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Cation Distribution and Equilibration Temperature of Amphiboles from the Sittampundi Complex, South India

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1. Introduction

Amphiboles are utilized as indicators of temperature, pressure, volatile content, and oxidation state by the petrologist; while to a mineralogist the amphiboles provide instructive examples of structural phase transitions, cation ordering over multiple crystallographic sites, and a variety of atomic substitutions (Ghiorso & Evans, 2002). Relating the chemistry of calcic-amphibole to the *P-T* conditions of crystallization is a daunting task, because the amphibole crystal structure consists of a variety of cation sites, accommodating elements of a wide range of ionic radii and valences.

Amphibole has the general formula $A_{0-1}X_2Y_5Z_8O_{22}(OH,F,Cl)_2$, where $A = Na, K$; $X = Na, Li, Ca, Mn, Fe^{2+}, Mg$; $Y = Mg, Fe^{2+}, Mn, Al, Fe^{3+}, Ti, Li$; $Z = Si, Al$. Calcic amphiboles crystallize in the monoclinic crystal system (space group $C2/m$) (Hawthorne, 1983). An essential feature of amphibole is the presence of $(Si,Al)O_4$ tetrahedra linked to form chain which has double the width of those in pyroxenes. The four octahedral sites (M1, M2, M3 and M4) present in amphibole are neither symmetric nor of equal energy. The M1, M2 and M3 octahedra, which accommodate Mn, Fe^{2+} , Mg, Fe^{3+} , Cr, Al and Ti cations, share edges to form octahedral bonds parallel to *c*-axis. The M1 and M3 (regular octahedra) are co-ordinated by four oxygens and two (OH,F) groups. The M2 (slightly distorted octahedra) is co-ordinated by six oxygens. The cations Ti, Al and Fe^{3+} usually prefer the M2 site, especially when Na occupies the adjacent M4 site (e.g., Papike et al., 1969; Papike, 1988). The M4 site, which accommodates Na, Li, Ca, Mn, Fe^{2+} , Mg cations, is commonly a slightly-distorted octahedron when occupied by Mn, Fe^{2+} and Mg; and it is eight co-ordinated when occupied by Na or Ca (Papike, 1988). Two independent chains of tetrahedral sites, T1 and T2 cross-link the octahedral strips. The T1 site shares three oxygens with other tetrahedra and is more regular and smaller than T2. The T2 site shares two oxygens with adjacent tetrahedra. Papike et al. (1969) observed the preference of Al for the T1 site relative to the T2 site in hornblende, based on $\langle T1-O \rangle$ and $\langle T2-O \rangle$ lengths. The large and highly distorted A – polyhedron occurs between the tetrahedral chain. This site can be vacant, partially filled, or fully occupied by Na and/or K in $C2/m$ structure.

The present work was undertaken to investigate the chemical characteristic of calcic and magnesian amphiboles from upper amphibolite to granulite facies rocks in the Sittampundi complex. In addition, main objective of this work is to characterize iron ions of different valence states and their distribution within the crystal lattice, both in calcic and magnesian amphiboles of amphibolite, meta-anorthosite and chromitite respectively, using ^{57}Fe Mössbauer spectroscopy and estimate the equilibration temperature attained by the host rock.

2. Geological setting

Sittampundi Anorthosite Complex occurs as a layered igneous body. The area forms a part of the granulite terrain of South India. Major rock types are chromitite bearing meta-anorthosite, amphibolite, basic granulite, two pyroxene granulite, leptynite, biotite gneiss and pink granite. In this complex, amphibolite and chromitite occur as small discontinuous bands/lenses within the meta-anorthosite. Basic granulite occurs as discontinuous but conformable layers within meta-anorthosite. Two-pyroxene granulite and pink granite occur within the biotite-gneiss. The former two are the later intrusive phases (Subramaniam, 1956).

In the study area amphiboles commonly occur throughout the complex as mosaic aggregates, rarely as inclusion in plagioclase. The mineral assemblages of the major amphibole-bearing rocks are as follows:

- i. Amphibolite: calcic amphibole + plagioclase \pm garnet \pm cpx \pm chlorite \pm sphene \pm opaque
- ii. Meta-anorthosite: plagioclase \pm calcic amphibole \pm anthophyllite \pm clinozoisite \pm clinopyroxene \pm garnet \pm corundum \pm epidote \pm scapolite \pm carbonate \pm hercynite \pm spinel \pm sillimanite \pm muscovite \pm chlorite \pm sphene \pm opaque.
- iii. Chromitite: chromite \pm calcic amphibole \pm anthophyllite \pm rutile \pm chlorite.

