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THE OLIVINE-SPINEL INVERSION IN FAYALITE¹A. E. RINGWOOD,² *Dunbar Laboratory, Harvard University,
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The author has previously described the synthesis under high pressure of a spinel polymorph of fayalite (Ringwood, 1958*a, b*). The early work on the transition was essentially of an exploratory nature. It revealed that fayalite was stable at pressures below 30,000 bars in the temperature range 400–600° C., while the spinel was stable above 40,000 bars in the same temperature range. Within the pressure range 30,000–40,000 bars, conflicting results were obtained. These may have been caused by metastability or by insufficient experimental accuracy. In an attempt to resolve the conflict, further runs have been carried out. The position of the transition has been approximately located at 600° C. and 500° C., but the accuracy is not sufficient to provide a reliable slope (dp/dt). It is clear that many additional runs would be required in order to find the slope. Since the author has now ceased working on this problem, it seemed advisable to record the results of runs carried out (Table 1).

Charges were prepared by weighing out ferric oxide, metallic iron powder, and Mallinckrodt silicic acid in the proportions required to give the fayalite composition plus a 10–20% excess of iron. These components were then intimately mixed together by grinding under acetone. The purpose of the excess iron was to maintain oxidized iron in the divalent state. The charges were subjected to the desired temperature and pressure in a "squeezer" apparatus (Griggs and Kennedy, 1956) which was frequently calibrated for temperature and checked for piston distortion.

After completion of a run, temperature and pressure were swiftly lowered. The transition is sluggish and no difficulty was experienced in quenching the high pressure form to atmospheric pressure. Products were examined by x -ray and optical methods. Identification of the spinel form of fayalite was based upon its optical properties and powder pattern, which have been previously described (Ringwood, 1958*b*).

Appearance of the charges under the microscope varied from opaque, indefinite aggregates to completely crystallized, colorless to green masses of fayalite or spinel crystals. The powder patterns showed that the opacity and darkness of samples was due principally to the presence

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TABLE 1. SUMMARY OF EXPERIMENTAL RESULTS

| No. | Temperature ° C. | Pressure (bars) | Time of run (hours) | Phases present (a) (b) | Notes |
|-----|---------------------|--------------------|---------------------------|------------------------|-------|
| F3 | 500 | 20,000 | 7 | magnetite (fayalite?) | |
| F6 | 500 | 20,000 | 24 | fayalite, magnetite | (c) |
| F11 | 500 | 20,000 | 14 | fayalite, (magnetite) | |
| F14 | 500 | 25,000 | | fayalite, (magnetite) | |
| F2 | 500 | 30,000 | 16 | magnetite, spinel | (e) |
| F5 | 500 | 30,000 | 24 | spinel, magnetite | (c) |
| F15 | 500 | 30,000 | | fayalite, magnetite | |
| F16 | 500 | 35,000 | 12 | fayalite | |
| F1 | 500 | 40,000 | 8 | spinel, ((magnetite)) | |
| F4 | 500 | 40,000 | 24 | spinel, (magnetite) | |
| F13 | 600 | 25,000 | 3 | fayalite, magnetite | |
| F8 | 600 | 30,000 | 3 | fayalite | |
| F9 | 600 | 35,000 | 3 | fayalite | |
| F10 | 600 | 40,000 | 3 | spinel, ((fayalite)) | |
| F12 | 600 | 45,000 | 3 | spinel, (magnetite) | |
| F7 | 600 | 45,000 | 4 | spinel | (c) |
| F17 | 600 | 70,000 | 1 | spinel, ((fayalite)) | (d) |

- (a) In all runs, some unreacted iron remained in the product. This has not been recorded in the above table.
- (b) Phases are recorded in order of abundance. Single brackets denote rarity, double brackets denote extreme rarity.
- (c) In these runs, thermocouple behaved irregularly, preceding a corrosion break. Temperature could have been as much as 20° higher than that recorded.
- (d) Starting charge was moistened Rockport fayalite.
- (e) "Spinel" refers to the spinel polymorph of fayalite.

of magnetite. It was noticed that little or no magnetite was present when the charges had been prepared shortly before using. However if charges had been mixed more than one day, before being run, appreciable quantities of magnetite were likely to be found in the product. This may be due to oxidation of metallic iron in the charge by water from the silicic acid.

From Table 1 it is seen that the position of the transition at 600° C. is close to 38,000 bars. At 500° C. there appears to be a region of indifference where equilibrium is not always attained, with the transition probably between 30,000 and 35,000 bars.

A sample of moistened Rockport fayalite was run at 600° C. and 70,000 bars for one hour. Almost complete conversion to spinel was obtained.

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CORRELATION BETWEEN X-RAY EMISSION AND FLAME PHOTOMETER
DETERMINATION OF THE K₂O CONTENT OF POTASH FELDSPARS

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Using *x*-ray emission spectroscopy in the quantitative determination of chemical composition is still in the developmental stages. As a test of its accuracy the K₂O content of potash feldspars from granitic rocks of the Inyo batholith was determined both by flame photometry and by *x*-ray emission techniques. Comparison of the results shows that the simpler and non-destructive *x*-ray emission technique gives satisfactory estimates of K₂O values.

To prepare each sample, 0.3–0.4 gram of the feldspar separate was powdered in a Wig-1-bug by shaking 0.1- to 0.2-mm. grains for five minutes in a steel capsule with steel balls. These powders were tightly packed into the wells (3/4×1/2×1/16 inch) of aluminum slides that fit the sample holder of the *x*-ray spectrophotometer.

A General Electric XRD-5 *x*-ray spectrophotometer with a lithium fluoride analyzing crystal was used. The tungsten tube was operated at 50 kilovolts and 50 milliamperes. Helium was used in the optical path, and a gas-flow proportional counter was the detector. The analytical line used was K α . Background intensity variations of the different samples were insignificant in relation to the peak heights. Intensities were measured during a constant counting time of ten seconds. With a constant helium flow of eight cubic feet per hour the flushing time required to clear the system of air after the introduction of each sample was 120 seconds.

The standards used to establish a working curve were the chemically analyzed G-1 granite (Fairbairn *et al.*, 1951) and a potash feldspar megacryst of the Aiken facies of the McAfee adamellite (Emerson, 1959). The use of standards in a similar matrix, together with a narrow range in composition of samples, avoids significant deviations from the proportionality between analytical-line intensity and weight-fraction of the element being determined (Liebhafsky and Winslow, 1958).