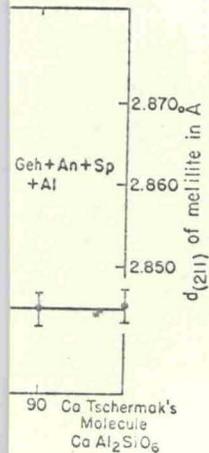


an + Sp + Fo, and $Di_{ss} + Fo$. The $d_{(211)}$ can be calculated as a function of composition in the $Geh + An + Sp + Fo$ phase where melilite coexists with these phases. For mixed compositions, $d_{(211)}$ of melilite varies with An + Fe and in general cannot be uniquely determined for these phase assemblages. The $d_{(211)}$ of melilite in $Geh + Di_{ss}$. To calculate $d_{(211)}$ in four-phase systems the critical planes between the critical planes must be determined. Iron must be present in $Geh + Sp + An$ to be possible to do this. The $d_{(211)}$ of melilite in these positions could be determined from a plot of bulk composition as shown in figure 2 as solid lines. The $d_{(211)}$ of melilite in these positions could be determined from a plot of bulk composition as shown in figure 2 as solid lines.

Invariant points have been determined for the phase-equilibrium series $Di-CTs$ and these points lie outside the $Geh + An + Sp + Fo$ phase volumes and the $Geh + An + Sp + Fo$ reaction point lies



composition along join $Geh + An + Sp + Fo$ actually determined in determinations

within the $Mel_{ss} + An + Sp$ volume at a temperature of $1360 \pm 5^\circ C$. This is only in fair agreement with De Vries and Osborn (1957), who measured a value of $1350^\circ C$ for this point. "Alumina" refers to corundum and/or " β alumina." These phases often occur together, although " β alumina" is predominant near liquidus temperatures, and only corundum peaks are observed in X-ray patterns of solidus assemblages.

The $Sp + An + Fo + Ak_{69}Geh_{31} + L$ invariant point lies within the $Mel_{ss} + An + Fo$ volume, and its temperature is $1225 \pm 5^\circ C$. The $Di_{ss} + An + Fo + Ak_{80}Geh_{11} + L$ invariant point also lies within the same volume. Its temperature is not known precisely. It is drawn in figure 1 at $1225^\circ C$, which probably represents a maximum value. Since these two invariant points have closely similar temperatures and compositions, it is difficult to decipher their mutual relationship. If they have the same temperature and different compositions, it is likely that some compositions along the $Di-CTs$ join will pass through neither point and will crystallize directly to $Mel_{ss} + An + Fo$ without forming any pyroxene or spinel. This possibility is depicted on figure 1. Another, equally likely, possibility is that the reaction point at which pyroxene is consumed occurs at a slightly lower temperature than the reaction point at which spinel is consumed. If this is so, some compositions along the join may pass through both points as they crystallize, before winding up as a mixture of melilite, anorthite, and forsterite. Since compositions along the "grossularite"-pyrope join (Chinner and Schairer, *Year Book 59*) appear to raise but not answer the same questions, the answers can be supplied only by the study of compositions lying off these joins in the $Ak-Ak_{70}Geh_{30}-An-Fo$ volume.

Chinner and Schairer observed that several compositions on the "grossularite"-pyrope join crystallized an aluminous pyroxene that reacted with liquid at lower temperatures to produce a melilite-

anorthite-forsterite assemblage. They suggested that extensive fractionation of diopside crystallized from a basaltic melt as the result of limestone syntexis would enrich the melt in Al_2O_3 . Aluminous pyroxene could then store up CaO and Al_2O_3 , which would contribute to the formation of melilite as pyroxene redissolved in the magma during the final stages of crystallization. This mechanism is in complete qualitative agreement with the classic contamination sequence described by Tilley and Harwood (1931) at Scawt Hill. It also receives excellent qualitative confirmation by the relations observed on the $Di-CTs$ join. The analogous reaction point in this system is $Di_{ss} + Ak_{80}Geh_{11} + An + Fo + L$, at which melts do indeed consume pyroxene and form melilite and other phases. This is presumably the same reaction point encountered by Chinner and Schairer. Their mechanism, however, has at least one serious limitation in the application to the melilite rocks of Scawt Hill. The pyroxene at this reaction point in the synthetic system has been shown to contain about 40 mole per cent $AlAl$ for $(Ca, Mg)Si$. This is more than twice as much Al_2O_3 as is found in Scawt Hill aluminous pyroxenes (Tilley and Harwood, 1931). Thus the analogy between the synthetic and the natural pyroxene reaction point is less direct than Chinner and Schairer inferred.