In the basic granulite and two-pyroxene granulite, calcic amphibole occurs as rim surrounding diopside. In meta-anorthosite, tschermakitic hornblende forms triple junction with plagioclase; and triple junctions are common between amphibole crystals in amphibolite.

3. Experimental methods

Electron microprobe analysis of the samples has been obtained by JEOL-733 superprobe (wavelength dispersive method) at 15 kV with beam current of 0.01 μA and 2 μm beam diameter and Cameca CAMEBAX SX 50 probe with digital computer PDP 11.53. Operating condition was 15 kV accelerating voltage with a beam current of 15 nA and beam size $\sim 1\mu\text{m}$. The average spectrum count was compared with the natural standards.

The room temperature (RT) Mössbauer spectra were recorded in a conventional constant acceleration spectrometer (Wissel GmbH, Germany) with a 25 mCi Co/Rh source. Two mirror-imaged spectra were obtained in 512 out of 1024 channels. The finely powdered samples were pressed tight in plexi holders, and based on the iron content (known from EPMA) determined amounts of the samples were taken to optimise the thickness effect. The amount used for the samples are: 60 mg (sample 3c), 68 mg (sample 15b) and 92 mg (sample 30a). The velocity calibration was performed with respect to pure metallic iron (99.99%). The spectra were then fitted to the sum of Lorentzian functions with a non-linear least square fitting programme of Meerwall (1975) on an IBM personal computer. For each doublet the intensity and line-width of the peaks were constrained to be equal.

4. Results and discussion

4.1 Chemical composition of Sittampundi amphiboles

Amphiboles have been classified according to Leake (1978). For this classification empirical Fe^{3+} in calcic amphibole was estimated from EPMA chemistry following Schumacher (1991). Site occupancy of different elements in amphiboles (viz. calcic amphiboles, anthophyllite) was assigned on the basis of 23 oxygens following Papike (1988). We present here the composition of only three amphiboles (3c, 15b, 30a), the chemistry of which were determined employing both the EPMA and Mössbauer spectroscopy (Table 1).

Oxides	3c	15b	30a
SiO ₂	42.49	42.48	56.44
TiO ₂	1.26	0.28	0.07
Cr ₂ O ₃	0.06	0.03	0.48
Al ₂ O ₃	11.47	16.82	2.86
FeO	18.78	10.80	7.80
MnO	0.34	0.15	0.20
MgO	9.04	13.09	28.35
NiO	0.01	-	0.11
CaO	11.57	11.52	0.60
Na ₂ O	1.91	2.52	0.30
K ₂ O	0.54	0.08	-
Total	97.47	97.57	97.21
Number of cations on the basis of 23 oxygen			
Si	6.459	6.145	7.699
Fe ³⁺	0.402	0.263	0.065
Al ^{IV}	1.139	1.592	0.236
Sum (T)	8.000	8.000	8.000
Al ^{VI}	0.916	1.290	0.224
Ti	0.144	0.030	0.007
Cr	0.007	0.003	0.052
Fe ³⁺	0.317	0.182	0.166
Fe ²⁺	1.293	0.675	0.368
Mg	2.048	2.820	4.148
Mn	0.044	-	0.023
Ni	0.001	-	0.012
Sum (M123)	4.770	5.000	5.000
Fe ²⁺	0.377	0.193	0.290
Mg	-	0.015	1.618
Number of cations on the basis of 23 oxygen			
Mn	-	0.018	-
Ca	1.884	1.794	0.087
Na	-	-	0.005
Sum (M4)	2.261	2.020	2.000
Na	0.502	0.709	0.075
K	0.104	0.014	-
Sum (A)	0.660	0.723	0.075

Table 1. Chemical composition and cation distribution of amphiboles studied by EPMA and Mössbauer spectroscopy

