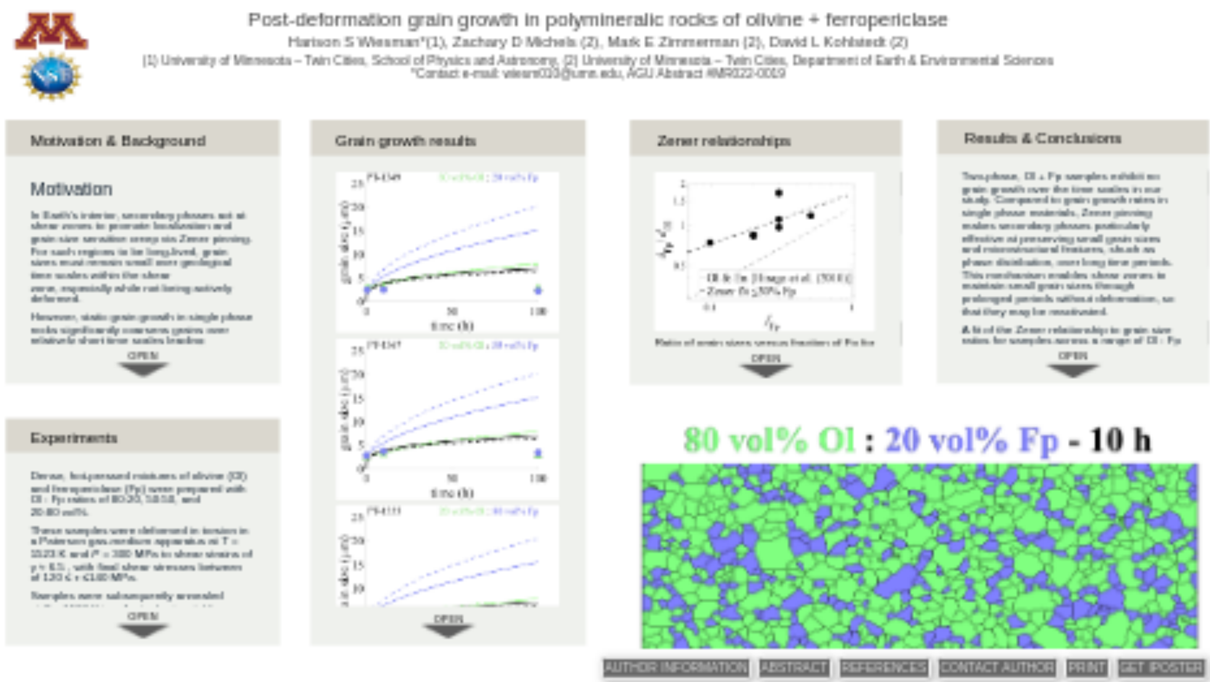
Post-deformation grain growth in polymineralic rocks of olivine + ferropericlase



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Motivation & Background

Motivation

In Earth's interior, secondary phases act at shear zones to promote localization and grain size sensitive creep via Zener pinning. For such regions to be long-lived, grain sizes must remain small over geological time scales within the shear zone, especially while not being actively deformed.

However, static grain growth in single phase rocks significantly coarsens grains over relatively short time scales leading to strengthening of the material. Slower rates of grain growth occur in two-phase materials, but these experiments have typically been carried out on undeformed materials.

In this study, grain growth experiments were carried out on two-phase samples to observe the effects of secondary phases on grain growth kinetics in materials that have undergone large shear strain deformation.

Theory

Static grain growth in single- and multi-phase systems is a process whereby a polycrystalline material reduces energy stored in grain and phase boundaries by increasing its grain size. Grain growth is typically described by the equation

$$d^n - d_0^n = kt,$$

where d_0 the initial grain size, d is the grain size at time t, n is the grain growth exponent, and k is a growth rate coefficient.

In polymineralic rocks, grains of a secondary phase inhibit grain growth via grain and phase boundary pinning. Grain size is then described by the Zener relation,

$$d_{\mathrm{i}} / d_{\mathrm{ii}} = C / f_{\mathrm{ii}}^{\,m},$$

where d_{i} is the grain size of the primary phase, d_{ii} is the grain size of the secondary phase, f_{ii} is the fraction of the secondary phase in the system, m is the Zener exponent, and C is a constant.

Experiments

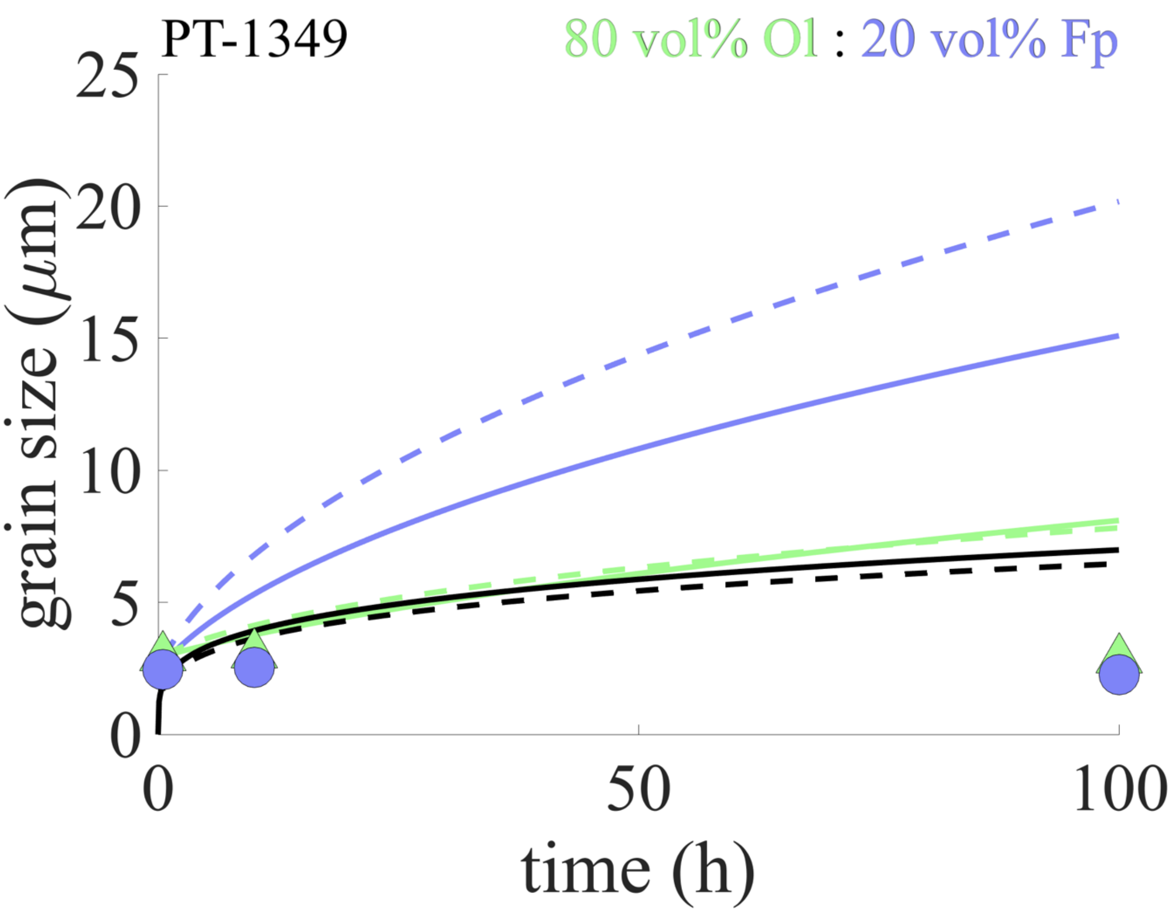
Dense, hot-pressed mixtures of olivine (Ol) and ferropericlase (Fp) were prepared with Ol : Fp ratios of 80:20, 50:50, and 20:80 vol%.

