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# Evolving microstructure during experimental deformation of Maryland diabase



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#### A R T I C L E I N F O A B S T R A C T

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Dynamic boundaries comprised of brittle faults and ductile shear zones are an essential feature of Earth-like plate tectonics. The intermixing of mineral phases that have undergone dynamic recrystallization and deform via grain size sensitive creep may be an essential mechanism for maintaining weakness in shear zones over long time intervals. However, the strain and timescales necessary for creating this microstructure are not well understood. To determine the magnitude of shear strain necessary for phase mixing in mafic rocks, diabase was deformed in the Large Volume Torsion Apparatus at Washington University in St. Louis at high temperature (1270 - 1350 °C) and high pressure (1.9  $\pm$  0.1 GPa) conditions, to shear strains up to  $\gamma=9.5$  at strain rates of 2.1  $\times$   $10^{-6}$  – 3.4  $\times$  $10^{-5}$  s<sup>-1</sup>. Microstructural analysis of undeformed and deformed samples was conducted using optical and electron microscopy and electron backscatter diffraction (EBSD). The microstructure evolved from an igneous texture characterized by relatively large grains and widely spaced secondary phases to one of narrow compositional bands comprised mainly of small, recrystallized grains. Percent recrystallization, domain width, and degree of phase mixing was measured for each sample. None of the microstructures analyzed in this work were found to be in steady state by a strain of  $\gamma = 9.5$ . This evolution in microstructure was also accompanied by an inferred partial transition in deformation mechanism to a grain size sensitive diffusion creep regime. This study demonstrates that microstructure and therefore rheology of mafic rocks is expected to evolve over large strain and time scales. Strains greater than 60 may be necessary to create a steady state microstructure and hence a steady state rheology. Even at rapid geologic strain rates of 10<sup>-12</sup> s<sup>-1</sup>, achieving steady state rheology may require millions of years.

# 1. Introduction

An essential feature of Earth-like plate tectonics is the presence of dynamic boundaries, composed of brittle faults and ductile shear zones, which accommodate the relative motion of plates (e.g., Bercovici and Ricard, 2012; Montési, 2013; Ramsay, 1980). Ductile shear zones are observed in the geologic record as mylonites and ultramylonites, and are characterized by the development of strong foliation, grain-size reduction of two to three orders of magnitude, and the progressive intermixing of different mineral phases (e.g., Allard et al., 2021; Linckens et al., 2015; Stenvall et al., 2019; White et al., 1980). Ductile shear zones have been observed in numerous crustal and mantle lithologies including granitic (e.g., Stenvall et al., 2019), peridotitic (Linckens et al., 2015; Linckens and Tholen, 2021; Précigout et al., 2017), and gabbroic compositions (e.g., Allard et al., 2021; John et al., 2004; Mehl and Hirth, 2008). The evolution of mylonitic microstructure is expected to

be sensitive to the magnitude of strain (Cross and Skemer, 2017), the composition and proportion of minerals in the host rock (Linckens et al., 2015), the rheological properties of the minerals themselves (Cross et al., 2020), and the mechanisms of deformation (Gilgannon et al., 2020; Precigout et al., 2007; Warren and Hirth, 2006).

The evolution of deformation microstructures, in particular grainsize reduction and phase mixing, can create shear zones that are weaker than the surrounding rock and remain weak over long time intervals, allowing these shear zones to be reactivated repeatedly through multiple cycles of tectonism (e.g., Bercovici and Ricard, 2012; Stern and Gerya, 2018). Grain-size reduction can induce a transition from grain-size insensitive creep to grain-size sensitive (GSS) creep, leading to a reduction in strength (Rutter and Brodie, 1988). Phase mixing suppresses grain boundary migration and limits growth through grain boundary pinning forces according to the Zener pinning phenomenon (Evans et al., 2001; Olgaard, 1990; Smith, 1948). By inhibiting grain