*Phase Relations in the System
CaMgSi₂O₆-CaAl₂SiO₆-SiO₂
at Low and High Pressure*

*Sydney P. Clark, Jr., J. F. Schairer, and
John de Neufville*

There is substantial indication that basaltic magmas are generated in the mantle, perhaps at considerable depths. Some of this evidence is seismic, some geothermal, some geologic. With the exception of the seismic activity associated with Hawaiian eruptions, it is indirect and perhaps capable of other

interpretations. But enough evidence points in the same direction to make a study of the effect of pressure on melting relations in systems of petrological interest worth while. Furthermore, an upper limit to the temperature in the mantle is set by the liquidus of whatever material is down there, for superheated liquid must either move relatively rapidly toward the surface or lose its superheat by reaction with surrounding solid material. Effects of pressure on liquidus relations must be studied in systems of moderate complexity before inferences about melting in the mantle can be drawn with any confidence. It has been found that a pressure of 20 kb produces large effects on the liquidus that could not have been predicted from data obtained at atmospheric pressure alone.

The quaternary system $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$, which contains the join $\text{CaMgSi}_2\text{O}_6$ (diopside)- $\text{CaAl}_2\text{SiO}_6$ (lime Tschermak's molecule)- SiO_2 , is of great importance, because it is sufficiently complicated to represent qualitatively the phase relations of the basic igneous rocks, particularly basalts, and rocks arising from their metamorphism. The main constituents commonly present in such rocks and absent from this quaternary system are iron in both its valence states, soda, water, and to a lesser extent K_2O , TiO_2 , and MnO . In this simplified system it is impossible to study such important relationships as the effects of the fugacities of water and oxygen or changes in composition of feldspars. Experimental difficulties occasioned by the various possible oxidation states of transition elements and the volatility of alkalis and water at high temperatures, however, are avoided. Despite the simplifications, a number of reactions of petrological importance take place in this system; because of this and its relative chemical tractability the system is well suited for a beginning to the study of complex chemical equilibria at high pressures.

Among the important phases lying in this system are the oxides spinel and

corundum, forsterite, the melilites akermanite and gehlenite, wollastonite, diopside, enstatite and its polymorphs, the aluminosilicates andalusite, kyanite, sillimanite, and mullite, the garnets pyrope and grossularite, cordierite, anorthite, and the polymorphs of silica. During the report year a new phase, with the composition of lime Tschermak's molecule ($\text{CaAl}_2\text{SiO}_6$), was synthesized for the first time at a pressure of 20 kb.

Because of the importance of this quaternary system, much previous work has been done to elucidate phase relations in it. Most were investigations of lines and planes joining two or three of the phases listed above. In this way the tetrahedron is crossed in many directions, and, given enough such studies, it should be possible to deduce with high precision the quaternary equilibrium relations at atmospheric pressure.

For initial study in this system at high pressure we selected the join diopside-anorthite. As Bowen recognized, this is a simple, pseudobinary representation of many basalts and diabases. The system was first shown not to be truly binary by Osborn (1942). We expected that the nonbinary behavior would be accentuated by pressure, and this has proved to be so. Part of the join is quaternary because of the incongruent melting of anorthite at high pressures. We have, however, only studied compositions lying in the plane diopside-lime Tschermak's molecule-silica, which contains the join diopside-anorthite.

For purposes of orientation it is helpful to consider the composition plane enstatite-wollastonite-corundum (fig. 3). All phases shown in the figure lie precisely in this plane; none are projected. Of particular interest are the intersecting joins diopside-lime Tschermak's molecule and grossularite-pyrope. The pyroxene join is characterized by complete solid solution at 20 kb; the garnet join, by complete solid solution above 30 kb. Magnesian Tschermak's molecule, shown on the diagram, has never been synthe-

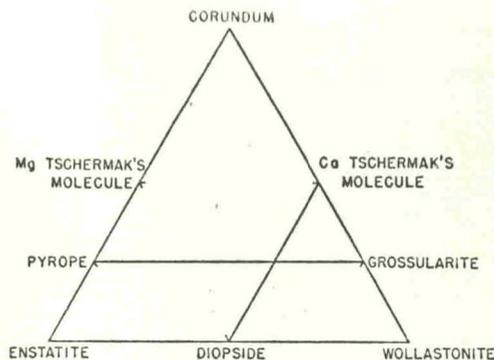


Fig. 3. Solid phases in the plane $\text{CaSiO}_3\text{-MgSiO}_3\text{-Al}_2\text{O}_3$.

sized, although Boyd and England (*Year Book 59*, p. 49) have made enstatites with at least 15 mole per cent Al_2O_3 in solid solution.