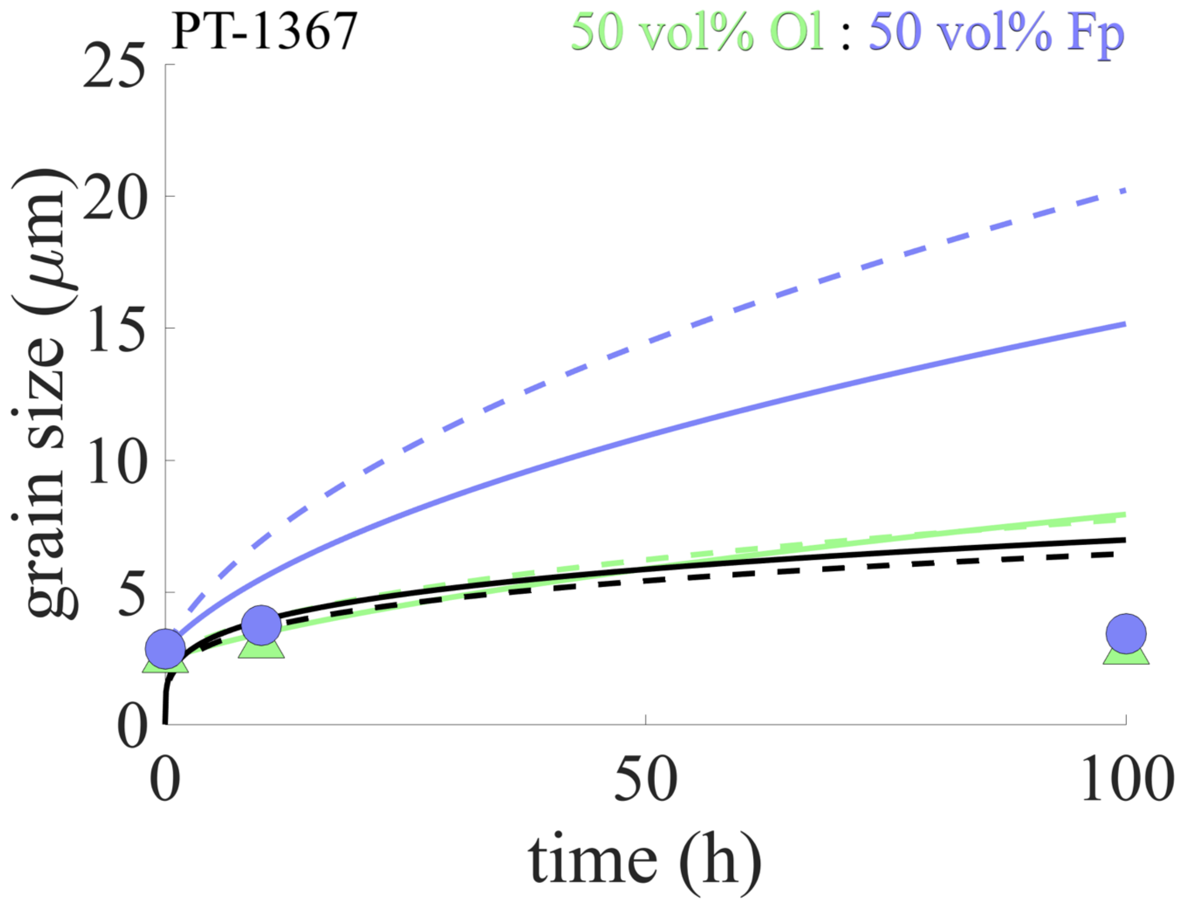
These samples were deformed in torsion in a Paterson gas-medium apparatus at $T = 1523$ K and $P = 300$ MPa to shear strains of $\gamma \approx 6.5$, with final shear stresses between of $120 \leq \tau \leq 140$ MPa.

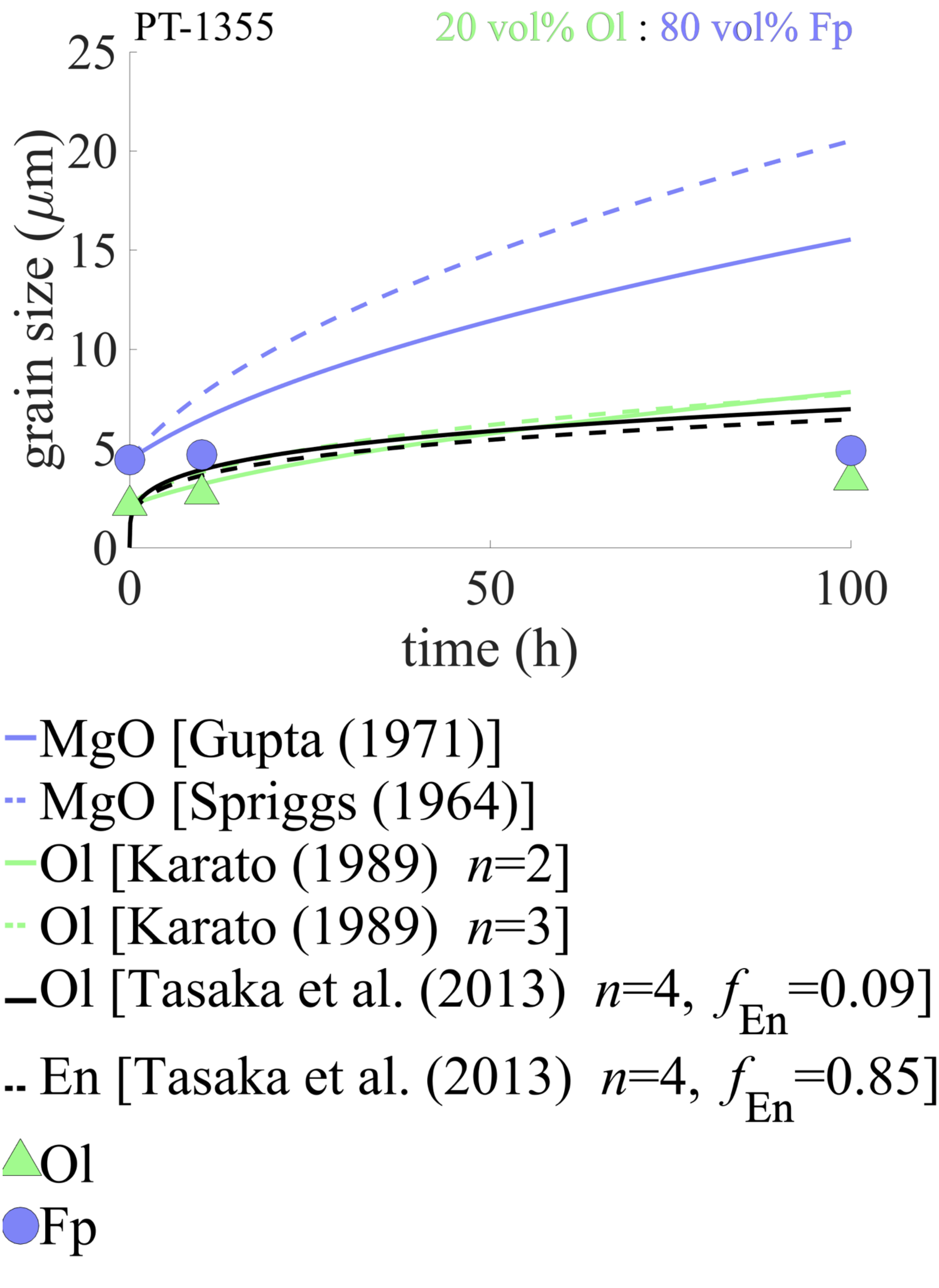
Samples were subsequently annealed at $T = 1523$ K in a 1-atm horizontal furnace for 10 h and 100 h with the oxygen fugacity controlled via mixed CO/CO_2 gas at the Ni/NiO buffer.

