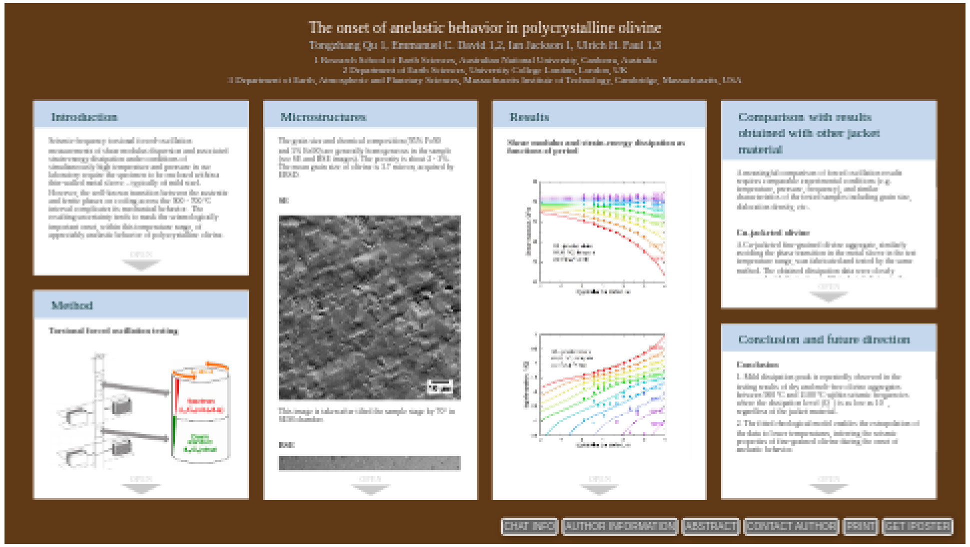
# The onset of anelastic behavior in polycrystalline olivine

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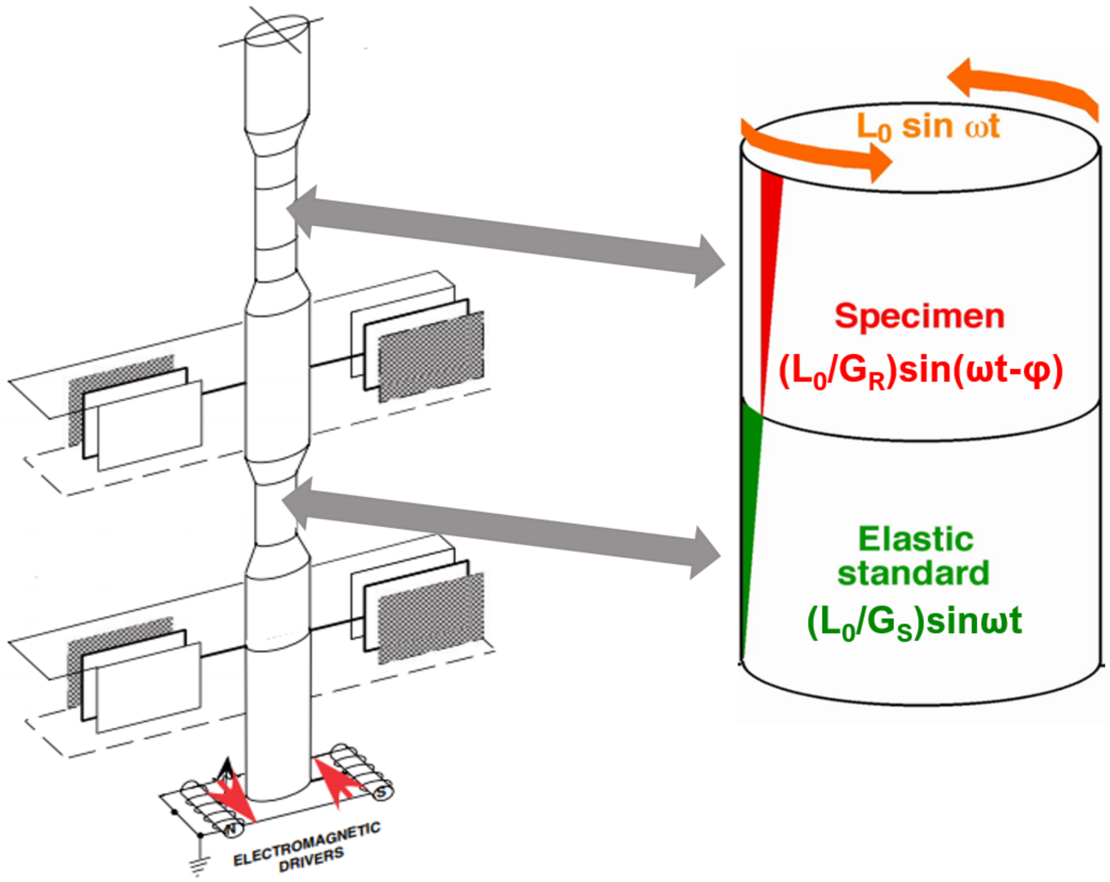
# INTRODUCTION

Seismic-frequency torsional forced-oscillation measurements of shear modulus dispersion and associated strain-energy dissipation under conditions of simultaneously high temperature and pressure in our laboratory require the specimen to be enclosed within a thin-walled metal sleeve – typically of mild steel. However, the well-known transition between the austenite and ferrite phases on cooling across the 900 - 700 ºC interval complicates its mechanical behavior. The resulting uncertainty tends to mask the seismologically important onset, within this temperature range, of appreciably anelastic behavior of polycrystalline olivine.