4.2 Characterization of samples

Studied amphiboles were selected from different metamorphic rocks, such as amphibolite (sample 3c), meta-anorthosite (sample 15b), chromitite (sample 17c, 18b, 25, 29, 30a) and basic granulite (sample 6b, 13). Mössbauer analysis of two calcic amphiboles (sample 3c and 15b) and one anthophyllite (sample 30a) were considered in the present study. Sample 3c was taken from amphibolite occurring in the eastern part of the Sittampundi complex, near the Cauvery river bed of Kettuvellanpalaiyam. Sample 15b was taken from meta-anorthosite in the western side of Pamandapalaiyam, near the road between Cholasiramani and Tottiyamthottam. Sample 30a was taken from chromitite in the western side of Karungalpatti. The crystal chemistry of the last three amphiboles was determined from microprobe analyses combined with the results of Mössbauer spectroscopy. The studied amphibole species present in different rock types are identified as follows:

- i. Meta-anorthosite: ferroan pargasite
- ii. Chromitite: edenitic hornblende, magnesio-hornblende, tchermakitic hornblende, anthophyllite
- iii. Basic granulite: edenite, edenitic hornblende, pargasite, pargasitic hornblende, ferro-hornblende, actinolite
- iv. Two-pyroxene granulite: edenitic hornblende, magnesio-hornblende
- v. Amphibolite: ferroan pargasitic hornblende

The variational range of X_{Mg} [$Mg/(Mg+Fe)$], X_{Ca} [$Ca/(Ca+Mg)$], X_K [$K/(K+Na)$], TiO_2 and Cr_2O_3 of calcic amphiboles and anthophyllite in different rock types are given in Table 2. TiO_2 content of calcic amphibole in meta-anorthosite is low (0.21-0.36 wt%) but it is high in some basic granulite ($\cong 2.07$ wt%). TiO_2 content of calc amphiboles in two-pyroxene granulite, amphibolite and chromitite range between 0.90-1.42 wt%, 1.02-1.47 wt%, 0.47-1.09 wt%, respectively. Cr_2O_3 content of calcic-amphiboles and anthophyllite in chromitite show wide variation (0.00-1.12 and 0.38-0.83 wt%, respectively), but in other rock types it is low (<0.34 wt%). The plot of Si vs. (Ca+Na+K) of Sittampundi calcic amphiboles, following Shido & Miyashiro (1959) and Binns (1965), show a cluster in the field of green and brown hornblende (belonging to the high amphibolite and granulite facies). In anthophyllite, the ratio of (Na+K) to tetrahedral Al is remarkably constant and it lies close to $\frac{1}{4}$. Thus, for each 0.1 Na ion per formula unit, there are 0.4 tetrahedral Al ions substituting for Si.

Amphiboles	Host rock	X_{Mg}	X_{Ca}	X_K	TiO_2	Cr_2O_3
	Basic granulite	0.36-0.89	0.29-0.55	0.00-0.30	0.13-2.07	0.02-0.33
Calcic-amphibole	Two pyroxene granulite	0.68-0.78	0.33-0.37	0.00-0.02	0.90-1.42	0.00-0.33
	Anorthosite	0.68-0.69	0.38-0.40	0.02-0.11	0.21-0.36	0.00-0.35
	Amphibolite	0.45-0.47	0.47-0.49	0.14-0.17	1.02-1.47	0.02-1.12
Anthophyllite	Chromitite	0.84-0.87	0.01-0.02	0.02-0.05	0.03-0.14	0.38-0.83

Table 2. The range of the values of X_{Mg} , X_{Ca} , X_K , TiO_2 and Cr_2O_3 for calcic amphiboles and anthophyllite in the studied samples based on the EPMA

4.3 Mössbauer study and assignment

The room temperature (298 K) Mössbauer spectrum of the calcic amphiboles and anthophyllite show a close range of isomer shift (IS) of octahedral Fe²⁺ ion (Table 3) and the assignment is made from the quadrupole splitting (QS) values. The quadrupole splitting of M1, M2 and M3 sites which are regular octahedra, completely overlap in the case of Mg-Fe amphibole, but chemistry becoming more complex with Ca, Na, Al and Fe³⁺ the QS values tend to decrease (Goldman, 1979).