Grain size was measured with electron backscatter diffraction (EBSD) and calculated using the equivalent area method with a correction factor of $4/\pi$ applied. EBSD phase maps of each sample at each stage of annealing are presented in the slideshow.

Grain growth results




PT-1367
50 vol% Ol : 50 vol% Fp
grain size (μm)
time (h)
0
5
10
15
20
25
0
50
100



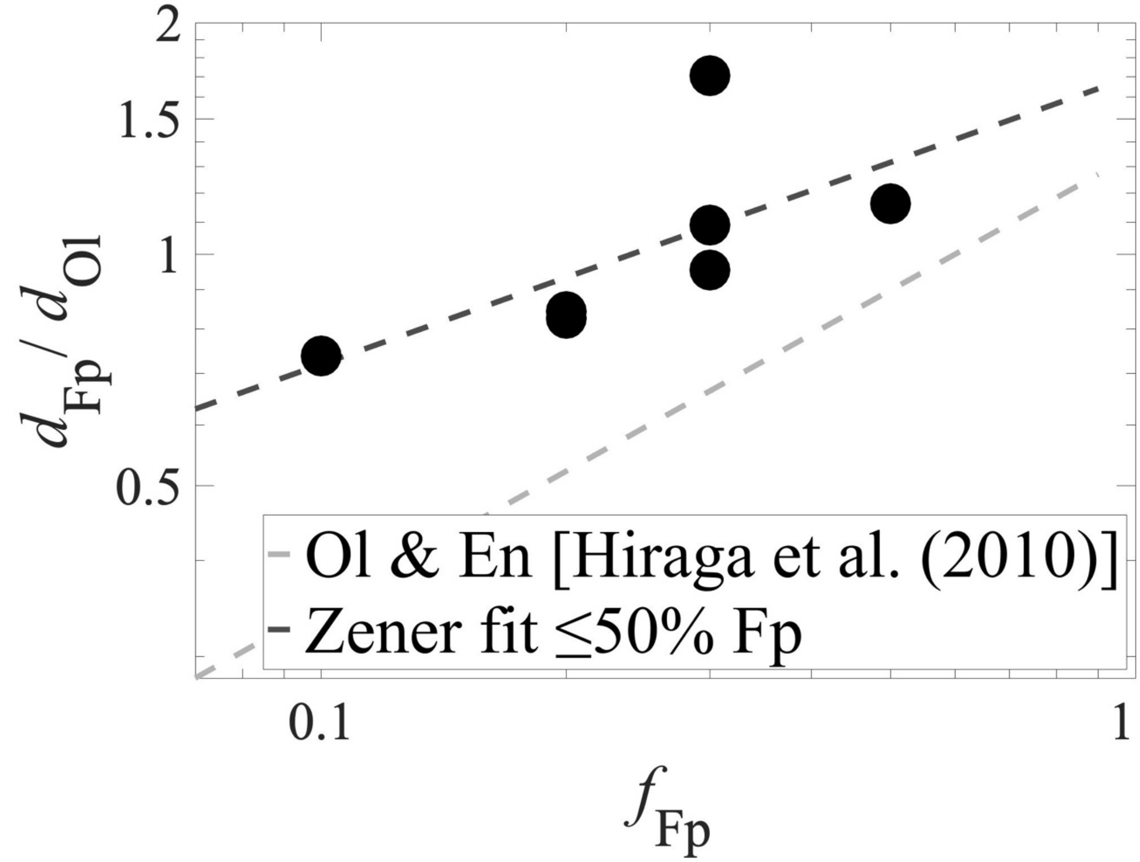
PT-1355
20 vol% Ol : 80 vol% Fp
grain size (μm)
time (h)
0
5
10
15
20
25
0
50
100
MgO [Gupta (1971)]
MgO [Spriggs (1964)]
Ol [Karato (1989) n=2]
Ol [Karato (1989) n=3]
Ol [Tasaka et al. (2013) n=4, f_En=0.09]
En [Tasaka et al. (2013) n=4, f_En=0.85]
Ol
Fp

Grain size of Ol and Fp versus annealing time for each of the three two-phase mixtures used in this study. Mean grain sizes reported are determined from a log-normal fit to the grain size distribution. Error in grain size is determined from the variance in the distribution, and is approximately the size of the data points. In each case, for the Ol + Fp system, little to no grain growth occurs across experimental time scales. Representative microstructures are presented in the slideshow.

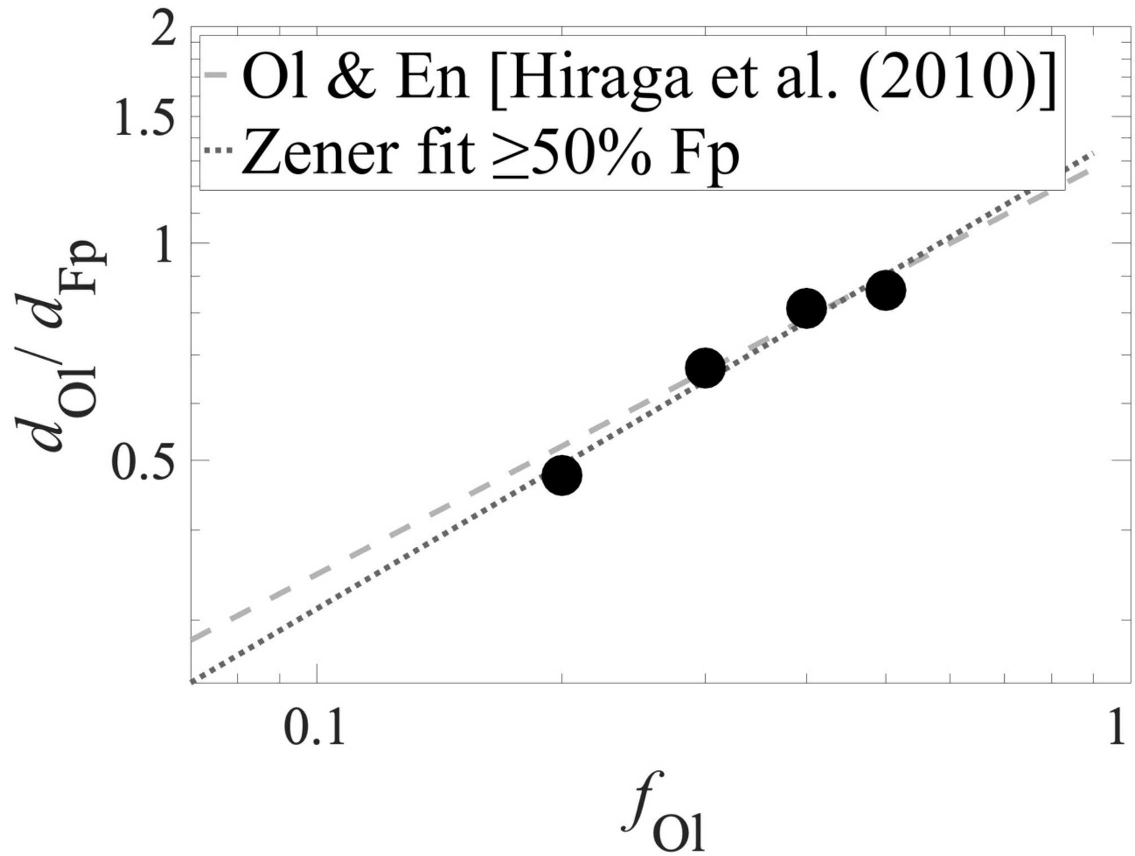
Grain growth laws for MgO (the iron-free end member of Fp) and Ol are plotted in each figure for comparison. Spriggs (1964) and Gupta (1971) grain growth laws are for dense and porous MgO aggregates respectively. The Ol grain growth laws were determined by Karato (1989) from porous olivine aggregates.