In order to more closely document this important transition in mechanical behavior, we have conducted an unpublished study in which a specimen of polycrystalline olivine is jacketed within a copper sleeve which retains its face-centered-cubic (fcc) structure throughout the range of the measurements limited to 1050 ºC by the proximity of the melting point. Here we report measurements to a higher temperature (1200 ºC) in which we employ austenitic (fcc) stainless steel (SS) as alternative jacket material.

# METHOD

**Torsional forced oscillation testing**




Experimental conditions

Temperature: 200 - 1200 °C

Confining pressure: 200 MPa, Ar medium

Oscillation period: 1 - 1000 s

Max shear strain: $2*10^{-5}$

This mechanical testing is a simulation of seismic wave propagation at low frequencies and low shear strain. The oscillation is generated by a pair of electromagnetic drivers at the bottom, transmitted to an elastic standard with known mechanical properties, and the specimen embedded within a furnace. The strain of the elastic standard and the specimen assembly was measured by two pairs of capacitance transducers.

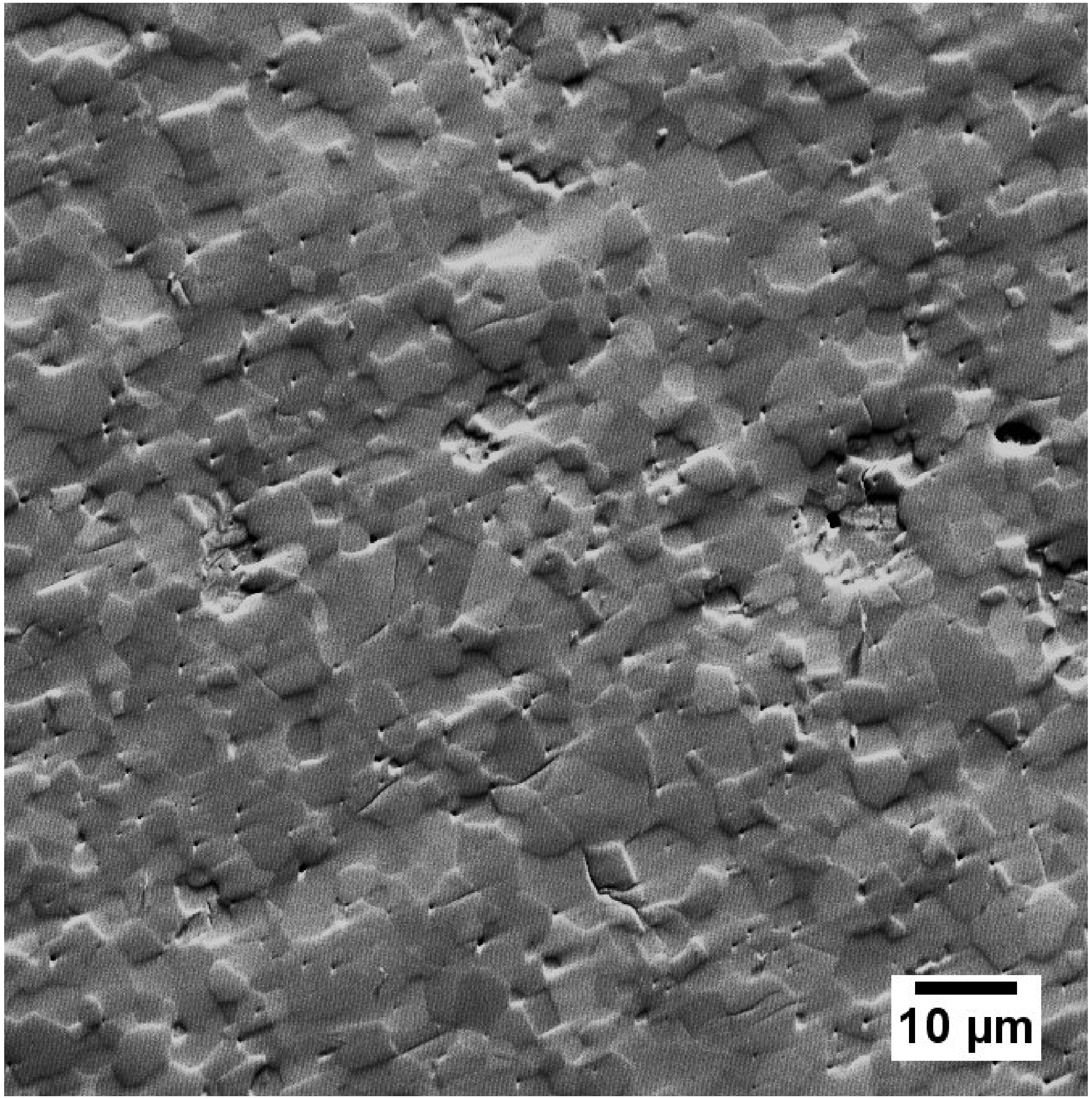
The olivine specimens (Fo90 ) were fabricated by the solution-gelation method within $Fe^{2+}$ stable field. A few % pyroxene (En90) exist in the sample to buffer the activity of silicon. During the high-temperature testing, a Ni70Fe30 liner wraps the cylindrical surface of the olivine specimen to stabilize logfO2 at -11.1 (CCO buffer) and prevents intrusion of Cr and Mn from the stainless-steel jacket.

Two parallel reference experiments with an SS-jacketed SS specimen and an SS-jacketed sapphire specimen were also conducted to obtain the mechanical properties of the jacket material. Such information concerning the viscoelastic behavior of SS is used to subtract the torsional stiffness of the SS jacket from the properties of SS-jacketed olivine and thereby isolate the mechanical behavior of olivine.

