EXPERIMENTS ON ULTRAMAFIC ROCKS AND VOLATILES AT HIGH TEMPERATURES AND PRESSURES

by

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A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF M. A. Sc.
in the Department of Mining and Geological Engineering

We accept this thesis as conforming to the required standard

THE UNIVERSITY OF BRITISH COLUMBIA
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ABSTRACT

The mechanism of emplacement of ultramafic intrusions has been a controversial subject for many years. This thesis summarizes evidence favoring the intrusion of ultramafic rocks as magmas. Experiments were conducted to study the effect on the melting behavior of ultramafic rocks of carbon dioxide, sodium, sulphur and water at temperatures up to 925°C. and pressures up to 2125 atmospheres. No melts were produced but it is felt that the evidence presented justifies further work on the problem.
ACKNOWLEDGEMENTS

Financial support from the Department of Geology, University of British Columbia, is gratefully acknowledged.

Dr. H. J. Greenwood, of Princeton University, provided design drawings for the pressure vessel, helped obtain the special alloy needed, and gave advice on the operation of high pressure equipment.

The Department of Metallurgy, University of British Columbia, supplied the furnace, controller and variable transformer used in the experimental work.

Messrs. J. D. Donnan and G. E. Montgomery, technicians for the Department of Geology, assisted the author in many ways throughout the course of the study.

Dr. D. A. Dunell, Department of Chemistry, helped the author with the pressure calculations.

The pressure vessel was made by Modern Engineering, Ltd. of Vancouver, of an alloy that proved very difficult to machine. The job was performed with skill and dispatch.

This study was suggested to the author by Dr. K. C. McTaggart, Department of Geology, who gave the author much valuable advice and assistance during the study.
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INTRODUCTION

Certain field evidence suggests that alpine-type ultramafic bodies are amplaced as magmas, other evidence, both experimental and observed in the field, supports the hypothesis that ultramafic bodies are injected as solid bodies or as masses of crystals lubricated by residual fluids. The problem is unresolved. The author, accepting the statement of Davis and Hess, (1949, p. 857), "It would appear that a further search for a chemical basis for an ultramafic magma is in order.", carried out a part of this search as described in this thesis.

The object of the experimental work was to investigate the effects of carbon dioxide, sodium, sulphur and water, alone and in combinations, on the melting behavior of ultramafic rocks. Experiments were conducted at 850 and 925°C., at pressures of 850 to 2125 atmospheres.

The results of this study were inconclusive. Within the range of temperature, pressure and composition investigated ultramafic melts were not produced. It is possible that at higher temperatures or pressures, or with compositions not tested, ultramafic melts may exist.

The literature dealing with the emplacement of ultramafic rocks is summarized and a bibliography of these papers is presented.
THE PROBLEM OF ULTRAMAFIC INTRUSIONS

A) History of the problem:

This discussion deals with bodies of ultramafic rock which have no proven or obvious connection with feldspathic rocks. Many of these intrusions belong to the "Alpine-type" of Benson (1926) or the "member of the ultramafic magma series" of Hess (1938) (see Smith, 1958, pp. 74-77). The complex intrusions of southeast Alaska (Taylor and Noble, 1960) do not fit into either of these classes but are included in the discussion because they have not been shown to be genetically connected with feldspathic rocks.

For over 75 years there has been controversy over the origin and mode of emplacement of ultramafic rocks. In the later part of the nineteenth century and the early part of the twentieth some authors advocated a metamorphic origin for ultramafic rocks (Benson, 1918, p. 695; Cairnes, 1924, p. 36). Most geologists later accepted the view that ultramafic rocks crystallized from a melt at some stage in their history. The controversy now is over the mode of emplacement of these rocks. During the past 30 years the principal antagonists in the controversy have been N. L. Bowen and H. H. Hess. Bowen and his associates, basing their arguments primarily on laboratory evidence, believed that ultramafic rocks are intruded either as crystal mushes or as essentially solid masses. Hess and his supporters, on the basis of field evidence, concluded that most ultramafic bodies were intruded as magmas.

J. H. L. Vogt (1924) believed that peridotite melts were formed by remelting of olivine crystals settling under gravity as differentiates of a basaltic magma. Vogt also suggested that
there was a "sag" in the forsterite-fayalite liquidus solidus loop that would allow olivines of composition Fo$_{90}$ to melt at a temperature as low as 1600° C. (Vogt, 1921). Bowen and Schairer (1935, 1936) showed that the equilibrium diagram was of the normal solid solution type and that olivines of composition Fo$_{90}$ required a temperature of 1850° C. for complete melting.

These high temperatures are the main objection to the existence of ultramafic magmas. Geologic thermometry indicates temperatures of 1200° C. (Turner and Verhoogen, 1960, p. 440) are the maximum obtainable in the crust, therefore a dry ultramafic melt is not possible.

In reply, Hess (1938a, b, c) postulated a hydrous ultramafic magma which would solve the problem of the origin of serpentinites as well as the problem of the emplacement of ultramafic magmas. Hess argued that the presence of 5 to 15 percent of water in an ultramafic magma (i.e. a "serpentine magma") would lower the freezing temperature of the melt sufficiently to allow intrusion of the melt at a temperature less than 1,000° C. As the melt cooled, the water would serpentinize the olivine and pyroxene, thus forming a serpentinite.

