SOUND VELOCITY STUDY OF NATURAL ORTHOPYROXENES
WITH VARYING IRON CONTENT

BY

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THESIS

Submitted in partial fulfillment of the requirements for the degree of Master of Science in Geology in the Graduate College of the University of Illinois at Urbana-Champaign, 2015

Urbana, Illinois

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ABSTRACT

Determination of the acoustic wave velocities of orthopyroxene as a function of composition is essential to identify the underlying causes of seismic heterogeneity. Such laboratory measurements are essential to determine the structure, composition and temperature variability of the upper mantle. This study presents a systematic characterization of orthopyroxene elastic moduli as a function of composition in the enstatite-orthoferrosilite system. For each sample and composition, the sound velocities are measured by Brillouin scattering in three orthogonal crystallographic planes. Inverting sound velocity data allows determination of the complete elastic modulus tensor, as well as the adiabatic bulk ($K_s$) and shear ($\mu_s$) moduli. Samples were naturally occurring with approximate compositions: En90, En79, En74, and En55. The results establish nearly linear trends of $V_P$ and $V_S$ as a function of mole fraction En between the end members enstatite (Jackson et al 2007) and orthoferrosilite (Bass and Weidner 1984). Analysis of previous studies on orthopyroxene and the present work confirms that aluminum has the effects of increasing $K_s$ while also confirming no discernable effect from Ca substitution. $K_s$ is poorly correlated to Mg-Fe substitution whereas $\mu_s$ correlates in a more linear between the end-members. These findings will help better characterize chemical, namely Mg and Fe, heterogeneity in the upper mantle.
For Grandma and Papa
ACKNOWLEDGMENTS

This thesis is not the product solely of the author, but a collection of very dedicated research scientists at the University of Illinois at Urbana-Champaign, namely my adviser, Jay D. Bass and my coworker Jin Zhang. I’m greatly indebted to them for my growth as a research scientist during my time in graduate school. Your knowledge, support, and endless enthusiasm to help me succeed is, without question, the critical ingredient in my graduate career.

Additionally, my time in the Department of Geology contained many experiences external to my thesis work, but nonetheless instrumental in my development as a geophysicist. Particularly, I’m grateful to the collaborative efforts of the Donut’s and Dynamics group. Our weekly meetings have been a highlight since the group started meeting in the summer of 2014. I especially acknowledge Lijun Liu, Trish Gregg, Craig Lundstrom, Stephen Marshak, Willy Guenther, and Michael Stewart. Your comments were insightful and contributed greatly to the discussions of the Earth system. To my fellow students, Quan Zhou, Jiashun Hu, Vlad Iordache, and Kelsey Kehoe: you’ve helped make the discussion group an expanding and interesting forum to share our work and passion for the Earth with each other. Keep it going!

Without the support and friendship of Katelyn Zatwarnicki, Vincent Brice, and Kati Pryzybylski, I could not have accomplished this.
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CHAPTER 1: INTRODUCTION

1.1 Motivation and Overview

The upper mantle contains olivine, orthopyroxene, clinopyroxene, and garnet. Melt fractionation at mid-ocean ridges alters the chemical composition of these phases through production of an iron-rich melt and a magnesium-rich residue. This leads to a melt residuum more buoyant than fertile mantle. Over time, melting at ridges partitions a buoyant, depleted mantle below cratonic lithosphere as proposed by T. H. Jordan’s Tectosphere (1975). Over time, the depleted mantle cools and contracts. However, Jordan also recognized the buoyancy effect of melt depletion must counteract thermal subsidence to produce an isopycnic (neutrally buoyant) Tectosphere. Cratonic xenolith samples (T. H. Jordan 1979) indicate an enrichment of orthopyroxene and olivine, relative depletion of clinopyroxene and garnet, and an increase in Mg:Fe. Thus, a systematic study Mg and Fe compositional effects on the elasticity of orthopyroxene could help constrain seismic velocity evidence for the Tectosphere.

This study utilizes Brillouin spectroscopy to determine elastic properties of orthopyroxene as a function of Mg and Fe composition and presents the first elastic characterization of orthopyroxene with orthoferrosilite content greater than 20%. Utilizing natural samples help characterize effects other than Fe-Mg substitution and provide a more realistic characterization of orthopyroxene elasticity. From the analysis of five natural samples, thorough examination, and comparison of 11 other studies, this study concludes the bulk modulus of orthopyroxene is insensitive to the Mg:Fe ratio. Meanwhile the shear modulus is negatively, linearly related to increasing iron content. Both $V_P$ and $V_S$ are found to decrease linearly with increasing iron. Additionally, this study finds a strong relation between the Al content and the bulk modulus of orthopyroxene while the Ca content appears to have little or no
effect. These findings are crucial in forming accurate upper mantle petrology models to predict seismic velocities as a function of chemical heterogeneity.

1.2 Importance of Elasticity Measurements

Understanding structure in the interior of the Earth, requires the interpretation of seismic velocity variations and comparing it to a reference, such as the Preliminary Reference Earth Model (PREM) (Dziewonski and Anderson 1981). Mantle mineral models (Ringwood 1962, Bass and Anderson 1984) may be compared and constrained by individual phase elasticity and acoustic velocity. Because of the critical link between mineral and seismic properties, a general description of elasticity is provided using conventions of Nye (1985).

Elasticity describes the instantaneous, recoverable deformation of a body under the influence of stress. If a crystal experiences a stress less than its elastic limit, it will return to its initial state in accordance with Hooke’s Law:

$$\sigma = c \varepsilon$$  \hspace{1cm} Eq. (1)

Where $\sigma$ is an applied stress (generally compressive for geophysical concerns), $c$ is the elastic stiffness, and $\varepsilon$ is the strain. A generalized form for three dimensions is:

$$\sigma_{ij} = c_{ijkl} \varepsilon_{kl}$$  \hspace{1cm} Eq. (2)

This produces 81 coefficients, though 21 are unique. Symmetry properties within a crystal may further reduce the number of unique coefficients. For instance, an orthorhombic crystal produces a set $c_{ij}$ coefficients (presented in matrix notation):

$$
c_{ij} = \begin{pmatrix}
c_{11} & c_{12} & c_{13} \\
c_{21} & c_{22} & c_{23} \\
c_{31} & c_{32} & c_{33} \\
& c_{44} & \\
& & c_{55} \\
& & & c_{66}
\end{pmatrix}$$  \hspace{1cm} Eq. (3)
It can be shown, \( c_{ij} = c_{ji} \). Thus, the matrix simplifies further to:

\[
c_{ij} = \begin{pmatrix}
  c_{11} & c_{12} & c_{13} \\
  c_{22} & c_{23} \\
  c_{33} & c_{44} \\
  c_{55} & c_{66}
\end{pmatrix}
\]

\[Eq. \ (4)\]

Thus, an orthorhombic symmetry defines 9 independent elastic coefficients (Nye 1985). From this expression of elasticity in three dimensions, the aggregate Young’s modulus, bulk modulus, and shear modulus for an isotropic polycrystalline sample with randomly oriented grains may all be derived. For instance, the bulk modulus (volumetric incompressibility) and shear modulus (rigidity) are defined as:

\[
K_V = \frac{c_{11} + c_{22} + c_{33} + 2(c_{12} + c_{13} + c_{23})}{9}
\]

\[Eq. \ (5)\]

\[
\mu_V = \frac{(c_{11} + c_{22} + c_{33}) + 3(c_{44} + c_{55} + c_{66}) - (c_{12} + c_{13} + c_{23})}{15}
\]

\[Eq. \ (6)\]

For the Voigt bounds (Carcione 2015). Similarly, Ruess and Voigt-Ruess Hill averages may also be determined. These moduli are paramount to the equations for seismic wave (acoustic wave) propagation. Thus, determination of elastic stiffness of a material allows for more accurate interpretation of observed seismic remote sensing.

The seismic velocity equations rely on accurate knowledge of the bulk modulus and shear modulus of a material. The bulk modulus expresses the normalized volumetric contraction when
pressure is applied. The shear modulus gives a measure of material rigidity. These apply to the seismic velocity equations:

\[ V_P = \sqrt{\frac{K_S + \frac{4}{3} \mu}{\rho}} \]  \hspace{1cm} \text{Eq. (7)}

and

\[ V_S = \sqrt{\frac{\mu}{\rho}} \]  \hspace{1cm} \text{Eq. (8)}

Where \( V_P \) is the velocity of the seismic wave, \( V_S \) is the velocity of the secondary seismic wave (transverse acoustic mode), \( K_S \) is the adiabatic bulk modulus, \( \mu \) is the shear modulus, and \( \rho \) is the density of the medium, defined as:

\[ \rho = \frac{m}{v} \]  \hspace{1cm} \text{Eq. (9)}

Where \( m \) is the mass and \( v \) is the volume of the medium. Thus, the fundamental seismic wave equations rely on accurate determination of the elasticity and density of the material the through which the waves propagate.