The plane in figure 3 contains a number of phases with a striking variety of crystal structures, all characterized by a metal-to-oxygen ratio of 2:3. Their densities are closely correlated with the structure. Densities of diopsidic pyroxenes mentioned in the ensuing paragraphs have been calculated from X-ray data discussed below. Cell edges of the grossularite-pyroxene series of garnets were given by Chinner, Boyd, and England (*Year Book 59*, p. 77), and the densities of other phases were taken from the literature, using X-ray data whenever possible.

Wollastonite and the pyroxenes are chain-type silicates. The lightest, wollastonite, has a density slightly greater than 2.9 g/cm^3 . The density of enstatite is 3.212 g/cm^3 , that of diopside is 3.281 g/cm^3 , and that of lime Tschermak's molecule is 3.437 g/cm^3 . The garnet structure is composed of isolated silica tetrahedra, connected by irregularly coordinated cations. The density of pyrope is 3.566 g/cm^3 ; that of grossularite is 3.603 g/cm^3 . The densest structure is that of the closely packed oxide corundum, 4.02 g/cm^3 .

It has been suggested that pyroxenes might undergo transitions to the corun-

dum structure at very high pressures, and this inversion has been reported in MgGeO_3 (Ringwood and Seabrook, 1962). It does not seem to have been remarked that garnets also have the metal-to-oxygen ratio appropriate to undergo a transition to a corundum structure. Such an inversion may take place deep in the transition zone in the mantle.

Two other comparisons of density are interesting to make. The first is between the density of crystalline lime Tschermak's molecule ($\text{CaAl}_2\text{SiO}_6$) and the densities of its low-pressure breakdown products gehlenite ($\rho = 3.038 \text{ g/cm}^3$), anorthite ($\rho = 2.765 \text{ g/cm}^3$), and either "β alumina" or corundum. The density of "β alumina" is not well known, but neither alumina phase is present in large amounts. The mean density of the breakdown products cannot be far from 2.9 g/cm^3 . Lime Tschermak's molecule is 18 per cent denser than this. The density change between the pyroxene and garnet at the intersection of the two joins shown in figure 3 is 6 per cent (3.368 versus 3.592 g/cm^3).

X-ray data for diopsidic pyroxenes. In order to set up suitable determinative procedures for complex solid solutions such as those shown by diopsidic pyroxenes, careful crystallographic work must be done. The fine-grained nature of synthetic crystals precludes single-crystal studies, and care must be taken that determinative peaks on powder patterns can be unambiguously indexed. Otherwise errors from effects of preferred orientation may influence measurements of unresolved multiple reflections.

In crystals of low symmetry it is all but impossible without the aid of a high-speed computer to be sure that all indexing allowed by the space group has been compared with the observed reflections. Only by being certain that all possibilities have been considered can one be sure that a reflection is not multiple. Such precautions have not always been taken in the past.

All data processing was carried out on

an IBM 7090 digital computer using programs written by Charles W. Burnham. His program for calculating unit-cell parameters by least squares is described elsewhere in this report. His program for calculating d values permitted by the space group from the parameters of the unit cell was used in indexing powder patterns.

TABLE 1. Miller Indices and d Values of Reflections Used in Calculating Unit-Cell Parameters of Diopsidic Pyroxenes

hkl	d Value	
	Diopside	Lime Tschermak's Molecule
221	2.9897	2.9412
310	2.9492	2.8997
311	2.8924	2.8613
311	2.3009	2.2739
330	2.1546	2.1062
331	2.1322	2.0902
421	2.1067	2.0752
132	1.9679	1.9468
150	1.7535	1.7026

The starting point in our investigation was a carefully indexed powder pattern of pure diopside. This pattern was compared with patterns obtained on material prepared by completely crystallizing glasses on the join diopside-lime Tschermak's molecule at 20 kb. The positions of the peaks were found to shift smoothly as a function of composition from one end of the join to the other. No peaks appeared that could not be traced into their counterparts in the diopside pattern;

this plus optical examination provides evidence that only one phase, a pyroxene, was present in these runs. As a check, the complete pattern for the composition 50 per cent diopside, 50 per cent lime Tschermak's molecule, was calculated. No unexpected interferences between peaks were found. The reflections used and their d values for diopside and lime Tschermak's molecule are given in table 1. These reflections were chosen because they can be indexed unambiguously and are sharp and strong—an important feature if they are to be used for determinative purposes in mixtures of phases that do not contain very much pyroxene.