Bowen had pointed out that no ultramafic extrusive rocks had ever been found. In answer to this objection, Hess argued that when the ultramafic melt approaches the surface, they would lose its volatiles and freeze before reaching the surface.

Bowen and Tuttle (1949) investigated the system MgO - SiO$_2$ - H$_2$O and were able to show that water under pressures equivalent to several miles of depth in the earth's crust (15,000 to 30,000 pounds per square inch) had no effect on the melting of olivine
at temperatures up to 1,000° C. and that a "serpentine magma" could not exist at temperatures less than 1,000° C. Bowen and Tuttle believed that ultramafic rocks are intruded as essentially solid bodies, moving by "gliding translations and perhaps also by granulation and some major fracturing." (1949, p. 455). Water is believed to have some lubricating effect on the movement of these crystalline bodies.

Davis and Hess (1949) admit that the idea of an hydrous ultramafic magma at low temperatures is untenable, but point out that further research into the chemistry of ultramafic magmas is necessary.

During and after this period of controversy numerous papers have been published giving evidence in favor of magmatic emplacement of ultramafic rocks. This evidence is summarized in the following section.

B) Evidence for ultramafic magmas:

1. External features:
   a) Lack of deformation of the country rock; - If solid intrusions or intrusions of crystal mushes were the only ways in which ultramafic rocks could be emplaced, there should always be evidence in the form of faults, contortions and crumpling of the country rock to show that strong forces have been operative during intrusion of the ultramafic rocks. Ultramafic rocks in California (Kramm, 1910, p. 321; Taliaferro, 1943, p. 155; Chesterman, 1960, p. 215), the Bridge River district, B. C. (Cairnes, 1937, p. 30), the Tulameen district, B. C. (Camsell, 1913, p. 67), Aiken Lake, B. C. (Roots, 1954, pp. 146, 149), and in the Teslin district, Yukon Territory (Mulligan, 1963, p.63) are noted as having pro-
duced little deformation of the country rock upon intrusion.

Davis and Hess (1949, p. 857) state that many ultramafic rocks were intruded at shallow depth into weak, but relatively undisturbed sediments. Hess (1938b, p. 326) describes such an intrusion in Cuba where an ultramafic body was intruded into practically unconsolidated sediments without much deformation. The shearing strength of serpentine (180-340 kg/cm²) is somewhat greater than that of limestone (100-200 kg/cm²) or sandstone (50-150 kg/cm²) (Billings, 1954, p. 17). Dunite is probably stronger than serpentinite. Therefore, forceful intrusion of an ultramafic mass should sometimes be accompanied by "intrusion" of sandstones, limestones and other sediments. The author has found no description of such occurrences.

Hess (1938b, p. 326) describes examples of ultramafic rocks intruded at shallow depths, which have smooth, nearly horizontal contacts. If these ultramafic rocks were emplaced as essentially solid bodies, there should be extensive deformation and contortion of these contacts.

Leech (1953, p. 38) states that the main mass of peridotite in the Shulaps Range, B. C., is a structural unit. He believes there would have had to have been movement of a block of rock about seven miles by two miles in cross-section to account for the features seen in this body if it was emplaced as a solid or near solid body.

Hess (1955, pp. 401-402) points out that alpine-type ultramafic rocks appear to have been emplaced before the main period of deformation. Also, many of these ultramafic bodies are located near the margins of the disturbed areas where the stresses
were not high. Taylor and Noble (1960, p. 175) believe that the ultramafic rocks of southeast Alaska were intruded after the folding and metamorphism of the Paleozoic and lower Mesozoic rocks and before the emplacement of the Coast Range rocks.

Roots (1954, p. 149) argues that the ultramafic rocks of the Aiken Lake area, B. C. were emplaced after the regional deformation of the country rocks.

Chesterman (1960, p. 215) describes an ultramafic intrusion at Leech Lake Mountain, California, where there is little shearing parallel to the contacts of the ultramafic rock. Instead, the country rocks show tension joints perpendicular to the contacts.

b) **Geometry of the ultramafic bodies:** - The alpine-type of ultramafic rock is generally thinly tabular or lenticular. It is difficult to imagine how these thin sheets could forcefully intrude rocks, especially at shallow depths, without being intensely crumpled and deformed by the pressure forcing them into the country rock. Armstrong (1949, p. 86) working in the Ft. St. James map area, B. C., and Cooke (1937, p. 66) in the Thetford, Disraeli, Warwick map area, Quebec, both comment on this point.

c) **Connections with the magma chamber:** - Armstrong (1949, p. 86) observed two pipe-like, peridotite masses in the Ft. St. James map area, B. C. which may represent feeders to some of the nearby ultramafic sills.