Along with the single phase grain growth laws, a two-phase grain growth law, determined by Tasaka et al. (2013) for the iron-free Ol + enstatite (En) system, is plotted for comparison. Here the grain growth laws are displayed for two different mixtures with 9 vol% En and 85 vol% En.

Zener relationships



Ratio of grain sizes versus fraction of Fp for samples containing ≤ 50% Fp fit using the Zener relation. This fit yields *m* = 0.38 indicating secondary phase particles occur predominately at three- and four-grain junctions [Evans et al. (2001)].



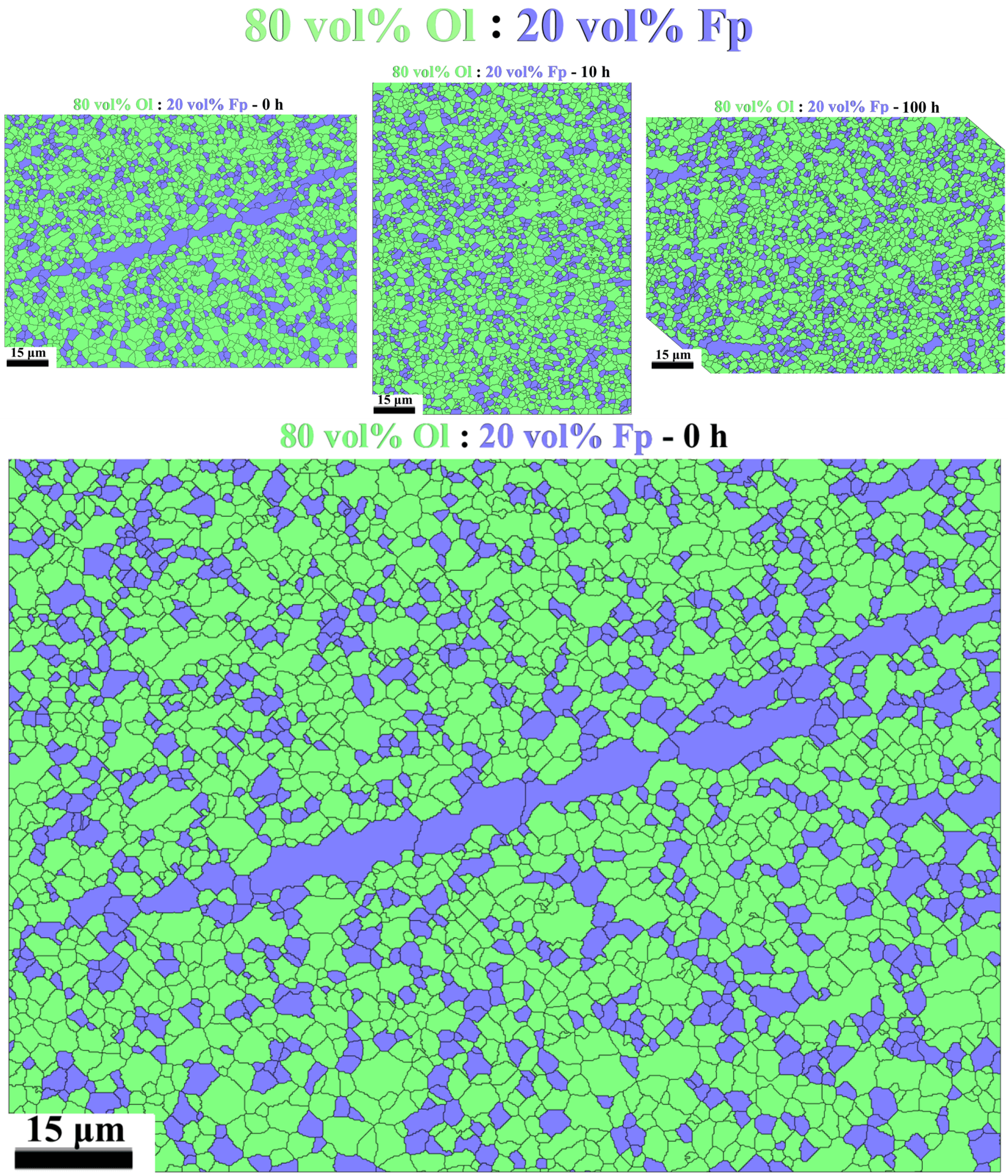
Ratio of grain sizes versus fraction of Ol for samples containing ≥ 50% Fp fit using the Zener relation. This fit yields *m* = 0.66 indicating secondary phase grains are small relative to the primary phase and occur along grain boundaries or are randomly distributed throughout the matrix [Evans et al. (2001)].

In each case, the Zener relation for the Ol + En system determined by Hiraga et al. (2010) with *m* = 0.59 is shown for comparison.

