Kinetics of Melting in Peridotite from Volume Strain Measurements

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Abstract

Partial melting in the Earth's mantle involves a combination of processes such as ionic diffusion and solid - liquid transition. These processes yield an array of characteristic times for the melting kinetics. Both the magnitude and time constant of melt-induced volume strain affect seismic velocity, attenuation, and buoyancy. In situ monitoring of samples during melting are enabled by multi-anvil high-pressure devices coupled with synchrotron x-ray radiation. In this study, the volume strain induced in a sample from melting is measured as a function of time using x-ray images. We find a doubling of the effective thermal expansion for only 2% melt both from our data and from thermodynamic models of peridotite. The characteristic time of melting is determined to be less than one second. These findings have a direct impact on the effect of melting on seismic velocities.

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23	Key words:
24	Partial melting
25	Mantle peridotite
26	Low velocity zone
27	Elastic softening
28	Melting kinetics
29	

1. Introduction

Melting is a profound phase transformation that is accompanied by significant changes in strength, volume, and viscosity. Because of the chemical complexity, most rocks melt over a wide temperature range. Changes in pressure, temperature, or stress will promote a change in the amount of melt. Since the melt is inherently less dense than the solid, these changes in pressure, temperature, or stress will result in an enhanced effective compressibility, thermal expansion, or elastic compliance and, ultimately affect sound velocity and buoyancy.

This effect is simply illustrated for changes in pressure or temperature as they are accompanied by a change in the amount of melt in the system. Melting induces a volume change of the sample because the melt volume is generally significantly different from that of the host crystals. The volume change, in turn, affects both the effective thermal expansion and the effective compressibility as the fraction of melt (F) changes due to changes in P and T. The resulting thermal expansion α and the compressibility β are:

$$\alpha_{\text{effective}} = \frac{1}{V} \left(\frac{\partial V}{\partial T} \right)_X + \left(\frac{\Delta V}{V} \right)_{\text{melting}} \left(\frac{\partial F}{\partial T} \right)_X \quad (1)$$

$$\beta_{\text{effective}} = -\frac{1}{V} \left(\frac{\partial V}{\partial P} \right)_X - \left(\frac{\Delta V}{V} \right)_{\text{melting}} \left(\frac{\partial F}{\partial P} \right)_X \quad (2)$$

where $(\Delta V/V)_{\text{melting}}$ is the relative volume change associated with melting. X represents the P-T path of the perturbation. These terms are typically isothermal (T), adiabatic (S), or constant pressure (P), or, as in a realistic high pressure experiment, is a combination of these. The first term on the right is the intrinsic thermal expansion (compressibility). This

term includes the properties of all of the phases present as a mechanical ensemble. The case where this term is defined for a solid – liquid mixture has been studied in depth e.g. (Hammond and Humphreys, 2000a, b; Mavko and Nur, 1975; Sato et al., 1989; Spetzler and Anderson, 1968) and it has been concluded that the compressibility is relatively insensitive to the presence of a small amount of melt, while the shear modulus can vary significantly as a function of the amount of melt present (F) but the magnitude of this effect also depends on the geometry of the melt.

The second term on the right side of these equations is the volume strain resulting from melting as P or T change. It depends on the melt productivity ($\partial F/\partial T$ or $\partial F/\partial P$) and is independent of the amount of melt present (F). The effective thermal expansion (compressibility) defined in equations (1) and (2) are the zero frequency value of the variables as they represent thermodynamic equilibrium. The kinetics of the melting process will make these terms time dependent.

Thermodynamic calculations with the MELTS program (Smith and Asimow, 2005) for a peridotite composition suggest that melting will double the thermal expansion or the compressibility for about 2% melt. The impact of melting on seismic velocities (Anderson, 1989; Li and Weidner, 2013) or mantle convection and post-glacial uplift (O'Connell, 1976) depends on the time that is required for significant melting to occur, that is, the kinetics of melting.