Sample No	IS (mm/s)	QS (mm/s)	Distribution	Area (%)	Width (mm/s)	χ^2	Fe ³⁺ / Σ Fe
Calcic-amphibole							
3c	1.23	2.93	Fe ²⁺ (M1M3)	33.59	0.35	1.38	0.30
	1.25	2.47	Fe ²⁺ (M2)	20.52	0.41		
	1.47	1.41	Fe ²⁺ (M4)	15.77	0.49		
	0.41	0.82	Fe ³⁺ (M2)	13.29	0.37		
	0.02	0.35	Fe ³⁺ (T)	16.83	0.38		
15b	1.22	2.84	Fe ²⁺ (M1M3)	32.05	0.35	3.00	0.20
	1.25	2.35	Fe ²⁺ (M2)	19.35	0.44		
	1.42	1.40	Fe ²⁺ (M4)	14.75	0.41		
	0.47	0.86	Fe ³⁺ (M2)	13.83	0.41		
	0.07	0.38	Fe ³⁺ (T)	20.02	0.41		
Anthophyllite							
30a	1.28	2.88	Fe ²⁺ (M1M3)	33.51	0.38	4.94	0.26
	1.13	2.42	Fe ²⁺ (M2)	7.89	0.39		
	1.26	1.93	Fe ²⁺ (M4)	32.60	0.40		
	0.50	0.75	Fe ³⁺ (M2)	18.71	0.45		
	0.01	0.55	Fe ³⁺ (T)	7.29	0.25		

Table 3. Mössbauer parameters of amphiboles with Fe³⁺/ Σ Fe ratio

The M1 and M3 sites of the calcic amphibole are usually occupied by the Fe²⁺ and Mg whereas the M2 site is occupied by Fe³⁺, Al³⁺, Fe²⁺ and Mg (Skogby, 1987). With incorporation of more Al and Fe³⁺ in amphibole structure the M–O bond length of M2 site become smaller (Papike et al., 1969) and the QS for Fe²⁺ in the M2 site would manifest a reduction compared to that of the M1 and M3 sites. However, Della Ventura et al. (2005) showed that because the M2 site is adjacent to the M4 site, the QS value change correlated directly with the Li–Na substitution. In aluminous amphibole Fe²⁺ is enriched in M3 site relative to M1, which host Mg. In calcic amphibole, the M4 octahedral site is most distorted and is preferentially occupied by larger cation such as Ca, Na, Mn although occurrence of Fe²⁺ and Mg at M4 site has been postulated by some authors (Goldman & Rossman, 1977; Goldman, 1979; Skogby & Annersten, 1985; Skogby, 1987). Difficulty in determining Fe²⁺ at M4 site arises because Ca occupies 85-95% of this site (Leake, 1968). However, a high degree of distortion of the M4 site is expected to produce large Fe²⁺ crystal field splitting analogous to that seen in the distorted M2 site of orthopyroxene (Goldman & Rossman, 1977).

4.3.1 Calcic amphiboles

The room temperature (298 K) Mössbauer spectra (Figs. 1a, b) of the ferroan pargasitic hornblende (from amphibolite, sample 3c) and ferroan pargasite (from meta-anorthosite, sample 15b), show two asymmetric paramagnetic doublets of which the more intense peak lies at lower velocity region ($\cong -0.13$ mm/s). The fitting of the Mössbauer spectrum was approached in two ways: viz. (a) four doublet fit (three doublets for octahedral Fe^{2+} ions, and the other for octahedral Fe^{3+}), and (b) five doublet fit (three doublets for octahedral Fe^{2+} ions, and two for octahedral and tetrahedral Fe^{3+}). The five doublet fit improves the chi-square value and narrows down the line-width (0.35-0.49 mm/s) from wider line widths of the four doublet fit. The Mössbauer parameters as well as the assignment of the doublets are presented in Table 3. The basis of assignment is stated below.