The first three reflections listed in table 1 fall at 2θ angles less than 31° for copper radiation. Hence the d values cannot be determined with high accuracy. The parameters of the unit cells were calculated by least-squares adjustment both with and without these peaks. The resulting parameters do not differ significantly, but the standard errors are usually smaller if the low-angle peaks are rejected.

The unit-cell parameters of lime Tschermak's molecule and diopside are given in table 2, along with parameters for diopside from other observers. The agreement is good. The change of parameters along the joins diopside-lime Tschermak's molecule and diopside-enstatite is shown in figures 4 and 5. The data in figure 5 were obtained by applying the procedures described above to a series of glasses that had previously been crystallized at 1 atmosphere. Compositions containing more than 40 per cent

TABLE 2. Unit-Cell Parameters of Lime Tschermak's Molecule and Diopside

	Lime Tschermak's Molecule (present)	Diopside (present)	Diopside (Sakata, 1957)	Diopside (H. H. Hess, unpublished)
a , Å	9.615 ± 0.003	9.745 ± 0.001	9.743	9.741
b , Å	8.661 ± 0.002	8.925 ± 0.001	8.923	8.924
c , Å	5.272 ± 0.003	5.248 ± 0.001	5.251	5.247
β , deg	73.88 ± 0.03	74.13 ± 0.01	74.07	74.15
V , Å ³	421.79 ± 0.28	439.08 ± 0.07	438.98	438.77

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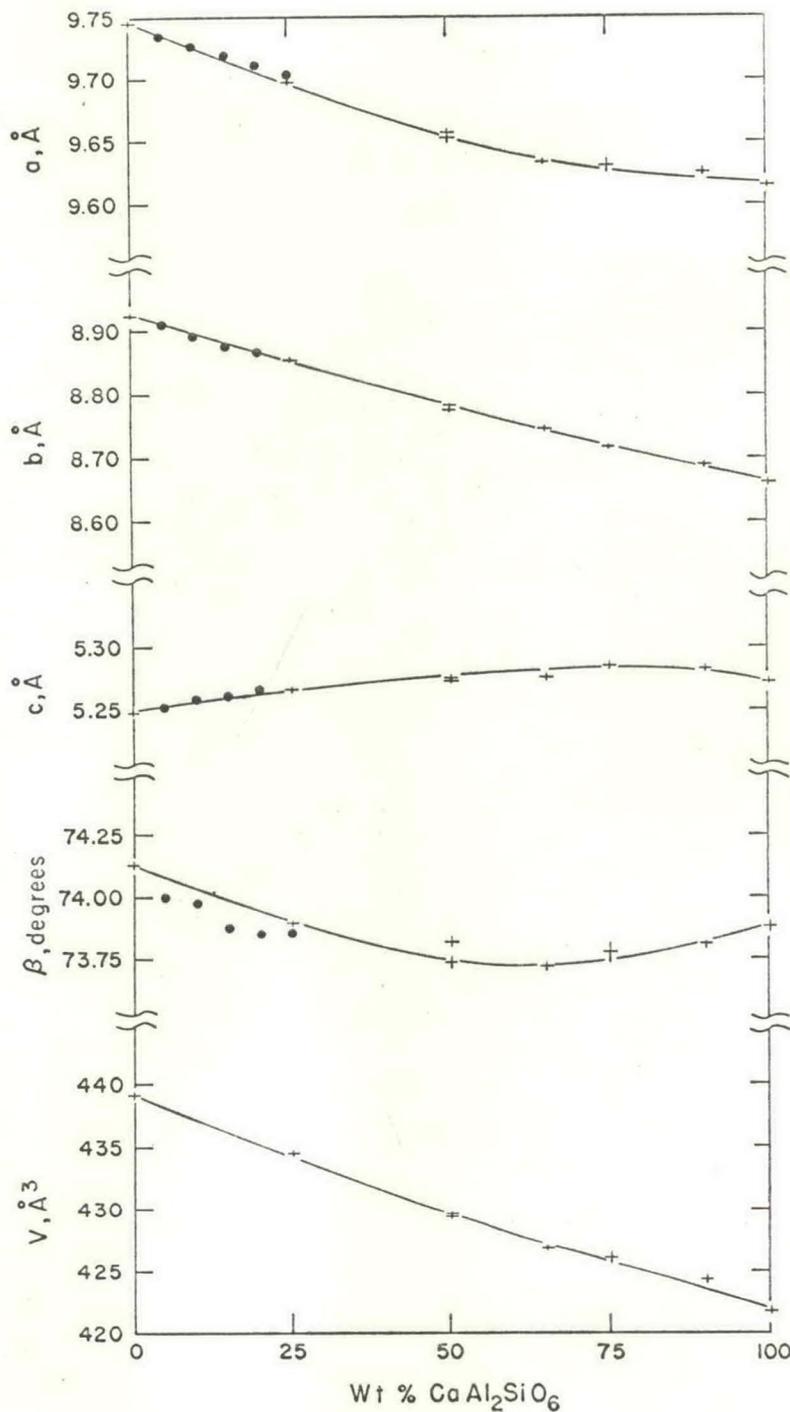


Fig. 4. Unit-cell parameters along the join diopside-lime Tschermak's molecule.

