MAJOR AND MINOR ELEMENT SITE OCCUPANCIES IN HEATED NATURAL FORSTERITE

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Abstract. Using a new analytical transmission electron microscope technique
known as Channelsing Enhanced X-ray Emission (CHEXE) spectroscopy, the
M-site occupancies of Fe, Ni, Mn, and Ca have been determined in a natural
forsterite olivine (FeO,Mg2O) heated at different temperatures. The sample
was taken as a single olivine grain from a spinel peridotite inclusion in an alkali basalt
and contains 0.36 wt% NiO, 0.07 wt% MnO, and 0.09 wt% CaO. In the non-
heat-treated sample, 49 ± 4% of the Fe, 17 ± 3% of the Ni, and 1 ± 5% of the
Mn is the sample occupy the M1 site. In the present study of samples
quenched from different temperatures, the fraction of the Ni present at M1 is 87
± 5% (6 days at 350°C), 83 ± 5% (4 days at 600°C), 83 ± 5% (45 h at 1000°C)
and 80 ± 5% (24 h at 1500°C). We thus observe a lesser tendency for Ni to
order in the heattreated samples. For Mn, typically
85% of the atoms occupy M1 in the heat treated samples. No significant
deviation from complete ordering into M2 was observed for Ca. The Fe atoms
are completely disordered with 50 ± 1% at each M-site, except for a weak
dependence on a temperature of 350°C with 47 ± 1% at M1. The study indicates that exchange
between M-sites begins as low as 300°C. This implies that Ni and
Mn distributions in natural olivines may be a useful indicator of cooling rate in
rapidly cooled rocks.

Introduction
The yield of electron-induced characteristic X-ray emission from a thin crystal
depends on the crystallographic direction of the incident electron beam [Collins
et al., 1973]. For brevity we shall denote this phenomenon CHEXE
(Channelsing Enhanced X-ray Emission). A new crystallographic technique
based on this phenomenon has recently proved capable of locating cation
concentrations as low as 0.1 ppm [Taffé, 1982]. Using this technique, Taffé and
Spence [1982] were able to determine with a relative high degree of precision,
the distribution between M1 and M2 sites for Fe and low concentrations of Ni
(0.2 ppm) and Mn (0.05 ppm) in a natural olivine (Fo95) from the San Carlos
Indian Reservation, Arizona. The ability to locate such low concentrations,
which is beyond the capability of any established technique, suggests that site
distributions of minor elements may be used as petrogenetic indicators. In order
to explore the possibility of using CHEXE for this purpose, we have studied the
cation distributions in heat-treated olivines from this same locality.

Experimental Procedure
A large single crystal of olivine (Fo95) approximately 10 x 5 x 5 mm in size was
selected from a spinel peridotite nodules in an alkali basalt from Peridot
Mesa on the San Carlos Apache Reservation, Gila County, Arizona. The crystal
was crushed and milled to 0.8 mm. The less than 0.2 mm fraction was
removed and the remaining grains washed in acetone to remove residual fines. A
few impurity grains from the surface of the original grain were removed by hand
picking, yielding a pure sample of approximately 300 mg. Four splits containing
approximately 50 mg each were wrapped in Pd foil and placed in
silica glass ampules of a volume estimated to 2 cm3 each. The ampules were
evacuated to a pressure of 10⁻¹ mm Hg and sealed.

The four sample splits were heated at different temperatures: 300 ± 5°C
for 6 days, 600 ± 5°C for 48 h, 950 ± 5°C for 45 h, and 1000 ± 10°C for 34 h.
Each sample was quenched in air so that it cooled to less than 100°C within
30 seconds. Temperature was monitored by a calibrated chromel-alumel
thermocouple attached to the outside of the ampule. Several grains from the
unheated portion and each heated sample were analysed using a Cameca
Camex electron microprobe. Results of these analyses are reported in Table 1
and indicate no evidence of Fe or Ni loss during heating.