Several processes can control the kinetics of melting. In order for the equilibrium portion of melt to form, atoms must diffuse. The construction/deconstruction of crystal structures must occur and can create a delay between the time of the change in state and the attainment of equilibrium. Understanding the process will assist us in relating

laboratory measurements to planetary-scale issues. Indeed, melting will impact the density even before equilibrium is attained. Thus, it is important to know the time dependence of the density change in order to define the impact of melting on these Earth issues.

The relaxation time τ of an activated process is generally represented by $\tau(P, T) = \tau_0 \exp[(E^* + PV^*) / RT]$ (Anderson, 1989), where E^* and V^* are activation energy and volume, τ_0 is a reference characteristic time, P and T are the pressure and temperature departure from the reference state. For partial melting, τ_0 has multiple values. In the case for the bulk sample to reach compositional equilibrium, the ionic diffusion is rate-limiting, yielding a τ_0 that depends on a characteristic length and the diffusion coefficients. On the other hand, the atomic level process of moving the crystal - liquid interface could be characterized with a different τ_0 , where this latter characteristic time is probably shorter than the diffusion characteristic time. τ impacts the seismic velocity by:

$$c^2(\omega) = c_0^2 (1 + \omega^2 \tau^2 c_\infty^2 / c_0^2) / [(1 + \omega^2 \tau^2)^2 + 2\omega^2 \tau^2 Q_{\max}^{-1}]^{1/2} \quad (3)$$

where $2Q_{\max}^{-1} = \frac{c_\infty^2 - c_0^2}{c_0 c_\infty}$, c is the phase velocity, ω is seismic frequency. c_∞ and c_0 are

velocities at infinite and zero frequency (Anderson, 1989). The goal of this study is to define the time scale for significant volume change associated with melting.

Seismic velocities $V_p = \sqrt{(K + 4G/3)/\rho}$ and $V_s = \sqrt{G/\rho}$ depend on elastic moduli, where K and G are bulk modulus (or inverse of compressibility) and shear modulus and ρ is density. Both bulk and shear moduli are affected by melting due to the transformation volume as discussed elsewhere (Li and Weidner, 2013). The crucial factor concerning the effect of melting on seismic velocities is the value of $\omega\tau$. If $\omega\tau \ll 1$, the seismic wave

will experience a media with reduced elastic moduli, resulting in a slowing of the seismic velocity. For $\omega\tau \gg 1$, maximum values of seismic propagation will occur. If $\omega\tau \approx 1$, then the velocity will be frequency dependent and seismic attenuation will result.

In our previous study (Li and Weidner, 2013), we probed the significance of equation (2) on the effective elastic moduli. The results indicate a significant melting-induced softening caused by a non-negligible $\partial F/\partial P$ (Young's modulus softened to less than half of the intrinsic value). That study (Li and Weidner, 2013) measured the modulus at a period of 720 seconds to mimic seismic frequency. The study reported here is designed to measure the time scale of melting through the viewpoint of equation (1) by varying temperature to investigate the time response of the system, i.e. the term $\partial F/\partial T$. The experiment uses x-ray images of the sample from a CCD camera that can capture sample images at tens of frames per second in which we can resolve volume strain at 10^{-4} precision. The uniqueness of this study is to use image-resolved volume strain at a sub-second time scale to yield effective thermal expansion during melting in situ at high P-T. This study by changing $\partial F/\partial T$, is complementary to the study varying $\partial F/\partial P$ (Li and Weidner, 2013), both provide strong arguments to support a short characteristic time and softened elastic modulus of peridotite in partially molten regions of the Earth's mantle.

2. Experimental Methods

Our measurement of thermal expansion centers on measuring sample dimensions (Li et al., 2003; Li and Weidner, 2007; Li and Weidner, 2013) using synchrotron x-ray absorption images of the sample in the D-DIA high pressure device. Details of the experimental setup are further described in (Li and Weidner, 2014).

