

# THE SYNTHESIS AND CHARACTERIZATION OF Mg-Fe-Mn GARNETS

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## INTRODUCTION

Garnet is a common mineral in metamorphic rocks that can be used with other minerals both as a geothermometer and as a geobarometer. The general structural formula for garnet is  $X_3Y_2Z_3O_{12}$  where X is in eight-fold coordination, Y is in six-fold coordination and Z is in four-fold coordination. Winchell (1933) divided the garnets into two groups: ugrandites, where Ca is in the X site, and pyrospites, where Ca is not in the X site. The ugrandites include: uvarovite ( $Ca_3Cr_2Si_3O_{12}$ ), grossular ( $Ca_3Al_2Si_3O_{12}$ ), and andradite ( $Ca_3Fe_2Si_3O_{12}$ ). The pyrospites include: pyrope ( $Mg_3Al_2Si_3O_{12}$ ), almandine ( $Fe_3Al_2Si_3O_{12}$ ), and spessartine ( $Mn_3Al_2Si_3O_{12}$ ).

Most garnets have compositions that lie near but between the two series (end member garnets rarely occur in nature). Various methods have been used to determine the compositions of natural garnets as a function of their physical properties. Historically certain properties such as unit cell size ( $a_0$ ), specific gravity (G), and refractive index (n) are most often used because they are comparatively easy to measure. Ford (1915) was the first to try to relate the refractive index (n), specific gravity (G), and lattice constant (a) to composition. Since Ford (1915) many other diagrams have been compiled, Stockwell (1927), Fleisher (1937), Kennedy (1947), etc. These earlier predictions were made by measuring the properties of natural garnets and then extrapolating to the pure end-members. Skinner (1956) used synthetic end-member garnets to determine the lattice constant (a), molar volume (V), density (r), and refractive index (n). He found that his data were comparable to those of Stockwell (1927) and Fleischer (1937) for the unit cell sizes of the pyrospite series. Sriramadas (1957) put the data from Skinner (1956) into diagram form relating the chemical compositions as a function of the unit cell edges and refractive indices. Winchell (1958) added the specific gravity (G) to the diagrams made by Sriramadas. This project extends this earlier work into the pyrospite ternary.

## EXPERIMENTAL

Garnets of composition  $Alm_{60}Pyr_{20}Sps_{20}$ ,  $Alm_{70}Py_{30}$ ,  $Alm_{30}Pyr_{10}Sps_{60}$ ,  $Alm_{50}Pyr_{10}Sps_{40}$ , and  $Alm_{60}Pyr_{40}$  were synthesized using a 3/4" piston-cylinder device from glasses made by J. B. Brady. End member glasses were made by melting mixes of oxides in graphite crucibles at 1 atm for 30 minutes at 1500°C. They were then crushed and ground under acetone for one hour. Crushed glass from each end member composition was weighed in appropriate amounts and ground together for an hour to achieve homogeneity. Two hundred to four hundred milligrams of the powder were then packed into a graphite container. The graphite container with lid was placed inside a fired pyrophyllite container and lid, which was held at the midpoint of the furnace. The furnace consisted of a graphite cylinder inside of a pyrex sleeve. The remaining space is filled from the bottom by a graphite plug, a glass plug, and a corundum plug. Above the sample container is another corundum plug with a hole for the thermocouple. The whole furnace was then placed inside a halite sleeve as shown in Figure 1.

The furnace assembly was then wrapped in lead foil and inserted into the piston-cylinder device along with a W-Re thermocouple to monitor temperature. The endload was applied, and the ram pressure was raised to about 7 kilobars. The temperature was then raised to 1250°C using the hot piston technique (Bohlen 1984). First the temperature was raised to 800°C then during a dwell time of three minutes the

the energy present in the system that is sufficient to break bonds in the melt. The effect of the pressure is potentially the most interesting part of the problem. Previous work has suggested coordination changes of the metals present in the melt structure with higher pressure (Stolper, 1987). Within the system, a greater amount of diopside has the effect of depolymerizing the melt and reducing the viscosity. Viscosities of natural basalts have been measured as 40 poise at 1350°C and 15 kbar (Kushiro et al., 1976). We can expect similar results for the synthetic compositions under these conditions, and predict lower viscosities with higher pressures and temperatures. The relative rates of change in viscosities with these changes will probably differ from the changes in other compositions and will be the most interesting and useful results.