In five doublet fit, the outermost doublets (1-1) with IS 1.23 mm/s (for sample 3c) and 1.22 mm/s (for sample 15b) and corresponding QS values of 2.93 mm/s, 2.84 mm/s are assigned to Fe^{2+} in M1 and M3 sites (Goldman, 1979; Schumacher, 1991). However, the IS values (at RT) for silicate phases range from 1.3 to 1.43 mm/s for octahedral Fe^{2+} (vide Mitra, 1992, Table 1.3). The doublets (3-3) showing IS 1.25 mm/s (for sample 3c), 1.25 mm/s (for sample 15b) and the corresponding QS 2.47 mm/s, 2.35 mm/s are assigned to Fe^{2+} in the M2 site (Goldman, 1979; Schumacher, 1991). The innermost doublets (5-5) with IS 1.47 mm/s (for sample 3c), 1.42 mm/s (for sample 15b) and corresponding QS 1.41 mm/s, 1.40 mm/s are assigned to Fe^{2+} in the M4 site (Goldman, 1979; Skogby & Annersten, 1985; Skogby, 1987; Schumacher, 1991). Differing QS value arises from difference in magnetic field generated by the orbital momentum of Fe^{2+} ion in those octahedral (M) sites giving rise to different orbital contributions H_{orb} and H_{hf} and also to the differences in covalency (Linares et al., 1983). The M4 site is bonded more covalently with Ca ion, with consequent decrease in the QS value of Fe^{2+} at this site. The decrease can also be accounted by considering the NNN effect where one Fe^{2+} is substituted by cations such as Mg^{2+} or Ca^{2+} and the super exchange in Fe–O–Fe bond, between octahedral sites (Goodenough, 1963). The entry of Al^{3+} ion in the M2 site could also affect the QS value of the M4 site. The lower QS value of 1.41 mm/s for sample 3c ($\text{Al}^{\text{VI}} = 0.916$ p.f.u.) and 1.40 mm/s for sample 15b ($\text{Al}^{\text{VI}} = 1.290$ p.f.u.) of M4 site may also signify greater distortion caused by faster rate of its formation (Jenkins, 1987; Skogby, 1987) during cooling.

4.3.1.1 Fe^{3+} in calcic amphiboles

The doublets (4-4) showing IS 0.41 mm/s (for sample 3c), 0.47 mm/s (for sample 15b) and corresponding QS 0.82 mm/s, 0.86 mm/s are assigned to Fe^{3+} in the M2 site (Goldman, 1979; Skogby & Annersten, 1985; Skogby, 1987; Schumacher, 1991). Fe^{3+} at distorted polyhedron shows larger QS than the regular one. So the larger QS value of Fe^{3+} doublet represents its occupancy at distorted M2 site.

The IS value of the other Fe^{3+} doublet (2-2) suggests a tetrahedral co-ordination rather than Fe^{3+} at octahedral site. So the doublets with IS 0.02 mm/s (for sample 3c), 0.07 mm/s (for sample 15b) and the corresponding QS 0.35 mm/s, 0.38 mm/s are assigned to Fe^{3+} in the less-distorted tetrahedral site. Extremely low value of IS for sample 3c is due to Fe^{3+} at an almost regular tetrahedron.

