LETTER

Trace element heterogeneity in hydrothermal diopside: evidence for Ti depletion and Sr-Eu-LREE enrichment during hydrothermal metamorphism of mantle harzburgite

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We discovered a new kind of dyke in the mantle section of the Oman ophiolite composed mainly of diopside that we called diopsidite, although traces of forsterite, anorthite, titanite or andradite may be also observed. The texture and chemical composition, and the relationship between antigorite and diopside or rare forsterite, suggest that they formed by high-*T* metamorphism of serpentine (antigorite) and are the result of interaction between hydrothermal fluid and percolating magma in the shallow mantle. Chemical data, obtained by electron microprobe and in situ laser ablation coupled to ICP-MS, show large heterogeneities from one sample to another and between diopside crystals in each sample. The trace element patterns are characterised by strong positive anomalies in Sr and Eu and a negative anomaly in Ti. Small-scale heterogeneities reflect the disequilibrium conditions in the dyke formation and show that a relatively Ti-rich- and Al-Eu-Sr-LREE-poor phase may be included in the diopside. The exact nature of this phase is difficult to determine but the Eu negative anomaly, and the low Sr and LREE concentration suggest that it is probably some partially altered, relict protolith mineral.

Keywords: Diopsidite, Oman ophiolite, Mantle dyke, Trace element heterogeneity, Hydrothermalism

INTRODUCTION

Dykes are ubiquitous in the mantle section of the Oman ophiolite, the large majority showing mineralogical and chemical compositions and textures compatible with crystallisation from common silicate melts (Allemann and Peters, 1972; Python and Ceuleneer, 2003). They are interpreted as the relics of liquid that circulated in the mantle and fed the overlying gabbroic crust. However, a few of the dykes exhibit characteristics that cannot be explained by partial melting/melt crystallisation process. Samples taken from these dykes show textures and mineralogical and chemical compositions that suggest that they formed by the metamorphism of antigorite and minor tremolite (Python et al., 2007) under nonequilibrium conditions. Their field occurrence as well as their mineral assemblage (diopside $\pm$ forsterite, anorthite and/or

traces of andradite and various hydrous minerals such as antigorite or tremolite) resembles some skarn deposits developed in contact metamorphic aureoles or rodingite dykes found in serpentinite bodies of ophiolitic complexes (Frost, 1974; Schandl et al., 1989; Puschignig, 2002; Palandri and Reeda, 2004). Contrasting with these examples, however, the studied rocks are usually very poor in Fe, Al, Cr, Ti, Na and in all trace elements except Sr and Eu. These are features that can be simply explained by their derivation from a mantle protolith that is rather depleted in all these elements when compared to the crustal lithologies from which rodingite derives.

We interpreted these dykes as the product of high-temperature hydrothermal circulation in the upper mantle section of the Oman ophiolite. The strong combination between the provision of hydrothermal fluids and heating by mantle upwelling led to the partial transformation of mantle olivine into serpentine and its subsequent metamorphism into diopsidite. In a previous paper (Python et al., 2007) we presented the petrographic and major element chemical characteristics of these diopsidite dykes. In this paper, we give preliminary trace element data obtained by

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in situ laser ablation coupled to ICP-MS, allowing us to decipher the origin of trace element heterogeneity in the hydrothermal diopside.

ANALYTICAL METHODS

Each mineral was analysed by electron probe micro-analysis using a standard program at Toulouse University (Cameca SX50) and Kanazawa University (JEOL JXA-8800R), using an acceleration voltage of 20 kV, a probe current of 20 nA and a probe diameter of 3 μm . In each case, Cameca or JEOL software was used for ZAF corrections. Three samples were analysed for their trace element compositions by inductively coupled plasma mass spectrometry coordinated with laser ablation (LA-ICP-MS, Agilent 7500S, 193 nm ArF excimer: MicroLas GeoLas Q-plus) at the Incubation Business Laboratory Centre, Kanazawa (Ishida et al., 2004); the global settings of the laser and the mass spectrometer are given in Appendix 1. Analyses were performed by ablating 132 μm diameter centres on diopside with 240 laser pulses at 6 Hz with an energy density of 6 J/cm² per pulse. The total duration of the analyses was 120 seconds and the background (detection limit) was counted during the first 40 seconds. At the commencement and end of each batch of three or four sample analyses, measurements under the same conditions were carried out on the primary calibration standard (NIST SRM 612 glass). To avoid Pb or Ba contamination occurring during sample preparation (thin section polishing), one preliminary laser shot was given at each analysis point before analysis.