### 1.3 Solid Solution and Phase Relations

In the natural world, many minerals exist as a solid solution – two components which completely mix in a system. For instance, orthopyroxene and olivine may each be thought of as a two-component system of MgSiO₃-FeSiO₃ and Mg₂SiO₄-Fe₂SiO₄, respectively. Most natural orthopyroxene exists as a solid solution of \((\text{Mg},\text{Fe}_{(1-x)})_2\text{Si}_2\text{O}_6\). Consideration of expected phases
and compositions at varying conditions, requires an equilibrium phase diagram. See Figure 1.1 for an example of an equilibrium phase diagram for the orthopyroxene system. Experimental petrologists, like Norman Bowen, established phase relations for many systems in the early part of the 20th century. At atmospheric pressure, observing phase and composition relations with varying temperature could establish how magmas and parent rock evolve over sequences of heating, cooling, and partial melting or crystallization.

Fractional crystallization is a fundamental paradigm of igneous petrology. Within in this framework, the melt and residuum can become isolated systems and not react to any appreciable extent. Fractional melting is the equivalent term for a melting system (Presnall 1979). In the simplest terms, melting a source rock containing orthopyroxene and olivine is a three component system of \( \text{MgO-FeO-SiO}_2 \) at a given pressure (Figure 1.2). Experiments on this melting in the \( \text{MgO-FeO-SiO}_2 \) show Mg-rich phases (Forsterite, Enstatite) are in equilibrium with a liquid at greater temperature than their Fe-rich counterparts (Fayalite, Ferrosillite) (Bowen and Schairer 1935). Considering a partially melting system, the Mg-rich phase would compose the residue in equilibrium with an Fe-rich melt. This is typified by the iron-enrichment trend of Tholeiitic basalts (like MORB). Thus, melting in the Earth’s upper mantle produces an Mg-rich residuum and Fe-rich liquid. Noting \( \rho_{\text{Mg}} < \rho_{\text{Fe}} \), this melting system produces a less dense residuum.
Figure 1.1
Schematic equilibrium phase diagram of the orthopyroxene solid solution. After Bowen and Schairer (1935).
Figure 1.2
The MgO-FeO-SiO$_2$ System (Bowen and Schairer 1935). Temperature is decreasing in the direction of the arrows. This illustrates liquids evolving along a thermal gradient will be enriched in Fe end members while Mg end members will form the residue. This holds for both olivine and orthopyroxene.
1.4 Magnesium Number and Melt Depletion

Common chemical analyses of rocks and minerals report composition as oxide weight percent (Na₂O, K₂O, Al₂O₃, Fe₂O₃, FeO, MgO, SiO₂). For primitive mafic systems, FeO and MgO ratios are of particular interest as other oxide components fluctuate little, relative to MgO, in the early stages of magma differentiation. In general, melting produces a refractory residue with a higher MgO content (lower Fe content = [Fe]/[Fe]+[Mg]) than the liquid. This is due to the phase relations of both olivine and orthopyroxene Fe-Mg solid solutions discussed in Section 2.0.2. However, strict comparison of MgO ignores the crystallization of phases which contain no Mg, namely plagioclase and magnetite. To make a more useful interpretation of composition, Mg is compared in relation to Fe. This is known as the Mg#:

\[
Mg\# = \frac{[MgO]}{[MgO] + [FeO]} \tag{10}
\]

This ratio is unaffected by the crystallization of plagioclase. However, crystallization of magnetite may alter or reverse trends observed. Nonetheless, to the first order observation, a higher Mg# implies a more primitive material while a lower Mg# implies a greater amount of melt removal (Cox, Bell and Pankhurst 1979).

1.5 Orthopyroxenes

Orthopyroxene is a subgroup of the Pyroxene group of the chain silicates and contains species in the orthorhombic crystal system and Mg-Fe solid solution with the chemical formula, \((Mg, Fe)_2Si_2O_6\). Mineral names depend on composition as follows:

Enstatite: \(Mg_{(0.88-1.0)}Fe_{(0.12-0)}SiO_3\)
Bronzite: \( \text{Mg}_{(0.70-0.88)}\text{Fe}_{(0.30-0.12)}\text{SiO}_3 \)

Hypersthene: \( \text{Mg}_{(0.50-0.70)}\text{Fe}_{(0.50-0.30)}\text{SiO}_3 \)

Ferrohypersthene: \( \text{Mg}_{(0.30-0.50)}\text{Fe}_{(0.70-0.50)}\text{SiO}_3 \)

Eulite: \( \text{Mg}_{(0.12-0.30)}\text{Fe}_{(0.88-0.70)}\text{SiO}_3 \)

Orthoferrosilite: \( \text{Mg}_{(0.12-0.30)}\text{Fe}_{(1.0-0.88)}\text{SiO}_3 \) \hspace{1cm} \text{(Deer, Howie and Zussman 1972)}

For simplicity, hypersthene may be used for all intermediate compositions. All intermediate compositions maintain the Pbca space group with 3 mirror planes, each with two-fold rotational symmetry. Tetrahedra and octahedra form alternating layers within the (100) plane. Each sharing two corners, tetrahedra (TO\(^2\)) form chains extending along the \(c\) axis with their bases oriented subparallel to the (100) plane. Cations in octahedral \(\text{M}_1\) sites are coordinated with 6 anions while \(\text{M}_2\) sites may coordinate with 6, 7, or 8 anions. In edge sharing configuration with other octahedra, the \(\text{M}\) sites form discontinuous beams in the (100) plane extending along \(c\) (Cameron and Papike 1981). See Figure 1.3-1.5 for structural diagrams of Enstatite projected along \(a\), \(b\), and \(c\) (Downs and Hall-Wallace 2003). From these figures, it can be determined each unit cell contains 8 formula units (\(Z = 8\)). Cleavage forms parallel \(\{210\}\) with partings in \(\{100\}\) and \(\{010\}\) which may form from diopside lamellae (Deer, Howie and Zussman 1972). Refractive indices and density correlate linearly with increasing Fe composition (Deer, Howie and Zussman 1972).

Orthorhombic symmetry implies there are 9 unique, non-zero components of the \(c_{ij}\) stiffness coefficients. Elasticity is influenced by a slight \(\text{Fe}^{2+}\) affinity for the \(\text{M}_2\) site. Thus, one might expect that along the En-Fs join, Fe-Mg substitution should have a strong effect on \(c_{22}\) relative to \(c_{11}\) and \(c_{33}\). (Bass and Weidner 1984).
Figure 1.3
Enstatite structural model as viewed down $a$. $M_1$ sites in green. $M_2$ sites in red. Unit cell outlined in grey. Rendered with XtalDraw (Downs and Hall-Wallace 2003).

Figure 1.4
Enstatite structural model as viewed down $b$. Unit cell outlined in grey. Rendered with XtalDraw (Downs and Hall-Wallace 2003).
Figure 1.5
Enstatite structural model as viewed down \( c \). Unit cell outlined in grey. Rendered with XtalDraw (Downs and Hall-Wallace 2003).
1.6 Pyrolite Model of the Upper Mantle

After nearly a century of experimental petrology and seismic imaging, upper mantle composition remains undetermined. Primary indicators of upper mantle mineralogy include experiments on midocean ridge basalts (MORB) and more fertile candidate mineral assemblages, and natural samples of mantle xenoliths from Kimberlite pipes. Experiments assert the upper mantle contains a mineral assemblage which can, upon partial melting, form MORB. Assuming the average upper mantle composition is similar beneath oceans and continents, Ringwood (1962) recognized a mixture of 20% basalt and 80% dunite corresponds to oxide composition analysis of chondritic bulk silicate earth models. This mixture is called pyrolite—a theoretical rock composition similar to a lherzolite. At ambient upper mantle conditions, pyrolite contains a mineral assemblage of olivine, orthopyroxene, and clinopyroxene. However, beneath continents with low heat flow (particularly Archean cratons) where a cooler geotherm is predicted, iron-rich garnet (pyrope) is stable. Bulk chemical composition would remain the same provided the aluminum components are accommodated by spinel and plagioclase at lower pressures (Frost 2008). Thus, beneath stable continental cratons, a garnet (or spinel) pyrolite is expected as the principal mantle rock (Ringwood 1962). As predicted by the pyrolite model, mantle xenolith samples from Kimberlite pipes in cratons contain garnet lherzolites (equivalent to theoretical garnet pyrolite), and within the ocean basins, peridotite xenolith samples, approximate a system with an extracted basaltic component (Ringwood 1991). Jordan (1979) also explored the continental xenolith sample in great detail and its implications are discussed in Section 2.3. The piclogite model of the upper mantle proposes a lower olivine content which better matches seismic velocity data in the transition zone (Bass and Anderson 1984). However, pyrolite is
consistent with shallow (<400 km) upper mantle seismic data and cratonic xenolith inclusions in kimberlite.