The sample, fine ground KLB1 peridotite powder (Takahashi, 1986) (~5 micron grain size), was cold pressed to 1.5 GPa, then heated to subsolidus conditions (c.a. 850 °C) and annealed for 30 minutes. The melting relations of this peridotite have been thoroughly studied as a function of pressure (Herzberg et al., 1990; Takahashi, 1986; Takahashi and Scarfe, 1985) as a model mantle composition. Rather than a conventional cell assembly as used in (Li and Weidner, 2007), a rhenium capsule is used to wrap the cylindrical sample (1 mm diameter, 1.5mm long) to serve as the lateral strain marker, as illustrated in Figure 1. The sample, S, is located between two dense corundum rods, A1 and A2, also decorated with Re foils above and below. In our experiment, the thermocouple is placed outside of the capsule, yielding a measured temperature that could be 100°C lower than the sample temperature.

In the DIA geometry, the x-ray beam passes between two of the anvils on it's entrance and two more on it's exit from the sample. Tungsten carbide anvils truncate the beam width to less than one millimeter, thereby obscuring the lateral dimension of the sample. Thus, we used sintered diamond as the lateral anvils as they are transparent to the x-ray beam allowing us to obtain a full 2-D image of the sample as delineated by the rhenium foils.

Measurements from the image of the longer corundum rod, A1, are used as a reference for the thermal expansion measurements. This thereby serves to qualitatively compare a material with a known and well-behaved thermal expansion with the thermal expansion of the sample.

The post – annealing sample temperature, as a function of time, recorded by the thermocouple is illustrated in Figure 2. Over time, the median temperature was stepped

from 850°C to 1300°C with 50°C per step. About each median temperature, the temperature was oscillated, first in a sinusoidal fashion, then in steps as illustrated in the inset of Figure 2. The oscillations and steps are used to condition the cell assembly to yield reversible temperature – volume relationships with time. Our experience is that cell pressure drops during the first heating cycle, but reversibly increases and decreases as temperature is cycled thereafter as long as the peak temperature is not exceeded. Once the peak temperature is exceeded, the pressure will decrease through a temperature cycle. We infer that the cell relaxes on heating, but the relaxation stabilizes to the level defined by the maximum temperature.

A CCD camera records images of the sample during changes in temperature. Images of the sample were taken during the temperature steps at a rate of 20 frames per second during some of the temperature changes. The symmetry of the cell assembly suggests that the axial and radial directions may exhibit different amounts of strain, requiring the analysis of both axial and radial strain. Since the boundaries in both the radial and axial directions are present in the images, we determine both radial strain and axial strain yielding the volume strain assuming that the lateral strain is cylindrically symmetric.

The time dependence of the volume strain is fitted as though it is controlled by a characteristic time. The time function is that the volume strain is a constant until the time $t = t_0$, then increasing by $\epsilon(1 - e^{-(t - t_0)/\tau})$. The strain jump, ϵ , is used to calculate the thermal expansion of Figure 3, and the characteristic time, τ , is evaluated to estimate the characteristic time of melting. The last heating step is used to define the effective thermal expansion. After the last temperature increase, the sample was quenched down to room

temperature in a few steps. Using the departure of the strain from the fitted form, we conclude the standard deviation of the volume strain to be less than 10^{-4} .

The fitted characteristic time for all temperature steps is about 1.5 seconds with no significant variation. This time is consistent with the expected heating time for a peridotite specimen that is heated by a cylindrical furnace. The consistency of this characteristic time between samples that are melting during the heating phase with those that are not melting implies that the melting time must be less than one second.

An SEM image of the recovered sample is given in Figure 4. The sample was recovered from the highest temperature of this experiment and cut along the axis of the cylinder. Significant melting is indicated in the image from both the texture and the chemical composition (as determined from energy dispersive analysis of fluorescence). Significant clinopyroxene is identified throughout the sample suggesting that the melt fraction was less than 10% during the experiment.