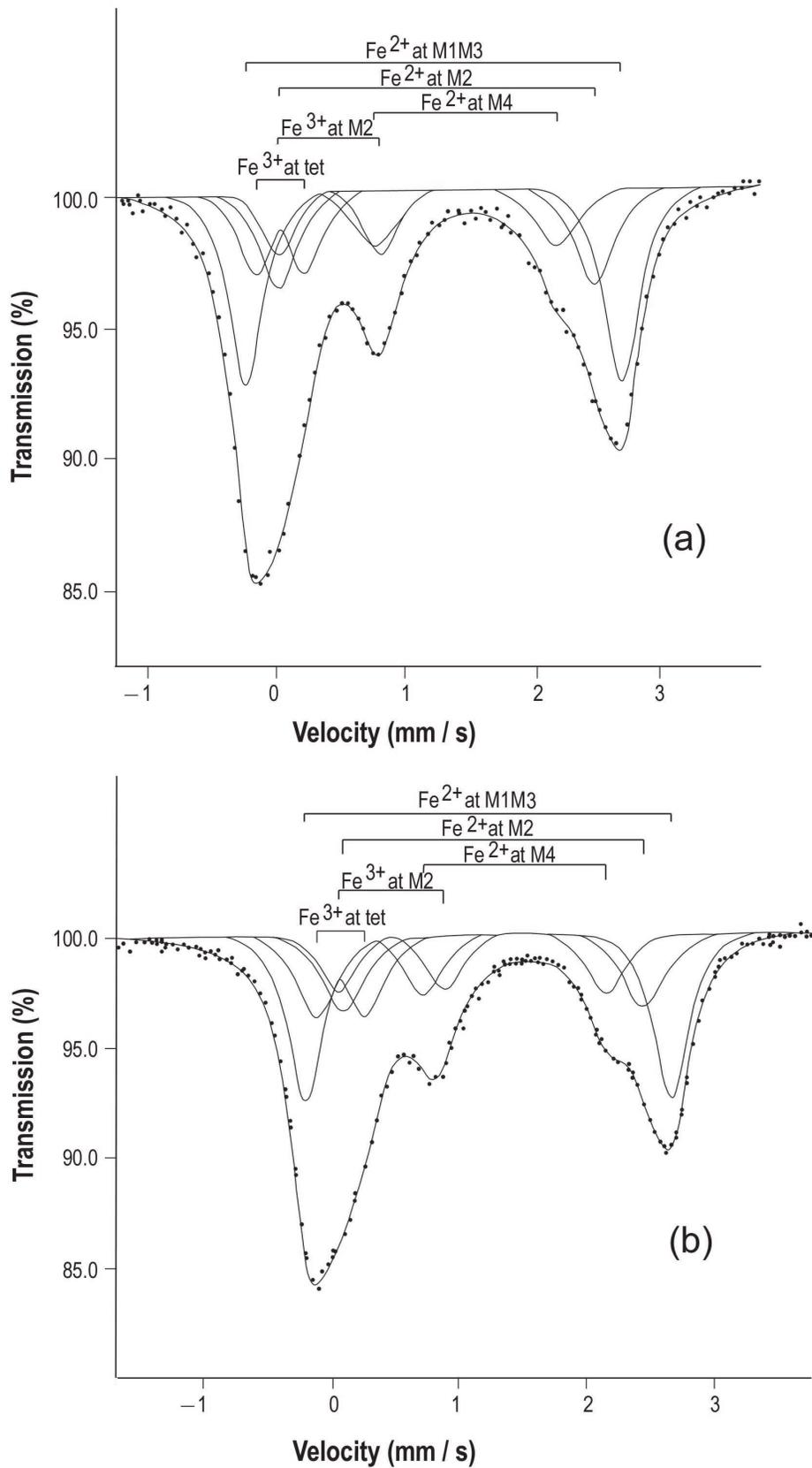
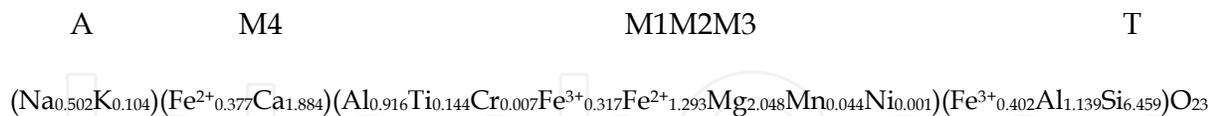


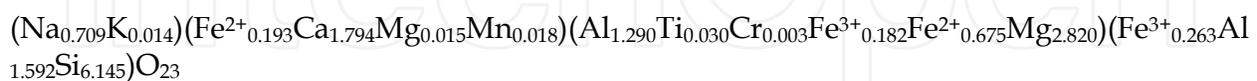
Fig. 1. Room temperature ⁵⁷Fe Mössbauer spectra of calcic amphiboles (a) sample 3c (b) sample 15b (Dots: Observed MS counts; continuous lines: fit with Lorentzian line shape)

The structural formulae of calcic amphiboles determined from EPMA and Mössbauer analysis are