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growth, phase mixing can keep minerals in the weaker GSS creep regime. The intermixing of mineral phases may occur via mechanical mechanisms (e.g., Ashby and Verrall, 1973; Bercovici and Skemer, 2017; Cross and Skemer, 2017) or chemical mechanisms such as metamorphism, chemical diffusion, or precipitation of secondary phases in cavities (Gilgannon et al., 2017; Precigout and Stunitz, 2016; Tasaka et al., 2017). Chemical phase mixing can be very efficient, especially under evolving metamorphic conditions. However, at high confining pressures (where cavitation is suppressed) (Brace and Kohlstedt, 1980), or where pressure, temperature, and composition are largely constant, mechanical mixing mechanisms may dominate. This may be the case for active shear zones in the lower crust as well as shear zones on other planetary bodies where conditions are expected to be dry, such as Venus.

The focus of the present study is rocks of mafic composition. Mafic rock is formed primarily through the partial melting of ultramafic mantle, is abundant on terrestrial planetary bodies in our solar system (Basilevsky et al., 1985; Ringwood and Essene, 1970; Surkov et al., 1983), and has been detected on rocky exoplanets (Kreidberg et al., 2019). Therefore, shear localization in rocks of mafic composition may be important to the initiation of plate tectonics on Earth and the development of stable tectonic regimes on other planetary bodies (Bercovici, 2003; Bercovici et al., 2015). The rheology of mafic rocks at low strains has been well-characterized through previous experiments on diabase (Caristan, 1982, 1980; Kronenberg and Shelton, 1980; Mackwell et al., 1998). However, with progressive deformation to greater strains, microstructure and consequently rheology are expected to evolve further. Rheology is assumed to reach steady state when additional strain does not result in significant change in microstructure. In natural rocks, mylonites and ultramylonites exhibit a pattern of evolution, with ultramylonites approaching a steady state microstructure characterized by a fully recrystallized, fine grained, well-mixed matrix (e.g., Linckens et al., 2015). In this study, we seek to understand the mechanisms and timescales for mylonite formation in rocks of mafic composition.

#### 2. Methods

#### 2.1. Starting material and sample preparation

The starting material for the deformation experiments in this study is diabase collected near Frederick, Maryland from a complex of dikes and sills (pers. comm. Terry Tullis). It is compositionally and texturally similar to the specimens referred to as 'Maryland diabase' deformed in previous studies by Caristan (1980, 1982), Kronenberg and Shelton (1980), and Mackwell et al. (1998). Our starting material is comprised of ~54 % plagioclase feldspar (Ab30An70 cores, Ab50An50 rims), ~41 % pyroxene in the form of augite (Wo38En44Fs18 cores, Wo36En40Fs24 rims), pigeonite, and inverted pigeonite (enstatite). High-calcium pyroxenes contain lamellae of low-calcium pyroxene and vice versa. Lamellae in augite grains are very narrow (< 0.5  $\mu$ m) whereas lamellae in inverted pigeonite grains have widths of up to 3  $\mu$ m. Less than 5 % of the specimen is tertiary phases including quartz, magnetite, and ilmenite. The starting material has an igneous texture, characterized by randomly oriented, equant grains 10s to 100s  $\mu$ m in diameter.

Cylindrical cores of the starting material were prepared using a diamond coring drill and low speed wafering saw to achieve dimensions of 4 mm diameter and 5.2 mm height. Specimens were sliced in half lengthwise and sputter coated with a thin layer (< 1  $\mu$ m) of gold on the inner surface to create a passive strain marker. Sample half-cores were put together to create a full sample core. The sample assembly (Fig. 1) was dried overnight in a vacuum oven at 110 °C to remove any adsorbed water.

#### 2.2. Experimental methods

Deformation experiments were conducted in the Large Volume



**Fig. 1.** Schematic cross section of deformation assembly. SFP = soft-fired pyrophyllite; HFP = hard-fired pyrophyllite; hBN = hexagonal boron nitride; Cu = copper.