2. Contact features:

a) **General contact features:** - Although many ultramafic intrusions are roughly concordant with the regional structure, on close examination they are shown to be cross-cutting in detail. Roots (1954, p. 149) describes the sharp contact at the
roof of the ultramafic rocks in the Aiken Lake area, B. C. This contact truncates the regionally tilted but locally undeformed country rock. Hess (1938b, p. 324) mentions having collected some specimens of chilled contact rocks.

b) Lack of thermal effects: The lack of thermal effects around many ultramafic intrusions has always troubled geologists who favor the hypothesis of an ultramafic magma. Hess (1938b, p. 323) points out that good exposures of the contacts of ultramafic rocks with country rocks are rare. He attributes this to zones of talc-carbonate and serpentine alteration found at the contacts of many ultramafic rocks and to the fact that many of these contacts weather easily and are concealed.

Little (1949) describes the ultramafic rocks of the Middle River range, a part of the Ft. St. James area, B. C. Using the method of Ingersoll and Zobel (1913), he determined that if the original temperature of an ultramafic sill 400 feet thick was 1200° C., and the original temperature of the country rock 100° C., the temperature of the country rock 15 feet from the ultramafic contact would be less than 600° C. and 100 feet away it would only be about 350° C. Therefore, he explains the lack of thermal effects as being due to the alteration of the country rock near the ultramafic body to talc-carbonate. He describes one occurrence of a spotted slate 50 feet from a contact.

In southeast Alaska, Ruckmick and Noble (1959, p. 1013) believe that the lack of contact effects is due to the fact that the ultramafic rocks were intruded into a gabbro which is itself stable at high temperatures and shields the country rock from the effects of high temperature. They also believe that the dry-
ness of the intrusion would limit the distance from the contact at which metamorphic effects would be found. Taylor and Noble (1960, p. 177) describe a similar gabbro zone around other ultramafic intrusions in this area.

Hess and others have been mapping the ultramafic intrusions in Venezuela. One of Hess' coworkers (Shagam, 1960, p. 297) explains the lack of contact effects around one group of these ultramafic bodies by explaining how the ultramafic magma could have been intruded as a single mass to a level a few hundred feet below its present position and then broken up and moved along faults to its present positions.

c) Dikes and apophyses: - There are numerous examples of ultramafic rocks with small dikes and apophyses intruding the country rock. Chesterman (1960, p. 215) describes such features at Leech Lake Mountain, California, and notes that the metamorphic aureole around these smaller features is less than that around the sill. In the Bridge River district, B. C., Cairnes (1937, p. 30) found pyroxenite dikes cutting diorite and showing chilled contacts against the diorite. Hess (1938b, p. 326) says that complex diking of the country rock is common around ultramafic rocks.

d) Descriptions of metamorphic rocks around some ultramafic intrusions: - Chesterman (1960, p. 210-215) describes contact rocks at Leech Lake Mountain, California, which have been metamorphosed to a glaucophane schist to greenschist facies assemblage of diopside-jadeite, antigorite and chlorite with rare glaucophane. This metamorphism, according to Turner and Verhoogen (1960, p. 534, 544), is indicative of temperatures of 300 to 500° C. and pressures greater than 8,000 bars.
Kramm (1910, pp. 321, 324) describes bodies of ultramafic rock in the Central Coast Range of California which have baked and indurated sandstones. Fairbanks (1892, quoted by Taliaferro, 1943, p. 204) describes a serpentinite in the Knoxville shales of central California which has metamorphosed these shales over a width of one hundred feet, obliterating bedding and causing noticeable hardening of the shale. Taliaferro (1943) describes numerous occurrences of serpentinized ultramafic rocks in the Franciscan and Knoxville rocks of California which have baked the country rock. Taliaferro also describes areas of glauconophane schist metamorphism, as does Durrell (1940).

Roots (1954, pp. 133-139) gives a detailed description of the metamorphic rocks formed by the intrusion of ultramafic rocks into volcanics and sediments in the Aiken Lake area, B. C. The country rocks are andesitic flows and tuffs, medium-to-fine-grained greywacke, minor argillite and limestone. The metamorphic aureole is 1,000 to over 8,000 feet wide around the largest ultramafic body. This aureole consists of a variety of contaminated, hybrid and contact metamorphic rocks. Argillite has been recrystallized to a coarse rock with a porphyroblastic texture. This rock in places contains andalusite porphyroblasts up to one-quarter inch long. Fine-grained, carbonaceous sandstones have been "cleansed", leaving fresh detrital quartz grains in a carbonaceous matrix. The andesitic rocks are composed mainly of saussuritized plagioclase and chlorite. In general in the outer zone these rocks are recrystallized and reconstituted to a biotite-feldspar gneiss. The gneissosity becomes more pronounced nearer the ultramafic body and the rocks approach the compo-
sition of a biotite diorite. Near the contacts the rocks are hybrid rocks consisting of hornblende or biotite bearing basic and ultrabasic rocks which Roots thinks formed from contaminated ultramafic magma. The limestones are recrystallized and about half of the limestone contains irregular patches up to several feet across of diopsidic pyroxene. Much of the limestone has been serpentinized. About one-fifth of the limestone is porphyroblastic, containing grossular garnet crystals up to three millimeters in diameter. In all of the limestone detrital quartz is unaffected.

The metamorphism of these contact rocks belongs to the hornblende hornfels or pyroxene hornfels facies, corresponding to temperatures of 500 to 700° C. and pressures of 0 to 4,000 bars. The stability of the detrital quartz in the associated limestones limits the temperature of these rocks to a maximum of 400 to 650° C. Aikens believes that the metamorphism was predominantly thermal, with little material added from the ultramafic magma.