Data reduction was carried out for all points (including the heterogeneous ones), following a protocol essentially identical to that outlined by Longerich et al. (1996), and three times the standard deviation of the values obtained during the first 40 seconds were used as detection limits. Concentrations in ppm for elements were computed using three steps: 1) the average of the count per second measured during ablation; 2) application of a linear drift correction by using the calibration value obtained on the glass NIST SRM 612 (Pearce et al., 1997; Morishita et al., 2004); 3) using the concentration of SiO₂ measured with the microprobe as an internal standard.

For the representation in (Fig. 2) a “concentration-normalised intensity” was computed by normalising the instantaneous intensity in counts per second (cps) with the ratio of the calculated concentration in parts per million (ppm) for each element to the average intensity during the analysis time. This concentration-normalised intensity was used as the ordinate axis. For representation commodity, the concentration-normalised intensity for Si and Ca was divided by 10⁴, that for Co multiplied by 10, and

that for Y by 100.

TRACE ELEMENT COMPOSITIONS OF DIOPSIDE

We analysed three samples of a diopsidite dyke from Wadi Sudum in the Oman ophiolite. The dyke is approximately one kilometre in length with a highly variable width from a few centimetres to almost one metre, striking at 130° and dipping at 70° to the northwest. Two core samples of the dyke (05SDM2a and 05SDM2b, noted 02a-# and 02b-# in Table 1) were respectively taken from a ~ 1 m and a ~ 15 cm width section. The samples show textural, mineralogical and chemical characteristics typically observed for the diopsidite dykes, (Python et al., 2007) with a 100% diopside monomineralic composition having a fibrous texture in the specimen from the thicker part and a partly cataclastic texture for the thinner section. The third sample (05SDM1a, labeled 01a-# in Table 1) was taken from the contact with the host rock in the thin (~ 15 cm) zone. This shows a unique texture with needles of diopside crystals in a fine-grained mixed matrix of antigorite and tremolite. In this sample, globular ghosts of olivine may still be identified (see Fig. 5 in Python et al., 2007 for a microphotograph of this sample). In all three samples, the X_{Mg} (= Mg/(Mg + Fe) atomic ratio) of the diopside is abnormally high (95%-99%) and the Al, Cr, and Ti concentration is very low (Table 1).

The Strongly Depleted Chemistry Of Clinopyroxene In Diopsidite

The shaded area in Figure 1 shows the trace element com-

Figure 1. Chondrite (Sun and McDonough, 1989) normalised abundance pattern for moderately to highly incompatible elements. Most of the diopside specimens plot in the shaded area (12 analysis on three thin sections for three different parts of the same dyke), the black line with open circles and the dashed line with grey squares show the profile for two highly heterogeneous diopside crystals of sample 05SDM2b1 (point labeled 01a-1 and 01a-2 on Table 1, see Figure 2 for variations during laser ablation) and represent the average trace element concentration for the duration of the ablation.

Table 1. Chemical characteristics of the analysed diopside in each sample

Label	02a-1	02a-2	02a-3	02b-5	02b-6	02b-7	01a-9	01a-8	01a-6	01a-7	01a-3	01a-4	01a-1	01a-2
Microprobe														
Al*	918	1879	1307	910	852	759	868	214	924	1545	1352	1799	794	13
Ti*	0	46	28	30	40	0	30	0	0	0	78	0	156	36
Cr*	0	0	48	0	44	0	137	268	250	157	92	274	181	0
Xmg*	95.48	96.05	96.96	96.94	97.58	97.24	97.33	97.97	98.22	95.80	97.04	93.64	99.07	95.89
La-ICP-MS														
Al	726.9	868.3	710.4	578.5	760.2	815.4	1357.1	463.7	515.3	1006.7	1986.2	1895.7	158.1	71.2
Cr	17.44	80.18	60.71	25.57	7.41	9.59	223.37	549.19	419.97	573.81	249.14	670.54	121.22	99.34
La	0.01	0.01	0.02	0.03	0.01	0.01	0.07	0.02	0.05	0.04	0.01	0.01	0.00	0.00
Ce	0.05	0.04	0.07	0.09	0.06	0.04	0.16	0.08	0.20	0.15	0.04	0.04	0.03	0.03
Sr	4.58	4.59	5.20	4.96	4.88	4.61	4.73	4.01	5.02	5.11	4.48	4.47	1.16	1.12
Pr	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.03	0.03	0.01	0.01	0.01	0.01
Nd	0.07	0.05	0.06	0.09	0.07	0.06	0.07	0.06	0.15	0.11	0.05	0.07	0.05	0.07
Sm	0.04	0.02	0.02	0.03	0.03	0.02	0.02	0.02	0.03	0.03	0.02	0.04	0.04	0.04
Eu	0.04	0.02	0.03	0.04	0.04	0.03	0.03	0.05	0.15	0.07	0.03	0.04	0.01	0.01
Gd	0.06	0.04	0.03	0.04	0.05	0.04	0.03	0.03	0.01	0.03	0.03	0.06	0.05	0.06
Ti	29.88	42.80	57.13	19.18	47.58	21.50	35.11	8.25	5.91	16.62	52.27	20.05	110.10	97.53
Tb	0.01	0.01	0.01	0.01	0.01	0.01	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.01
Dy	0.08	0.04	0.04	0.05	0.09	0.04	0.02	0.02	0.02	0.04	0.04	0.07	0.06	0.07
Ho	0.02	0.01	0.01	0.01	0.02	0.01	0.01	0.00	0.00	0.01	0.01	0.02	0.01	0.02
Y	0.43	0.21	0.16	0.24	0.41	0.22	0.11	0.09	0.07	0.20	0.19	0.36	0.32	0.35
Er	0.05	0.02	0.02	0.03	0.05	0.02	0.01	0.01	0.01	0.02	0.02	0.03	0.04	0.04
Tm	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01
Yb	0.08	0.03	0.02	0.04	0.05	0.03	0.01	0.02	0.01	0.03	0.02	0.03	0.04	0.04
Lu	0.02	0.00	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.01	0.00	0.01	0.01	0.00