# MICROSTRUCTURES

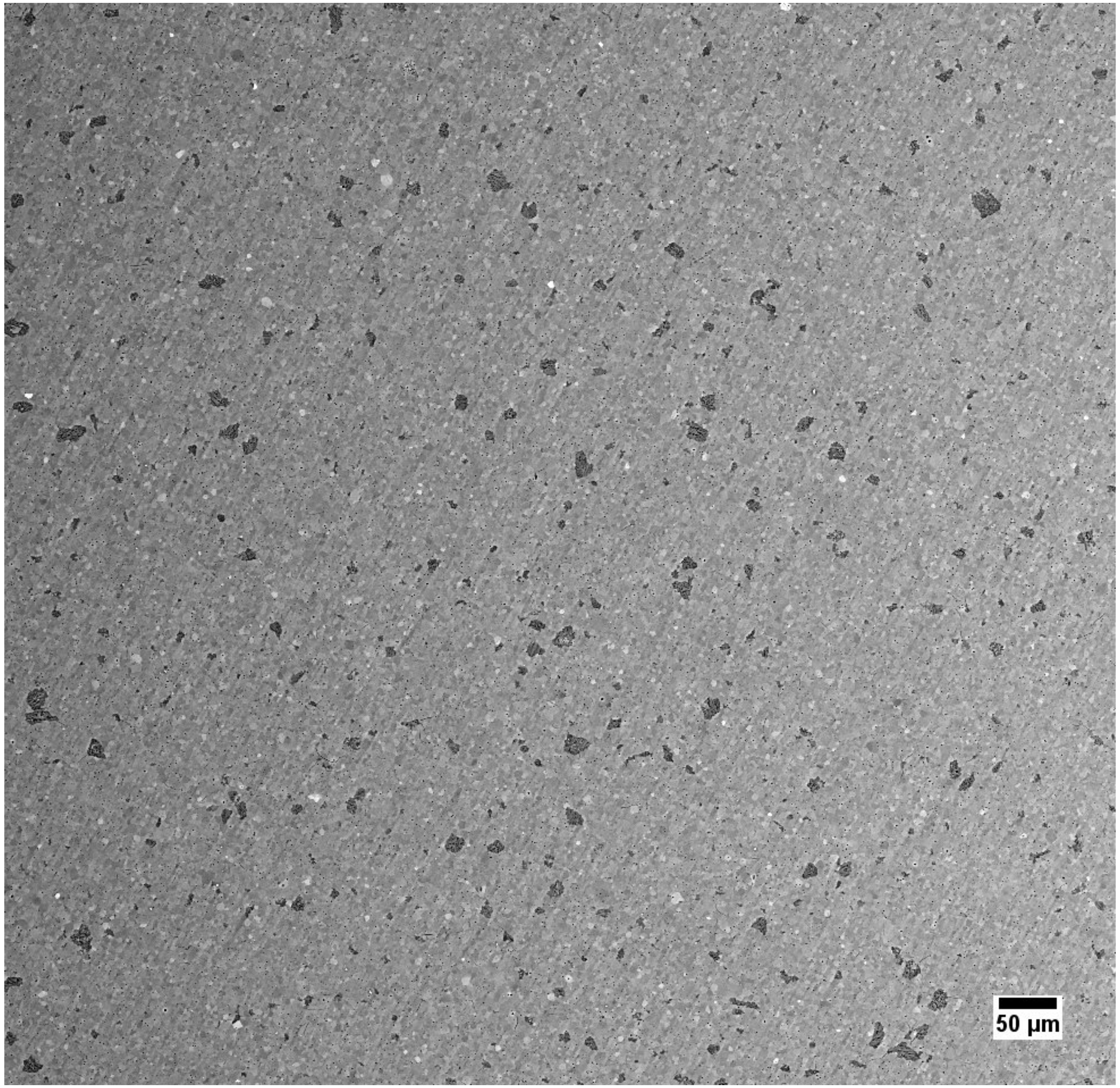
The grain size and chemical composition (95% Fo90 and 5% En90) are generally homogeneous in the sample (see SE and BSE images). The porosity is about 2 - 3%. The mean grain size of olivine is 3.7 micron, acquired by EBSD.