MacKenzie (1960, pp. 309-313) describes the contact metamorphic rocks associated with the Tinaquillo peridotite in Venezuela. The metamorphic aureole is 1500 to 2,000 meters wide. The country rock is a hornblende-quartz-plagioclase gneiss. Away from the contacts this rock is metamorphosed to a garnet-pyroxene-hornblende-plagioclase gneiss with pyralspite garnets becoming larger and more numerous towards the contact. At the contact there is a zone of augite-hypersthenepyrope-almandine-hornblende rock which cannot be correlated with any of the established metamorphic facies. Because of the development of pyroxenes and garnets, MacKenzie believes the temperature of the country rock was 800 to
1,000 C. MacKenzie postulated that the ultramafic rocks were intruded as a mass of crystals lubricated by vapor, but Hess (1960, p. 239) believes that the body probably moved only a few hundred feet as a solid - only enough to cause the undulatory extinction in the olivine and the cataclastic texture which MacKenzie described. Hess does not believe that the ultramafic rocks could have moved very far as a nearly solid body and still have a temperature of about 900° C. when it reached its present position.

Walton (1951, p. 322) believes the gabbro rim around the Blashke Island ultramafic complex in Alaska is the product of metasomatism and partial remobilization of the country rocks caused by the introduction of hot solutions from an ultramafic magma. This rim of gabbro is up to 1400 feet wide. Outside this rim the country rock has been converted to hornfels of gabbroid composition for about 100 feet.

Mulligan (1963, p. 63) mentions pyroxene hornfels facies metamorphic rocks near ultramafic rocks in the Teslin area, Yukon Territory.

3. Internal features:

a) Undulatory extinction and granulation: - Undulatory extinction and the cataclastic texture of olivines in some ultramafic rocks has been used by many authors as evidence for solid or near solid intrusion of the rocks. Hess (1960, p. 239) believes that only a few hundred feet of movement is sufficient to produce these features. Turner (1942), after a study of the orientation of olivine crystals in ultramafic rocks, concluded that no change in fabric can be attributed solely to grain deformation that has
been sufficient to cause marked undulatory extinction in olivines. He did find examples where the orientation of olivine crystals has been affected by stronger forces which he believes resulted from intrusion of the ultramafic body as a solid or near solid mass.

Many ultramafic rocks exhibit little or no evidence of strain or cataclasis. Taylor and Noble (1960, p. 184) found no evidence of strain or granulation in the 35 ultramafic complexes in southeastern Alaska. Leech (1953, p. 38) found no evidence for intense deformation in the Shulaps ultramafic rocks. Others have also noted a lack of deformation (Wilkinson, 1953, p. 319). (Cooke, 1937, p. 65; Ruckmick and Noble, 1959, p. 1006; Mulligan, 1963, p. 63).

Wilkinson (1953, p. 319) describes peridotites in Queensland, Australia which have myrmekite-like intergrowths of picrotite in olivine and pyroxene.

b) Zonal structure and layering: - The ultramafic intrusions in southeastern Alaska are characterized by similar compositional zoning (Taylor and Noble, 1960, p. 176, 177). All of these intrusions have dunite or peridotite cores surrounded by pyroxenites. All of them show cryptic layering; the Mg/Fe ratio increases in the olivine and pyroxenes towards the outside of the intrusion; in the pyroxenes Al/Si and Fe$^3$/Si decrease towards the outside.

Findlay (1963) describes a similar compositional layering in the Tulameen, B. C. ultramafic complex. He finds, however, that there is a decrease in the Mg/Fe ratio towards the edge of the complex.

Leech (1953) estimates that 10 percent of the rocks of the main Shulaps ultramafic body are compositionally layered. The
sequence of rock types is not constant. Thickness of the layers ranges from a few inches to a few hundred feet and the layers often die out and recur along strike. Dunite dikes cross-cut pyroxenite and peridotite layers but merge with dunite layers.

Armstrong (1949) describes a rough gradation from dunite at the bottom to peridotite or pyroxenite at the top of the ultramafic bodies in the Ft. St. James map area, B. C. He found irregular masses of dunite cross-cutting the layering and narrow dikes of a very coarse-grained pyroxenite cutting the dunite and peridotite. In some places the pyroxene crystals are oriented perpendicular to the walls of the dikes. These features are difficult to explain except as magmatic features.

In none of the papers was feldspar reported as a primary constituent of the ultramafic rocks. Therefore it is unlikely that these rocks could have been formed by differentiation of a feldspathic magma. In addition, none of the ultramafic rocks are associated with feldspathic rock types which can be shown to be genetically connected with the ultramafics.

c) **Inclusions**: Wilkinson (1953, p. 311) described peridotites in Queensland which contain inclusions of an older peridotite. He believes that these inclusions were incorporated into an ultramafic magma.

Armstrong (1949, p. 86) found numerous inclusions of country rock near the contacts of ultramafic rocks.

4. Ultramafic volcanic rocks

Three recent papers have been written which describe ultramafic extrusives. Bailey and McCallien (1953) describe serpen
tinized peridotite lavas in the vicinity of Ankara, Turkey.
Gass (1958) has found ultramafic pillow lavas on the island of Cyprus. Maxwell and Azzaroli (abst. 1962) presented a general discussion of extrusive ultramafic rocks in the Alpine-Himalayan system.