Torsion (LVT) Apparatus at Washington University in St. Louis (Table 1). The LVT is a solid-medium apparatus capable of achieving pressures of up to 3 GPa, temperatures of up to 1500  $^\circ$ C, and shear strain rates of  $10^{-4} - 10^{-8} s^{-1}$ . The torsional deformation geometry produces deformation that is nearly simple shear, with no geometric limit to the magnitude of strain (Paterson and Olgaard, 2000). Samples were deformed between two tungsten carbide Drickamer-style anvils (Fig. 1) with grooved culets, one of which is fixed and the other which rotates at a constant rate. The duration of the experiments was 4 - 12 days yielding strain rates of  $2.1 \times 10^{-6} - 3.4 \times 10^{-5} s^{-1}$ . Experiments were performed at a constant furnace power (920 W) equivalent to a maximum temperature of 1430  $\pm$  50 °C. Relationships between furnace power, temperature, and position are determined through static experiments with precisely positioned thermocouples (Couvy et al., in prep). Due to the cylindrical furnace geometry, there are axial and radial temperature gradients. We compensate for the radial temperature gradient by accounting for slight differences in depth of the regions examined for microstructural analysis. As such, the temperature range across all experiments is 1270 - 1350 °C. The variation in temperature of a single experiment due to axial gradients is small (less than  $\pm$  30 °C). Deformation experiments were performed at constant axial load equivalent to a pressure of 1.9  $\pm$  0.1 GPa at the positions of the regions used for analysis. Pressure is calculated assuming a linear pressure gradient from the edge of the assembly to the center. The maximum pressure at the center (3 GPa) was determined using the Bismuth I-II transformation (Bridgman, 1940). The minimum pressure at the interior edge of the gaskets (0.5 GPa) was calibrated assuming the load from the hydraulic ram is distributed uniformly across the entire 40 mm diameter gasket.

Table 1					
Summary	of	ext	beri	me	ents

Name	IGSN	Duration (Days)	Measured Strain <sup>b</sup>	Max Strain <sup>c</sup>	Strain Rate (s <sup>-1</sup> )
LVT371	IESPM0091	0.1	-	-	$-2.1 \times 10^{-6} \\ 5.5 \times 10^{-6} \\ 1.6 \times 10^{-5} \\ 3.4 \times 10^{-5} \\ 2.4 \times 10^{-5} \\$
LVT375	IESPM0094	11.9	2.0	2.1	
LVT380	IESPM0098	10.2	-	4.8	
LVT381	IESPM0099	10.9	2.9	15.3	
LVT383	IESPM009B	11.9	9.5	35.0	
LVT387	IESPM009G	4.2	6.7	8.6	

 $^a$  All experiments were conducted at a power level of 920 W (equivalent to  $T_{max}$  of 1430  $^\circ\text{C}$ ) and a confining pressure of 1.9 GPa.

<sup>b</sup> Determined from angle of deflected strain marker.

<sup>c</sup> Determined from the thickness of deformation and total degrees of twist.

Once the target strain was reached, or the furnace appeared to be failing (as interpreted by an accelerating increase in measured furnace resistance) the experiment was quenched, the torsional actuator was stopped and electrical power through the furnace was turned off. The sample is quenched below 200  $^{\circ}$ C within a few seconds. The sample was then decompressed over several hours to minimize decompression crack formation.

#### 2.3. Microstructural analysis

After unloading, the sample assembly was encased in a two-part epoxy. Specimens were cut with a diamond band saw at a tangent to the edge of the specimen core. Most specimens had a torus-shaped region of melt near the hottest part of the assembly (the outermost edge of the core, near the vertical center). The specimens were sanded with SiC sandpaper through these partially melted regions to a distance of 1.18 - 1.40 mm from the central axis of the cylinder to analyze regions where melt accounts for much less than 1 % of the material. In these regions, we do not observe evidence of reactions with melt. Samples were mounted in a 1-inch round of Buehler TransOptic Compression Mounting Compound in the Buehler SimpliMet 4000 Mounting System. The specimens were polished using progressively finer diamond grit (6, 3, 1,  $\frac{1}{4}$  micron). Finally, they were polished with colloidal silica on a Buehler Vibromet I polisher for two hours.