**SE**



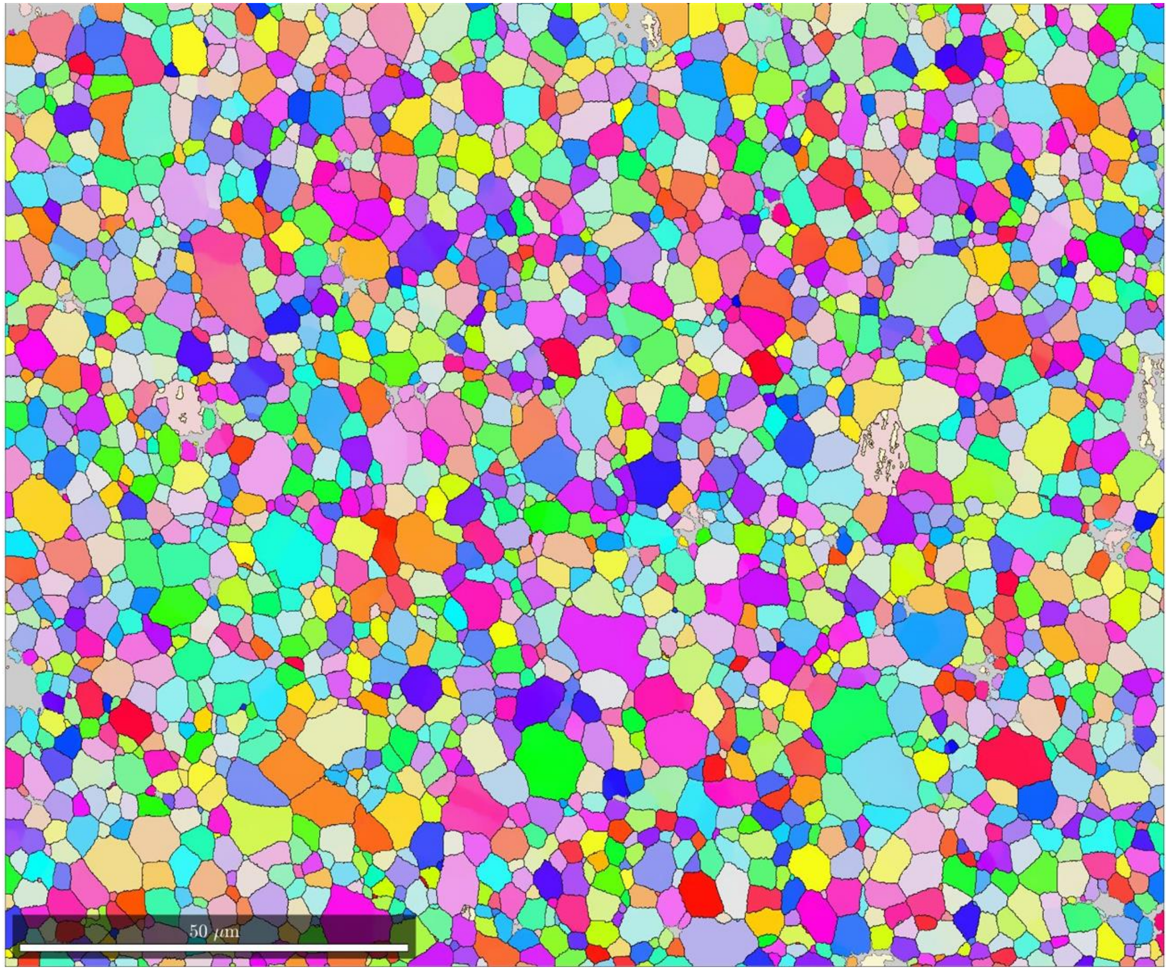

This image is taken after tilted the sample stage by 70° in SEM chamber.