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pressure was increased to 80% of its value, 16 kbars. The temperature was then raised to 1000°C at 200°/min. Finally the pressure was increased to 20 kbars and the temperature to 1250°C at 100°/min where it remained for 24 hours. After the 24 hours the sample was quenched rapidly. See Table 1 for program data and Table 2 for a list of the runs completed. The samples that do not follow this program include sample DYV1 which was held at 1500°C and did not produce a useable amount of garnet crystal because it was above the melting temperature and sample DYV8 which had an extended run time of 72 hours to try to produce more homogeneous garnets.

After the run was completed the sample was extracted. A portion of the sample was ground and examined under a petrographic microscope using immersion oils. This gave a preliminary look at garnet yield. Another portion of the powder was packed into a glass tube to find the lattice spacing using the Debye-Scherrer powder diffraction technique, JCPDS data cards 9-427, 15-742, and 10-354, and the cubic worksheet (Novak and Colville 1989). An aluminum plug was drilled for use with the Scanning Electron Microscope and Kevex energy dispersive spectroscopy. The garnets were held in place with epoxy and then polished at Amherst College.

## DISCUSSION

Figure 2 is a back scatter photograph of DYV2 that had not been polished. The zoning is a topographic effect, it is difficult to distinguish grain boundaries in the polished samples. The garnets are about ten microns in diameter. No additional phases were detected in the x-ray diffraction film. Figure 3 shows the ternary diagram of the synthesized garnets. Preliminary analysis of Debye-Scherrer photos of DYV2 and DYV3 gave lattice constants of 11.198 and 11.499. An initial look at the homogeneity with the Scanning Electron Microprobe showed some chemical zoning is present and needs to be characterized more fully. Molar volume and absorption bands as a function of composition are important for the calibration of garnet geothermometers and geobarometers. Additional XRD and FTIR spectroscopic study of the samples is planned.

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FIGURE 1 ASSEMBLY FOR GARNET SYNTHESIS