The polished samples were coated with a 3.5 nm layer of carbon for scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) analysis. For microprobe analysis, the 3.5 nm carbon coating was removed with colloidal silica and the sample was coated with 30 nm of carbon.

Samples were analyzed using the Thermofisher Quattro S Environmental Scanning Electron Microscope (ESEM) at the Institute of Materials Science and Engineering at Washington University in St. Louis operating at an accelerating voltage of 20 kV and a beam current of 8.2 nA under high vacuum conditions. Backscatter electron (BSE) images were collected at 120x magnification for undeformed starting material and 500x magnification for deformed specimens due to the different scale of microstructural features. All BSE images were taken at a resolution of 2048  $\times$  3072 px, resulting in a minimum resolution of  $\sim$ 0.5  $\mu$ m in 120x images and  $\sim$ 0.1  $\mu$ m in 500x images. Electron dispersive spectroscopy (EDS) was conducted on each sample as well using the Oxford Instruments AZtec Energy Dispersive X-ray Spectrometer. EDS maps were created of the relative elemental abundances of Al, Au, Ca, Fe, Mg, Na, Si, and Ti.

Electron backscatter diffraction (EBSD) data were collected of the starting material and deformed specimens using Oxford instruments Symmetry Electron Backscattered Detector and AZtec software. Maps of the starting material were constructed at 120x magnification with a spot size of 4 µm to determine starting grain size, orientations, and phase distributions. Maps at two different magnifications, 650x and 1500x with spot sizes of 1 µm and 0.5 µm respectively, were made of deformed specimens to capture different grain size populations. EBSD data was processed in the MTEX toolbox for MATLAB (Bachmann et al., 2010). Pixels with high mean angular deviation (MAD) (> 1°) and/or low band contrast (BC) (< 38.25) were removed. Small or non-compact non-indexed regions with  $\frac{area}{number of boundary segments} < 0.8 µm^2$  were filled. Grain boundaries are defined as any surface between two domains of the same mineral phase with a misorientation angle of > 10°.

The composition of phases was determined from Wavelength

Dispersive Spectroscopy (WDS) analysis on the JEOL 8200 Electron Microprobe in the Department of Earth and Planetary Sciences at Washington University in St. Louis (See Supplemental Information Tables S1 and S2). Samples were analyzed at an accelerating voltage of 15 kV and a beam current of 25 nA. Pyroxenes were analyzed using a 1 µm beam size. Plagioclase grains were analyzed using a 3 µm beam size. The composition was determined separately for both the cores and rims of individual phases.

#### 2.4. Strain estimation

For most samples, shear strain was determined by measuring the rotation of the passive gold strain marker (Table 1). Strain can be related to this angle of rotation:

$$\gamma = \tan(\psi) \tag{1}$$

Where  $\gamma$  is shear strain and  $\psi$  is the angle between the strain marker and the shear plane normal. Error in shear strain is determined assuming a measurement error of  $\pm 1^{\circ}$ 

Maximum possible strain is calculated from the total amount of twist, the position of the analyzed section and the thickness of the deformed layer:

$$\gamma_{max} = \frac{\left(\pi d \frac{\varphi}{360}\right)}{z} \tag{2}$$

Where *d* is the diameter,  $\varphi$  is the twist in degrees, and *z* is the thickness of the deformed layer (Paterson and Olgaard, 2000).

The thickness of the zone of deformation was determined using a petrographic microscope by determining where the strain marker is deflected by  $\psi > 15^\circ$ , which corresponds to a shear strain  $\gamma \sim 0.5$  where microstructural change is readily observed. The thickness of decompression cracks is excluded from this measurement. The strain measured from the passive strain marker is sometimes much lower than this maximum value. We interpret this difference to be a result of slipping along the anvil-specimen interface. In subsequent analysis and figures, strain values are those determined by measuring rotation of the passive strain marker, with the exception of LVT380, where the value of maximum possible strain is used instead because the strain maker in that sample was absent or too disaggregated to measure (Table 1).