**BSE**



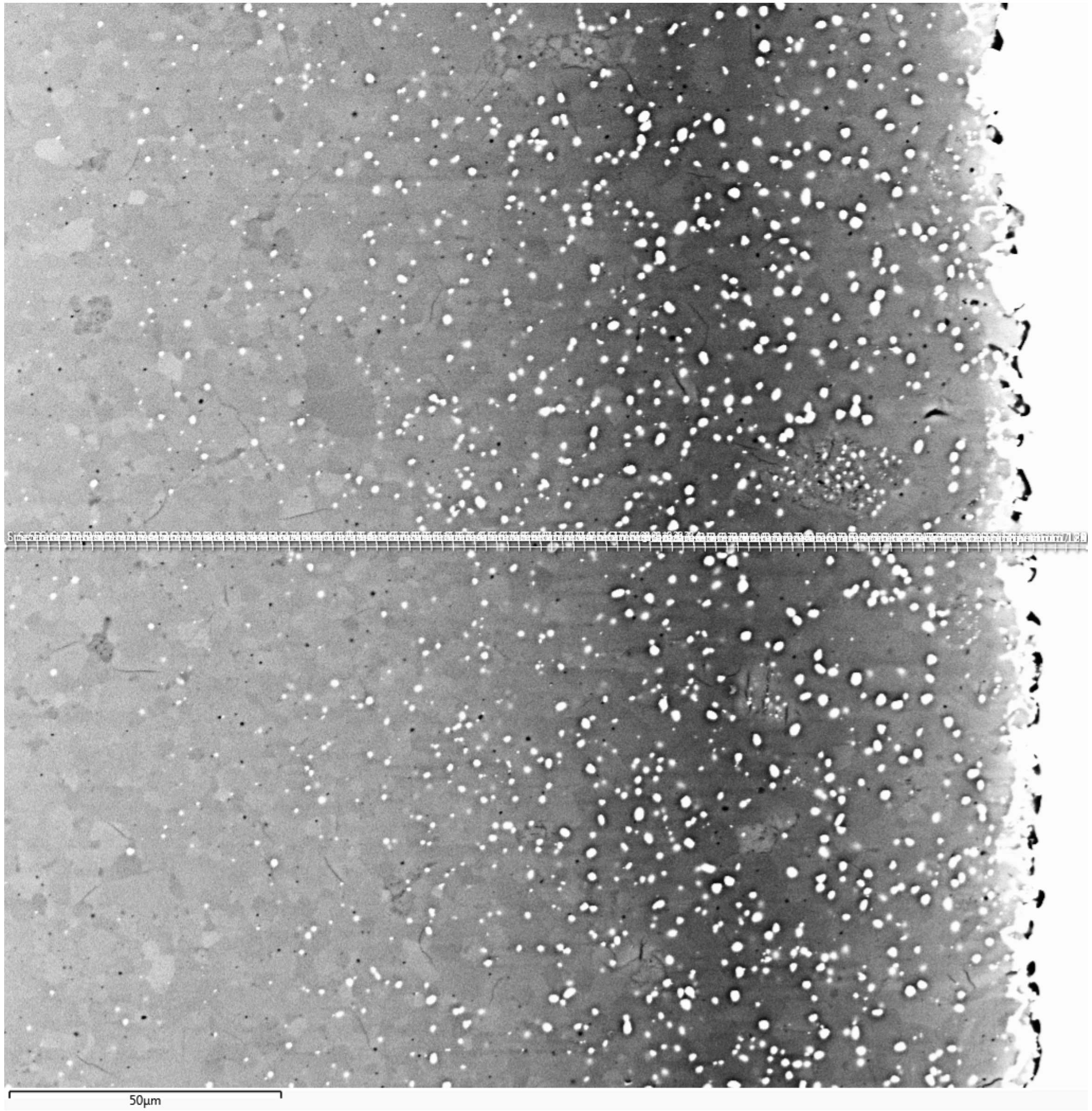

The streaking is a polishing artifact. The dark phase is En90. The light phase is Fo90.

**EBSD**



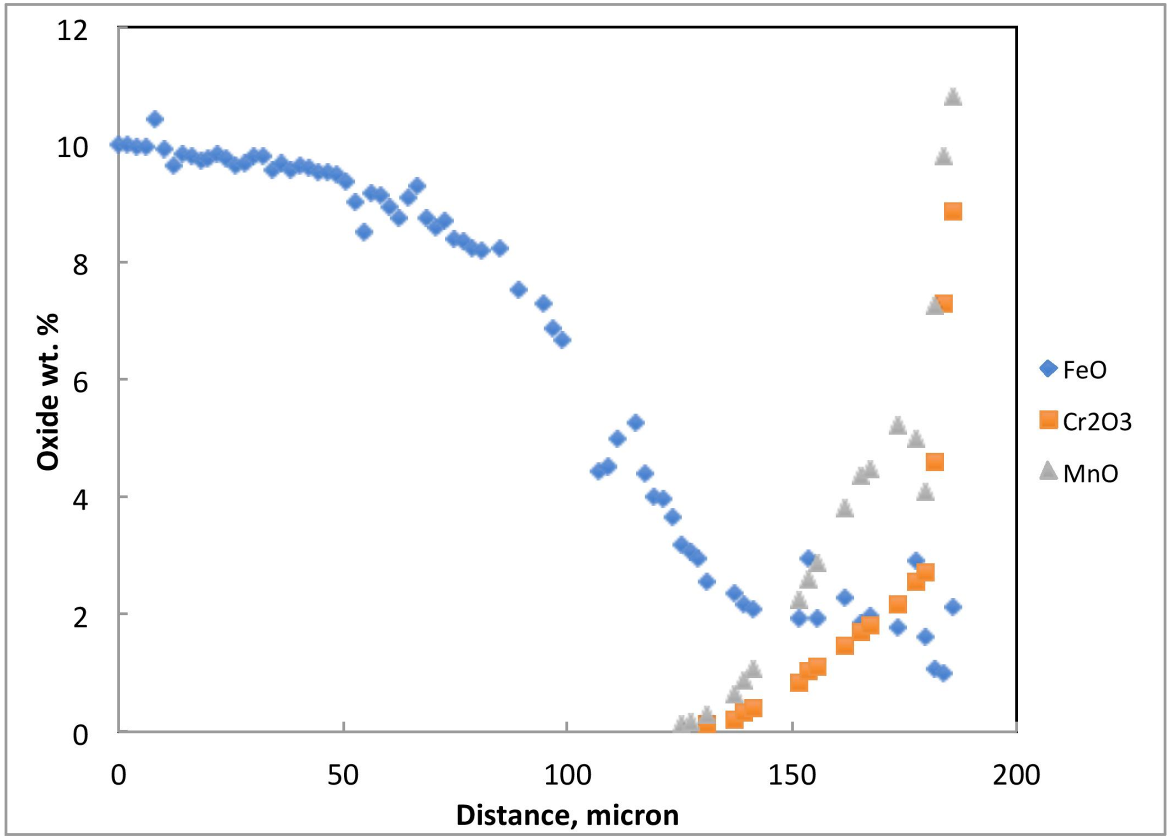

The map was acquired with a step size of 100 nm, the indexing rate for olivine was > 90%. There are over 2700 grains on the map.