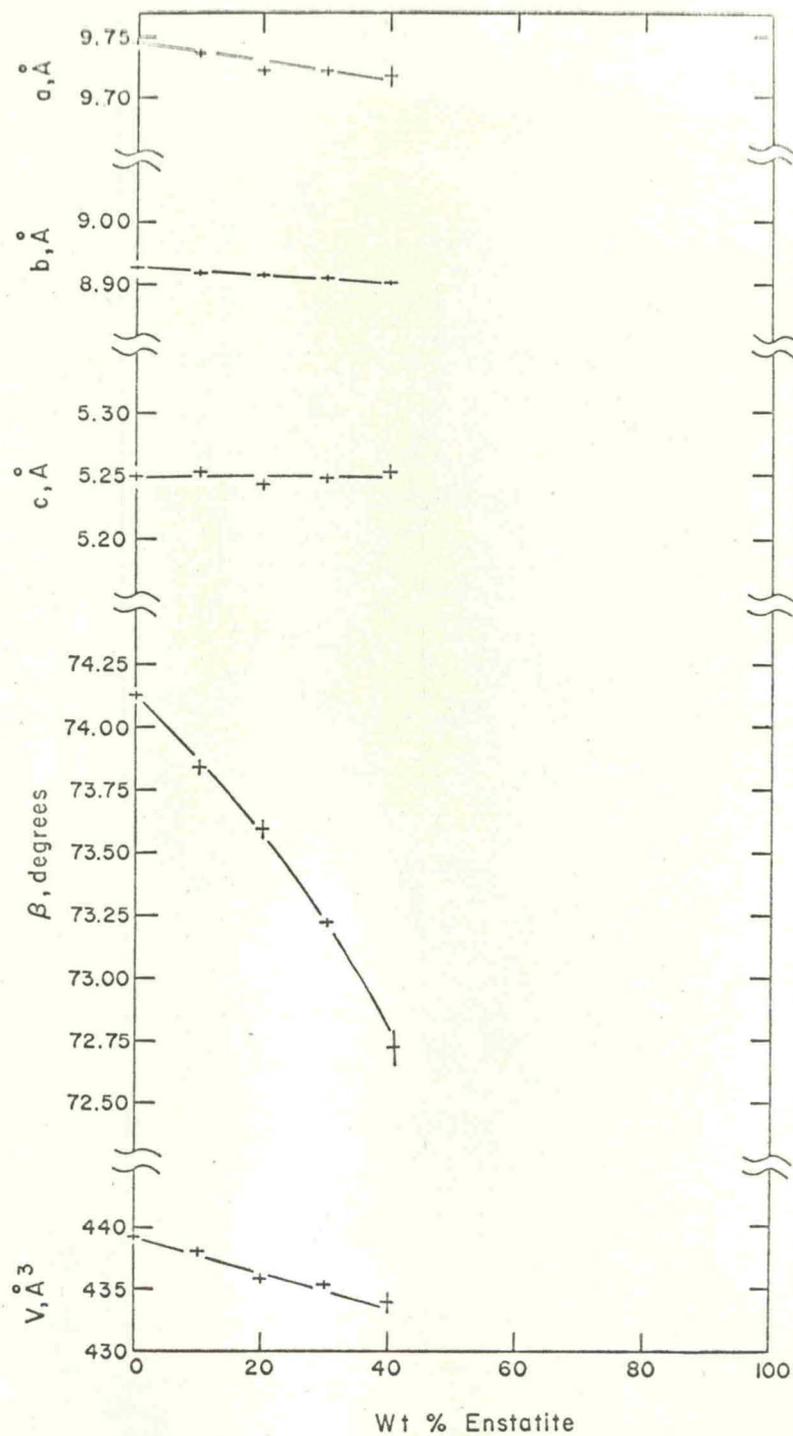


Fig. 5. Unit-cell parameters along the join diopside-enstatite.

enstatite do not crystallize to a single phase under these conditions.