# 2.5. BSE image thresholding

BSE images were used to calculate the spatial extent and boundaries between phase domains. Before analysis, BSE data were thresholded in MATLAB according to brightness. The relative intensity in BSE images is correlated with atomic weight and therefore can be related to mineral phase. Decompression cracks and holes that result from plucking of grains during the polishing process result in surficial topography that can result in an area of higher intensity of backscatter along the edge of the crack. To prevent this topographic effect from being erroneously classified as a mineral phase, large cracks and holes were masked with black in Adobe Photoshop. Areas of very high or very low intensity were excluded from the final thresholded images since they represent metallic phases (magnetite, ilmenite, or gold strain marker) and decompression cracks, holes, and masked areas respectively.

# 2.6. Phase domain width and phase boundary density

Phase domains are defined to be contiguous areas of a single mineral phase, which can be composed of either single or multiple grains. Thresholded BSE images were used to measure the phase domain width, defined as the width of phase domains measured perpendicular to the shear plane, following the method of Cross and Skemer (2017). For the starting material, transects were drawn ~30 pixels apart oriented both vertically and horizontally, resulting in 170 transects per image. In deformed samples, vertical transects ~30 pixels apart were drawn perpendicular to the shear plane, resulting in 102 transects per image. The width of plagioclase and pyroxene phase domains along these transects was measured. Domains with widths of less than or equal to 2 pixels (1.12  $\mu$ m or less in 120x magnification images and 0.27  $\mu$ m or less in 500x magnification images) are considered low confidence and are excluded in subsequent analysis.

Phase boundary density is a quantitative measure of phase mixing and is defined as the total length of the interface between two phases normalized by area (Cross and Skemer, 2017):

$$\delta = \frac{L_{pb}}{A} \tag{3}$$

Where  $\delta$  is the phase boundary density (in m/m<sup>2</sup>),  $L_{pb}$  is the length of the phase boundary (in m), and A is the total area (in m<sup>2</sup>) of both phases of interest.

Phase boundary length and total area of the phases of interest were measured from thresholded BSE images. Phase boundary density is dependent on the resolution of the images. Although a lower magnification was used for the starting material than for the deformed specimens, phase domain boundaries in the starting material are generally smooth, meaning that imaging at a lower magnification does not obscure boundary roughness in the undeformed material.

# 2.7. Dynamic recrystallization

Mineral phase distributions of the undeformed starting material were determined from EBSD maps taken at 120x magnification with a 4 µm step size. EBSD maps (650x magnification, 1 µm step size) were used to determine the relative abundance of relict and recrystallized grains in deformed samples. Recrystallized grains are defined as grains with an equal area diameter of less than or equal to 6  $\mu$ m. Relict grains are defined as grains with an equal area diameter of greater than 6  $\mu m.$  We estimate a recrystallized grain size of 2  $\mu m$  using the flow law for Maryland Diabase (Mackwell et al., 1998) and the plagioclase piezometer (Post and Tullis, 1999), excluding any effect of strain partitioning between mineral phases. We then set the threshold to be 3x the expected recrystallized grain size, in order to account for uncertainties in our recrystallized grain size estimation and natural variation in recrystallized grain size. Visual inspection of EBSD maps confirmed this to be a good division between recrystallized and relict grains. Percent recrystallization at a given strain was determined by taking the total area of recrystallized domains of each mineral phase over the total area covered by those mineral phases. No stereological correction was applied. Tertiary phases were excluded from these calculations due to their low abundance.