The samples were prepared for electron microscopy by crushing to approxi-
mately 1 mm grain size. The small grains were dispersed on a copper grid coated
with a holey carbon film. For the CHEXE studies, a Philips 400T electron
microscope equipped with a Trace-Northern energy dispersive X-ray analyser
was used. X-ray spectra were taken while illuminating thin, wedge-shaped
crystal areas of approximate diameter 500A and thickness sp to about 1000A
with 100 keV electrons.

As in the previous work on the non-heated treated olivine, the (010)-systematic
row (002) plane was examined, i.e. the crystal was so oriented that Bragg
reflection only from the (010) planes occurs. Two X-ray spectra were taken from
each sample, one with the incident beam nearly parallel to the (010) planes (θ < 10°)
and one by tilting the crystal about one half degree such that the angle between
the (010) planes and the incident beam was slightly larger than the Bragg angle
for the first-order reflection along the (010) direction, i.e. the 20° reflection (θ > 10°).
Figure 1 shows spectra from the same area of the sample heat-treated at
600°C for the two directions of incidence. Using the intensity of the Kα line of
Mg as a reference, we notice that the intensity of the Kα line of Si decreases on
changing from θ < 10° (Fig. la) to θ > 10° (Fig. 1b). The same is true for Fe and
Ca. The Fe Kα line does not change whereas the Ni Kα  line increases for θ > 10°.
The ratio, R(Si/X) is defined by:

\[ R(Si/X) = \frac{I(Si)}{I(X)} = \frac{I(Fo)}{I(Mg)} \]

where N₀ is the integrated number of Kα counts for Si and Nₓ for element X.

CHEXE spectrometry

The trends apparent in Table 2 for all measurements are similar: R(Si/Mg) >
R(Si/Ca) > θ < 10°, R(Si/Fe) > R(Si/Ni). For an understanding of these
rations, we have to consider the atomic arrangement along the (010) direction,
where there are two types of alternating atomic planes as shown in Figure 2. One
type of plane, the most densely populated one, contains the Si atoms, half of the
oxygen atoms, and the cations in the M2 sites, whereas the alternate planes
contain the other half of the oxygen atoms and the cations of the M1 sites. It has
been established previously that the electron wavefield averaged over thickness
has intensity maxima at the densely populated planes for θ < 10°, and at the alternate
planes for θ > 10°. In Figure 2, because only electrons travelling close to the atomic planes are capable of producing Kα X-rays, the
X-ray emission is proportional to the intensity of the electron wavefield at
the atomic planes. The observed change of the intensity of the Kα line of Si is
consistent with this simple consideration.

As in the case of olivine, the intensity of the wavefield at each type of plane
can be inferred from the observations. The Kα emission from Si gives us
information about the atomic arrangement and the wavefield density.

Ni Ordering

From the data in Table 2, we conclude that Ni is ordered in the olivine
experiments. The Kα emission from Ni is observed in the experiments with the
observations of Rajamani et al. [1973] and Bish [1981] for natural and synthetic

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Paper number 2L1255.
0094-8276/82/0202L-125$3.00
TABLE 1. Microprobe Analyses of Heat-treated San Carlos Olivines

<table>
<thead>
<tr>
<th>Number of</th>
<th>300°C</th>
<th>600°C</th>
<th>900°C</th>
<th>1050°C</th>
<th>Average</th>
</tr>
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<tr>
<td>analyses</td>
<td>6</td>
<td>5</td>
<td>4</td>
<td>4</td>
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<td>.00</td>
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<td>.02</td>
<td>.02</td>
<td>.02</td>
<td>.02</td>
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<td>.06</td>
<td>.00</td>
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<td>.32</td>
<td>.38</td>
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<td>.07</td>
<td>.07</td>
<td>.06</td>
<td>.06</td>
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<td>MgO</td>
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<td>49.48</td>
<td>49.96</td>
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<td>CaO</td>
<td>.11</td>
<td>.09</td>
<td>.09</td>
<td>.09</td>
<td>.09</td>
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<tr>
<td>Total</td>
<td>100.24</td>
<td>99.54</td>
<td>100.17</td>
<td>99.79</td>
<td>99.43</td>
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</table>