The Turkish ultramafic lavas are associated with spilitic lavas and radiolarian cherts. Serpentine is overlain by carbonated serpentine, limestone, greywacke and radiolarian chert. Bailey and McCallien believe that peridotite was intruded as a submarine lava, moving as a crystal mush lubricated by a carbon dioxide-rich fluid. The peridotite was altered in bulk to serpentine and superficially to carbonated serpentine. A deposit of fine-grained crystalline limestone fills fissures and cavities in the carbonated serpentine and in part is bedded over the serpentine. The authors give no evidence to support their belief that the ultramafic rocks were extruded as a crystal mush. The present author believes these lavas may have been extruded as magmas.

Gass describes two types of lavas. The first contains olivine phenocrysts in an ultramafic groundmass, the average composition of which is peridotitic. The other type is anhomogenous rock of ultramafic composition. The lavas show vesicles and pillow structures with chilled margins.

Maxwell and Azzaroli discussed large areas of ultramafic rocks which seem to represent pre-orogenic extrusion of ultramafic magma from fissure zones parallel to developing eugeosynclines. The magmas were fluid and are thought to have moved with high velocities. Radiolarian cherts and siliceous shales lie in sedimentary contact on the uneroded top surface of the lavas. Later orogenic movement is believed to be responsible
for the formation of the apparently rootless masses, usually interpreted as "cold intrusions".

C. Summary

The above discussion has presented considerable evidence in favor of the hypothesis that certain ultramafic rocks are emplaced as magmas. It is possible that the conclusions drawn by Bowen and Tuttle (1949) do not hold if other components are substituted for or added to water in the system MgO-SiO₂-H₂O. An account of experiments testing this possibility follows.
SELECTION OF COMPONENTS

The general effect of volatile components of lowering the freezing temperature of silicate systems is well known. The problem is to select components which will have the desired effect on the silicate system being studied and which can be shown by field evidence to be reasonable components of the original melt.

A) Carbon dioxide

Almost every serpentinized ultramafic rock has associated with it carbonate or carbonate-talc rocks. If, as some authors have suggested (Hess, 1933, p. 635; Bowen and Tuttle, 1949, p. 457), serpentinization is accomplished by residual fluids of the ultramafic rock, it may be that the carbonate rocks are also a deuteric effect.

B) Sodium

Sodium, although not a volatile, was chosen because of the association of glaucophane schist facies metamorphic rocks with ultrabasic rocks in California (Durrell, 1940, pp. 88-94; Taliaferro, 1943, pp. 159-175; Chesterman, 1960, p. 215). Spilites are common associates of serpentinites (Turner and Verhoogen, 1960 pp. 258-261; Hess, 1955, pp. 392-393).

C) Sulphur

Kullerud (1963, p. 187) states that "The melting points of certain silicates are considerably lowered by sulphur and its reaction products". He believes that the introduction of sulphur may lead to the formation of silicate-sulphide immiscible liquids at temperatures well below the melting temperatures of the sulphides or the silicates. Hawley (1962) has presented evidence to show that immiscible sulphide liquids were important in the
formation of the Sudbury ores. Nickel sulphides are also quite common in ultrabasic rocks.

D. Calcium, carbonate, potassium, sodium and carbon dioxide

Work on carbonatites has led to the hypothesis that carbonatites may separate from an alkali-rich ultramafic melt by differentiation (King and Sutherland, 1960). The major components of a carbonatite lava analyzed by Dawson (1964, p. 106) were mixed with olivine in an attempt to produce a melt.

E. Water

Water is the most common volatile in natural melts. Since the work of Bowen and Tuttle (1949) dealt only with the effects of pure water on the melting behavior of olivine, it was decided to study the effect of water plus other components.

Benson (1918, p. 709) mentions experimental work which showed that olivine and serpentine were soluble in carbonic acid to the extent of 3 to 4 percent.
MATERIAL

Vessel - Haynes R-41 alloy - 1/4" diam. x 12"
Cap - Cold rolled steel - 1 1/2" hex. x 2 3/4"
Filler rod - 303 stainless steel - 1/4" diam. x 9.525"
Seal - 303 st. st. - 3/4" diam. x 2"

fig. 1

COLD SEAL PRESSURE
VESSEL

(3/4 scale)
APPARATUS

A) Pressure Vessel (Figs. 1 and 2)

The vessel used is quite similar to that described by Tuttle (1949, p. 1728) as a cold-seal bomb. The vessel was built from plans provided by Dr. H. J. Greenwood (personal commun., 1964). Some changes in dimensions were made to facilitate construction.