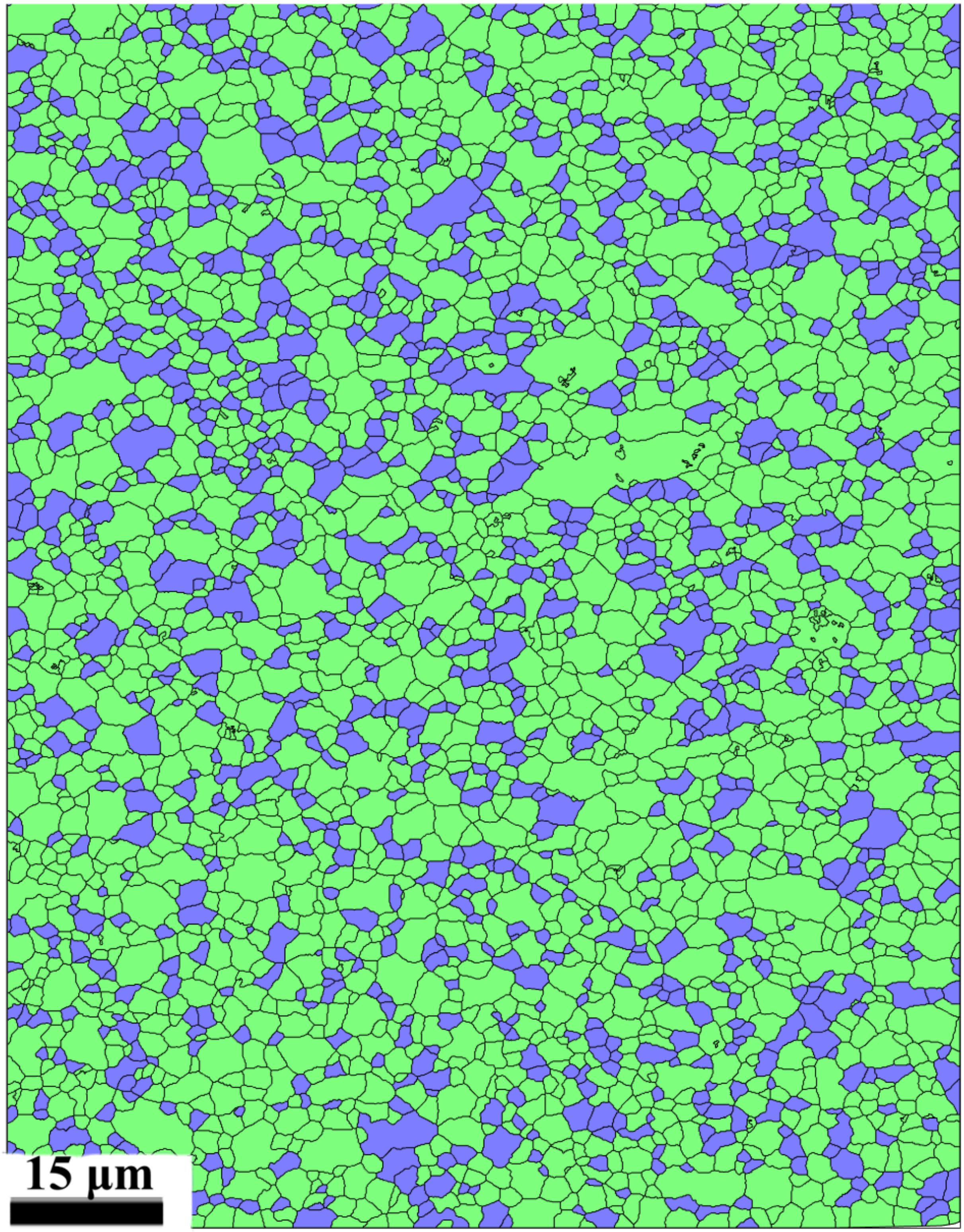
Results & Conclusions

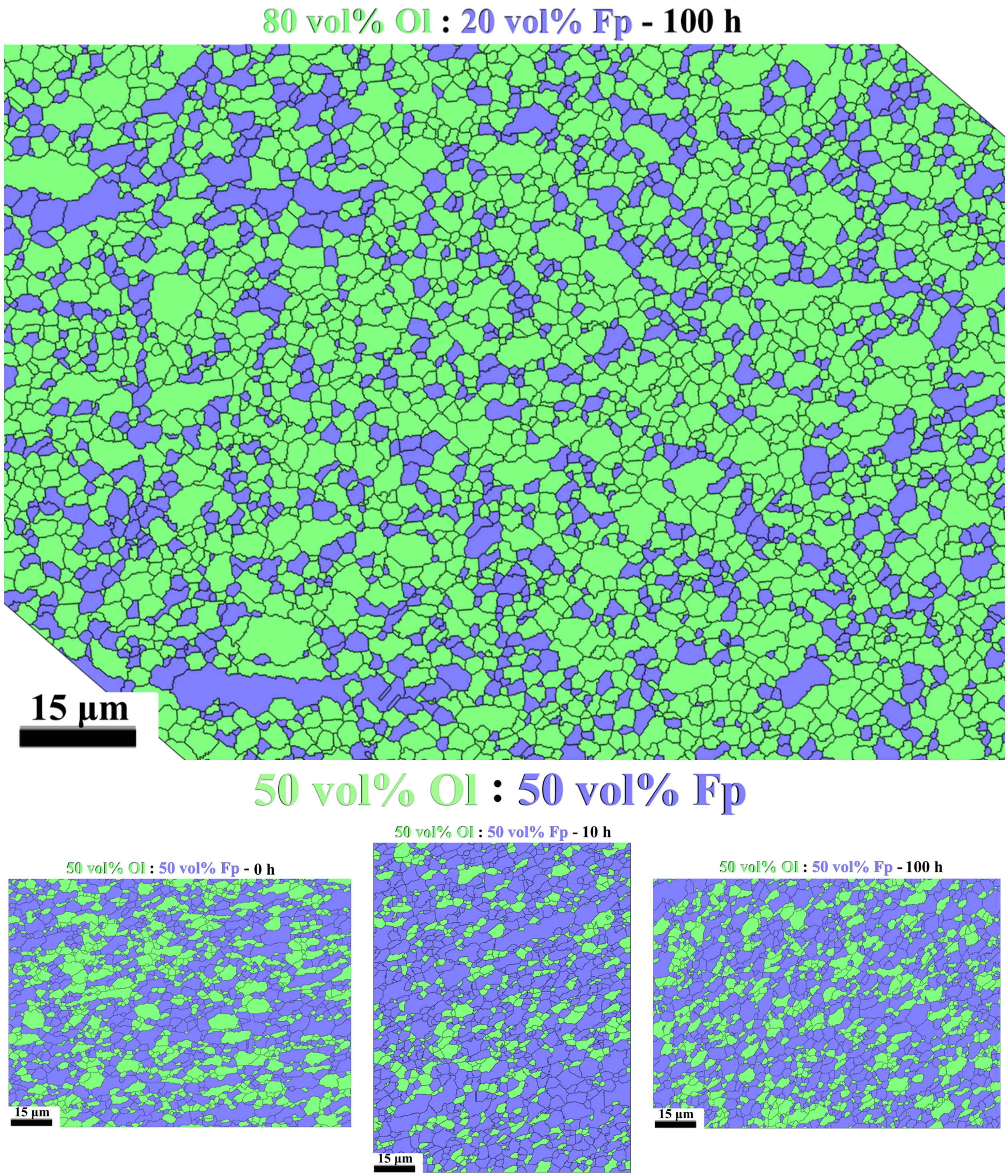
Two-phase, Ol + Fp samples exhibit no grain growth over the time scales in our study. Compared to grain growth rates in single phase materials, Zener pinning makes secondary phases particularly effective at preserving small grain sizes and microstructural features, shuch as phase distribution, over long time periods. This mechanism enables shear zones to maintain small grain sizes through prolonged periods without deformation, so that they may be reactivated.

A fit of the Zener relationship to grain size ratios for samples across a range of Ol : Fp volume ratios yields information about the spatial distribution of secondary phase particles among the matrix grains. In our case, the spatial distribution depends on whether Ol or Fp is the dominant phase; Fp particles occur primarily at three- and four-grain junctions in the Ol matrix, while Ol particles reside along grain boundaries in the Fp matrix. Microstructural observations support both of these conclusions.