Assuming a pyrolite composition, petrologists have examined phase and composition relationships as a function of partial melting. By definition, partial melting of a pyrolite produces a basaltic liquid. To estimate an oxide composition, several studies examine petrogenetic relationships between MORB and Harzburgite, komatiities and dunités, and lherzolites. Compositions determined from different relationships were in close agreement:

\[
\begin{align*}
\text{SiO}_2 &= 44.87 \pm .2 \\
\text{CaO} &= 3.46 \pm .05 \\
\text{MgO} &= 38.36 \pm .05 \\
\text{FeO} &= 7.93 \pm .3
\end{align*}
\]

Of particular interest, Mg\# = 89.6 for these model mantle compositions. As discussed in Section 2.0.3, the Mg\# establishes a proxy for melt depletion. Considering an extensive analysis of mantle samples, undifferentiated upper mantle rock should have an Mg\# \(\approx 89\). Greater amounts of melt depletion would produce a proportionally lower Mg\# parent rock. After establishing compositions and comparing with seismic data, a pyrolite assemblage of 61\% olivine, 14\% garnet, 15\% orthopyroxene, and 10\% clinopyroxene fits upper mantle conditions best (Ringwood 1991, Duffy and Anderson 1989).

Mantle petrologists must consider the consequences of a pyrolite model for the upper mantle. Discovery of a low velocity zone near 150 km in the early 1960’s and subsequent interpretation as a region of partial melt, prompted investigation by A.E. Ringwood to model the nature of upper mantle mineralogy. Without direct sampling, a petrologic model needed to satisfy seismic observation and melt to produce a basaltic composition. Holding all else equal,
seismic velocity decreases as a material is heated and increases as a material is compressed. Thus, a melting point gradient within the Earth may be extrapolated from seismic data and pressure calculations. Pyrolite, a 20% basaltic component and 80% dunite component, best addresses these observations. Partial melting of pyrolite produces basaltic magma and peridotite residue less dense than the fertile pyrolite. Within this model, residual mantle would not remix with fertile mantle, and would create a density stratification in the upper mantle. Over time, low density residue would accumulate below even lower density continental material. Chemical zoning would produce different mineral assemblages under cratons and ocean basins. Consistent with this model, cratonic mantle xenoliths in Kimberlite pipes contain an assemblage of garnet pyrolite, and MORB xenoliths contain plagioclase pyrolite. The differing source regions for these materials implies a temperature and pressure-induced transition between garnet pyrolite and plagioclase pyrolite. Indeed, mid-ocean ridge melting schemes along a mantle geotherm require the melting of plagioclase pyrolite rather than garnet pyrolite. Such a model in the oceanic upper mantle, would produce a seismic low velocity zone where adiabatic decompressive melting of plagioclase occurs – the asthenosphere. Precambrian shields exhibit very low heat flow. This implies the geotherm beneath cratons never reaches the plagioclase pyrolite stability region. Thus, the subcratonic mantle should be a garnet pyrolite stability field with no low velocity observed (Ringwood, A Model for the Upper Mantle 1962). Establishing an upper mantle petrologic model also implies geophysical behavior which must be consistent with observation. Accurate determination of density and single-crystal elastic moduli can more accurately constrain chemical composition and heterogeneity. To date, experiments on olivine elastic moduli as a function of iron composition, temperature, and pressure are better constrained at upper mantle conditions with bulk modulus errors of ~1.5% and shear modulus errors of 4%
To a lesser extent garnet, the elastic moduli of orthopyroxene, and clinopyroxene are well determined for end-member compositions for temperature and pressure. However, significant elasticity variations between species of these phases introduces much greater error bounds. For instance, Mg-Tschermak’s orthopyroxene estimated errors are 9% for the bulk modulus and 11% for the shear modulus (Stixrude and Lithgow-Bertelloni 2005). Melting phase relation models of the upper mantle with a pyrolytic composition typically rely on end member compositions alone, and poor accounting of complex species can produce a significant portion of error (Schutt and Lesher 2006).

1.7 The Tectosphere

The concept of the Tectosphere draws upon observations and concepts from geophysics, petrology, and mineralogy as established in prior sections. Jordan postulated that the chemical variation modeled by A.E. Ringwood manifested itself through geologic time as a cohesive Tectosphere or deep continental root. Cratonic heat flow and seismic velocity data indicate that chemically differentiated mantle may extend to 400 km or deeper (T. H. Jordan 1975). However, these observations require a mechanism to stabilize the lower cratonic mantle geotherm to great depths without convective overturn. The age versus depth relation of the sea floor implies the lithosphere-asthenosphere boundary coincides with the mantle solidus. Combining sea floor topography and seismic data, the thermal profile may be considered adiabatic below 100 km with a gradient of 0.6°C/km (T. H. Jordan 1975). In the ocean basins, horizontal variations in ambient mantle temperature are assumed negligible.

In relation to the continents, particularly cratons, heat flow measurements require a thermal boundary layer at least twice the thickness of ocean basins (200 km) – assuming the mantle below the thermal boundary layer is adiabatic and convective. This implies observed
shear velocity variations between 100-200 km are due to chemical heterogeneity rather than thermal. However, geothermometry of pyroxenes in kimberlite mantle xenoliths has shown subcratonic mantle temperatures are significantly depressed from the suboceanic. Thus, lower temperatures suggest a colder subcratonic mantle with greater viscosity, density, and shear velocity. Considering these data, the thermal boundary layer between subcratonic mantle and the ambient mantle may extend to a depth of 400 km. In contrast to the oceanic lithosphere coinciding with the thermal boundary layer, the cratonic thermal boundary layer would coincide with the boundary of the Tectosphere. However, this would produce a convective instability. Given the billion-year stability of cratons, a counteracting mechanism must exist.

To counteract thermal subsidence, Jordan (1978) sought to provide an opposing buoyancy force to create an isopycnic (uniform density) Tectosphere. Considering the differing geothermal gradients, mantle temperatures 150-200 km beneath a craton may be 300-500 K cooler than in the ocean basins. Thus, effects on density from thermal expansion could be countered by a 0.9-1.5% decrease in density by chemical depletion below the cratons (T. H. Jordan, Composition and development of continental tectosphere 1978). Garnet lherzolite xenoliths from kimberlite pipes are thought to be the best sample of cratonic mantle. Kimberlite systems are volatile-rich, mantle-sourced magmas with rapid eruption rates and a general association with cratons. Therefore, kimberlites are a unique tool to sample the upper mantle. It remains unclear if the sampled mantle is indeed average cratonic mantle or tapping a localized kimberlite source region. However, this bias would be towards a fertile upper mantle. If such a bias exists, any conclusions are muted from the natural systems. Analysis of 78 such continental garnet lherzolites (equivalent to theoretical subcontinental pyrolite) confirms the average density could overcome thermal contraction. Normative mineralogy comparisons based on oxide weight
percent of theoretical pyrolite, St. Paul’s Rocks (spinel peridotite), calculated Hawaiian basalt parent rocks, and spinel hherzolite xenoliths, confirm the average continental garnet lherzolite (ACGL) is 1.3% less dense than model pyrolite composition. For the ACGL, Mg# = 91.1. 

Further, a linear decrease in density with increasing Mg# was observed (T. H. Jordan 1979). Additionally, normative mineral calculations for ACGL suggest ~10% less garnet and clinopyroxene and relative enrichment in orthopyroxene and olivine. Olivine, orthopyroxene, and garnet are all important carriers of iron and magnesium for the depths and pressures relevant to the Tectosphere hypothesis. (T. H. Jordan 1979). This makes iron and magnesium variation in olivine, orthopyroxene, and garnet an ideal candidate for testing the plausibility of the Tectosphere.

Considering the garnet pyrolite model, basalt genesis produces a residue less dense than the primitive material. Early in Earth’s history, depletion of basalt components would partition less dense material to below convectively stagnant cratons. Remaining laterally cohesive over billions of years, the Tectosphere explains the long-term stability of cratons. Basalt depletion is expected to lower garnet and clinopyroxene content of the residuum as well as Mg#. Thus, the Tectosphere is a self-consistent hypothesis to explain the stability of cratons over geologic time with implications for the upper mantle’s chemical and thermal variation. At present, the Tectosphere remains plausible but unverified. Observation of mantle major element composition through seismic sensing would provide a means to verify to existence of the Tectosphere. In order to utilize seismic imaging, a more clear relation of Mg/Fe composition and single-crystal elastic moduli must be determined. Any remaining perturbation in seismic velocity could then be assigned to thermal effects.
Figure 1.6
Generalized Tectosphere Model after Jordan (T. H. Jordan 1978). General properties are given. The Tectosphere comprises brown regions representing relative depletion of basaltic components (dark brown, more depleted).
1.8 Modeling composition and temperature in the Upper Mantle

For decades, geophysicists have sought to quantify the physical properties of the upper mantle. Of particular interest, composition and temperature relate directly to mantle density. Compositional variation, pressure, and temperature will alter the elastic properties of a given mineral component of the upper mantle. Composition influences the density of the mineral through the differences in atomic masses of substituting components. However, this also influences the volume of the unit cell. In the En-Fs system, complete substitution of Fe for Mg increases density ~25%. Further, increasing pressure reduces unit cell volume and increases density. Thermal expansion increases the volume at constant mass and thus decreases density. Each effect must be accounted for to realistically model the Earth’s dynamic mantle.