Fig. 2 - Photograph of pressure vessel

The vessel was machined from Haynes No. R-41 alloy. This is a vacuum-melted, nickel-base alloy supplied by the Metals and Carbon Division, Union Carbide Stellite Corp. Its approximate composition is Ni^{50} Cr^{20} Co^{11} Mo^{10} Fe^{5} Ti^{3} Al^{1.5}. This alloy has "exceptionally high strength at temperatures in the range of 1200 to 1800°F". (650 to 980°C.) (Union Carbide Corp. 1963, p. 2) 0.2 percent Yield Strength at 925°C. is 48,000 to 55,000 psi. The alloy is corrosion resistant and very tough; machining must be done with tungsten carbide tools.
The filler rod and cone-seal were machined from stainless steel (S.A.E. 303). The maximum temperature of the seal during the experiments was about 400° C. and stainless steel has adequate strength at this temperature. A small amount of scale formed on the stainless steel after the first few runs. A different type of stainless steel might prevent such corrosion.

The cap, which remains relatively cool (see Fig. 5), is machined from cold-rolled steel.

Platinum foil crucibles, crimped with pliers, held the ultramafic rock material. To prevent any reaction between the platinum and the alloy of the pressure vessel, the platinum crucibles were placed inside silica glass tubes. This glass tubing also served to reduce the volume of the vessel chamber. (Fig. 3).

---

fig. 3- CHAMBER of PRESSURE VESSEL

(Diagrammatic)

- Filler rod
- Pt foil
- Silica glass tubing
  17/64" O.D.
  7/32" I.D.
B) Furnace and Control Equipment (Figs. 4 and 5).

The furnace consists of four nichrome resistance coil elements, formed around a cylindrical chamber 13 inches long and 3 inches in diameter. The coils are insulated by at least 3 inches of fire brick. Each coil has a resistance of 35 to 40 ohms. The four coils are connected in series-parallel (Fig. 5), giving an overall furnace resistance of about 37.5 ohms. This load draws about 6 amperes at 220 volts.

Towards the end of the study, one side of the furnace burned out, leaving only two coils on one side of the furnace. These two coils were reconnected in parallel, giving a total resistance of about 20 ohms and drawing about 11 amperes current. With this arrangement a temperature of 925° C. was still attainable.

The controller is a Barber-Colman type 401, with a range of 0 to 1200° C. This controller would maintain the temperature to within 10° C. of the desired setting.
fig. 5

SCHEMATIC DIAGRAM of
FURNACE CONNECTIONS
A variable transformer was used to adjust the current to the furnace after the desired temperature had been reached. It was possible to adjust the current so that the power input nearly balanced the heat losses from the furnace, thus keeping the temperature nearly constant and putting little strain on the furnace coils.

The thermocouple was glass insulated, chromel-alumel, No.20 gauge wire. The insulation on the length of thermocouple within the furnace soon burned off, and was replaced with asbestos paper. The thermocouple was inserted in the well machined into the base of the pressure vessel.

The pressure vessel was inserted through the bottom of the furnace rather than from the top so that the seal remained relatively cool (Fig. 5). The top of the furnace chamber was closed by a plug except for a small hole for the thermocouple wires. This plug allowed rapid heating of the furnace and helped keep the temperature constant during the runs.
CALCULATIONS

For any gas pressure, the amount of an ideal gas required to generate this pressure at a given temperature can be calculated from the ideal gas law. To allow for the non-ideality of gases, it is necessary to use a compressibility factor, "z".

In this study the amount of gas was first calculated assuming a pressure of 1,000 atmospheres and a constant temperature in the pressure vessel chamber equal to the temperature at the thermocouple. When the temperature gradient along the chamber was determined, and the average temperature in the chamber estimated, accurate calculation of pressure could be made.

A) Determination of compressibility factor, z:

Fig. 6 is a chart showing compressibility factors of gases and vapors over a wide range of temperatures and pressures. To use the chart the reduced pressure $P_R$, and the reduced temperature, $T_R$ are required.

$$ P_R = \frac{P}{P_c} \quad \text{(1)} $$

$$ T_R = \frac{T}{T_c} \quad \text{(2)} $$

where: $T$, $P$ = assumed temperature and pressure

$T_c$, $P_c$ = critical temperature and pressure of the gas.

All temperatures are in K.

From Fig. 6, the intersection of $P_R$ and $T_R$ gives $z$.

B) Calculation of amount of gas; n

Ideal gas formula: $PV = nRT$

for non-ideal gases: $PV = znRT \quad \text{(3)}$

where: $P$ = desired pressure

$V$ = volume of pressure vessel

$z$ = compressibility factor
\[ n = \text{amount of gas} \]
\[ R = \text{gas constant} \]
\[ T = \text{assumed temperature} \]

Dimensions: \( P \ V = z \ n \ \frac{R \ T}{\text{atmos}(\text{cc}) = \left( z \right) \left( \text{gm-moles} \right) \ \frac{\text{cc-atmos}}{\text{\( ^\circ \)K}} \left( \text{gm-mole} \right)} \)

Therefore: \[ n = \frac{PV}{zRT} \] \( \ldots \ldots \ldots \ldots \) (4)
C) Calculation of actual pressure:

Because one end of the pressure vessel is outside the furnace, there is a temperature gradient along the chamber of the vessel. The temperature of the outside of the vessel near the seal was about 400° C. The temperature in the chamber at the seal is estimated to have been about 450° C. Since about half of the volume of the chamber, including the portion containing the charge, was probably at the indicated temperature, the average temperature in the vessel chamber is believed to have been close to the indicated temperature. For runs at 850° C, the average temperature is estimated to have been 750° C, and for runs at 925° C, the average temperature is estimated to have been 810° C.