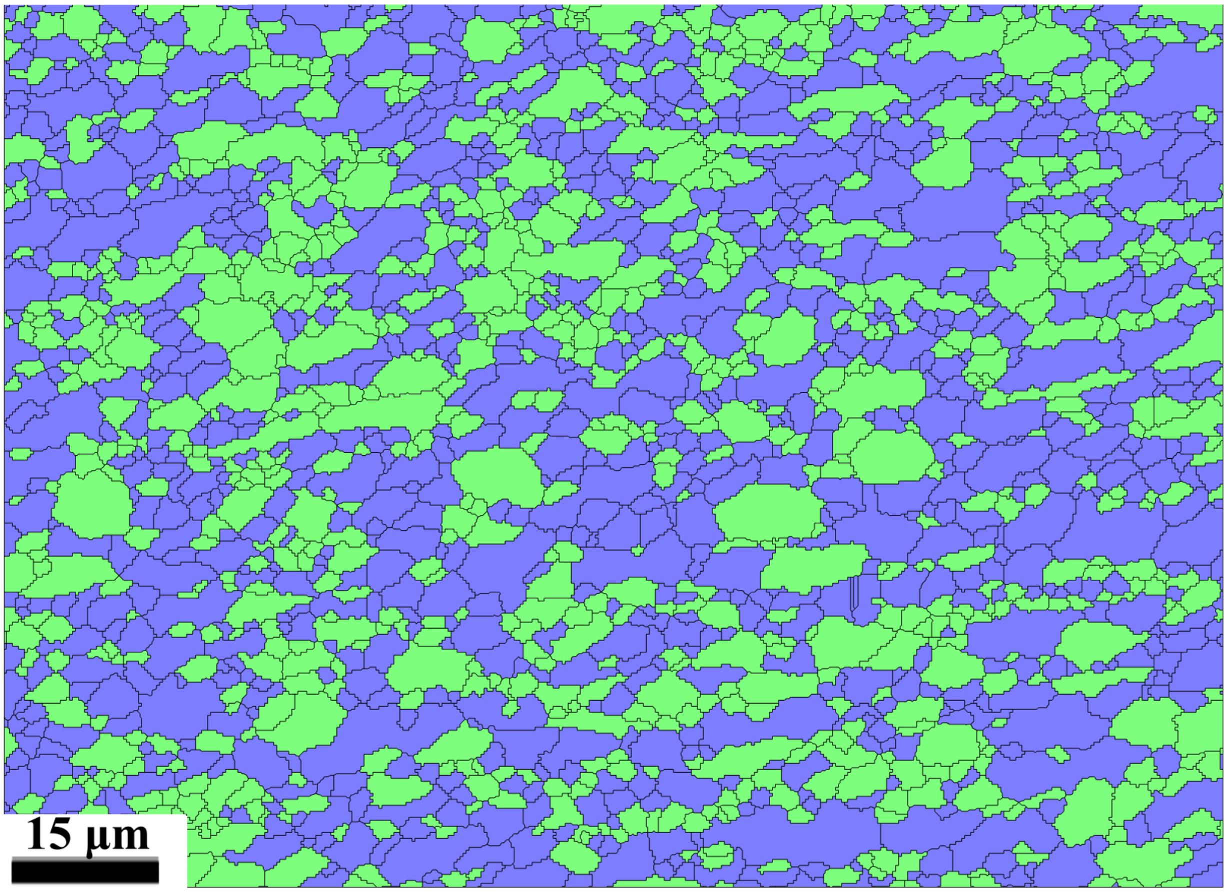


80 vol% Ol : 20 vol% Fp - 10 h

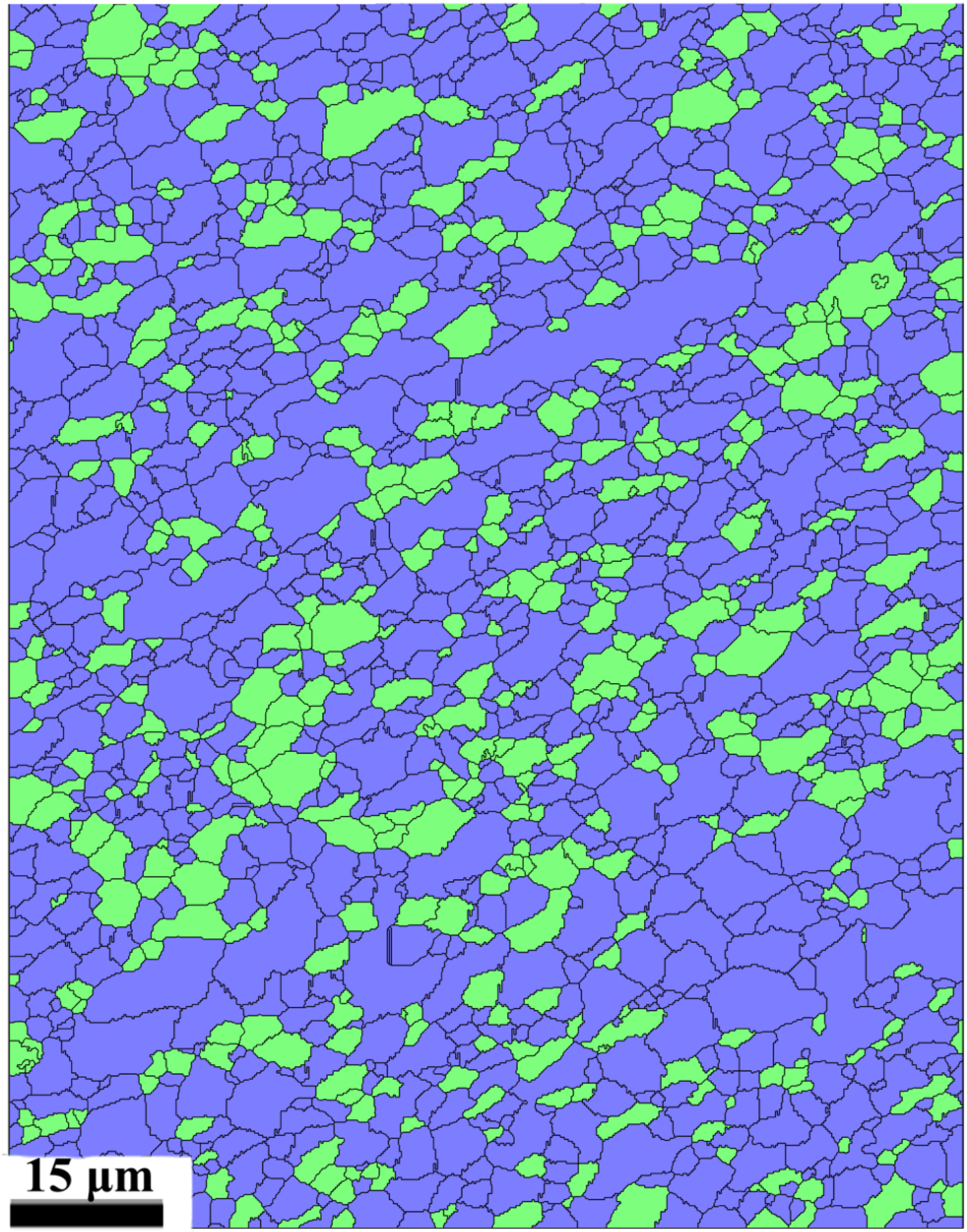


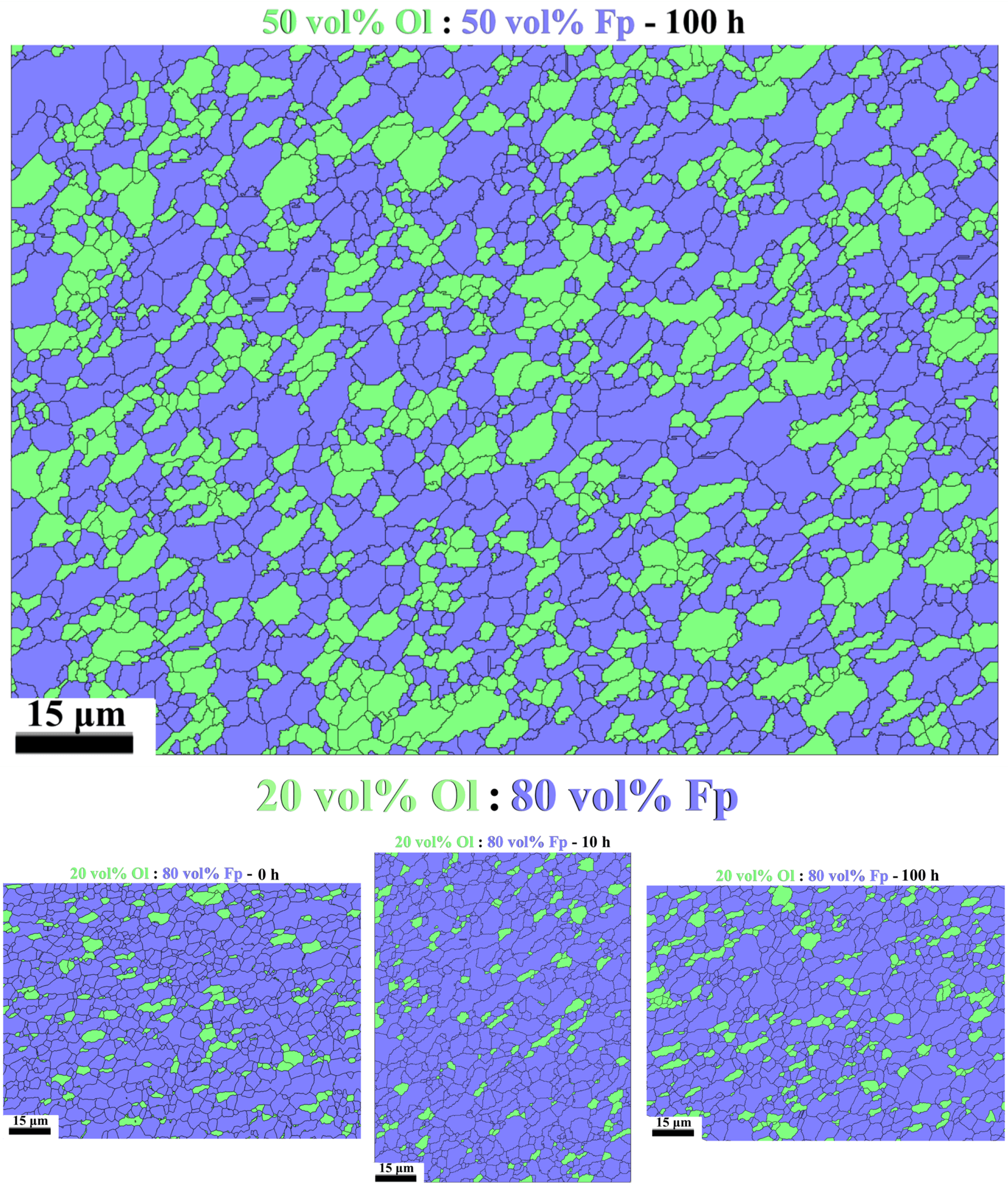

80 vol% Ol : 20 vol% Fp - 100 h
15 µm
50 vol% Ol : 50 vol% Fp
50 vol% Ol : 50 vol% Fp - 0 h
15 µm
50 vol% Ol : 50 vol% Fp - 10 h
15 µm
50 vol% Ol : 50 vol% Fp - 100 h
15 µm