Sample 3c



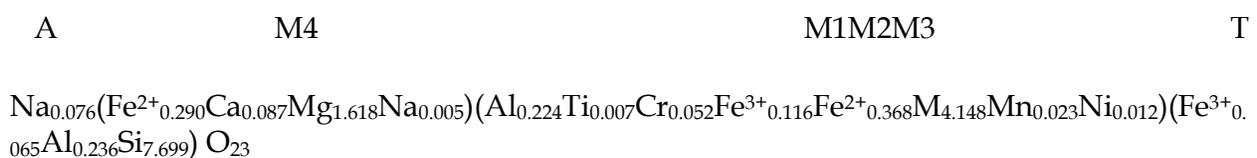
Sample 15b



The Fe²⁺ content (15.76% of total iron) of M4 site for sample 3c (total iron content is greater than sample 15b) determined by spectral fitting gives oversaturation of M4 site with vacancies in M1, M2 and M3 sites. The reduction of Fe²⁺ at M4 site in computer fitting causes increase in chi-square value of fitting which is not agreeable. So the enrichment of Fe²⁺ at highly distorted M4 site (QS = 1.41 mm/s) is evidently related to the substitutional Fe³⁺ at tetrahedral site. This coupled entry of Fe²⁺ at M4 site and Fe³⁺ at tetrahedral site point to a higher ordering by slow cooling. However, in natural calcic amphibole cation, M4 site totaling greater than 2.00 p.f.u. is not uncommon (Deer et al., 1978). This type of over saturation (in M4 site) and vacancies (in M1, M2 and M3 sites) are present in sample 3c (Table 1).

4.3.2 Anthophyllite

The room temperature (298 K) Mössbauer spectrum (Fig. 1c) of anthophyllite occurring in chromitite (sample 30a) consists of two asymmetric, paramagnetic doublets with more intense peak at lower velocity region ($\cong 0.25$ mm/s). The inner, more intense doublet can, on the basis of the crystal structure determination, be assigned to Fe²⁺ in M4; and the outer doublet to Fe²⁺ in M1, M2 and M3 sites (Seifert & Virgo, 1973). Similar to the calcic amphiboles, the fitting of Mössbauer spectrum was approached likewise in two ways: (a) four doublet fit and (b) five doublet fit. The five doublet fit here again showed better Mössbauer parameter (χ^2) than those of the four doublet fit. The Mössbauer parameter as well as the assignment of the iron doublet to specific crystallographic site of anthophyllite, following calcic amphibole assignment, is presented in Table 3. The distribution of Fe²⁺, Fe³⁺ in the different sites of anthophyllite (sample 30a) determined by spectral fitting indicates the absence of vacancies in different sites. The structural formula of anthophyllite determined from EPMA and Mössbauer analysis is



The higher QS value (0.55 mm/s) of Fe³⁺ doublet (2-2) of tetrahedral site suggests that this site is more regular (as it should be) than the tetrahedral site of calcic amphiboles (0.35 mm/s for sample 3c and 0.38 mm/s for sample 15b).