From equation (1)

\[ P = P_R P_C \]........(1a)

Substituting in (4)

\[ n = \frac{P_R P_C V}{zRT_{av}} \]........(4a)

where : \( T_{av} \) = average temperature of chamber

Therefore: \( z = \frac{P_C V}{nRT_{av}} \frac{P_R}{P_R} \)........(5)

\( P_C, V, n, R \) and \( T_{av} \) are known;

Therefore: \( z \propto P_R \)

\[ T_R = \frac{T_{av}}{T_C} \]........(2a)

For any value of \( P_R \), a value for \( z \) can be calculated. Also, from Figure 6 or from tabulated values (e.g. Hougen, Watson and Ragatz, 1959, Table 49), another value of \( z \) is obtained from \( T_R \) and the arbitrary value of \( P_R \). From a number of arbitrary values of \( P_R \), two series of values for \( z \) are obtained. A plot of these two series of \( z \) against \( P_R \) gives a unique point of intersection at the true value of \( z \) and \( P_R \).
Substituting the unique value of $P_R$ in equation (1a) gives the actual pressure in the chamber (see example of calculations, section D).

D) Example of Calculations:

For run 2, a pressure of $\text{CO}_2$ of 1,000 atmos. was desired.

For $\text{CO}_2$: Molecular weight = 44

\[ T_c = 31.1^\circ \text{C}. \]

\[ P_c = 73 \text{ atmos.} \]

Temp. of charge = $850^\circ \text{C}$. 

a) \[ PR = \frac{1000}{73} = 13.7 \] \[ TR = \frac{1123.2}{304.3} = 3.70 \]

From Fig. 6, \( z = 1.23 \)

b) \[ n = \frac{PV}{zRT} = \frac{1000 \times 3.53}{1.23 \times 82.05 \times 1123.2} \]

\[ = 0.0311 \text{ gm-moles} \]

\[ = 0.0311 \times 44 = 1.36 \text{ gms. CO}_2 \]

To allow for the lower average temperature 1.50 gms. of $\text{CO}_2$ were used \( (n=0.0341 \text{ gm-moles}) \)

c) \[ z = \frac{P_cV}{nRT_{av}} P_R = \frac{73 \times 3.53 \times PR}{0.0341 \times 82.05 \times 923.2} \]

Actual reduced temperature, \( T_R = T_{av}/T_c \) \[ \quad \text{(2a)} \]

\[ T_R = \frac{923.2}{304.3} = 3.36 \]

<table>
<thead>
<tr>
<th>$P_R$</th>
<th>$z$ (eq. 5)</th>
<th>$z$ (Fig. 6)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.90</td>
<td>1.12</td>
</tr>
<tr>
<td>12</td>
<td>1.08</td>
<td>1.18</td>
</tr>
<tr>
<td>13</td>
<td>1.17</td>
<td>1.22</td>
</tr>
<tr>
<td>14</td>
<td>1.26</td>
<td>1.26</td>
</tr>
</tbody>
</table>

Point of intersection; \[ z = 1.26 \]

\[ P_R = 14 \]

Therefore: \( P = 14 \times 73 \) \[ \quad \text{(1a)} \]

\[ = 1020 \text{ atmospheres} \]
EXPERIMENTAL PROCEDURE

A) Materials:

1. Olivine: - collected from the Twin Sister dunite, Washington (Ragan, 1963). The olivine is clear, pale green, medium-to-coarse-grained. The dunite was ground to -100 mesh, 200 mesh and passed through a Frantz isodynamic separator to remove chromite and pyroxene.

2. Serpentinite: - a sample of serpentinized peridotite from the Lizard area, Cornwall (Green, 1964) was ground to -100 mesh, 200 mesh.

   In Runs 11 and 14 a sample of carbonate-bearing serpentine from Boston Bar, B. C. (Coquihalla serpentine belt) was used.

3. Carbon dioxide: - commercial dry ice. No attempt was made to keep the CO₂ from combining with atmospheric water. The effect of water would be to raise the total pressure slightly.


5. Sodium, potassium: - from their hydroxides (A. C. S. reagent grade).


B) Method:

A weighed amount of olivine or serpentinite (0.1 to 0.3 grams) was crimped in a platinum foil crucible. The crucible was slipped inside a silica glass tube and the tube was placed in the chamber of the vessel. All other components were dissolved in water or placed loose in the chamber. The filler rod and seal were put on and the cap tightened down hard on the seal with 18-inch wrenches.
The pressure vessel was inserted through an opening in the bottom of the furnace and held in place by a clamp attached to a stand. The thermocouple was placed in its well in the end of the vessel and the plug fitted into the top of the furnace chamber. The controller was set to the desired temperature and the line switches to it and the furnace was turned on.

The furnace reached the set temperature in about one hour. Runs were held at temperature for about 4 hours. At the end of a run the furnace was turned off and the plug taken out. After about thirty minutes the vessel was cool enough (below 500° C.) to be safely quenched in cold water. The vessel was then opened and the platinum crucible was recovered and opened. After each run the chamber of the vessel and the filler rod were cleaned thoroughly with solvent and hot water and then dried.