50 vol% Ol : 50 vol% Fp - 0 h

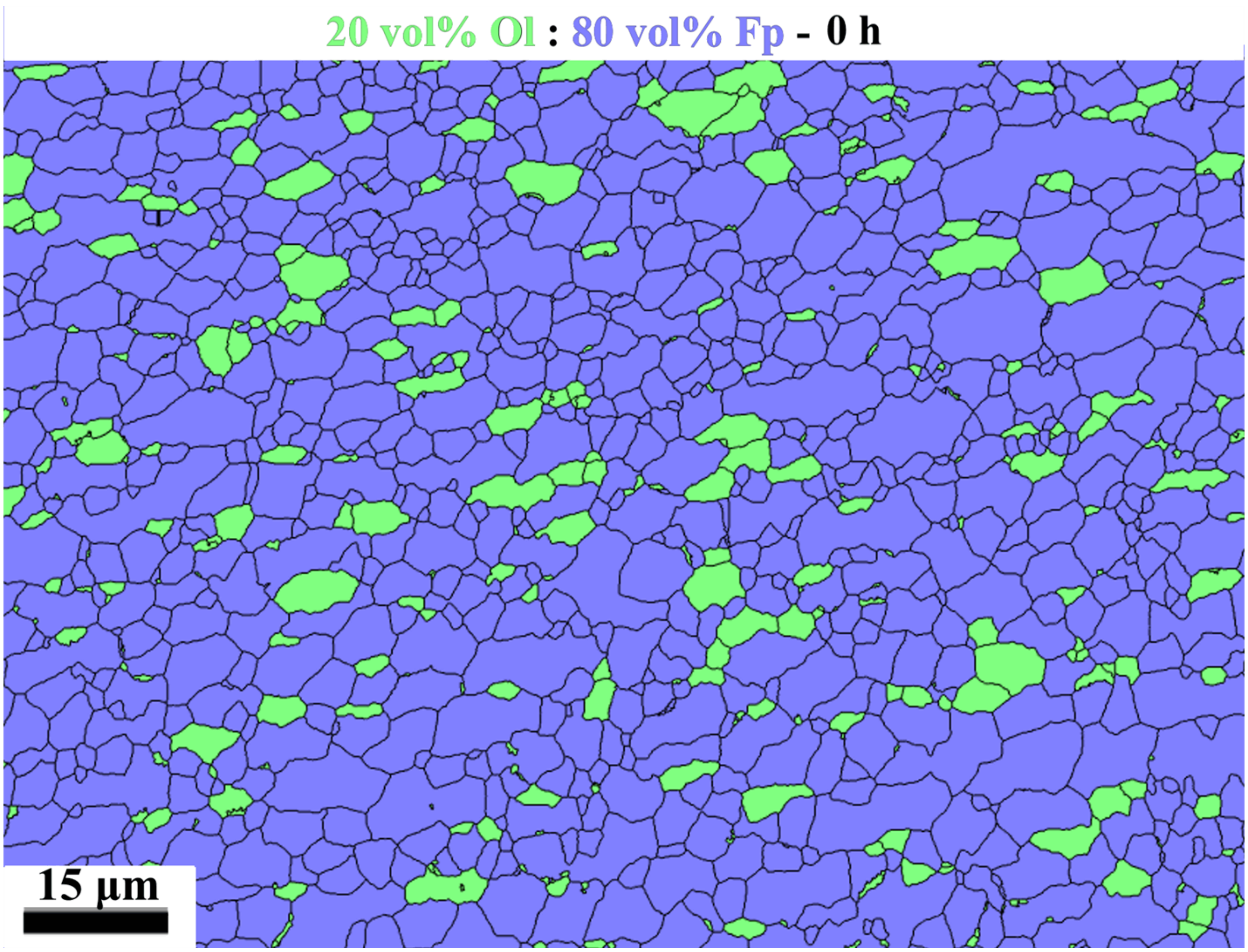


50 vol% Ol : 50 vol% Fp - 10 h

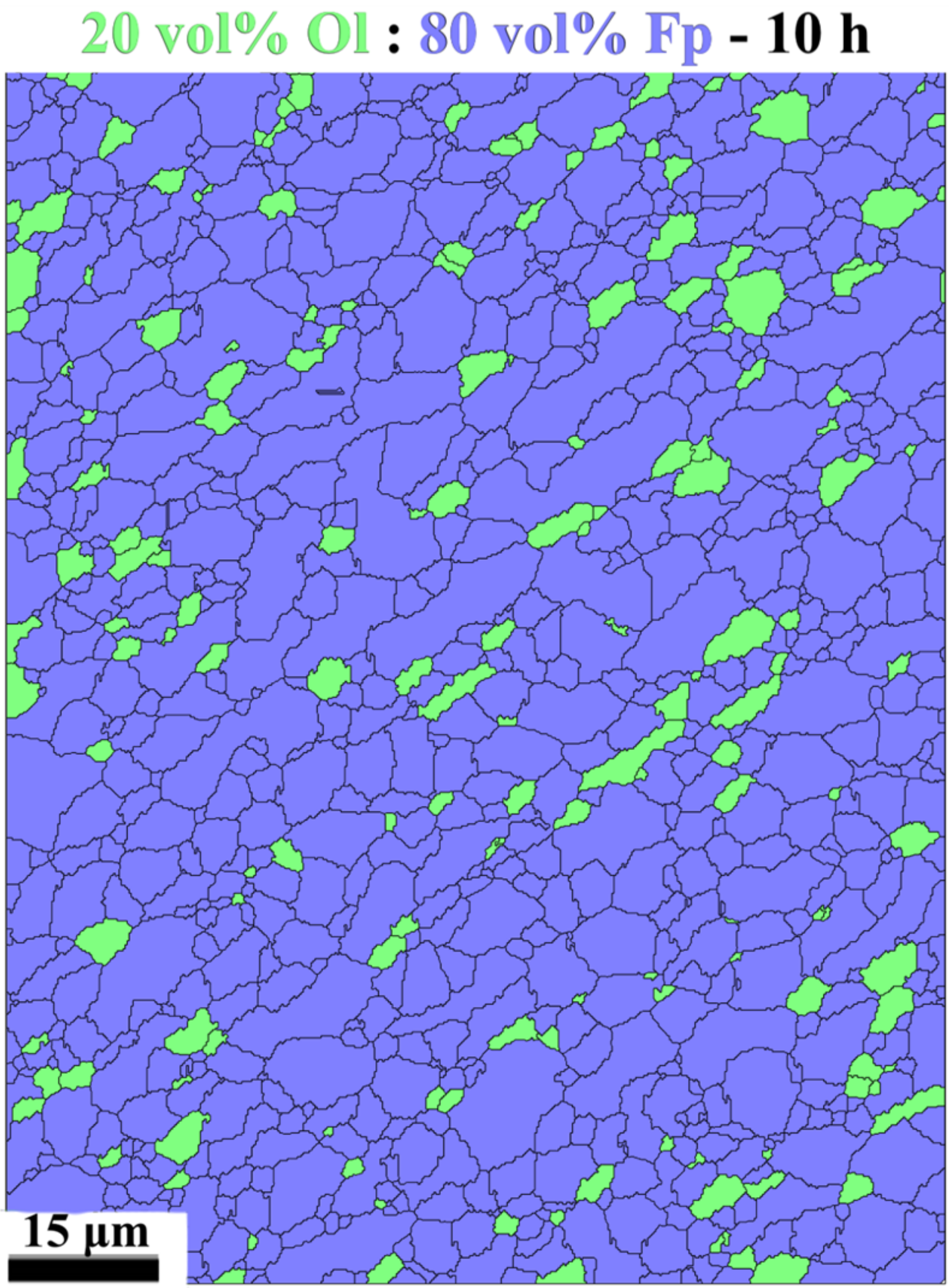


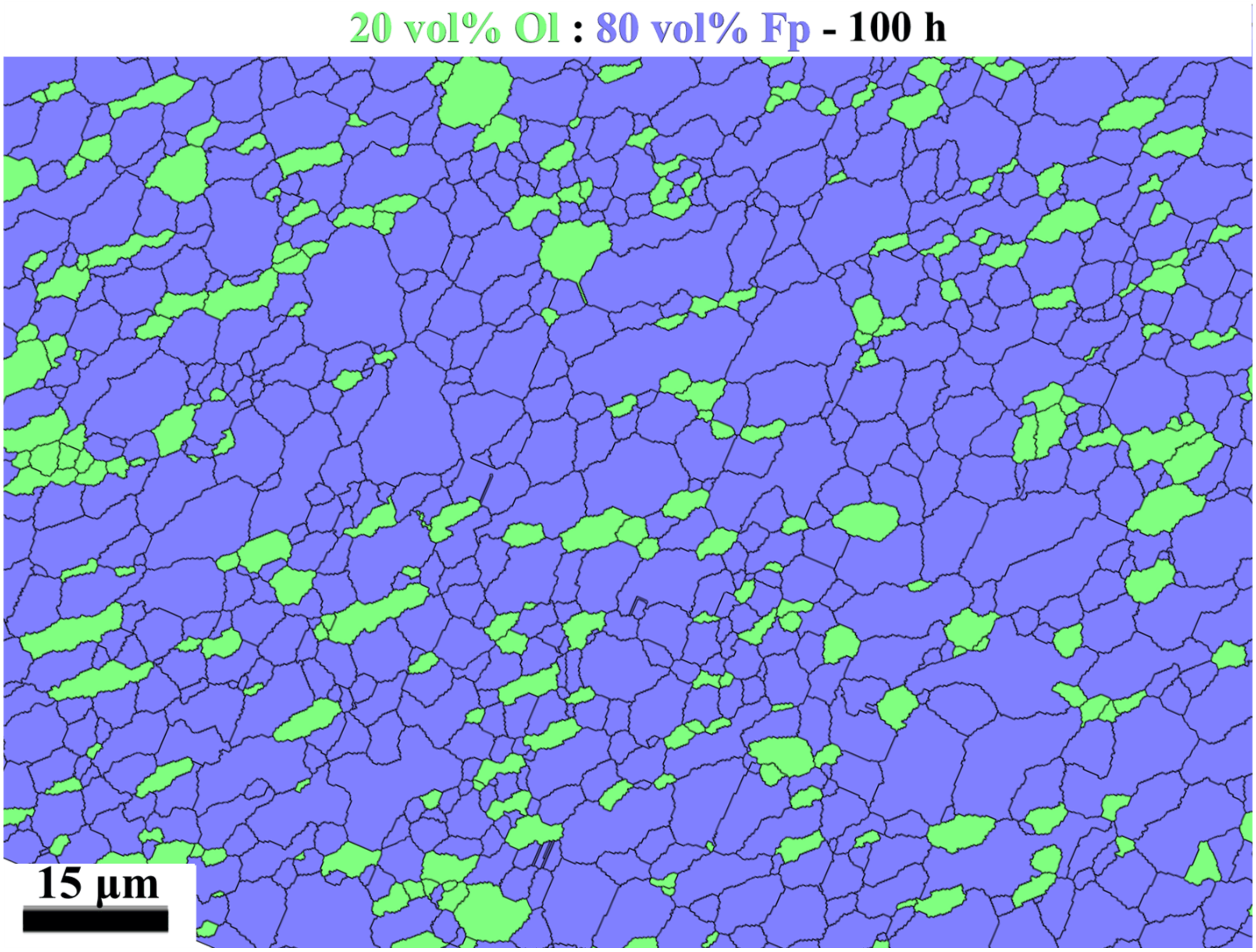


50 vol% Ol : 50 vol% Fp - 100 h
15 µm
20 vol% Ol : 80 vol% Fp
20 vol% Ol : 80 vol% Fp - 0 h
15 µm
20 vol% Ol : 80 vol% Fp - 10 h
15 µm
20 vol% Ol : 80 vol% Fp - 100 h
15 µm



20 vol% Ol : 80 vol% Fp - 0 h
15 µm


20 vol% Ol : 80 vol% Fp - 10 h
15 µm



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Abstract

Observations of rocks from exhumed shear zones clearly reveal that secondary phases strongly influence the mechanical and microstructural evolution of materials undergoing large-strain shear deformation. Through Zener pinning, secondary phases promote grain size sensitive creep, allowing deformation to localize in fine-grained regions. For the longevity of such shear zones over geological times, fine grain sizes must be maintained between episodes of deformation such that localization will continue in these regions in subsequent deformation events. Experimental studies of static grain growth on single phase materials demonstrate relatively fast rates of grain growth that would serve to undo grain size refinement under natural conditions. Although static

grain growth experiments on samples composed of two or more phases indicate slower growth rates of each phase, such studies have typically been carried out on undeformed material.