One of the most influential dynamic processes occurring in the upper mantle is mid-ocean ridge melting. Melting at the ridge produces a residuum more buoyant than the parent rock. This is a foundation for the Tectosphere hypothesis discussed in Section 1.7. Schutt and Lesher (2006), Matsukage (2005), and Lee (2003) have modeled the effects of melt depletion on seismic velocity. Using end-member elastic moduli, Jordan (1979) calculated seismic velocities based on the average continental garnet lherzolite, pyrolite, and an alkali olivine basalt. Partial melting (20%) of a basaltic component from pyrolite produces a .1% increase in P-wave velocity in the residue relative to the parent rock while S-wave velocity increased .5%. As predicted, 20% partial-melting decreased density 1.6% in the residue. These early models are consistent with an isopycnic Tectosphere. However, modeling is dependent on the accuracy of the parameters used. Utilization of end-member composition data relies on unverified trends expected in intermediate compositions.
Modeling the melting of spinel and garnet lherzolite requires accurate knowledge of phase relations as melt is extracted. Model compositions reflect pyrolitic models (discussed in Section 2.1). Phases progress from a fertile lherzolite, to harzburgite, and depleted dunite. As partial melting removes a greater proportion of parent mantle, the residual Mg# increases for both orthopyroxene and olivine components. Bulk rock density is largely controlled by Fe removal in olivine and orthopyroxene. At greater depths, greater melt extraction produces less of an effect on the Mg# of olivine, orthopyroxene, and clinopyroxene, and thus density (Schutt and Lesher 2006). Current end-member mineral data gives a 5.6% (Stixrude and Lithgow-Bertelloni 2005) decrease in adiabatic bulk modulus, and 25% increase in density from Enstatite to Orthoferrosilite (Schutt and Lesher 2006). Forsterite to Fayalite produces a 5% increase in adiabatic bulk modulus (Stixrude and Lithgow-Bertelloni 2005) along with a 26% increase in density (Schutt and Lesher 2006). Realistic proportions in the Earth could produce a 1.3% density contrast between low Mg# parent material and high Mg# refractory material (T. H. Jordan, Composition and development of continental tectosphere 1978). Schutt and Lesher (2006) found density decreases of similar magnitude with the most pronounced effects between 3.5 GPa and 6 GPa.

Temperature variations in the mantle may accompany basalt depletion of garnet lherzolite. Removing basalt components increases the solidus and therefore heat capacity of the refractory material. Seismic shear wave velocities decrease as temperature approaches the solidus of the transit material. This effect is less in Vp. Therefore, shear velocity is frequently considered to infer mantle thermal anomalies with decreasing shear wave anomalies corresponding to higher temperatures (closer to solidus temperature). Currently, discerning
between thermally or chemically induced seismic velocity variation is poorly constrained in the orthopyroxene component.

Models indicate 30% mantle depletion at 110 km and 10% partial melting below 125 km can stabilize the cratonic lithosphere, creating the hypothesized isopycnic Tectosphere (Schutt and Lesher 2006). Melt depletion causes a seismically detectable increase in velocity of the residue of .25% per 10% melt depletion of a spinel peridotite. This effect is unresolvable for garnet peridotite. This is much lower than previous estimates of 1% velocity increase per 10% melt depletion. Thus, seismic velocity variation due to major element composition is likely lower than 1%. For this model, orthopyroxene constitutes nearly 20% of the total error in estimating the whole rock bulk modulus (Schutt and Lesher 2006). Many other studies (Claire Perry and Forte 2010) confirm shear velocities best reflect temperature variations. To complement the great deal of work modeling composition, temperature, and pressure effects on upper mantle shear velocities, shear velocities for orthopyroxene as a function of Fe (a proxy for melt depletion) must be determined.

1.9 Brillouin Scattering

From the quantum theory of physics, light energy propagates as quantized packets of energy, known as photons. When light interacts with a medium, photons may transmit through it, reflect off of it, or transfer heat energy to it. Brillouin scattering is concerned the thermal effects causing vibrations called phonons in a medium.

Brillouin light scattering relates a sound wave vector of a dielectric crystal to a light wave vector, according to the equation:

\[ 2|k_s| \sin \frac{\theta}{2} = |q| \]  

Eq. (11)
where \( k_s \) is the wave vector of the scattered light, \( q \) is the wave vector of the phonon (acoustic wave), and \( \theta \) is the scattering angle. The absolute values relate to the Stokes and anti-Stokes vectors of a phonon. In the Stokes case, phonon creation from interaction with the incident light scattering in a dielectric solid, reducing the magnitude of \( k_s \). Meanwhile, the magnitude of \( k_s \) increases during the anti-Stokes event as phonon annihilation transfers energy to the scattered light. This process is analogous to the Doppler shift in classical physics (Sandercock 1982, Speziale, Marquardt and Duffy 2014). Combining this relationship with Snell’s Law, this equation can determine the velocity of the phonon:

\[
v = \frac{\Delta \nu |\lambda|}{2 \sin \theta}
\]

Eq. (12)

Where \( \Delta \nu \) is the frequency shift of the scattered light, \( \lambda \) is the wavelength of incident light, and \( \theta \). Utilizing platelet symmetry simplifies the geometry of scattering light and does not require precise knowledge of the refractive indices of a sample. Further, utilizing 90° scattering geometry simplifies the acoustic velocity equation to:

\[
v = \frac{\Delta \nu \lambda}{\sqrt{2}}
\]

Eq. (13)

Thus, an acoustic velocity through a medium is determined with the scattering of light (Whitfield, Brody and Bassett 1976). See Figure 2.5 for a schematic geometry of 90° platelet symmetric Brillouin scattering.

The frequency shift produced by photon-phonon interaction can be measured. A Fabry-Perot interferometer allows such measurements. The most basic interferometer consists of two highly parallel mirrors with a variable distance, \( L_1 \) (referred herein as plate spacing). This allows the discrimination between light with wavelength, \( \lambda_1 \), and \( \lambda_1 + \Delta \lambda \) according to:
\[ L_1 = \frac{n\lambda_1}{2} \]

\[ n\lambda_1 = (n - 1)(\lambda_1 + \Delta\lambda) \]

where \( n \) is an integer. This also means the plate spacing parameter of the spectrometer directly impacts the resolution of the instrument. This is the simplest description of the concepts utilized by a Fabry-Perot interferometer (Sandercock 1982).

Improvements to the system allow for a greater ability to analyze small shifts in wavelengths. Increasing the intensity of shifted light relative to noise, or contrast, is critical to resolve small differences between wavelengths with a Fabry-Perot interferometer. Creating a light path which travels multiple times (multi pass) through the same set of mirrors increases the contrast (or signal-to-noise ratio) many times over a single-pass instrument. Using a stabilizing, piezoelectric transducer allows small changes in the plate spacing to scan over >5000 Å.

Likewise, a strong reference peak allows electronic stabilization to maintain parallel mirror alignment. Creating a tandem system (utilizing two sets of parallel mirrors) further increases the contrast, by suppressing neighboring orders of light (Sandercock 1982).
Figure 1.7
Light paths for 90° platelet symmetric scattering. $\vec{k}_i$ indicates the vector of incident, reflected, transmitted, and scattered light. $\vec{q}$ indicates the phonon vibration directions with wave fronts normal to this vector shown in blue. After Whitfield, Brody, and Bassett (1976).
CHAPTER 2: METHODS

2.1 Overview

Chapter 1 motivated a systematic characterization of orthopyroxene elasticity as a function of Mg:Fe ratio. Sample descriptions of the five, natural orthopyroxenes are provided with chemical compositions including Mg, Fe, Ca, and Al cations (Section 2.2). Sample preparation techniques of crystals are discussed with particular attention to polishing planes normal to the c axis (Section 2.3). Elasticity analysis was performed with Brillouin spectroscopy – an excellent tool to determine single-crystal elastic properties (Section 2.4). Seismic velocity calculations follow programs well established in the mineral physics literature (Section 2.5). Together, these methods provide a thorough determination of the orthopyroxene elasticity and its relation to composition.

2.2 Sample Description

Tanzania Orthopyroxene

Tanzania (OPX TANZ) orthopyroxene samples are from the Morogoro Region with composition \((\text{Mg}_{0.897}\text{Fe}_{0.097}\text{Ca}_{0.004}\text{Al}_{0.002})_2(\text{Si}_{0.996}\text{Al}_{0.002})_2\text{O}_6\) (Zhang, Reynard, et al. 2013). Specimens are of high optical quality with no detectable inclusions or lamellae by optical polarized light microscopy. All sample sections were ground and then polished with abrasive films of successively smaller grit sizes down to 0.3 µm. Transmission of laser light \((\lambda=514.5 \text{ nm})\) through the crystals produced no observable scattering, allowing for crisp focus of incident laser light and excellent quality Brillouin spectra.

Bamble Orthopyroxene
Bamble, Norway (OPX BAM) orthopyroxene samples are from the Bamble metamorphic region with composition (Mg$_{0.849}$Fe$_{0.142}$Ca$_{0.005}$Al$_{0.002}$)$_2$Si$_2$O$_6$ (Goldman and Rossman 1979). This sample contains a significant number of sheet-like inclusions aligned roughly parallel to the (100) plane (Figure 3.X). Samples were polished down to 1 µm surface roughness. Areas free of inclusions produced excellent quality Brillouin spectra. However, intense scattering of λ=532 nm laser light prohibited collection of Brillouin spectra in the (001) plane.