The trace and minor elements concentrations (in ppm) were measured by laser ablation coupled to ICP-MS. In the two last columns, the compositions of the heterogeneous points represented in Figure 1 are shown, the profiles of Figure 2 correspond to the point labeled "01a-2" in this table.

* Microprobe analysis (ppm).

positional range for the dyke core diopside. The results of microprobe and ICP-MS analyses are given in Table 1. Trace and minor element concentrations is very low compared to the Cpx from a mantle or magmatic lithology (Benoit et al., 1996; Tamura and Arai, 2006), generally ranging from 0.01 to 1 $\times$ the chondrite value depending on the element (Fig. 1). Other than a marked positive Eu anomaly, the general shape of the REE distribution pattern of the diopside is close to that of a mantle Cpx, with depletion in LREE relative to HREE. When adding to the pattern Sr, Ti and Y that have a REE-like behaviour in Cpx, it appears that Y behaves like other HREE as is usual for Cpx. Conversely, strong anomalies positive for Sr and negative for Ti are rather unusual for this mineral. Sr values lie between 4 and 5.2 ppm (i.e., 0.65–0.7 $\times$ the chondrite concentration), and considering the variability of the other trace elements, they are remarkably constant in all the sample grains regardless of the size or other textural feature.

Eu and Ti concentrations show much larger varia-

tions than Sr, their content respectively being between 0.02 and 0.15 ppm and 5 and 50 ppm (i.e., 0.2 to 1 $\times$ and 0.01 to 0.1 $\times$ the chondrite value, Fig. 1). Ti, Cr and Al concentrations indicated by the LA-ICP-MS technique may show a substantial difference to those measured by microprobe analysis (Table 1), highlighting that these concentrations are close to the microprobe detection limit and that, in this case, the error bars must be seriously considered. The concentrations are lower than those usually measured in Cpx from the Oman "magmatic" lithologies, especially when taking account of the very high Xmg of these minerals (Table 1). Moreover, the strong Ti negative anomaly observed in the trace element pattern (Fig. 1) shows that its content in diopside cannot be totally explained by the incompatible character of Ti in Cpx and that the material crystallised in a particularly Ti-poor environment. In the same way, the systematic positive anomaly in Eu, which is rather unusual in Cpx, suggests its growth in a Eu-rich system.

In the context of diopsidite formation within a mag-

matic-hydrothermal interaction, these two features can be explained invoking a hydrothermal fluid rich in Eu and Sr and poor in Ti. Hydrothermal seawater that had leached crustal gabbro lithologies rich in plagioclase would contain a relatively large amount of Eu and Sr yet be strongly depleted in Ti. Such a fluid could be a suitable reaction candidate to achieve the diopside chemistry. The Sr concentration was probably buffered by the concentration in the hydrothermal fluid, as the contribution of the mostly serpentinised olivine protolith was not significant.