Along the join lime Tschermak's molecule-diopside, the substitution is Al-Al for Mg-Si. One would expect that replacing an Mg atom with a relatively small Al would cause a and b to decrease. Likewise, replacing a Si atom with a relatively large Al atom in the silica chains would cause a slight increase in c . These are the observed effects. β changes little in this series. The internal consistency of the data for this parameter, i.e., the lack of scatter of the points about the curve in figure 4, is remarkable considering the scale of the diagram. The parameter that changes most is b , and hence reflections with large k are most satisfactory for determinative purposes along this join.

Volumes in this solid solution series depart systematically from a straight line connecting the end members in a way that implies that they are nonlinearly related to composition. The departure from linearity, although apparently real, is not large. A straight line would fit the data within 0.5 per cent.

Edges of the unit cell change little along the diopside-enstatite join; the most conspicuous feature of figure 5 is the large decrease in β with increasing content of enstatite. The volume is essentially linear with composition over the limited range of the data.

There is a systematic difference between our results and those of Hytönen and Schairer (*Year Book 60*, p. 136). They based a determinative procedure for diopsidic pyroxenes on the positions of the (150) and (510) reflections. We did not read (510) because of possible interferences with (422) and (332), but we can calculate its position from our data. For both reflections our 2θ angles are about 0.1° larger than those reported by Hytönen and Schairer. By assuming a value for β , it is possible to calculate a and b for diopside from their data. Using the extreme values of β in table 2, it is found that $a = 9.751$ to 9.755 Å and $b = 8.937$ Å. These values are sufficiently

larger than those found by other workers to cast serious doubt on the determinative curves given by Hytönen and Schairer.

Melting relations in the system diopside-anorthite-silica. Liquidus data for this system at atmospheric pressure are shown in figure 6. Dots indicate the compositions studied by the quenching method. Except for compositions near the diopside-silica join, the figure has approximately the appearance of the simplest type of ternary diagram, that is one in which only three pure solid phases exist and liquid miscibility is complete. That this is only approximately true was first shown by Osborn (1942), who demonstrated that the join diopside-anorthite is not binary owing presumably to solid solution of alumina in the pyroxene. This result has been confirmed by Hytönen and Schairer (*Year Book 60*). To obtain more precise information on the composition of the pyroxene, careful X-ray work was done on a composition lying on the diopside-anorthite join that was equilibrated with liquid at 1260°C and on a composition lying in the ternary plane that was equilibrated with liquid at 1220°C . In both, the departure of the unit-cell parameters from those of pure diopside was small; it was greater for the composition crystallized at the higher temperature. Hytönen and Schairer (*Year Book 60*, p. 137) indicate that at 1135°C in this system (a temperature well below the solidus) the pyroxene contains about 3 per cent lime Tschermak's molecule. They considered it probable that this amount of solid solution was metastable, and our results suggest the same. Because of the small shift in properties relative to experimental error, it is not possible to determine the direction in which these pyroxenes differ from pure diopside.

In all the sixteen compositions within the triangle diopside-anorthite-silica, the third solid phase first appeared on cooling at temperatures between 1218° and 1225°C . This implies that the system diopside-anorthite-silica is very nearly ternary, and that the stable pyroxene

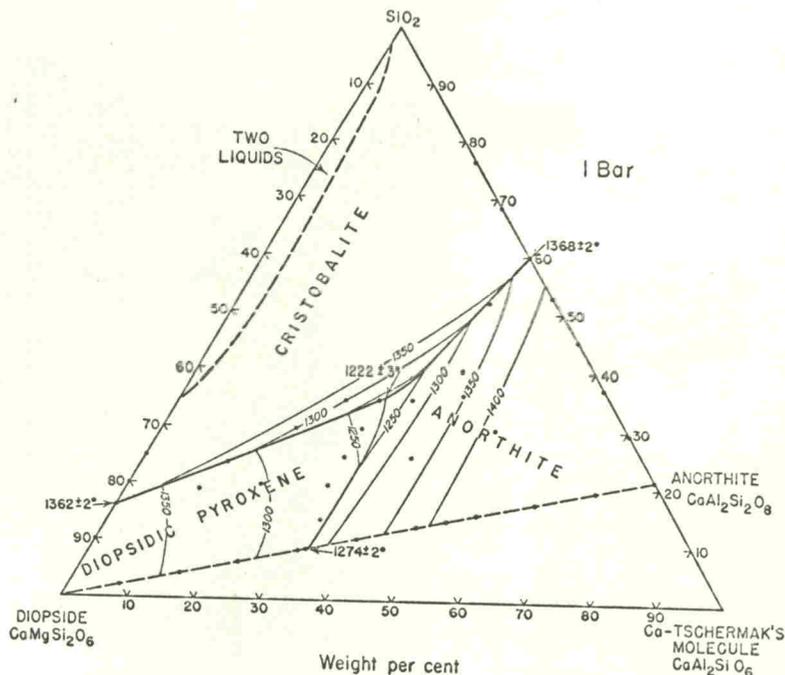


Fig. 6. Equilibrium diagram for the system diopside-lime Tschermak's molecule-silica at 1 atmosphere.