**Diffusion of Cr and Mn from jacket into olivine**



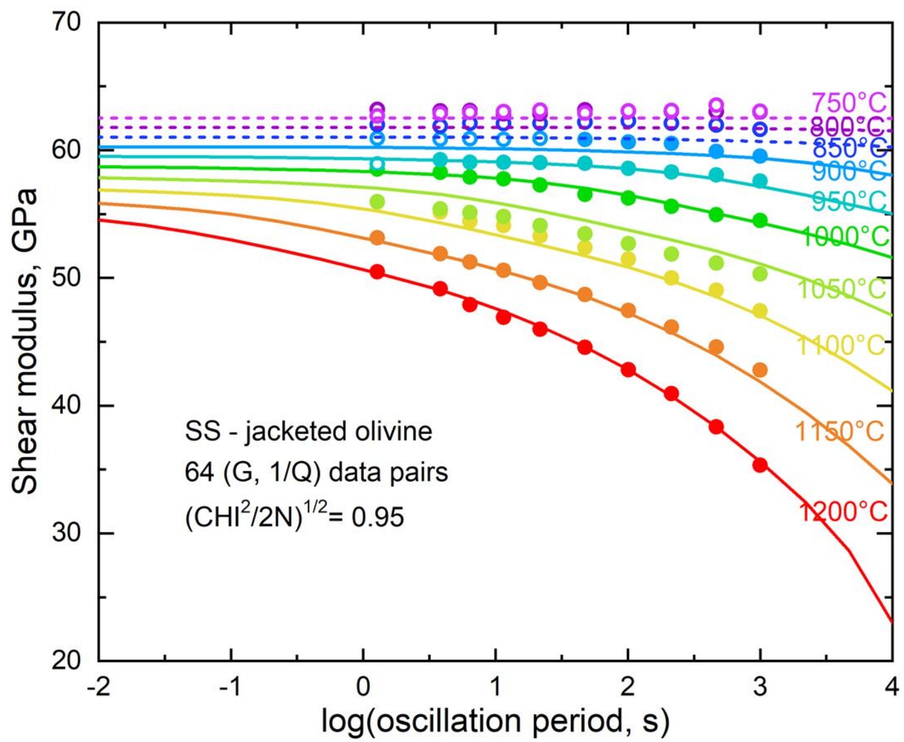

The Fe in olivine adjacent to the jacket was reduced and precipitated as blebs. The variation of FeO, Cr2O3 and MnO content in olivine grains is shown below, obtained by EDS. There is no Ni diffusion into the sample. The metal blebs are too small to get meaningful 'pure' metal analyses, but there is always Mg and Si present.

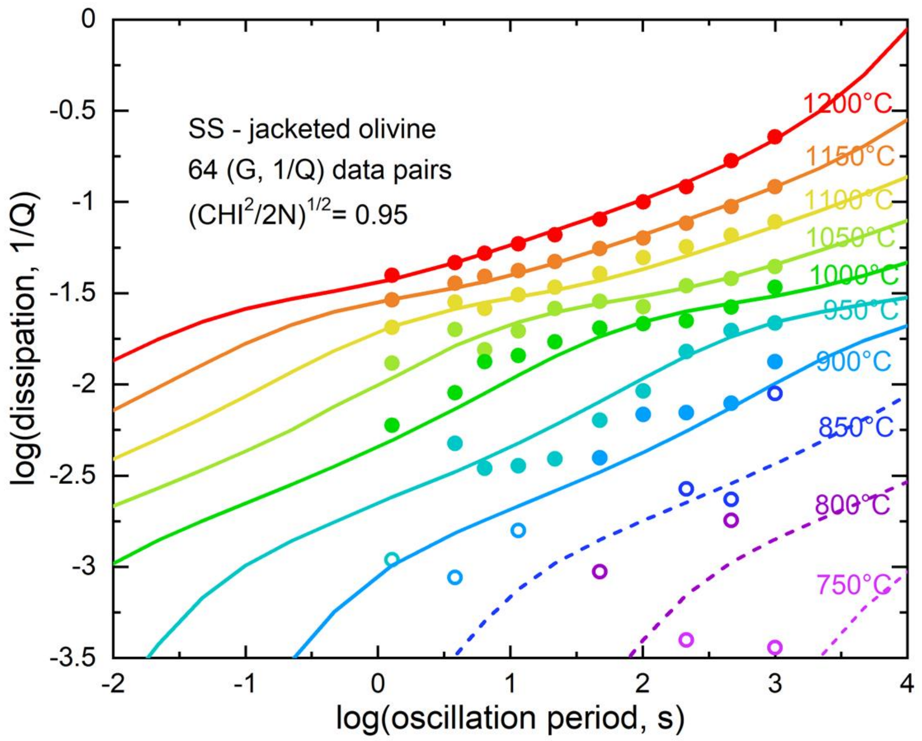
An interpretation of the Fe diffusion profile could be that this reflects fO2 changes. Presumably, oxygen has to diffuse from olivine to oxidize Cr and Mn diffusing from the liner/jacket into the sample, forming a rim of Fe loss in olivine and metal blebs. Sufficient isolation of olivine sample limits the width of the rim within 100 µm, provided by the NiFe liner.