To investigate the effectiveness of secondary phases at inhibiting grain growth after large-strain deformation, analog samples were synthesized from olivine (Ol) and ferropericlase (Fp) powders with Ol:Fp ratios of 1:5 to 5:1. Samples were deformed in torsion in a triaxial gas-medium apparatus to shear strains of $\gamma = 3 - 7$ at $P = 300$ MPa and $T = 1523$ K to induce mixing between the two phases; specifically, the distribution of phase boundaries followed a random binomial distribution. Subsequently, sections of each sample were statically annealed at $P = 0.1$ MPa and $T = 1523$ K for 10 h or 100 h. Grain size measurements obtained via electron backscatter diffraction indicate that after 10 h of post-deformation annealing Ol grains are smaller by a factor of 1.5 and Fp grains are smaller by a factor of 3 than predicted from single-phase grain growth laws. Comparing the ratio of grain sizes of the two phases to the secondary phase fraction yields a power law fit with an exponent of ~ 0.4 in Ol-rich samples and ~ 1 in Fp-rich samples. These results, along with microstructural observations, indicate that secondary phase particles are primarily distributed along grain boundaries in the Ol-rich samples but are randomly dispersed in Fp-rich samples. Our results demonstrate that secondary phases are highly effective at pinning grain size during static annealing following significant deformation.

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