must lie close to the plane of figure 6 at 1222°C . The X-ray evidence implies that it is essentially pure diopside. The piercing point, or ternary eutectic, must be close to or at the thermal maximum on the quaternary univariant line connecting two quaternary eutectics. At one, wollastonite, diopsidic pyroxene, anorthite, and a silica phase coexist with liquid, and at the other enstatitic pyroxene, diopsidic pyroxene, anorthite, and a silica phase coexist with liquid. Determination of the composition of the latter eutectic is of great geologic significance, since it represents the goal of crystallization of a simplified silica-saturated basalt at low pressures.

High-pressure studies of the liquidus in this system have been carried out in a "single-stage" type of apparatus similar to that described by Boyd and England in *Year Book 60*. Results at 20 kb are shown in figure 7; a large number of runs have also been made at 30 kb, but this

work is not yet ready for presentation. In all the work described the load pressure has been decreased by 3 per cent to allow for the effect of friction.

The accuracy with which temperature can be measured is much lower at high pressures than at atmospheric pressure. At high pressures the uncertainty in temperature ranges from $\pm 10^{\circ}\text{C}$ in favorable cases to $\pm 20^{\circ}\text{C}$ or so. These estimates are based on the internal consistency and the reproducibility of some of our results. There is in addition a correction for the systematic effect of pressure on the emf of a thermocouple; this has been omitted because the elusive problem of quantitative determination of the correction remains to be successfully attacked. In contrast, at atmospheric pressure an accuracy of $\pm 2^{\circ}\text{C}$ can be achieved with care.

The eutectic temperature in the binary system diopside-silica is raised by slightly more than 200°C by a pressure of 20 kb.

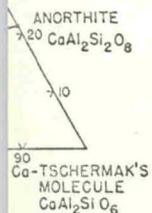
This is essentially the same as the change in melting point of diopside itself. The composition of the eutectic is not measurably affected by pressure. In this system, as in all the work at 20 kb, quartz is the silica phase stable on the liquidus. The effect of pressure on the two-liquid region in this system has not been investigated.

The system anorthite-silica is not binary at 20 kb because of the incongruent melting of anorthite, probably to corundum + liquid (Boyd and England, *Year Book 60*, p. 119). Between the fields of corundum and quartz on the liquidus there is a field of sillimanite. The temperature of lowest point on the liquidus, between the quartz and sillimanite fields, is 1540°C, and the composition is 48 weight per cent SiO₂. At atmospheric pressure the binary eutectic lies at 1368°C and 59 weight per cent SiO₂. (Both silica contents are determined relative to lime Tschermak's molecule.)

Changes produced by pressure in the system diopside-anorthite are greater

than in the other limiting systems. Not only does anorthite melt incongruently at high pressures but also the amount of alumina in the pyroxene increases dramatically. At compositions near anorthite, corundum and "β alumina" both appear at high temperatures, with and without other crystalline phases. One of these alumina phases must be metastable on the liquidus, but it is not clear which. There is some evidence that, although corundum is stable at the anorthite composition, "β alumina" is the stable liquidus phase at neighboring magnesian compositions. It will be difficult to work out the correct relationship between these phases because of the stubbornness with which they both persist metastably.

The nature of the minimum on the liquidus in this system has not yet been determined. It may be a cusp, resembling a eutectic, or it may be a smooth trough, depending on whether the minimum lies within the pyroxene field or at its boundary. Figure 7 is drawn as if this



ak's molecule-silica at 1

ready for presentation. described the load pres- eased by 3 per cent to of friction. th which temperature s much lower at high atmospheric pressure. s the uncertainty in es from ±10°C in ±20°C or so. These sed on the internal he reproducibility of s. There is in addition e systematic effect of of a thermocouple; ed because the elusive ative determination of ains to be successfully rast, at atmospheric cy of ±2°C can be