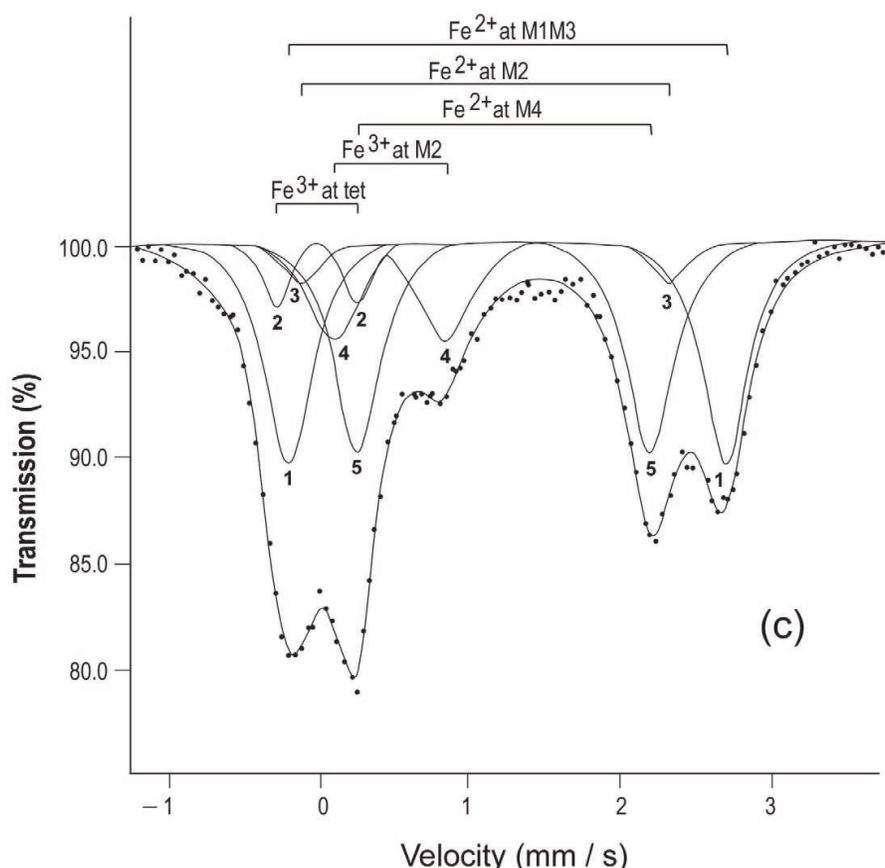


Fig. 1. (c) Room temperature ^{57}Fe Mössbauer spectra of anthophyllite sample (30a, Dots: Observed MS counts; continuous lines: fit with Lorentzian line shape)

It should be mentioned that the chi-square values of samples 15b ($\chi^2 = 3.84$) and 30a ($\chi^2 = 4.94$) are not within the usual limits. X-ray study shows that the sample 15b is inhomogeneous in character. However, the inhomogeneity could be seen in backscattered electron (BSE) images but the fineness of the lamellae evaded analysis. The spectral fitting of these samples show higher chi-square values, but the fitting logic and Mössbauer parameters are in conformity with the earlier reported values (e.g., Schumacher, 1991).

5. Equilibration temperature of the amphiboles

Mineral assemblage of the studied calcic amphibole bearing samples indicates that the rock underwent amphibolite to lower granulite facies metamorphism. Amphiboles are good indicators of temperature and pressure over wide range of metamorphic conditions (Shido & Miyashiro, 1959; Engel & Engel, 1962; Binns, 1965; Leake, 1965, 1971; Bard, 1970; Ernst, 2002). We have used Holland & Blundy's (1994) amphibole-plagioclase thermometry to calculate the equilibration temperature of the studied Sittampundi calcic amphiboles. In the studied samples, the magmatic Ca-amphibole probably equilibrated with calcic plagioclase but may not have been silica-saturated, and the mineralogical assemblages of the samples do not show any free quartz, so the thermometer (B) was utilized. A pressure of 10 kbar has been estimated using Newton & Perkin's (1982) geobarometry for the basic granulites and this is considered for calculating the equilibrium temperature of the studied samples. The

estimated equilibration temperatures of the calcic amphiboles are presented in Table 4. Due to the absence of suitable coexisting mineral pairs, for the anthophyllite associated with the Sittampundi chromitites, we used the Plyusnina's (1982) curve to calculate the equilibration temperature and found to range from 530-560°C. We have also used Graham & Powell's (1984) hornblende-garnet geothermometry to calculate the equilibration temperature of the basic granulites. The temperatures calculated from the two methods are falling within the same range.

Rock type	Sample no.	Temperature (°C)	Method
Basic granulite	11b	800	Holland & Blundy (1994)
		780-800	Graham & Powell (1984)
	12c	650-680	Graham & Powell (1984)
	33	695-750	Holland & Blundy (1994)
	41a	680-700	Graham & Powell (1984)
Two-pyroxene granulite	6b	540	Plyusnina (1982)
	13	540	Plyusnina (1982)
Meta-anorthosite	15b	890	Holland & Blundy (1994)
Amphibolite	3c	800-820	Holland & Blundy (1994)
Chromitite	17c	530	Plyusnina (1982)
	18b	560	Plyusnina (1982)
	25	550	Plyusnina (1982)
	29	560	Plyusnina (1982)
	30a	540	Plyusnina (1982)

Table 4. Equilibration temperatures of Sittampundi amphiboles

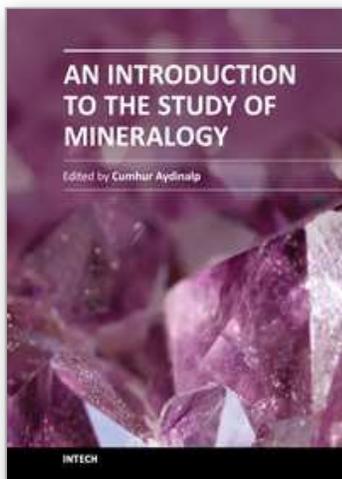
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