The material in the crucible, as well as any material seen on the filler rod was collected after each run, dried and examined. All olivine and serpentine charges were examined under oil with a petrographic microscope and then X-rayed. Products found on the filler rod were only X-rayed because of the small amounts present. X-ray powder photographs were taken using iron radiation and a manganese oxide filter. Cameras were 57.4 mm. diameter. All olivine X-ray powder photographs were read and the forsterite content determined from the d-spacing of the 174 back reflection (Jambor and Smith, 1964).
G) Experiments performed:

Group 1: Olivine - all runs 4 to 5 hours duration.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Volatile Components</th>
<th>Calc. Press. (atmos.)</th>
<th>Temp. of Charge (°C)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CO₂</td>
<td>890</td>
<td>850</td>
<td>Leakage</td>
</tr>
<tr>
<td>2</td>
<td>CO₂</td>
<td>1020</td>
<td>850</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>CO₂, H₂O</td>
<td>920</td>
<td>850</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>H₂O, Na</td>
<td>935</td>
<td>850</td>
<td>01 on filler rod</td>
</tr>
<tr>
<td>5</td>
<td>CO₂, S</td>
<td>890</td>
<td>850</td>
<td>Troilite and 01 on filler rod</td>
</tr>
<tr>
<td>6</td>
<td>H₂O, S</td>
<td>2100</td>
<td>850</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>H₂O, S, Na</td>
<td>2125</td>
<td>850</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>H₂O, Na</td>
<td>850</td>
<td>925</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>CO₂, H₂O, K, Na, CaCO₃</td>
<td>50</td>
<td>925</td>
<td></td>
</tr>
</tbody>
</table>

Group 2: Serpentinite

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Volatile Components</th>
<th>Calc. Press. (atmos.)</th>
<th>Temp. of Charge (°C)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>Lizard serp.</td>
<td>1</td>
<td>1150</td>
<td>2 hours</td>
</tr>
<tr>
<td>11</td>
<td>Coquihalla serp.</td>
<td>1</td>
<td>1050</td>
<td>Furnace coils failed</td>
</tr>
<tr>
<td></td>
<td>open crucible</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>Lizard serp.</td>
<td>905</td>
<td>850</td>
<td>01 on filler rod</td>
</tr>
<tr>
<td></td>
<td>CO₂</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>Lizard serp.</td>
<td>885</td>
<td>850</td>
<td></td>
</tr>
<tr>
<td></td>
<td>CO₂, H₂O</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>Coquihalla serp.</td>
<td>850</td>
<td>925</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H₂O, Na</td>
<td></td>
<td></td>
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</tbody>
</table>
RESULTS

Melts were not produced in any of the 14 runs.

In group 1, the olivines were unchanged physically and chemically. Under the microscope the grains still showed sharp, irregular edges caused by grinding the material. Compositions, determined from X-ray powder photographs, ranged from Fo89.1 to Fo93.5. This spread in compositions is thought to be largely due to reading errors. An average of the olivine compositions gives Fo90.0. This is in agreement with published compositions for Twins Sisters olivine. Ragan (1963) reports Fo90 by optical methods. Davis and Hess (1949) report Fo91.8 from a chemical analysis.

In group 2, the serpentinites were converted to olivine and a small amount of enstatite. A few grains of an orthopyroxene were observed under the microscope and X-ray powder photographs showed lines not belonging to olivine which corresponded to the enstatite pattern. No pyroxene was observed in thin sections of the serpentinites.

Scrappings from the filler rod in all runs but one proved to be olivine, which is believed to have been carried out of the crucible and deposited on the rod by the vapor. In Run 5 the deposit on the filler rod was determined to be troilite, FeS, plus olivine. The troilite formed by reaction of the sulphur in the fluid phase with iron of the filler rod.

Two runs at atmospheric pressure were attempts to duplicate a report by England and Adams (1951) that a serpentine began to melt at about 1150° C. No signs of melting were observed at 1050 or 1150° C. No further work could be done on this idea as
two furnace coils burned out at 1050 °C during the second attempt.

RECOMMENDATIONS

Although the results of this thesis were inconclusive, it is believed that much work remains to be done before the hypothesis of ultramafic magmas is abandoned.

Within the range of composition of the present experiments, the effect of higher pressures and temperatures up to the melting point should be examined. Other components may prove to be important in lowering the melting temperatures of ultramafic melts. Work done by Wyllie and Tuttle (1960, 1961, 1964) on the effects of HF, SO₃, P₂O₅, HCl and Li₂O on the melting temperatures of albite and granite showed that such components as HF, P₂O₅ and Li₂O, in combination with water, considerably reduced the melting temperatures at a total pressure of 2750 bars.

The effects of CO₂ and H₂CO₃ should be further investigated, possibly with experiments of longer duration. The experimental work on the solubility of olivine and serpentine in carbonic acid (Benson, 1918, p. 710) was done for periods of up to six months.

In any further work with equipment similar to that described in this thesis, the author believes that a gas pressure intensifier system would be more suitable than the present closed system of obtaining pressures. The intensifier allows more precise control of pressure and allows direct reading of pressures within the vessel during a run. The present vessel could be modified to work with an intensifier.
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