#### 2.8. Crystallographic preferred orientation (CPO)

Orientation data for relict grains ( $d > 6 \ \mu$ m) and recrystallized grains ( $d \le 6 \ \mu$ m) was collected from the 650x and 1500x magnification EBSD maps respectively in order to avoid double counting of grains. The M-index, which ranges from 0 (completely random) to 1 (perfect single crystal texture) (Skemer et al., 2005), was calculated for relict grains using all available data (405 – 1104 grains). Error was determined to be  $\pm$ 0.01, two standard deviations as determined from 100 random samples of 400 grains. M-indices for recrystallized grains and the starting material were calculated from a random sample of 1000 grains since these data sets are larger and have weaker textures, which requires a larger number of grains to resolve. Error was determined for each sample, representing two standard deviations as determined from 100 random samples of 1000 grains.

#### 3. Results

#### 3.1. Microstructural evolution

Deformation microstructures evolve significantly with strain (Fig. 2). The starting material is characterized by relatively large phase domains (10s to 100s of microns across) composed of multiple grains (each 10s of microns in size) (Fig. 2a). With increasing strain, grain size is reduced by dynamic recrystallization and phase domains elongate and flatten into thin sub-horizontal compositional bands (Fig. 2e).

#### 3.2. Modal abundance evolution

With increasing strain, the modal abundance of pigeonite increases while augite and enstatite decrease (Fig. 3). In the starting material, 19 % of pyroxene is the pigeonite phase. At the highest strain ( $\gamma = 9.5$ ), nearly half (48 %) of pyroxene is pigeonite. Over this same strain scale, enstatite decreases from 28 % to 5 % of pyroxene and augite decreases from 53 % to 48 %. This process appears to be enhanced by the dynamic recrystallization process, as there is little change in the relative abundance of each phase in relict grains.

#### 3.3. Grain-size reduction and dynamic recrystallization

We observe grain-size reduction at all strains (Fig. 4a and b). Dynamically recrystallized grains are  $\leq 6 \,\mu m$  in size, however many are smaller than 2 µm, one to two orders of magnitude smaller than the initial grain size. The median grain size of measured grains for plagioclase is 1.5 µm and for pyroxene is 1 µm. Henceforth, we will refer to these values as the median grain size; however, due to resolution limitations these values should be considered an upper limit. We do not observe significant flattening or elongation of relict grains. The overall proportion of recrystallized grains increases with strain (Fig. 3). At the maximum strain achieved in this study ( $\gamma = 9.5$ ), the rock is 78 % recrystallized but relict grains of all mineral phases remain. Plagioclase, the most abundant phase, is 30 % recrystallized at the lowest strain ( $\gamma =$ 2) and is 78 % recrystallized at the highest strain ( $\gamma = 9.5$ ). Pigeonite is the most recrystallized phase at all strains; however, this evolution is enhanced by the reaction that increases its abundance with strain. Pigeonite is 70 % recrystallized at the lowest strain ( $\gamma = 2$ ) and is 91 % recrystallized at the highest strain ( $\gamma = 9.5$ ).



**Fig. 2.** Backscattered electron images (left) and color composite energy dispersive spectroscopy maps (right) (Mg in blue channel, Ca in green channel. low-calcium pyroxene (dark blue) is pigeonite or enstatite and cannot be resolved from one another at this resolution) of representative regions in undeformed and deformed specimens. (a, b) Starting material, characterized by large grains and large phase domains 10s to 100s of  $\mu$ m in size. (c, d) Elongated phase domains composed of recrystallized grains and relict grains, common at intermediate strains ( $\gamma = 2 - 7$ ). (e, f) Compositionally banded microstructure with narrow, sub-horizontal bands dominated by recrystallized grains. Shear sense in c-f is dextral (top to the right). White scale bar is 50  $\mu$ m in all images. Areas of low BSE intensity are decompression cracks or holes caused by plucking during polishing.



**Fig. 3.** Areal percent of relict (light colors) and recrystallized (dark colors) domains relative to bulk strain of the sample as determined from EBSD maps. Total percent recrystallization is denoted by the black line and bold percentages.

