°C and a $\log_{10}f(O_2)$ of -24.6. This is indicative of the temperature at which ionic re-equilibration between coexisting ilmenite and magnetite ceased and provides a lower limit for the temperature and $f(O_2)$ conditions that existed during regional metamorphism. This also provides a lower temperature limit for the deformational twinning of the ilmenite. The existence of a low oxygen fugacity at this stage is supported by the presence of magnetite-reduction lamellae instead of hematite-exsolution bodies in the ilmenite.

Ilmenite composition 1 can be used to estimate the temperature and $f(O_2)$ conditions that existed after the thermal peak of metamorphism, if it is assumed that the Ti-poor magnetite composition was approached before a significant amount of cooling had taken place. This assumption is supported by the presence of abundant tectonically twinned granular ilmenite that is considered to have originated by extensive external granule exsolution of the primary Ti-magnetite. This process would have caused considerable depletion of the Ti-content of the magnetite prior to the deformational episode. Allowing for the uncertainties inherent in this assumption, it is significant that a temperature of approximately 550°C at a $\log_{10}f(O_2) = -18.5$ is obtained. These conditions are very close to the approximate ilmenite–hematite solubility limit (Spencer & Lindsley 1981) and support the existence of a near-saturated homogeneous rhombohedral phase at this stage of cooling. The coexisting magnetite would have held a larger amount of ulvöspinel in solid solution at the thermal peak of the metamorphic episode, so that the estimated temperature and $f(O_2)$ are probably both too low for this event. The extent of this difference is not known, but it is probably not significantly more than 50°C and one log $f(O_2)$ unit. This yields an upper temperature limit of between 550 and 600°C for the thermal peak of regional metamorphism and the development of deformational twinning in the ilmenite. High-energy dislocation sites were created within the ilmenite by the development of twin planes, particularly at their intersections. These provided suitable loci for the heterogeneous nucleation and growth of contemporaneous reduction-and-exsolution platelets of magnetite once suitable supersaturation levels were reached during subsequent cooling.

The maximum upper limit for amphibolite-facies regional metamorphism is placed at 650°C, beyond which anatexis occurs in felsic rocks (Winkler 1976, Turner 1981). Estimates of the lower temperature limit vary between 450°C (Turner 1981) and 500°C (Winkler 1976). The equilibrium temperatures obtained for the deformed ilmenite - Ti-magnetite pairs fall within this temperature interval and support the validity of the above approach.

Summary and Conclusions

The origin and microstructural evolution of the twinned granular ilmenite in certain Fe–Ti oxide ores from the southern margin of the Mambula Complex can be summarized as follows:

1) Ti-magnetite-rich layers containing minor coprecipitated ilmenite and silicate minerals developed during the later stages of fractional crystallization of the Mambula Complex. Primary subsolidus cooling resulted in the development of a range of ilmenite and Al-rich spinel microintergrowths in the Ti-magnetite crystals. A certain amount of granular ilmenite also formed by processes of oxidation and exsolution of external granules.

2) Increased temperatures during amphibolite-facies regional metamorphism resulted in the reactivation of contemporaneous oxidation and exsolution processes. This resulted in the development of abundant granular ilmenite by external granule exsolution and effectively purged the magnetite of the bulk of its titanium.

3) Temperature estimates based on compositions of coexisting ilmenite–magnetite pairs indicate maximum metamorphic temperatures of between 550 and 600°C at oxygen fugacities between $\log_{10}f(O_2)$ -18.5 and -17.5. The lower temperature limit at which ionic re-equilibration between ilmenite and magnetite ceased is placed at approximately 480°C at $\log_{10}f(O_2) = -24.6$.

4) Tectonic deformation of the ilmenite within this temperature range resulted in the development of twinning parallel to $\{10\bar{1}1\}$. The twinning episode probably occurred toward the upper limit of the 480–600°C temperature range during which the microstructures developed.

5) Heterogeneous nucleation of the larger platelets of magnetite occurred at energetically favorable sites that were created along twin planes, particularly at their intersections. Subsequent growth of the magnetite bodies was promoted by contemporaneous reduction with exsolution under conditions of low oxygen fugacity.

6) Nucleation and growth of the finer magnetite platelets were also activated by contemporaneous processes of reduction and exsolution as supersaturation was reached in areas away from the larger magnetite bodies during postmetamorphic cooling.

7) Partial martitization of both the magnetite platelets and the Ti-poor magnetite occurred during recent surface weathering.

The microstructural features of the ilmenite support the earlier conclusions of Ramdohr (1969) and Minkin & Chao (1971) that mechanical twinning on $\{10\bar{1}1\}$ is the most important mechanism of deformation of ilmenite at relatively low strain-rates and elevated temperatures. The loci and nature of the magnetite-exsolution bodies can be related to modern exsolution theory, as summarized by Champness & Lorimer (1976); the sequential development of these microstructures provides additional information on the conditions under which metamorphism occurred.

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