Diopsides contain Ti-rich Al-Eu-poor layers

The results of laser ablation at several sample points on the three analysed thin sections in most cases show a classical evolution with time as a slight decrease during acquisition. These points are homogeneous and their domain of composition is represented by the shaded area of Figure 1. The measurements were obtained from relatively thick diopside grains ($> 300 \mu\text{m}$) of either the coarse-grained fibrous zones or the cataclastic zones of the core of the dyke. Two points are distinct in exhibiting large variations in intensity during the analysis time that is diagnostic of heterogeneities present within the diopside grain (Fig. 2). They were measured in the sample taken from the edge of the dyke that show an unusual texture represented by $\sim 200 \mu\text{m}$ width diopside needles crystallised near an olivine ghost. The large ($> 300 \mu\text{m}$) prismatic diopsides of this sample, also plotted in the shaded area of Figure 1, are not heterogeneous and show similar profiles to those obtained from the fibrous and cataclastic samples.

At the two heterogeneous points of analysis, Si, Ca and Sr show slightly variable profiles but they exhibit the regular decrease usually observed during LA-ICP-MS acquisition (Fig. 2a). The intensities recorded for the other elements are highly variable, commonly showing a central peak diagnostic of a layer relatively enriched in Ti, Cr, Co, Y, and other elements in the centre of the analysed section (Fig. 2b). The data obtained for Pb, Gd (Fig. 2c) and Tm show small-scale variations that we can attribute to noise, but the average level of intensity follows the same evolution as the previously noted elements. Al, Cr and Co (Fig. 2b) show, close below the surface of the thin section and at the end of the analysis, a strong concentration on either side of the Ti-enriched layer. These intensity peaks are not observed for Ti and are more tenuous for other elements such as Y, Tb, Tm, Pb, and others. They appear to be characteristic of the chemical evolution of the analysed diopside and highlight layers of variable trace element concentration within this mineral.

In the case of the analyses corresponding to the two

Figure 2. Variation in the detection level for several elements during laser ablation of sample 05SDM2b1. The intensity (in counts per second) was normalised to the average concentration (in parts per million), see the text for details of the measurement and calculation methods. (a): black line, $\frac{\text{Si}}{10^4}$; grey line, $\frac{\text{Ca}}{10^4}$; dotted line, $\text{Sr} \times 10$. (b): black line, Cr; grey line, Ti; dotted line, Al; black dashed line, $\text{Co} \times 10$; grey dashed line, $\text{Y} \times 100$. (c): black line, Gd; grey line, Tb; dotted line, Er; black dashed line, Tm; grey dashed line, Pb.

profiles seen in Figure 1, the Ti, Al and Cr contents are all below the detection limit of the microprobe ($< 0.01 \text{ wt\%}$), and are respectively 100–110 ppm, 70–160 ppm, and 100–120 ppm (Table 1) when measured using the LA-ICP-MS technique. Ti concentration is also higher than that usually observed in the same sample ($\text{Ti} = 100\text{--}110 \text{ ppm}$ as against $5\text{--}50 \text{ ppm}$ at the other analysis

points). On the other hand, Al, Eu and Sr contents are lower than those of the other analysed points in the same sample with Al = 70–160 ppm, Eu < 0.01 ppm and Sr $\approx$ 1.15 ppm (respectively 500–2000 ppm, 0.03–0.15 ppm and 4–5.2 ppm for the other analysed points). This deficit or enrichment are illustrated in Figure 1: the Ti negative and Eu positive anomalies are not observed in the spider diagrams obtained from the two heterogeneous points, the Sr anomaly is less obvious than that seen elsewhere, and the HREE content is amongst the highest whereas the LREE content (Pr, La, Ce) is lower than that of the other points. The Eu anomaly is slightly negative for these two points, contrasting with the positive Eu anomaly generally observed in the homogeneous diopside, and no Ti anomaly is observed.

The heterogeneous points show the same patterns as that of Cpx contained in the troctolite or olivine gabbro dykes of the mantle section (Benoit et al., 1996), but with a depletion factor of 10 for all minor and trace elements. These features suggest some affinity with former magmatic Cpx rather than with residual ones that show a stronger fractionation for LREE–HREE than that observed, without a Eu negative anomaly (Takazawa et al., 2003; Tamura and Arai, 2006). Nevertheless, the global depletion in all minor and trace elements, especially Al, whose concentration is even lower than in the hydrothermal diopside (Table 1), cannot be explained in the frame of that hypothesis.