Atoms Per 8,000 Oxygenes

<table>
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<tr>
<th>Si</th>
<th>1.994</th>
<th>1.999</th>
<th>2.000</th>
<th>1.996</th>
<th>1.988</th>
<th>1.994 ± .006</th>
</tr>
</thead>
<tbody>
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<td>Al</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
</tr>
<tr>
<td>Ti</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
</tr>
<tr>
<td>Cr</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
<td>.000</td>
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<td>.000</td>
</tr>
<tr>
<td>Ni</td>
<td>.011</td>
<td>.011</td>
<td>.011</td>
<td>.011</td>
<td>.014</td>
<td>.013 ± .002</td>
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<td>.351</td>
<td>.354</td>
<td>.352</td>
<td>.352</td>
<td>.352</td>
<td>.353 ± .002</td>
</tr>
<tr>
<td>Mn</td>
<td>.002</td>
<td>.002</td>
<td>.002</td>
<td>.002</td>
<td>.002</td>
<td>.002 ± .005</td>
</tr>
<tr>
<td>Ca</td>
<td>.004</td>
<td>.004</td>
<td>.004</td>
<td>.004</td>
<td>.004</td>
<td>.004 ± .005</td>
</tr>
</tbody>
</table>

liebenbergites, however a significantly different interpretation may be possible based on the current data. Beek [1981] observed essentially complete ordering of Ni and M1 in a natural liebenbergite (Ni₉₋₁₇, Co₃₋₆, Fe₂₋₄, Mg₆₋₈, SiO₃) and partial order of Ni, Kp = [Mg(M2+)(Ni(M1))] / [Ni(M2+)+Mg(M1)] = 9.2, in a sample synthesized at 500°C. Rajamani et al. [1975] observed partial order of Ni on M1 (Kp = 9.9) in a sample synthesized at 1280°C and cooled to 1000°C in about 56 hours. Beek [1981] inferred that his natural sample showed Ni order consistent with equilibration at 730°C, and that the sample of Rajamani et al. [1975] showed Ni order representative of 1280°C, so that both would be consistent with a ΔG° of ~6.5 Kcal/mole as interpreted by Rajamani et al. Beek further suggested that his synthetic sample crystallized in a metastably

![Graph](image)

**Fig. 1. X-ray spectra with a θ < θ₀ and b θ > θ₀.**

TABLE 2. The Fraction C_X of Specie X of M1 Sites as Determined From the Different Measurements