**Labrador Orthopyroxene**

Labrador, Canada (OPX LBCA) orthopyroxene samples are from the Nain anorthosite complex with composition (Mg$_{0.79}$Fe$_{0.18}$Ca$_{0.03}$)$_2$Si$_2$O$_6$. Specimens include plate-shaped inclusions aligned parallel to the (100) plane. Sample sections (100) and (010) were polished to .3 µm. Difficulty of polishing Labrador (001) required a large sample dimension, approximately 1300 µm x 1000 µm x 250 µm polished to 3 µm surface roughness. Scattering of 514.5 nm and 532 nm laser light required long collection times (> 40 min). Although the signal/noise was relatively high for this data set, Brillouin peaks were clearly discernable.

**Bushveld Orthopyroxene**

Bushveld, South Africa (OPX BUSH) orthopyroxene samples are from the pyroxenite marker of the Upper Zone of the Bushveld Complex. OPX BUSH has composition (Mg$_{0.695}$Fe$_{0.266}$Ca$_{0.029}$)$_2$(Si$_{0.979}$Al$_{0.019}$)$_2$O$_6$. Sample sections were polished to .3 µm. Scattering of 532 nm laser light required long collection times (> 40 min), and the signal/noise was excellent. Brillouin peaks were clearly identifiable.
Summit Rock, Oregon Orthopyroxene

Summit Rock, Oregon (OPX SRO) orthopyroxene is from an andesitic host rock from the Cascades with composition \((\text{Mg}_{0.550}\text{Fe}_{0.395}\text{Ca}_{0.035}\text{Al}_{0.005})_2\text{Si}_2\text{O}_6\). (Figure 3.X2). Euhedral clear brownish single crystals are abundant and easily polished to .3 \(\mu\)m surface roughness. No inclusions were observed in any samples. Cutting normal to the \(c\) axis with the Princeton Wire Saw W22 produced a high quality sample in the (001) plane. Both 514.5 nm and 532 nm laser light was transmitted through each of the orthogonal sections with no observed light scattering. The quality of the Brillouin spectra was excellent.

2.3 Sample Preparation

Complete characterization of the elastic moduli for a sample with orthorhombic symmetry requires three orthogonal sections of the sample. Thus, this experiment requires three sample sections for each of the sample. Utilizing platelet symmetry requires two parallel, polished faces of each sample section – a total of 30 polished faces for this experiment.

Polishing samples into a platelet morphology proved more challenging than initially expected. In general, (100) and (010) planes polish with relative ease. In some cases, euhedral crystal forms allows approximate orientation by morphology. Samples in the (001) direction are normal to (100) parting of orthopyroxene. Thus, as the sample is polished, stress propagates through the sample – splitting it into thin sheets. The parted sheets have platelet geometry orthogonal to the desired orientation. See Figure 3.1. Parting occurred during cutting with a 300 \(\mu\)m wire saw at Materials Research Laboratory and the Brillouin lab’s 200 \(\mu\)m wafering saw. The strong parting of orthopyroxenes rendered conventional abrasive sawing unfeasible. The Princeton Wire Saw W22 utilizes a tungsten wire (diameter 30-60 \(\mu\)m) and an abrasive slurry to cut samples with little pressure on the sample surface. This tool is capable of cutting two parallel...
slices normal to the $c$ axis with minimal material loss, resulting in surfaces that required minimal polishing.

Once polished, samples are mounted onto a thin glass fiber (~100 µm) with UV-curing epoxy and placed into a goniometer. The orientation of each sample was determined by the George L. Clark X-Ray Facility using single-crystal X-Ray diffraction with a Bruker diffractometer. This method provides both an orientation matrix and the unit cell parameters. Using the orientation matrix the crystallographic identity of the polished faces was determined by optical goniometry. Specular reflections of a laser beam onto a pinhole were used as the optical goniometer.
Figure 2.1: Summit Rock Oregon orthopyroxene, looking down the c axis. Two parting planes are highlighted, approximately (100) and (-100).
2.4 Experimental Setup

This study utilizes Brillouin light scattering to analyze the acoustic velocities of natural orthopyroxenes with varying composition. Natural samples generally contain inclusions and other defects. Thus, a high-contrast Brillouin system with high resolution is required to observe the Brillouin scattered signal.

The Brillouin Spectroscopy Lab at the University of Illinois, Urbana-Champaign, utilizes a six-pass tandem Fabry-Perot interferometer (TFP), designed and manufactured by JRS Scientific Instruments. Benefits of the multi-pass and tandem system discussed in Section 2.5. The interferometer has an active vibration isolation system, also designed and manufactured by JRS Scientific Instruments. Vibration isolation virtually eliminates ambient mechanical noise. Laser light is produced by a solid-state, 532 nm Torus laser (Laser Quantum). The laser is tuned from the factory for single mode emission. Two optical paths provide the spectrometer with a reference light beam for stabilization of the TFP, and a separate path for light scattered by the sample. Light is detected with an avalanche photo-diode (APD) and accumulated with a multi-channel scaler (1024 channels). For a detailed drawing of the system, see Figure 3.2.

Sample sections are mounted onto a 3-circle, Eulerian cradle to allow for rotation along χ while maintaining platelet symmetric scattering geometry. For these experiments, an incident beam hits the sample 45° to the normal to the surface for the 90° scattering geometry used in these experiments. The precise external scattering angle (~90) is determined by Brillouin velocity measurements on silica glass that has been calibrated by ultrasonic interferometry (Steven Jacobsen, Northwestern University (Zhang, Bass and Taniguchi, et al. 2011). Scattering angles in this set of experiments range from 89.50° to 91.28°. Brillouin frequency shifts are measured every 15° over a range of 180° on one surface of the platelet. Measurements are then
made with the incident and scattered faces reversed (that is, with phi rotated by 180 degrees), but offset in chi by 7.5° relative to the first set of measurements. This offset provides a denser data set and redundancy in measurements. Also, any systematic differences in measurements for the two sample orientations can reveal possible systematic errors in the measurements. This process is continued for three orthogonal sections of a sample. Hence, for a typical sample 78 spectra are collected. With each spectrum containing at least two modes, 156 velocities are measured for each sample.

Each spectrum was individually analyzed. Peak positions were taken to be at the half-width at half maximum of a peak. The free spectral range calibration analyzed peak positions of light passing only through TFP1. This provides an accurate measure of the number of channels corresponding to a half wavelength of light. Plate spacing of 5 mm provided enough free spectral range to image both primary and secondary Brillouin shifts. Experiments conducted with an Ar laser at 514.5 nm used a scan amplitude of 550. Experiments conducted at 532 nm used a scan amplitude of 580. All data described was compiled for analysis via VEL-LAST (Vaughan and Bass 1983), ORTHO90 (Weidner and Carleton, Elasticity of Coesite 1977), and POLYXSTL (Watt 1987).
Brillouin Spectroscopy System in the UIUC Department of Geology

Figure 2.2: Illustration of the UIUC Department of Geology’s Brillouin spectroscopy system.
Figure 2.3: Illustration of raw Brillouin spectrum collected from the (100) plane of Tanzania orthopyroxene.
Features of a Raw Brillouin Spectrum

Figure 2.4: Illustration of raw Brillouin spectrum collected from the (100) plane of Tanzania orthopyroxene with notable features identified for the reader.
2.5 Calculations

Velocity

As described in Section 2.5, Brillouin shifts directly measure the acoustic velocity of the material. Rotational measurements along Chi maintain the same scattering geometry. As the sample rotates, phonons propagating in different crystallographic directions are sampled. To account for any deviation from parallel faces, the scattering angle, and other experimental parameters described above, all data was first analyzed using VEL-LAST. This program calculates velocity for each measured direction following the equations described in Section 2.5.

Density

Accurate knowledge of the density of each is required to accurately predict acoustic velocities and subsequently invert velocity data for $c_{ij}$. X-ray diffraction provides accurate determination of the unit cell volume for each sample. Mass is determined by chemical composition provided by electron microprobe data. Therefore, the density of the sample is determined by:

$$\rho = \frac{Z(M_{Mg} + M_{Fe} + 2M_{Si} + 3M_{O_2})}{VN_A}$$

Where $Z$ is the number of formula units contained within the unit cell, $M_i$ is the mass of each chemical component, $V$ is the unit cell volume, and $N_A$ is Avogadro’s number.