3. Results and Discussion

The effective thermal expansion of a peridotite as a function of time as temperature varies from subsolidus through partial melting is defined in these experiments. The measurements reflect the volume strain of the sample and a reference corundum sample placed adjacent to the sample in the center of the high pressure cell. Figure 5 illustrates volume strain as a function of time, in seconds, from a step increase in temperature at different temperatures that range from subsolidus into the partial molten zone. The gold colored curves are for the KLB-1 sample and the blue curves are for the companion corundum reference sample. We take the high frequency jitter to represent the background noise. In general all curves indicate a very crisp rise in strain with the onset of heating.

The strain more gradually reaches a maximum. All curves could be well fit with a strain rise time of about 1.5 seconds. This is the time we would calculate for the characteristic time of heating the sample from the outside. We expect that the furnace rise time is much faster in that we anticipate that the electronic rise time of the heater power supply is of the order of microseconds. The magnitude of the temperature increase in this step is approximately 100°C.

The volume strain jump is normalized to the thermocouple-defined temperature increase in Figure 3 defining the effective thermal expansion as a function of the baseline temperature. As indicated, the sample thermal expansion nearly triples for the highest two temperatures, while the corundum expansion slightly decreases for these last two temperatures.

Based on previous recovery experiments, as well as the SEM images of the recovered sample from this experiment, partial melting was present for the last two temperatures causing the sample's effective thermal expansion to increase. However, the time scale of the strain increase is the same for the last two temperatures as for the lower temperatures. Thus, the partial melting that is responsible for the volume increase occurs essentially in phase with the temperature increase and must occur within a second of the heating. Melting may be faster than a second, but a shorter time would be obscured by the finite rise time of the temperature.

The thermal expansion illustrated in Figure 6 is 'effective' not only because the volume change may include a contribution from melting, but also because the temperature change is not made at a constant pressure. The ram applies a constant load to the cell assembly, but the pressure within the assembly changes with temperature as a result of the

expansion of the cell and the pressure media. The actual P – T curve in the cell is not easily calculable. The increase in pressure with temperature will increase as melting begins because of the volume increase with melting. Of course, this will act to regulate the amount of melting. The decrease in the corundum thermal expansion coincides with the increase in KLB-1 expansion suggesting that the melting increased the pressure of both the sample and the corundum.

The thermal expansion of olivine at the experimental pressure and temperature is about $5.5 \times 10^{-5} \text{ K}^{-1}$, which serves as a good estimate of that of peridotite. Figure 6 illustrates the effective thermal expansion from equation (1) along with the intrinsic thermal expansion as a function of melt fraction for a peridotite that is calculated using the program MELTS (Smith and Asimow, 2005). The thermal expansion has doubled at about 2 vol% melt. Of course, small variations in bulk composition can change this relationship. The point here is that it is expected that small amounts of melt will have a significant effect on thermal expansion and elastic moduli. The example here indicates that 2% melt is sufficient to double the thermal expansion and cut the bulk modulus in half.

The 1 second state, with its change in the amount of partial melt and attendant change in thermal expansion, while representing an approach to thermodynamic equilibrium, has not attained equilibrium. Equilibrium will result when the chemistry within each phase is uniform, when this chemistry is properly partitioned between phases, and when the texture has reached an equilibrium state. Yet, this 1 second state is extremely important to defining all thermoelastic properties relevant to seismic wave propagation. Solid-state diffusion is necessary for the chemistry to reach equilibrium. And solid-state diffusion is slower than observed here. A considerable approach to equilibrium can be

obtained without solid state diffusion. Using the MELTS (Smith and Asimow, 2005) program, we (Li and Weidner, 2013) compared the melt fraction if only olivine and clinopyroxene are involved in the process rather than all of olivine, orthopyroxene and clinopyroxene. The calculation provides insight to the reaction if the orthopyroxene phase is isolated from the reaction. The presence or absence of diffusion to or from orthopyroxene did not substantially affect $\partial F/\partial P$ nor the effective elastic moduli. The chemistry that is available for a melt at the olivine – clinopyroxene interface is sufficient to promote melting short of obtaining a complete thermodynamic equilibrium. In contrast, our study of the kinetics of the olivine to spinel transition (Li and Weidner, 2008), for similar grain size and temperature yielded a time constant of a few thousand seconds. In that case, the time is consistent with solid state diffusion of iron – magnesium exchange.