<table>
<thead>
<tr>
<th>Temperature</th>
<th>R(B/Mg)</th>
<th>R(B/Fe)</th>
<th>R(B/Mn)</th>
<th>R(B/Ni)</th>
<th>R(B/Ca)</th>
<th>C_Mn(%)</th>
<th>C_Ni(%)</th>
<th>C_Mg(%)</th>
<th>C_Fe(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300°C</td>
<td>1.54</td>
<td>1.51</td>
<td>1.22</td>
<td>2.07</td>
<td>0.83</td>
<td>47.0</td>
<td>41.4</td>
<td>81.2</td>
<td>-23.2</td>
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<tr>
<td>6 days</td>
<td>1.66</td>
<td>1.585</td>
<td>0.945</td>
<td>2.60</td>
<td>1.07</td>
<td>45.5</td>
<td>-7.7</td>
<td>95.9</td>
<td>-7.7</td>
</tr>
<tr>
<td>1.88</td>
<td>1.91</td>
<td>1.30</td>
<td>1.40</td>
<td>1.19</td>
<td>50.7</td>
<td>21.1</td>
<td>92.9</td>
<td>14.4</td>
<td></td>
</tr>
<tr>
<td>1.665</td>
<td>1.575</td>
<td>1.11</td>
<td>1.47</td>
<td>1.16</td>
<td>49.5</td>
<td>10.8</td>
<td>88.6</td>
<td>14.4</td>
<td></td>
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<tr>
<td>Average</td>
<td>1.59</td>
<td>1.63</td>
<td>1.27</td>
<td>2.09</td>
<td>0.94</td>
<td>51.0</td>
<td>16.8</td>
<td>82.1</td>
<td>-7.5</td>
</tr>
<tr>
<td>604°C</td>
<td>1.715</td>
<td>1.715</td>
<td>1.11</td>
<td>2.72</td>
<td>1.21</td>
<td>50.9</td>
<td>14.6</td>
<td>81.7</td>
<td>26.5</td>
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<tr>
<td>48 hrs</td>
<td>1.60</td>
<td>1.58</td>
<td>1.22</td>
<td>2.00</td>
<td>0.99</td>
<td>48.8</td>
<td>22.3</td>
<td>73.1</td>
<td>-1.6</td>
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<td>1.66</td>
<td>1.67</td>
<td>1.23</td>
<td>1.62</td>
<td>0.85</td>
<td>50.3</td>
<td>20.8</td>
<td>94.1</td>
<td>-15.1</td>
<td></td>
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<tr>
<td>Average</td>
<td>1.595</td>
<td>1.50</td>
<td>1.87</td>
<td>1.42</td>
<td>1.28</td>
<td>43.0</td>
<td>7.0</td>
<td>90.0</td>
<td>28.0</td>
</tr>
<tr>
<td>915°C</td>
<td>1.56</td>
<td>1.60</td>
<td>1.89</td>
<td>2.21</td>
<td>0.90</td>
<td>53.5</td>
<td>11.1</td>
<td>76.9</td>
<td>-15.0</td>
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<tr>
<td>45 hrs</td>
<td>1.58</td>
<td>1.75</td>
<td>1.96</td>
<td>1.88</td>
<td>1.46</td>
<td>50.5</td>
<td>5.8</td>
<td>82.9</td>
<td>26.5</td>
</tr>
<tr>
<td>1.58</td>
<td>1.595</td>
<td>1.58</td>
<td>1.24</td>
<td>1.13</td>
<td>51.0</td>
<td>75.0</td>
<td>83.1</td>
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<tr>
<td>1.71</td>
<td>1.72</td>
<td>1.32</td>
<td>2.50</td>
<td>0.87</td>
<td>50.5</td>
<td>26.8</td>
<td>82.1</td>
<td>-13.3</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>1.595</td>
<td>1.50</td>
<td>1.87</td>
<td>1.42</td>
<td>1.28</td>
<td>43.0</td>
<td>7.0</td>
<td>90.0</td>
<td>28.0</td>
</tr>
<tr>
<td>49.7(3.9)</td>
<td>25.2(29)</td>
<td>83.5(8)</td>
<td>8.2(21)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| 1.50        | 1.52    | 1.23    | 1.76    | 1.18    | 52.0    | 25.7    | 76.9    | 20.0    |
| 1.050°C     | 1.57    | 1.575   | 0.92    | 2.27    | 0.87    | 50.5    | -10.9   | 91.9    | -15.1   |
| 25 hrs      | 1.55    | 1.565   | 1.14    | 2.00    | 1.02    | 51.0    | 15.7    | 78.2    | 2.2     |
| 1.66        | 1.655   | 1.075   | 1.38    | 1.07    | 49.5    | 7.2     | 82.7    | -7.2    |
| Average     | 50.6(1.0) | 9.1(15) | 88(8)   | 4.1(14) |          |          |          |          |

1Figure in parenthesis is calculated standard deviation.
disordered state and that Ni ordering proceeded slowly so that the observed Ni
order was not representative of the 500°C synthesis temperature.