Given the few microns thinness of these layers, the LA-ICP-MS technique is not accurate enough in terms of spatial resolution to determine unambiguously the trace element patterns of each layer. Taking into account the large variation in the intensity measurement during the laser ablation, the concentration calculation treatment by averaging cannot give a layer representative value or the interlayer element concentration (see Figure 2 for the interval of time used in the calculation of the average concentration). Accordingly, the spider diagrams in Figure 1 are likely to represent mixtures of depleted hydrothermal diopside and some other mineral, and the microprobe analysis can only give the composition of the layer exposed at the surface of the thin section (nevertheless, it is worth noting that, taking account of the relative stability of the signal for Si, the use of this element as an internal standard may still be valid).

Qualitatively, the trace element compositions of the layers are characterised by a higher concentration in Ti and a lower one in Al, Eu, Sr, and LREE (La, Ce, Pr) compared to that of the homogeneous diopside. In the context of strong influence by a hydrothermal fluid on Sr and Eu concentration in the diopside and taking into account that such fluids may be enriched in LREE as

compared to HREE (Douville et al., 2002), we suggest that these layers are relics of a mineral that was not totally altered by the hydrothermal fluid when the “diopsiditisation” process occurred. The exact nature of these layers cannot be determined as they are not visible under the microscope, but their presence within needles of diopside at the boundary of olivine ghosts suggests that these heterogeneous needles formed at a prograding metamorphic/hydrothermal front. This association between diopside crystals and relics of former minerals probably had a very restricted stability domain. A few microns distant from the olivine ghost the diopside becomes prismatic and shows no heterogeneity during laser ablation.

In light of the above, former amphiboles, partially altered Cpx, antigorite, fluid inclusions or Ti-rich accessory minerals (titanite, see Python et al., 2007) could have been good candidates for relic inclusions within the diopside. However, the marked depletion in Al cannot be explained by the two first-mentioned minerals, whereas antigorite or fluid inclusions would contradict the relatively constant Ca and Si and the high Ti concentration. Furthermore, with the inclusion of pure titanite, the concentration of Ti and other trace elements should be much higher than that actually measured. It is possible that the micro layers are a mixture of two or more of these features.

Whatever their origin, the micro layers show a LREE content lower than the homogeneous diopside and a HREE concentration that corresponds to the higher level of the latter. These observations, as well as the Ti, Al, Cr, Sr and Eu content of heterogeneous diopside when compared to that of the other, suggest a Eu, Sr and LREE enrichment and Ti depletion during the hydrothermal metamorphism. A HREE depletion effect may also have taken place, as the average HREE content of the homogeneous diopside is lower than that of the heterogeneous crystals (Fig. 1).

CONCLUSION

Trace element spider diagrams of the Cpx contained in diopside show large heterogeneities from one sample to another and from one mineral to another within a single sample. The study of profiles showing the evolution of intensity measured for each element during analysis shows that small-scale heterogeneities are frequent. We conclude that the heterogeneous diopside is some kind of sandwich structure in which a protolith (residual or cumulative) and hydrothermal minerals are interlayered. When compared to homogeneous diopside, the heterogeneous variety is more informative as it bears witness to a transient state of change from a serpentinised peridotite or a

magmatic lithology into diopsidite. Further studies should allow us to decide between these hypotheses and to more precisely determine the conditions for diopside formation. Although many uncertainties remain, whatever the heterogeneity of the system in REE, Ti, Al, and associated elements, the role of a hydrothermal fluid in buffering elements like Ca and Sr in the composition of diopside seems well established by our preliminary data.

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Appendix. LA-ICP-MS instrument and operational settings

(1) Instrumentation	
ICP-MS	Yokogawa Analytical Systems Agilent 7500s
Laser	MicroLas GeoLas Q-Plus
(2) ICP source	
Incident power	1.2 kW forward (< 1 W reflection)
Argon gas flow rate	Plasma: 15.0 L/min
	Aux: 1.0 L/min Carrier: 1.12 L/min
Mixing gas	He (0.8 L/min)
(3) Vacuum interference	
Expansion	2.5×10^{-2} Pa
Interface	2.5×10^{-2} Pa
Analyzer	2.3×10^{-4} Pa
(5) Mass spectrometer	
Scanning mode	Peak jump mode (point per peak)
Dwell time	10 msec
	5 msec ^{27}Al
Settled time	10 msec
Ion detection	Pulse counting
Detector type	ETP multiplier
Multiplier HT	12 kV
(6) Laser	
Model	Compex102
Wavelength	193 nm
Crater size	132 μm
Pulse width	20 nsec@FWHM
Pulse energy	6 J/cm ²
Repititation rate	6 Hz
Sensitivity Y	23000 CPS/ppm with 6Hz