#### 3.4. Domain width

In both plagioclase and pyroxene, domain width decreases with increasing strain (Fig. 5a). In the starting material, plagioclase domains are generally larger than pyroxene domains, with median domain widths of 31 µm and 23 µm respectively. In our lowest strain experiment ( $\gamma = 2$ ), domain width decreases by an order of magnitude in both phases. With increasing strain, phase domains continue to narrow and approach the width of a recrystallized grain (Fig. 5a). Plagioclase domains exhibit greater variation in domain widths than pyroxene domains at the same strain. Domains that are only one recrystallized grain across (termed "monolayers" by Cross and Skemer, 2017) become increasingly abundant at high strains (Fig. 2e and f).

#### 3.5. Phase boundary density

Phase boundary density increases monotonically with strain (Fig. 5b). Phase boundary density in the starting material is  $3.3x10^4 \frac{m}{m^2}$ . In the lowest strain experiment ( $\gamma = 2$ ), phase boundary density is  $2.1x10^5 \frac{m}{m^2}$ , six times greater than in the starting material. At the highest strain ( $\gamma = 9.5$ ), median phase boundary density is  $3.8x10^5 \frac{m}{m^2}$ , ten times



**Fig. 4.** Grain size distribution histograms for equivalent diameters of plagioclase (a) and pyroxene grains (b). Gray lines represent the analytical threshold (2 pixels) in 1500x magnification maps (dashed) and 120x magnification maps (dotted). Deformed samples (blue and green) were collected from 1500x magnification maps, which were biased towards recrystallized regions and underrepresent larger grain sizes. No stereological correction was applied.

#### greater than in the starting material.

Cross and Skemer (2017) developed a model for estimating the theoretical maximum phase boundary density of a two-phase material, based on recrystallized grain size and mineral phase proportion. For a 40/60 % phase mixture with a recrystallized grain size of 1.5, the theoretical maximum phase boundary density is  $6.3x10^5 \frac{m}{m^2}$ . Phase boundary density in our experiments approaches but has not yet reached this value (Fig. 5b).

#### 3.6. Crystallographic preferred orientation (CPO)

In plagioclase, the [100] axes cluster in the X direction (parallel to the shear direction) with some girdling parallel to the shear plane. The poles to (010) cluster in the Z direction, perpendicular to the shear plane (Fig. 6a). In pyroxene, at the highest strain, there is some girdling of the (001) and (010) perpendicular to the shear plane. The [001] axes cluster in the X direction (Figs. 6b and 5c).

In plagioclase, the strength of the texture generally increases with strain in both relict and recrystallized grains (Fig. 7a). The starting texture is nearly random (M < 0.01). At all strains, relict grains exhibit a stronger CPO than recrystallized grains (Fig. 7a). The difference in strength between relict and recrystallized grains also increases with strain.

In both clinopyroxene phases, the texture is weak and there is minimal strengthening of the texture with strain (Fig. 7b and c). In augite



**Fig. 5.** (a) Median domain width measured perpendicular to the shear plane of plagioclase (green) and pyroxene (blue) as a function of shear strain. Vertical error bars represent 25th and 75th percentiles of domain widths. Solid horizontal error bars represent error in shear strain angle measurement of  $\pm 1^{\circ}$  Dotted line represents the recrystallized grain size of plagioclase (green) and pyroxene (blue). (b) phase boundary density relative to shear strain. Vertical error bars are 25th and 75th percentiles of phase boundary density. Horizontal error bars are the same as in plot (a). Dotted blue line represents the theoretical maximum phase boundary density for the recrystallized grain size of plagioclase (calculated using the model developed by Cross and Skemer 2017).

relict grains, the texture strengthens slightly up to a strain of  $\gamma = 4.5$  before dropping to M = 0.01 (Fig. 7b). Augite recrystallized grains exhibit a weak texture (M < 0.02) at all strains and are within error of the starting texture strength (Fig. 7b). In pigeonite grains, there was not a statistically significant number of relict grains to measure texture strength. Recrystallized grains exhibit weaker textures than observed in the starting material (Fig. 7c).