$C_{ij}$

Once the density is determined for a sample, a theoretical seismic velocity may be predicted for each measured direction from a model elasticity tensor, $c_{ij}$. ORTHO90, a $c_{ij}$ inversion software, calculates a predicted velocity for each direction and refines the starting
model $c_{ij}$ according to the Christoffel equation and a least-squares algorithm (Weidner and Carleton, Elasticity of Coesite 1977). This allows determination of the 9 independent moduli of orthopyroxene. The $c_{ij}$ are refined until they converge to a stable solution. For the data sets in this study, the root-mean-square error was less than 40 m/s, with the exception of OEN LC with a RMS of 43 m/s. With a complete elastic tensor and sample density, bulk crystal properties were calculated with POLYXSTL providing Voigt and Ruess bounds on $K_S$, $\mu_S$, $V_P$, and $V_S$ as described in Section 2.0.1.
CHAPTER 3: RESULTS

Elastic properties were determined for five orthopyroxene samples. Results of this experiment are best presented as a series of tables and graphs in the following pages. Complete phonon direction, plane normal, and predicted and measured acoustic velocities are given in the appendix.

Single crystal properties are presented in Table 3.1. Bulk and shear moduli are plotted against Mg# in Figure 3.1. The trend of the bulk moduli was fit to a linear trend, with a moderate-poor fit of $R^2 = .5437$. Single-crystal elastic moduli are plotted against Mg# in Figure 3.2. Single crystal moduli, $C_{11}$, $C_{22}$, and $C_{33}$, fit a linear trends very well, with correlations of $R^2 = 0.9587$, $R^2 = 0.92265$, and $R^2 = 0.8624$, respectively. Similarly, single crystal moduli, $C_{44}$ and $C_{66}$, fit linear trends very well with correlations of $R^2 = 0.9807$ and $R^2 = 0.9641$, respectively. To a lesser degree, $C_{55}$ followed a linear trend with $R^2 = 0.6999$. The trends of the bulk, shear, $V_P$, and $V_S$ were also considered as a function of density. However, the strong relationship of density and Mg#, produces the exact same trends – noting an increase in Mg# corresponds to a decreasing in density (Figure 3.4 and Figure 3.6).

Predicted $V_P$ and $V_S$ seismic velocities are plotted against Mg# in Figure 4.5 and the ratio, $V_P/V_S$, is given in Figure 3.6. Both $V_P$ and $V_S$ follow linear trends very well, with $R^2=0.99167$ and $R^2 = 0.99787$. While these are very strong correlations, $R^2 = 0.91923$ for the $V_P/V_S$ ratio, may indicate some nonlinear systematics, as a ratio of two completely linear trends would produce another, completely linear trend.

A table of fittings is provided in Table 3.2. Implications for these results require comparison with a suite of other samples and may be found in the discussion.
**Single Crystal Properties of Orthopyroxene**

<table>
<thead>
<tr>
<th>% Iron</th>
<th>Tanzania</th>
<th>Bamble</th>
<th>Labrador</th>
<th>Bushveld</th>
<th>Summit Rock</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>En90Fs10</td>
<td>En85Fs15</td>
<td>En70Fs18</td>
<td>En74Fs26</td>
<td>En55Fs40</td>
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<tr>
<td>C11</td>
<td>10%</td>
<td>15%</td>
<td>18%</td>
<td>26%</td>
<td>40%</td>
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<tr>
<td>C22</td>
<td>169.7</td>
<td>165.4</td>
<td>165.7</td>
<td>171.4</td>
<td>156.6</td>
</tr>
<tr>
<td>C33</td>
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<td>205.7</td>
<td>219.4</td>
<td>204.9</td>
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<td>80.0</td>
<td>79.1</td>
<td>75.9</td>
</tr>
<tr>
<td>C55</td>
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<td>76.4</td>
<td>88.6</td>
<td>74.2</td>
<td>68.9</td>
</tr>
<tr>
<td>C66</td>
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<td>74.4</td>
<td>75.2</td>
<td>70.8</td>
</tr>
<tr>
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<td>72.6</td>
<td>76.2</td>
<td>73.2</td>
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<td>57.3</td>
<td>75.3</td>
<td>48.5</td>
<td>57.0</td>
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<tr>
<td>C23</td>
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<td>49.6</td>
<td>53.5</td>
<td>54.7</td>
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</tr>
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<td>3.367</td>
<td>3.415</td>
<td>3.536</td>
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<td>109.40</td>
<td>105.63</td>
<td>100.20</td>
</tr>
<tr>
<td>Ks,V</td>
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<td>102.30</td>
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<tr>
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<td>109.20</td>
<td>110.98</td>
<td>106.20</td>
<td>101.20</td>
</tr>
<tr>
<td>µs,R</td>
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<td>75.52</td>
<td>74.23</td>
<td>72.46</td>
<td>67.66</td>
</tr>
<tr>
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<td>75.88</td>
<td>73.90</td>
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<tr>
<td>µs,VRH</td>
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<td>76.13</td>
<td>75.06</td>
<td>73.18</td>
<td>68.34</td>
</tr>
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<td>7.94</td>
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<tr>
<td>Vp,VRH</td>
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<td>7.72</td>
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<td>4.76</td>
<td>4.68</td>
<td>4.61</td>
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</tr>
<tr>
<td>Vs,V</td>
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<td>4.80</td>
<td>4.72</td>
<td>4.65</td>
<td>4.42</td>
</tr>
<tr>
<td>Vs,VRH</td>
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<td>4.70</td>
<td>4.63</td>
<td>4.40</td>
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<tr>
<td>Vp/Vs</td>
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<td>1.663</td>
<td>1.677</td>
<td>1.669</td>
<td>1.678</td>
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</table>

**Table 3.1:** Single and bulk crystal properties of samples analyzed in this study in the enstatite-ferrosilite system
Figure 3.1: Comparison of the adiabatic bulk ($K_s$) and shear ($G$) moduli of the En-Fs system as a function of Mg# with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Figure 3.2: Comparison of $c_{11}$, $c_{22}$, and $c_{33}$ of the En-Fs system as a function of Mg# with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Figure 3.3: Comparison of $c_{44}$, $c_{55}$, and $c_{66}$ of the En-Fs system as a function of Mg# with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Figure 3.4: Comparison of the adiabatic bulk ($K_s$) and shear ($G$) moduli of the En-Fs system as a function of density with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Vp and Vs of Orthopyroxene

Figure 3.5: Comparison of $V_p, V_s$, of the En-Fs system as a function of Mg# with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Figure 3.6: Comparison of $V_p$, $V_s$ of the En-Fs system as a function of density with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Figure 3.7: Comparison of the $V_p/V_s$ ratio of the En-Fs system as a function of density with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Figure 3.8: $V_S$ of the En-Fs system as a function of density with end members from Bass and Weidner (1984) and Jackson et al. (2007).
Table of Linear Trends as a function of Mg#

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>Adjusted R²</th>
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<tbody>
<tr>
<td>$K_{s,VRH}$</td>
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</tr>
<tr>
<td>$\mu_{s,VRH}$</td>
<td>0.9886</td>
</tr>
<tr>
<td>$C_{11}$</td>
<td>0.9587</td>
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<tr>
<td>$C_{22}$</td>
<td>0.9227</td>
</tr>
<tr>
<td>$C_{33}$</td>
<td>0.8624</td>
</tr>
<tr>
<td>$C_{44}$</td>
<td>0.9807</td>
</tr>
<tr>
<td>$C_{55}$</td>
<td>0.6999</td>
</tr>
<tr>
<td>$C_{66}$</td>
<td>0.9641</td>
</tr>
<tr>
<td>$V_{P,VRH}$</td>
<td>0.9917</td>
</tr>
<tr>
<td>$V_{s,VRH}$</td>
<td>0.9979</td>
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<td>$V_{P}/V_{S}$</td>
<td>0.9192</td>
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<td>$V_{S}$ (as a function of $\rho$)</td>
<td>0.9970</td>
</tr>
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</table>

Table 3.2: Linear trends observed in the Enstatite-Orthoferrosilite system as a function of Mg#.
CHAPTER 4: DISCUSSION

4.1 Observations

This study of natural single crystal elastic moduli illustrates two critical ideas when considering implications of major element variation within the enstatite-orthoferrosilite system: (1) the bulk modulus, shear modulus, and predicted seismic velocities follow a linear decrease as Fe content increases, and (2) variations in less abundant elements (e.g., Al, Ca) may also contribute significantly to the observed elastic moduli.

To date, several studies have examined elastic properties of orthopyroxenes with orthoferrosilite (Fs) contents of 20% or less (Jackson, Sinogeikin and Bass 2007, Weidner, Wang and Ito 1978, Perrillat, et al. 2007, Duffy and Vaughan 1988, Webb and Jackson 1993, Chai, Brown and Slutsky 1997, Zhang and Bass, High-pressure Single Crystal Elasticity of Natural Iron-Bearing Orthoenstatite Across a High-Pressure Phase Transition n.d.) To constrain the elasticity systematics in the En-Fs system, Bass and Weidner (1984) studied a single crystal of synthetic end-member orthoferrosilite. Their Brillouin results were, however, compared with earlier results for the Mg-rich end of the solid-solution that were obtained using a variety of techniques from different laboratories on samples of uncertain quality. The varied sources of the data make comparisons of results susceptible to systematic errors that are difficult to assess. The present study provides the first experimental study of a suite of Mg-rich samples, which are most important for geophysical modeling, using a single technique from a single lab. The present results allow a more certain reexamination of the compositional dependence of elasticity between Fs_{10} and Fs_{20}. The following discussion reasserts the simple, linear trend observed in the shear modulus, but calls into question the assumption that the bulk modulus of intermediate En-Fs compositions may be explained with linear systematics as first noted by Bass and Weidner.
Indeed, the bulk modulus is not strongly correlated with changes in the iron content. The \( V_P/V_S \) ratio may also indicate a nonlinear trend in predicted seismic velocities.