# RESULTS

**Shear modulus and strain-energy dissipation as functions of period**



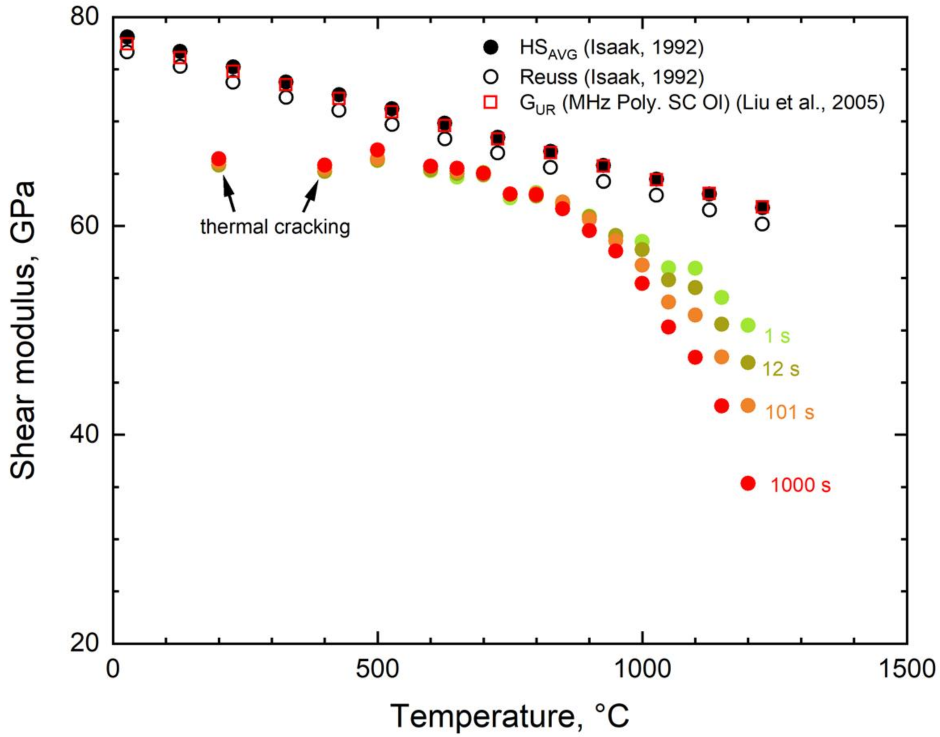



The main results from the testing are shear moduli and strain-energy dissipation at various temperatures (color-coded above) and oscillation periods (To), i.e. (G, 1/Q) = f(temp., To). In this stainless-steel jacketed olivine, a mild dissipation peak is observed from 900 to 1050 ºC, superimposed on the monotonic dissipation background. The dissipation peak and associated shear modulus dispersion, potentially attributable to elastically accommodated grain boundary sliding, display an Arrhenius dependence upon temperature - moving systematically to shorter periods with increasing temperature.

The transition between elastic and anelastic behavior is manifested as dissipation or dispersion of moduli at a trivial level, which occurs at medium frequencies (1 - 100 Hz) and/or medium temperatures (700 - 900 ºC). The minor anelastic relaxation brings challenges that the relatively high frequencies are mostly beyond the observational window of forced oscillation (0.001 - 1 Hz), while detection of the minor dissipation at the level of $10^{-2}$ to $10^{-3}$ at medium temperatures is demanding.

A rheological model, fitting the (G, 1/Q) data pairs at relatively high temperatures (solid lines), is used to infer the medium-temperature dissipation. Scattered medium-temperature dissipation data (hollow dots) largely agree with the extrapolation of the model (dashed lines).

**Shear modulus as functions of temperature**




In this plot, the oscillation periods are color-coded. The period dependence (dispersion) of shear moduli is notable from 850 to 1200 °C. Between 500 and 850 °C, the temperature dependence of shear moduli is similar to that of anharmonic values, but the absolute values are consistently lower by ~ 4 GPa.

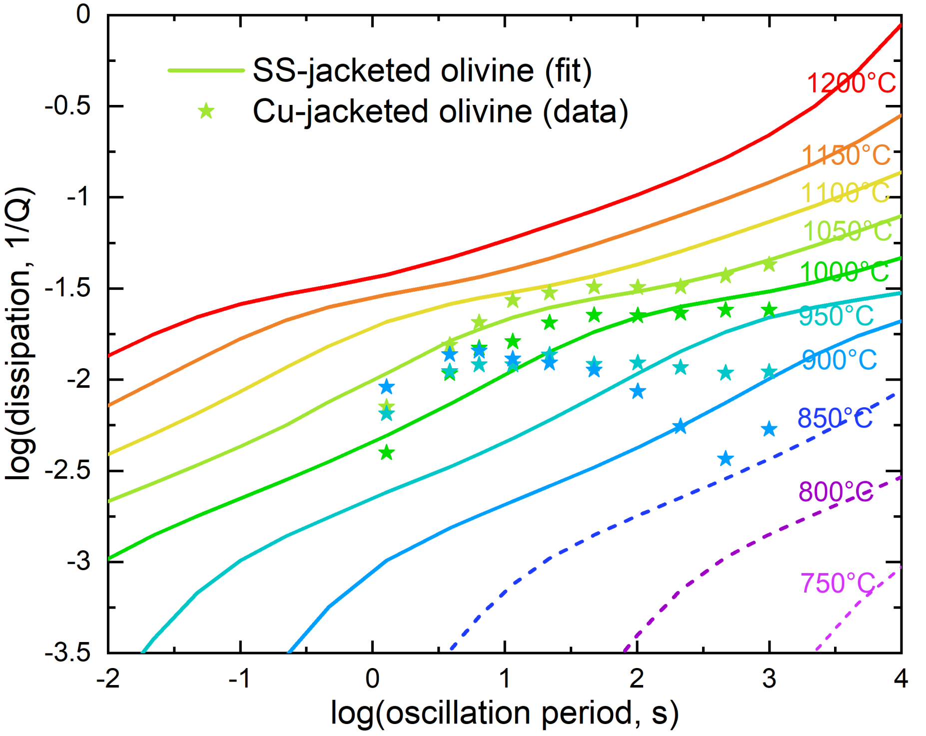
Such an offset is possibly caused by the porosity, thermal cracking during cooling, interfacial compliance at piston-piston or piston-sample interfaces, and/or bending of the vertical member (specimen assembly + steel standard).

# COMPARISON WITH RESULTS OBTAINED WITH OTHER JACKET MATERIAL

A meaningful comparison of forced oscillation results requires comparable experimental conditions (e.g. temperature, pressure, frequency), and similar characteristics of the tested samples including grain size, dislocation density, etc.