Rather, we suggest, the kinetics of this fast melting process is dominated by the time for melting to occur. We can model the melting process as the movement of the interface between the solid phases and the liquid phase. Melting or solidification is accomplished by movement of the boundary between the phases. This yields the distance that the phase boundary moves as a characteristic length. The characteristic time is related to a characteristic length by a velocity:

$$\tau = x / v \quad (4)$$

where τ is the characteristic time, x is the characteristic length, and v is the velocity of the rate process. Furthermore, since the structure of the melt and solid are different, a reconstruction front needs to move through this region. This process is often described by a velocity of the form:

$$v = AT \exp\left\{-\frac{\Delta H + PV^*}{RT}\right\} \left(1 - \exp\left\{\frac{-\Delta G}{RT}\right\}\right) \quad (5)$$

where A is a constant, T is temperature, ΔH is the activation enthalpy, P is pressure, V^* is the activation volume, and ΔG is the excess Gibbs free energy arising from the thermodynamic perturbation in the acoustic wave (Kubo et al., 2004; Turnbull, 1956).

Our results indicate that non-diffusion related melting occurred at the one second time scale and resulted in significant volume strain. Longer time will promote both chemical and textural equilibrium. Studies of melt – solid texture suggests that liquids tend to stabilize at triple junctions (Bussod and Christie, 1991; Faul et al., 2004; Jackson et al., 2006; von Bargen and Waff, 1986; Zhu et al., 2011) with some melt at grain interfaces. We have reported the evolution of the texture in (Du et al., 2014). Samples quenched after 60 seconds exhibit liquids at the interfaces between grains, while samples quenched after 30 minutes indicate melts at triple junctions between three grains, suggesting that on the short time scale, melting is driven by local thermodynamics and melting occurs at interfaces between grains that carry the optimum chemistry. As the system evolves, the melt surface tension concentrates the liquid. Further evolution will see the chemistry of the system move towards complete equilibrium with diffusion throughout the solid phases. The multi-process feature of partial melting requires that partial melting will greatly impact the effective thermoelastic properties of the system on a variety of time scales. The first stage is the fast solid-liquid transition at one second or less. The last stage reflects diffusion time scales, which may be 1000's of years.

An important purpose of identifying the process that is responsible for the experimental time scale is to allow extrapolation from the laboratory setting to the natural setting. These experiments probe melting with a significant thermodynamic perturbation, 100°C for relatively small grains (10 micron). A seismic wave imposes a much smaller

perturbation in the free energy, thus, we must be concerned as to how the magnitude of the effect scales with the size of the perturbing field. To first order, the effective thermal expansion, compressibility, or elastic moduli should be insensitive to the magnitude of the perturbing function. They all reflect the derivative of the strain with respect to the thermodynamic variable. Thus, thermal expansion is relatively independent of the size of the temperature perturbation. The second issue that may have an effect is whether the kinetics is sensitive to the magnitude of the driving force. In equation (5), the perturbation in Gibb's free energy is quite small, we can replace the term in parenthesis by $\Delta G/RT$ or by $\Delta V\delta P/RT$ for a pressure perturbation where ΔV is the volume change of the phase transition and δP is the excess pressure associated with the stress wave. Since all other values remain constant as the wave moves through the system, we have:

$$v = B\delta P \quad (6)$$

or simply, transformation rate, and hence velocity, is proportional to pressure perturbation. In addition, the distance, x , that the phase boundary must propagate to achieve equilibrium, is also proportional to δP since the amount of material that will change phase increases proportional to the pressure perturbation. Thus, the ratio of distance and velocity of equation (4) will be independent of pressure. Thus, this melting model suggests that the time constant of melting will be independent of the magnitude of the thermodynamic perturbation.