### 4.2 Comparison of Present Study with End-Member Elastic Properties

This following discussion compares data obtained in this experiment with end member values for enstatite (Jackson, Sinogeikin and Bass, Sound velocities and single-crystal elasticity of orthoenstatite to 1073 K at ambient pressure 2007) and orthoferroilite (Bass and Weidner 1984). Many models (Schutt and Lesher 2006, Lee 2003), mantle phase relations assume an ideal solid solution in which the relationship of \( K_S \) and \( \mu_S \) vary linearly with increasing iron content. This study finds linear correlation coefficients of .5437 for the bulk modulus and 0.9886 for the shear modulus. The degree of nonlinearity in the bulk modulus measurements prompted a closer examination of other studies (Section 5.2). As observed by Bass and Weidner (1984), addition of the Fs component affects the shear modulus to a greater extent than the bulk modulus (Figure 4.1). With regard to predicted seismic velocities, Fe content affects predicted \( V_P \) greater than \( V_S \), as primary waves are a function of both bulk and shear moduli. Linear correlation coefficients for the relation between Mg#, \( V_P \), and \( V_S \) are 0.99167 and 0.99787 (Table 4.2). This small difference may be significant when considering the ratio of these two supposedly linear functions produces a worse fit, of 0.91923 (though admittedly still strongly linear).

The ratio of \( V_P/V_S \) may provide meaningful insight on compositional heterogeneity in the mantle. Lee (2003) argues a 100°C temperature difference produces a change two orders of magnitude less than a unit change in Mg#. Other studies have disputed this (Schutt and Lesher 2006) finding no appreciable correlation. This work finds the orthopyroxene \( V_P/V_S \) ratio decreases ~0.1% per Mg# unit (Figure 4.4). Comparing results of this study and ranges relevant to the
Tectosphere, a pyrolite (Mg#=89.6) composition depleted to the ACGL (Mg#=91.1), would produce ~.14% decrease in $V_p/V_S$ for the orthopyroxene component, due only to Mg-Fe substitution. This seems inconsistent with a 1.7% decrease for bulk rock calculations (Lee 2003) as olivine $V_p/V_S$ is expected to increase. However, as Lee (2003) also points out, $V_S$ is also strongly correlated to Mg#, though effects of temperature must be considered. This study finds a similar $dV_S/dMg#$ value of .0133±.00013 kms$^{-1}$ to Lee (2003)’s calculation of .0150 kms$^{-1}$.

Depletion of the basaltic component of garnet lherzolite and thus a relative enrichment of orthopyroxene as suggested by Jordan (1979), could produce higher shear velocities and a higher $V_p/V_S$. Upper mantle enstatite enrichment has also been implicated in the production of kimberlite magmas (Russell, et al. 2012), making the relations determined by this study useful for the strong association of cratons, orthopyroxene enrichment, and kimberlites. Assuming the determined velocities following a completely linear trend, the findings of Schutt and Lesher (2006) appear to remain valid, in that the effect of iron depletion is overestimated while modal mineralogy has been poorly predicted. Evidenced by the poor fit of the bulk modulus data in this study, it is consistent that accounting for accurate thermal expansion values, may provide a better fit model than one which only accounts for density depletion relative to temperature, as suggested by Schutt and Lesher (2006).

Additionally, this study agrees with the work of Chai et al. (1997) on aluminous samples with Mg#=91. They found the effect of aluminum on the elastic properties of orthopyroxene could increase the overall seismic velocity profile in upper mantle mineral models at the expense of garnet content. While this present study does not argue against garnet content in the upper mantle, it does generally find a stiffening in elastic properties of orthopyroxene with increasing aluminum content. Further, the high degree of variability of orthopyroxene elastic properties
illustrated by this study, brings to light the pitfalls of oversimplifying upper mantle mineralogy as a function of Mg#. With garnets composing 6% of the AGCL and orthopyroxene 23%, aluminum distribution must also be considered. Complete understanding of upper mantle petrology requires the refinement of models such as Jordan (1979), Schutt and Lesher (2006), and Lee (2003), to account for the complex interplay of each mantle phase and multiple chemical components.

The discussion thus far has assumed the validity of the linear relationships established by regression of $V_P$, $V_S$, and the shear modulus. However, many of the trends observed by Bass and Weidner (1984) are also evident in the data of this study. Particularly, the elastic moduli show trends of maxima and minima, particularly around 20% iron content. The bulk modulus of SRO is within the margin of error of pure end member orthoferrasilite indicating no sensitivity to iron content for compositions greater than 40%. Building upon this earlier work, this study indicates some variation between the bulk modulus and iron content continue up to 26%, as opposed to 20% suggested by Bass and Weidner (1984). However, the overall observations of $C_{11}$ varying the least with iron content, $C_{33}$ varying the most remain valid. Likewise, the bulk modulus insensitivity to high iron content (particularly above 20%) is also validated. Also, the shear modulus is well correlated to iron content. Effects on $V_P$ and $V_S$ are found to be linear with $V_P$ being more sensitive to iron content ($dV_S/dMg# = .0166$ kms$^{-1}$ vs. $dV_S/dMg# = .0133$ kms$^{-1}$). However, this needs further testing, as the linear ratio, $V_P/V_S$, produces a worse fit.

4.3 Determination of the Influence of Minor Chemical Variation

A consequence of thorough examination of orthopyroxene elastic parameters initially studied for their En-Fs content, was the discernable relationships between minor chemical components, namely Ca and Al. Comparison of 15 other experiments on the system produces
more questions than answers for the complex interplay of minor elements and elastic moduli. Consideration of the orthopyroxene structure suggests the $c_{11}$ elastic constant is most sensitive to compositional variation (Weidner and Vaughan 1982). Calcium substitution may increase the density (relative to Mg), which may affect elastic constants, though this has been found to be minimal (Perrillat, et al. 2007). Aluminum has been suspected in the stiffening of tetrahedral chains of orthopyroxenes (Chai, Brown and Slutsky 1997, Webb and Jackson 1993), which would produce faster wave speeds along $c$ and significantly altering the bulk modulus.

Compared with 11 other studies, a new set of correlations was determined. Variations in Mg# produce a linear correlation to the $c_{11}$ modulus with a coefficient of .89. $V_P/V_S$ ratios for the entire data set also correlate well (-.92) with increasing Mg# strongly correlated to a decreasing $V_P/V_S$ ratio. However, bulk modulus and shear modulus coefficients of .44 and .95 strongly indicate there is a significant variable beyond Mg-Fe exchange.

Thus, to explain the significant scatter in elastic moduli in not only the samples of this study, but also in comparison with every other relevant measurement, an attempt has been made to produce a principal component analysis (PCA) to determine how Ca, Al, and Mg-Fe substitution relate to $c_{11}$, bulk modulus, and shear modulus (Figures 5.2-Figure 5.4). These analyses are provided as a first-order, qualitative assessment of the data set and are meant to provide a guide for interpretation and future studies rather than a rigorous, quantitative analysis. From a sample of 15 studies, with varying compositions, the PCA suggests that Al content has the most significant influence on the single-crystal elastic moduli and bulk elastic properties of orthopyroxene, in agreement with Chai et al. (1997). Further, as Perillat et al. (2007) suggested, there seems to be little effect of Ca on the single-crystal elastic moduli and bulk elastic properties of orthopyroxene.-While these analyses are generally consistent with other studies, it is
recognized that validity of comparison among them is limited by the characterization of sample composition. In general chemical compositions were provided by electron microprobe analysis. Flux grown enstatite (Weidner, Wang and Ito 1978), contained .25 wt.% of unknown impurities. Additionally, characterization of Al distribution between M1 sites and tetrahedral sites relied on charge balance calculations. While likely acceptable for these purposes, Al in each site as well as total Al in the sample was considered in the PCA.

While confirming several studies on the system, these findings motivate further study on the behavior of Al in orthopyroxene crystals structures at ambient pressure and temperature. Additionally, characterization of more orthopyroxene samples between 20-30% orthoferrosilite would better constrain the nonlinearity between 10-30% in the system. These data call into question the general assumption that the elastic properties of orthopyroxene are completely characterized by the Mg and Fe content and may be modeled as a linear relationship between end members. Particularly, the Al content affects the bulk modulus to a greater degree than the shear modulus. Reiterating the observations of Bass and Weidner (1984), the PCA confirms bulk modulus appears less sensitive to Mg-Fe substitution than the shear modulus.