In the present study, we observe significant disorder to have taken place at
300°C in as little as 6 days. This implies that cation exchange in olivines may
occur at rather low temperatures. If this is possible, the current observations
and those of Bish [1981] and Rajamanickam et al. [1975] may be readily reconciled. The
natural sample of Bish and the unheated sample in our study both show
essentially complete order of Ni and would represent equilibrium distributions
characteristic of very low temperatures (100°C or below). The sample
synthesized by Bish at 500°C may then represent an equilibrium distribution
characteristic of that temperature (K_un = 9.2, ΔG_un = −3.5 Kcal/mole), and that
of Rajamanickam et al. may have cooled slowly enough to show a cation distribution
characteristic of some temperature lower than 1280°C (say 550°C if ΔG_un = −3.5 Kcal/mole).
The distributions of Ni observed in the current study may then
represent more or less rough approximations to equilibrium at each temperature,
with K_un varying from 31 for the unheated sample to 4.0 for the sample
quenched from 1050°C. These exchange energies would then be in good
agreement with those obtained by Bish [1981] assuming that his synthetic
sample represents equilibrium at 500°C. Also, this interpretation would be
consistent with the high degree of order observed both in the present unheated
sample which occurred as an inclusion in the base of a 3 mm thick basalt flow and
the natural sample of Bish.

Fe Ordering

There have been numerous investigations of Fe ordering in Fe-Mg olivines
over the past 15 years. These have been reviewed recently by Brown [1980]. In
general, there is very little tendency for Fe to order on the M-sites of these
olivines and little agreement on the minor trends which have been observed. The
only effect apparent in the current investigation is a very slight preference of Fe
for M2 in the sample quenched from 300°C. This would be consistent with the
observation of Shinno et al. [1974] and Chase et al. [1976] who report a slight
preference of Fe for M2 at low temperatures and for M1 at higher temperature.
Certainly ordering effects observed in our experiments are much smaller than
those observed by Shinno et al. [1974] who report K_un(Fe) as high as 3.2 for a
sample quenched from 1150°C. If our observation of a slight order of Fe on M2
at 300°C is indeed significant, it would be further evidence of cation exchange at
relatively low temperatures in olivines. We believe that additional experiments
using CHESE spectrosopy on samples heated under carefully controlled
conditions and rapidly quenched hold promise of future clarification of these
complex effects.

Ms Ordering

The observed ordering of Mn on M2 of this olivine is consistent with reported
x-ray structure refinements of tephroite [Chase and Weidner, 1974; Francis and
Ribbe, 1980]. The very small amount of Mn present in the sample (0.07 wt% 
MnO), however, caused substantial uncertainty in the measurement of its
distribution. However, it does appear that the heated samples show significantly
greater disorder than the unheated samples, thus supporting our earlier
convention that significant cation exchange has taken place at temperatures as
low as 300°C. Natural Fe-Mg olivines typically contain substantially more Mn
than does the specimen from San Carlos studied here. The results of this study
indicate that substantial disorder of Mn may take place at relatively low
temperatures, and so determination of Mn distribution between M1 and M2 of
olivine by CHESE spectrosopy may become an important petrogenetic
indicator of temperature or cooling rate.

**TABLE 3. Inferred Cation Occupancies of M1 and M2 at Various Temperatures. Values are expressed as Cations per 4.000 Oxygen.**