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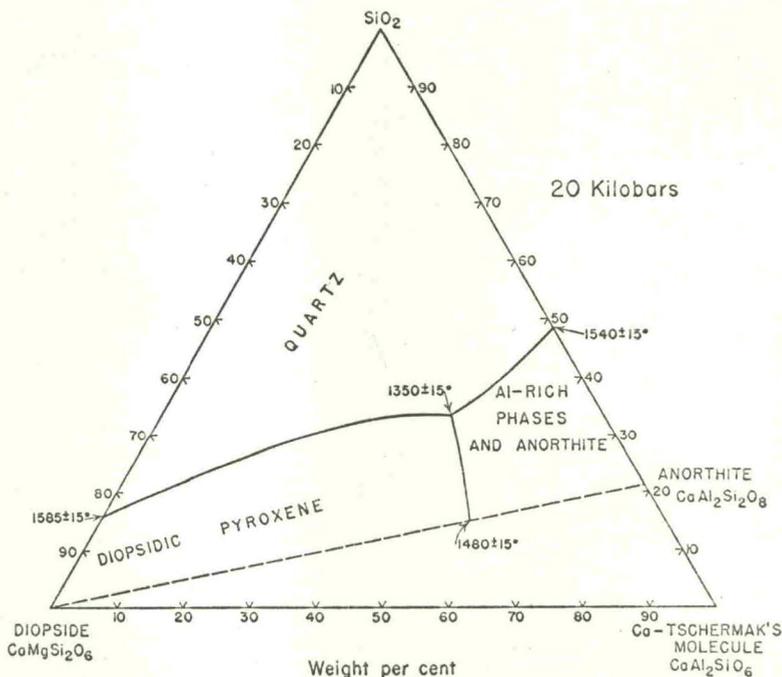


Fig. 7. Equilibrium diagram for the system diopside-lime Tschermak's molecule-silica at 20 kb.

minimum were a cusp at the boundary of the field, but future work may indicate the need for modification of this feature of the diagram. The temperature and composition of this point are 1480°C and 71 weight per cent anorthite. At atmospheric pressure this point lies at 1274°C and 43 weight per cent anorthite.

The complex relations at high pressures found in the systems diopside-anorthite and anorthite-silica continue into the triangle of figure 7. The fields adjacent to the anorthite composition have not yet been fully delineated. There must be a field of sillimanite, one of corundum, probably one of " β alumina," and, near the piercing point, one of anorthite itself. Although pyroxene, anorthite, and quartz are the solid phases present at the piercing point, the relationship there is not ternary. There is a melting interval of about 30°C. This point contains about 10 weight per cent more anorthite than its counterpart at atmospheric pressure, and its temperature is raised about 125°C by 20 kb. This is somewhat less than the increase in the minima in the diopside-anorthite and anorthite-silica systems.

That pyroxenes grown in this system at 20 kb do not lie on the join diopside-lime Tschermak's molecule is shown by the failure of compositions, as determined by X rays, to bear the relations to each other demanded by principles of phase equilibria, and by the fact that different parameters of the unit cell have values that would correspond to different amounts of lime Tschermak's molecule in solid solution. Correction for enstatite in solid solution, determined from β , improves the internal consistency of the data, but the remaining discrepancies are probably large enough to be considered real. Presumably there is also magnesian Tschermak's molecule (or corundum) in solid solution in the pyroxene.

These results should dispel any doubts that pressure, even in the absence of volatile constituents, can profoundly affect phase diagrams. In part of the range of compositions, the system at 20

kb is not even qualitatively similar to the system at atmospheric pressure, and quantitative differences in melting behavior occur at all compositions. The most striking new features caused by pressure are the incongruent melting of anorthite, the appearance of sillimanite on the liquidus, the appearance of quartz on the liquidus above 1000°C, and extensive solid solution in the pyroxene. None of these effects occurs at atmospheric pressure, and none of them could have been inferred without high-pressure experimentation.

There is an interesting possible geological consequence of the shift in composition of the piercing point with pressure. If a small amount of liquid were formed by fractional fusion at 20 kb in this system, it would have the approximate composition diopside₂₂-lime Tschermak's molecule₄₂-quartz₃₆. If this liquid were then decompressed suddenly, perhaps by rapid upward intrusion, it would arrive in a superheated condition and the composition of the liquid would be well inside the anorthite field at low pressure. The liquid would crystallize large quantities of feldspar before other solid phases appeared, which suggests a mechanism for the origin of anorthosites. It is to be expected that in the system albite-diopside-silica the piercing point will behave in a similar way because of solid solution of jadeite in the pyroxene and the eventual disappearance of albite from the liquidus. An important unexplored question is the behavior of intermediate plagioclases; it is not yet known whether the mechanism outlined can produce feldspars of the compositions found in anorthosites.

The System MgSiO₃-CaMgSi₂O₆

F. R. Boyd, Jr., and J. F. Schairer

Mineral assemblages containing two pyroxenes are of almost ubiquitous occurrence in mafic and ultramafic igneous rocks. The two pyroxenes are usually a calciferous pyroxene, augite or ferro-