4.4 Comparison of Orthopyroxene with Other Mantle Minerals

To accurately compare the orthopyroxene elasticity data of this study to other major mantle minerals, namely olivine, clinopyroxene, bulk and shear moduli end members are plotted in Figure 4.4. Data is from Scutt and Lesher (2006) and from this study. \( V_P \) and \( V_S \) (Figure 4.5) were calculated iteratively based on linear relations between end members of each phase, with the exception of orthopyroxene. Orthopyroxene calculations were fit to intermediate compositions of the present study with linear relations in between. Trends observed by Bass and Weidner (1984) are represented well in the present study. Particularly, the deviation in \( V_S \) of
olivine and orthopyroxene with Mg# >85. Also as expected, the seismic velocities of orthopyroxene and olivine are the most strongly related to Mg-Fe heterogeneity (based on dV_S / dMg#) compared to garnet and clinopyroxene. Inflections in the V_P trend of orthopyroxene may lead to misinterpretation of mantle velocity anomalies, as dV_P / dMg# may be very different from Mg#=85 to Mg# = 60. However, the consistent positive dV_P / dMg# trends of olivine and orthopyroxene make them ideal indicators of Mg-Fe variations. Different end member data may provide somewhat different plots, though the general trends are expected to be the same.

4.5 Future Directions

This study will continue with a generalized 2D seismic model relating the predicted seismic velocities of this study to those required to validate the Tectosphere hypothesis. Additionally, a buoyancy calculation will relate the observed density, thermal expansion, and proposed temperature variations of the Tectosphere. These two approaches intend to place first-order relations between this study and its implication to the Tectosphere.
4.6 Summary

These data illustrate a general linear trend in the shear modulus as a function of Mg-Fe substitution. Likewise, linear systematics apply to predicted $V_P$, $V_S$, and $V_P/V_S$. Though the lesser degree of correlation in $V_P/V_S$, may indicate $V_P$ is slightly nonlinear. However, the relationship between the bulk modulus and Mg-Fe substitution is far more complex and cannot be accurately predicted with a simple linear trend in neither the present study of five samples nor with the extensive suite of previous studies. Examination by principal component analysis confirmed the stiffening effects of Al on the bulk modulus while the Ca does not strongly influence it. Further, the sensitivity of Mg:Fe ratio to the shear modulus and relative insensitivity of the bulk modulus was reiterated by PCA. However, this analysis is not adequate to produce an accurate 3D surface relating the bulk modulus, Mg#, and Al content – more complete chemical and elastic characterization is needed in the system. Thus, this work supports the conclusions of Bass and Weidner (1984) and prediction of Anderson (1976) that the bulk modulus is independent of iron content.

Thus, $V_S$ is likely the best indicator of Mg-Fe content in the upper mantle, though it is more frequently used to infer temperature. Upper mantle petrologic models must take these observations into account when predicting seismic velocities and the influences of upper mantle heterogeneity. Upper mantle petrology models have attempted to produce an accurate mineralogy to compare with observed seismic velocities. However, in many cases, these models assume linear relationships of the elastic moduli in the solid solutions of olivine and orthopyroxene. Orthopyroxene may constitute up to 25% of the upper mantle beneath cratons. To better understand the elastic moduli of the orthopyroxene solid solution, this study examined five samples of natural orthopyroxene with Brillouin spectroscopy. To provide context for these
results, 11 additional studies were analyzed with statistical models and a principal component analysis. These comparisons supported the results of this systematic study.

This study finds linear trends are a poor predictor of the bulk modulus as a function of Fe content. However, the shear modulus as a function of Fe content is accurately predicted by linear systematics. This result helps constrain the sensitivity of shear wave velocities to chemical composition in the mantle. This study also illustrates the difficulties in accurately modeling the upper mantle bulk moduli of orthopyroxene. No definitive relation to the Tectosphere model is provided, though future work on this investigation will produce first-order models to provide direct implications.
### Comparative Data Set

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**Table 4.1:** Relevant studies on the orthopyroxene system with bulk modulus, shear modulus, V₉ P, and V₉ S provided.
Figure 4.1: Comparison of the adiabatic bulk ($K_S$) and shear ($G$) moduli of the En-Fs system as a function of Mg#. Sample set listed in Table 4.1.
Figure 4.2: Comparison of predicted $V_P$ and $V_S$ of the En-Fs system as a function of Mg#. Sample set listed in Table 4.1.
Principle Component Analysis

Figure 4.3: Principal component analysis illustrating the effects of Mg, Ca, Fe, and Al on the bulk and shear modulus.
Upper Mantle Mineral Elasticity

Figure 4.4: End member elasticity from parameters used in Schutt and Lesher (2006) for major upper mantle minerals. OPX contains data points from this study. OL – olivine, OPX – orthopyroxene, CPX – clinopyroxene, GT – garnet.
Figure 4.5: Seismic velocities predicted by iterative calculations based on linear substitution between end members. For OPX, linear relations are imposed between compositions of this study. End member elastic parameters from Schutt and Lesher (2006) for major upper mantle minerals. OL – olivine, OPX – orthopyroxene, CPX – clinopyroxene, GT – garnet.
CHAPTER 5: CONCLUSION

The Tectosphere hypothesis proposes chemical variations in the upper mantle produce a neutrally buoyant, or isopycnic, continental root. This root evolves from Mg-Fe partitioning as a product of melting at a mid-ocean ridge which produces a less dense residue. Over time, buoyant residue and continental craton would translate cohesively as a Tectosphere. In the ocean basins, the base of the Tectosphere would coincide with the LAB. However, the colder geotherm of the cratons could depress this boundary to 300-400 km. Comparison of seismic velocity models in the ocean basins and the cratons can help verify the existence of the Tectosphere.

The upper mantle (<400 km deep) is composed of olivine, orthopyroxene, clinopyroxene, and garnet. Partial melting at ridges will increase the Mg# as well as relative proportions of olivine and orthopyroxene while decreasing clinopyroxene and garnet in the residue. This chemical heterogeneity will have a characteristic seismic signature which can be identified through accurate knowledge of mantle mineral elasticity as a function of Mg#.

This study systematically characterized elasticity of orthopyroxene as a function of Mg# with Brillouin spectroscopy. The complete elastic tensor was calculated to provide single crystal moduli for the orthopyroxene system. From the tensor, bulk crystal properties were calculated. Statistical analysis found poor correlation of the bulk modulus with Mg# while the shear modulus was found to correlate linear and positive to increasing Mg#. Seismic velocities, $V_P$ and $V_S$, were also found to correlate linear and positive to increasing Mg#. Comparison of minor chemical variations in the samples and other studies found Ca produces no discernable effect on the bulk modulus while increasing Al content may significantly stiffen the bulk modulus. Comparison with other mantle minerals reaffirms the relation between the seismic shear velocity of olivine and orthopyroxene and the Mg content in the upper mantle.
These findings cannot yet support or refute the existence of a Tectosphere. However, this study will continue with fundamental modeling of the expected seismic velocity variations from these data and observations. Also, modeling of buoyancy differences from density data of this study may also help provide direct implications to the Tectosphere model. For now, it remains an eloquent and simple mechanism to explain the long-lived nature of the cratons.
REFERENCES


doi:10.1016/j.gca.2013.03.042.


Lee, Cin-Ty Aeolus. 2003. "Compositional variation of density and seismic velocities in natural peridotites at STP conditions: Implications for seismic imaging of compositional


### APPENDIX

**Table A.1 Tanzania Data (OPX TANZ)**

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| 0.974 | 0.1858 | -0.13 | 0.1285 | 0.0199 | 0.9915 | -0.187 | 0.9824 | 0.0045 | 8416.5 | 4922.3 | 4841.2 |  
| 0.8774 | 0.332 | -0.346 | -0.476 | 0.5078 | -0.718 | -0.476 | 0.5078 | -0.718 | 8391.3 | 4918.1 | 0 |  
| 0.8939 | 0.3208 | -0.313 | 0.3157 | 0.0453 | 0.9478 | -0.318 | 0.9461 | 0.0608 | 8340.4 | 4959.8 | 4730.2 |  
| 0.7241 | 0.4507 | -0.522 | -0.687 | 0.4041 | -0.604 | -0.687 | 0.4041 | -0.604 | 8266.4 | 5021.1 | 4637.7 |  
| 0.7569 | 0.4307 | -0.492 | 0.5082 | 0.0849 | 0.857 | -0.411 | 0.8985 | 0.1546 | 8217.9 | 4997.2 | 4625.6 |  
| -0.106 | 0.9928 | 0.0563 | 0.9942 | 0.1066 | -0.017 | 0.9942 | 0.1066 | -0.017 | 7267.3 | 4867.4 | 0 |  
| -0.174 | 0.9812 | 0.0841 | 0.9782 | 0.1818 | -0.101 | 0.1141 | -0.065 | 0.9914 | 7228.2 | 4868 | 5021.6 |  
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| -0.499 | 0.8636 | 0.0731 | 0.8594 | 0.5039 | -0.087 | 0.1121 | -0.019 | 0.9935 | 7558 | 4590.4 | 5012.5 |  
| -0.587 | 0.8077 | 0.0565 | 0.8093 | 0.5873 | 0.0095 | 0.8093 | 0.5873 | 0.0095 | 7977.6 | 4357.6 | 0 |
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