This model yields the value of x proportional to grain size. We thereby conclude that the characteristic time should be proportional to grain size. With possible mantle grain size being up to three orders of magnitude greater than the laboratory grain size implies that we need to improve our resolution of the laboratory melting time in order to accurately

predict the impact of the dynamic melting process on seismic velocity. In summary, X-ray image analysis of a peridotite sample at elevated pressure and temperature has captured the onset of partial melting. The sample, contained in a rhenium capsule, produces clear images that can be analyzed for volume strain as a function of time. The melting event is identified by a strong increase in effective thermal expansion. However, the time constant of the effective thermal expansion is the same whether or not melting is contributing, implying that the characteristic time of melting is less than one second. The dynamics of melting that occurs faster than the seismic period will significantly perturb the seismic velocity.

Acknowledgements

Use of the National Synchrotron Light Source, Brookhaven National Laboratory, Beamline X17B2 was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-98CH10886 and COMPRES, the Consortium for Materials Properties Research in Earth Sciences under NSF Cooperative Agreement EAR 11-57758 and by the Mineral Physics Institute, Stony Brook University. The authors acknowledge NSF for research funding for this program through EAR 0968823, EAR 1045629, and EAR 1141895. Discussions with Bill Huebsch were inspirational in defining the protocol of this experiment. We thank Wei Du, Matt Whitaker, Haiyan Chen, and Ken Baldwin for support. The authors thank Eiichi Takahashi for his help by providing the KLB1 sample.

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Figure Captions

Figure 1. Cell assembly for thermal expansion experiments. The diagram indicates the inner cylinder that is located within an amorphous boron - epoxy cube with 6 mm edge lengths. A1 and A2 are dense corundum rods; S is the KLB1 sample.

Figure 2. Temperature vs time as defined by the thermocouple in the thermal expansion experiment. At each temperature, a sin wave followed by a box-car function varied the temperature over a 100°C region. The two departures from this pattern were experimentalist error. Thermal expansion is measured on the last temperature increase. This illustration is modified from (Li and Weidner, 2014).

Figure 3 Effective thermal expansion of sample, KLB1 and a corundum rod, as a function of median temperature. The jump in thermal expansion for the highest two temperatures is consistent with the onset of partial melting. This illustration is modified after (Li and Weidner, 2014).

Figure 4. SEM image of recovered sample. This sample was quenched from the highest temperature in the experiment (see Figure 2). Olivine (ol), orthopyroxene (opx), clinopyroxene (cpx), and melt are indicated. These were identified on the basis of composition.

Figure 5. Volume strain of sample, KLB1 and a corundum rod, as a function of time from the time of a sudden increase of temperature. The temperature increase was about 100°C. The dark curves represent a model with volume strain constant for $t < 0$ and strain given by $\varepsilon(1 - e^{-t/\tau})$ for $t > 0$. ε represents the final strain reached after a characteristic

time of τ . The highest two temperatures demonstrate a sudden increase in volume strain for the sample, but not the corundum, consistent with the onset of partial melting.

Figure 6. Calculated effective thermal expansion for KLB1 as a function of fraction of melt, F . The intrinsic thermal expansion represents the thermal expansion of the mineral assemblage. The effective thermal expansion includes the contribution of melting to the thermal expansion (see equation 2). Values of F and $\partial F/\partial T$ are calculated using the MELTS program (Smith and Asimow, 2005) for the KLB1 composition at 2 GPa.

	Material
Al ₂ O ₃
carbon U5
carbon UF ₄ S
Al ₂ O ₃
Boron Nitride
carbon U5
crushable al ₂ o ₃
Re foil
Thermocouple

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