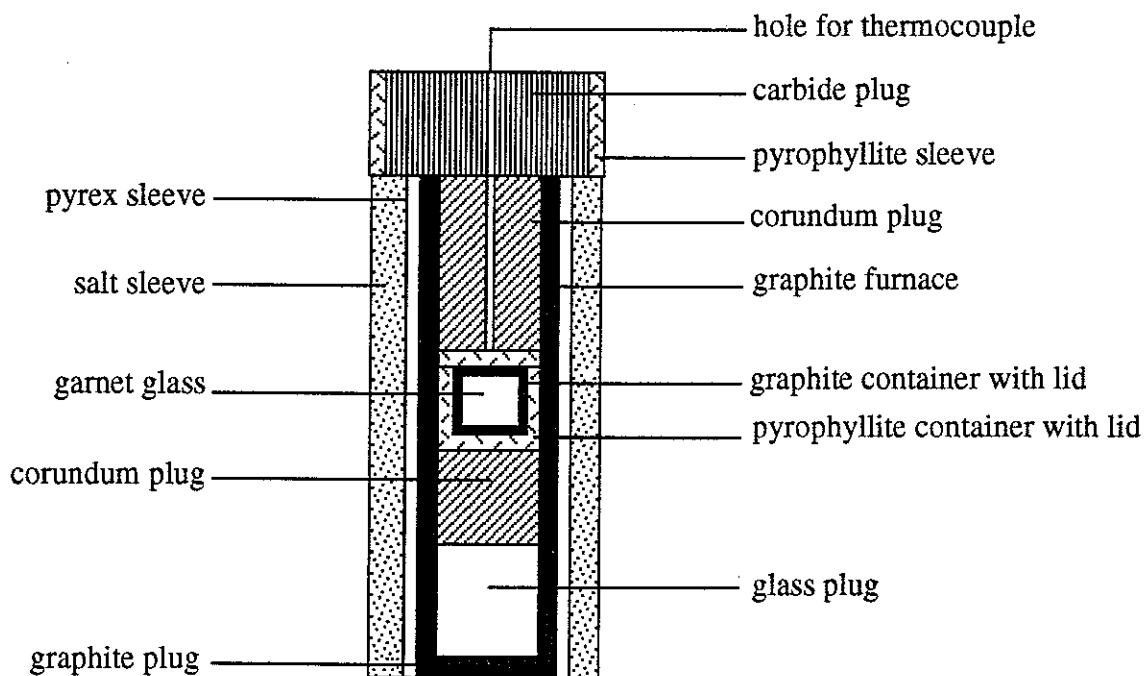


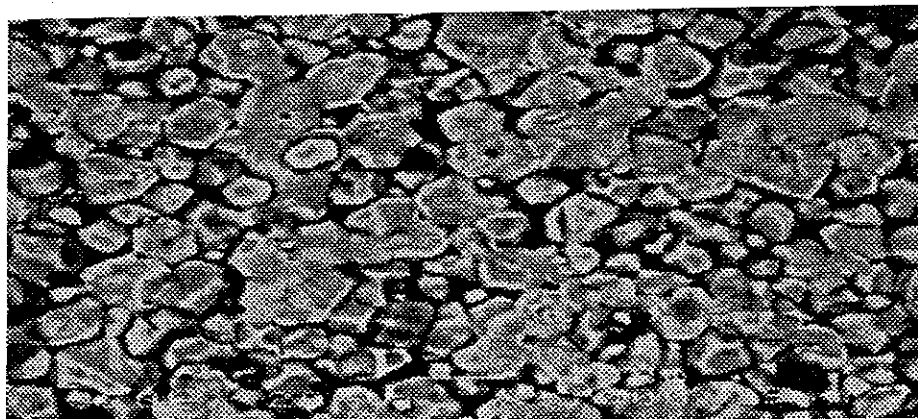
TABLE 1 PROGRAM FOR PISTON-CYLINDER

Ramp	1	2	3	4	5
Degrees/Minute	200	200	100	0	1000
Temperature	800	1000	1250	1250	0
Dwell time	3	3	980	460	end

TABLE 2 PISTON-CYLINDER RUNS

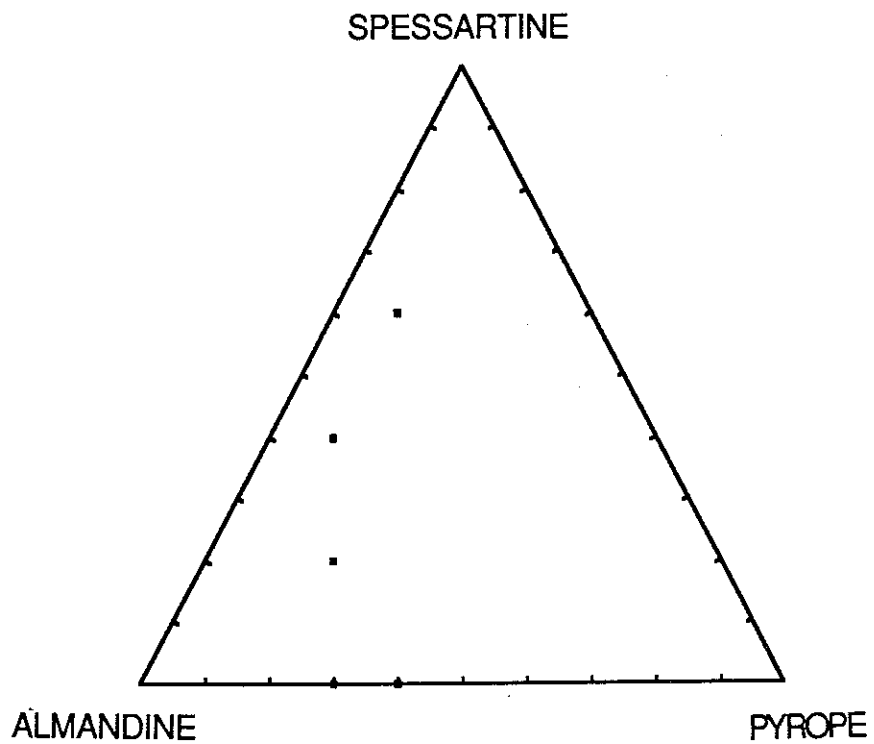
Run	Composition	Comments	Run	Composition	Comments
DYV1	60:20:20	Temperature too high	DYV5	30:10:60	
DYV2	60:20:20		DYV6	50:10:40	thermocouple failure
DYV3	70:30		DYV7	50:10:40	
DYV4	50:10:40	15 microliters of water	DYV8	60:40	72 hour run

FIGURE 2 BACK SCATTER ELECTRON IMAGE OF DYV2



— Ten Microns

FIGURE 3 TERNARY DIAGRAM OF SYNTHESIZED GARNETS



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