<table>
<thead>
<tr>
<th></th>
<th>Unheated</th>
<th>300°C</th>
<th>600°C</th>
<th>900°C</th>
<th>1050°C</th>
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<td><strong>M1 Site</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Ni</td>
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<td>0.057</td>
<td>0.054</td>
<td>0.054</td>
<td>0.052</td>
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<td>0.083</td>
<td>0.088</td>
<td>0.088</td>
<td>0.089</td>
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<tr>
<td>Ms</td>
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<td>0.002</td>
<td>0.002</td>
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<td>Mg</td>
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<td>0.911</td>
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<td>0.906</td>
<td>0.905</td>
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<tr>
<td>Ca</td>
<td>&lt;0.0005</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
</tr>
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<td><strong>M2 Site</strong></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ni</td>
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<td>0.008</td>
<td>0.011</td>
<td>0.011</td>
<td>0.013</td>
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<td>Fe</td>
<td>0.009</td>
<td>0.094</td>
<td>0.089</td>
<td>0.089</td>
<td>0.088</td>
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<tr>
<td>Ms</td>
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<td>0.008</td>
<td>0.008</td>
<td>0.008</td>
<td>0.009</td>
</tr>
<tr>
<td>Mg</td>
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<td>0.902</td>
<td>0.907</td>
<td>0.907</td>
<td>0.908</td>
</tr>
<tr>
<td>Ca</td>
<td>0.002</td>
<td>0.002</td>
<td>0.002</td>
<td>0.002</td>
<td>0.002</td>
</tr>
</tbody>
</table>

K_un(Ni) = 31.5

**Fig. 3.** The atomic fraction of Ni, Fe, Mn and Ca present in the sample that is
located at M1 for the different heat treatments. The indicated precisions are
based on counting statistics for the K_un lines and the background. The actual
observed variations in K_un (Table 2) are somewhat larger, suggesting an
inhomogeneous ordering of Mn in the heat-treated samples.

**Fig. 2.** Schematic diagram of the atomic arrangement along the (010) direction
in olivine. Large open circle = oxygen; medium open circle = M2; medium solid
circle = M1; and small solid circle = Si. The curves indicate typical intensity
modulations over the repetition unit for the two directions of incidence: solid
curve = θ < θ_R; broken curve = θ > θ_R.
Ca Ordering

The observation of nearly complete ordering of Ca on M2 is consistent with x-ray structure refinements of monoclinic and other enstatite olivines [Brown, 1980]. We observe no significant deviation from the fully ordered state at any temperature, and this is consistent with a very strong site preference for Ca for the larger M2 octahedron. However, at concentrations of 0.05 at% Ca in the sample, relative errors are large, and some disorder may have taken place within the experimental error.

Conclusions

It has been known for some years that cooling history has little influence on the ordering of Fe in magnesium olivines. Thus, there is a need for other parameters as indicators of temperature or cooling rate in olivines. We have demonstrated here that CHEX spectroscopy is capable of determining the site distribution of cations at concentrations of 0.2 at% or less, and that for Ni the precision is sufficient to observe a systematic trend dependent on heat treatment. At concentrations of 1 at% or greater, the precision is likely to be improved to 1 to 2 relative percent in the site occupancy. As compared to Mossbauer spectroscopy, CHEX is more versatile because it can be used to determine the distribution of any element with Z>11 as long as the K_{eq} line of the minor constituent does not overlap the K_{eq} line of an element of high concentration.

In addition to demonstrating the feasibility of using the minor element distribution between the two octahedral sites in natural magnesium olivine as an indicator of thermal history, this study has contributed to knowledge of cation exchange in olivines. The study indicates that significant cation exchange between M-sites may take place at temperatures of 300°C or less. The present results show a lesser tendency for Ni to order than postulated by previous workers for Ni-rich olivines. Additional studies by CHEX spectroscopy or by conventional x-ray techniques are desirable to confirm this observation. Also, additional studies by CHEX spectroscopy will be required to quantify the distributions and kinetics of the exchange reactions. We believe that the current results show that this technique offers considerable promise for the development of a cooling-rate geothermometer applicable to a wide range of rock-types.

Acknowledgments. Financial support from NSF Grant DMR 800108 and access to the NSF Facility for High Resolution Electron Microscopy at Arizona State University are gratefully acknowledged. Mr. Mark Beumus and Dr. John Holloway of Arizona State University aided with sample preparation and heating. Electron microprobe analysis and manuscript preparation were supported, in part, by Los Alamos National Laboratory.

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(Received July 2, 1982; accepted August 5, 1982.)