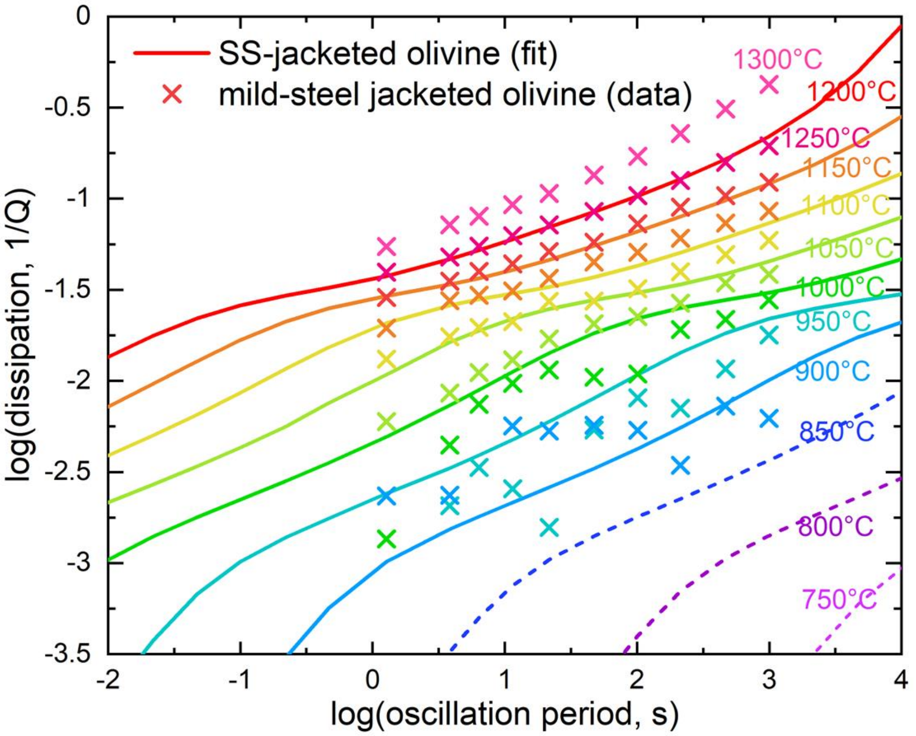
### Cu-jacketed olivine

A Cu-jacketed fine-grained olivine aggregate, similarly avoiding the phase transition in the metal sleeve in the test temperature range, was fabricated and tested by the same method. The obtained dissipation data were closely compared with dissipation in SS-jacketed olivine in the following figure. The peaks at 1000 °C and 1050 °C are repeatedly observed in the dissipation spectrum of Cu-jacketed olivine.



**Mild-steel-jacketed olivine**

Recycled from SS-jacketed specimen assembly, the same sample was jacketed in routine mild steel and tested from higher annealing temperature 1300 °C to 900 °C. Although the behavior is qualitatively similar, the olivine specimen is systematically less dissipative when tested within a mild-steel jacket than the fit for the SS-jacketed specimen. This difference is possibly due to grain growth during the long annealing at 1300 °C. There is again evidence of a broad dissipation peak - centered near 30 s period at 1050°C – superimposed upon the background.



# CONCLUSION AND FUTURE DIRECTION

**Conclusion**

1. Mild dissipation peak is repeatedly observed in the testing results of dry and melt-free olivine aggregates between 900 $^{o}$C and 1100 $^{o}$C within seismic frequencies where the dissipation level ($Q^{-1}$) is as low as $10^{-2}$, regardless of the jacket material.

2. The fitted rheological model enables the extrapolation of the data to lower temperatures, inferring the seismic properties of fine-grained olivine during the onset of anelastic behavior.

**Future direction**

The grain size sensitivity of the observed dissipation peak has not been explored. Whether the grain size sensitivity of the peak is similar to that of the background remains to be answered by follow-up tests on olivine aggregates with coarser grain size.

Acknowledgment

Many thanks to Hayden Miller and Harri Kokkonen (RSES, ANU) for technical assistance, Dr. Hua Chen and Dr. Frank Brink for the guidance of SEM (CAM, ANU). The EBSD and chemical composition analysis was conducted at SUNY New Paltz Analytical Facility (https://semsunynp.com/).

# AUTHOR INFORMATION

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# ABSTRACT

Seismic-frequency torsional forced-oscillation measurements of shear modulus dispersion and associated strain-energy dissipation under conditions of simultaneously high temperature and pressure in our laboratory require the specimen to be enclosed within a thin-walled metal sleeve – typically of mild steel. However, the well-known transition between the austenite and ferrite phases on cooling across the interval 900-700ºC complicates its mechanical behaviour. The resulting uncertainty tends to mask the seismologically important onset, within this temperature range, of appreciably anelastic behaviour of polycrystalline olivine. In order to more closely document this important transition in mechanical behaviour, we have conducted an unpublished study in which of a specimen of polycrystalline olivine is jacketed within a copper sleeve which retains its face-centred-cubic (fcc) structure throughout the range of the measurements limited to 1050ºC by the proximity of the melting point. Here we report measurements to a higher temperature (1200ºC) in which we employ austenitic (fcc) stainless steel (SS) as alternative jacket material. Two parallel reference experiments with a SS-jacketed SS specimen and a SS-jacketed sapphire specimen were also conducted to obtain the mechanical properties of the jacket material. Such information concerning the viscoelastic behaviour of SS is used to subtract the torsional stiffness of the SS jacket from the properties of SS-jacketed olivine and thereby isolate the mechanical behaviour of olivine. The resulting dissipation spectrum for the olivine specimen consists of a monotonic background dissipation with a superimposed peak located within the 1-1000 s period range for temperatures between 900 and 1050ºC. The dissipation peak and associated shear modulus dispersion, potentially attributable to elastically accommodated grain boundary sliding, display an Arrhenius dependence upon temperature - moving systematically to shorter periods with increasing temperature. Comparison of the results obtained for sol-gel $Fo_{90}$ olivine specimens enclosed within the alternative mild-steel, copper, and stainless-steel jackets will provide new insight into the nature of the seismologically important transition between the elastic and anelastic regimes.