#### 4. Discussion

#### 4.1. Previous work on deformation of mafic rocks

Several experimental studies (Kronenberg and Shelton, 1980; Mackwell et al., 1998) have explored the rheology of Maryland diabase, which is similar to the starting material of our experiments (Table 2). Additional experiments to greater strains have also been performed on synthetic plagioclase–clinopyroxene aggregates (Dimanov et al., 2007). The starting material used by Dimanov et al. (2007) has mineral phase proportions that are similar to those of our starting material; however, in their synthetic starting material, the grain size is much smaller (2 – 4  $\mu$ m), and the phases are well-mixed.

In addition, many studies have conducted microstructural analyses of naturally deformed mafic shear zones. Some of these studies analyze amphibole bearing rocks (e.g., Baratoux et al., 2005; Elyaszadeh et al., 2018); however, here we compare our results only to gabbroic mylonites with a similar mineralogical composition (plagioclase and pyroxene) to our starting material. These include microstructural analyses of mylonites from the Southwest Indian Ridge (SWIR) (Mehl and Hirth 2008; Allard et al., 2021), shear zones in the Hidaka metamorphic belt (Raimbourg et al. 2007; Kanagawa et al., 2008), the Kane oceanic core complex on the Mid-Atlantic Ridge (Hansen et al., 2013), and the Ivrea-Verbano Zone in Italy (Kenkmann and Dresen 2001). The rocks in these studies deformed at geologic conditions that are at lower temperatures (500 – 1100 °C) and slower strain rates ( $10^{-11} - 10^{-15} s^{-1}$ ) than the conditions achieved in laboratory experiments.

#### 4.2. Comparison of microstructures

# 4.2.1. Dynamic recrystallization and grain size evolution

Previous experimental work observed little to no reduction in average grain size (Dimanov et al., 2007; Kronenberg and Shelton, 1980; Mackwell et al., 1998). Below a strain of  $\epsilon = 0.1$ , no change in grain size or shape was observed (Mackwell et al., 1998). At a strain of  $\epsilon = 0.18$ , incipient dynamic recrystallization is seen in plagioclase grains (Kronenberg and Shelton, 1980). Despite reaching high shear strains of  $\gamma = 5$ , Dimanov et al. (2007) describe minimal grain-size reduction. However, the grain size of their synthetic starting material is similar to the recrystallized grain size at these experimental conditions, so little grain size evolution is expected. Dimanov et al. (2007) also observe wide-spread cavitation and the formation of micro-cracks, which we do not observe in our experiments. This can be attributed to the relatively low confining pressures (P<sub>c</sub> = 200 – 400 MPa) and the high shear-stress (up to 500 MPa) in some of these experiments.

In naturally deformed gabbroic shear zones, previous authors observe reduction in grain size by several orders of magnitude (from centimeters to microns) (Allard et al., 2021; Kanagawa et al., 2008; Kenkmann and Dresen, 2002; Mehl and Hirth, 2008; Raimbourg et al., 2008). The recrystallized grain size observed is one to two orders of magnitude larger than the recrystallized grain size observed in our experimental samples, which is expected due to the lower stresses within natural shear zones (Post and Tullis, 1999).

In our experiments, at a strain of  $\gamma = 9.5$ , no phase is fully recrystallized. Since grain size continues to evolve over the strains achieved in our experiments and no phase achieves > 90 % recrystallization at the highest strain, grain size has not yet reached steady state.

### 4.2.2. Changes in modal abundance of pyroxene

In our study, we observe an increase in the proportion of pigeonite and a decrease in the proportions of enstatite and augite with strain (Fig. 3). This change is most pronounced in recrystallized grains. Yund and Tullis (1991) showed empirically that deformation processes can promote compositional changes in plagioclase and suggested that deformation could accelerate compositional changes in other minerals as well. Their study demonstrated two processes by which metastable compositions can be homogenized: (1) reduced grain size can reduce the length scale of diffusion and (2) grain boundary migration can cause dissolution of the metastable phase and precipitation of the stable phase. When plagioclase is deforming via dislocation creep, they showed that grain boundary migration can change composition more efficiently than volume diffusion alone. Deformation has also been shown to enhance



**Fig. 6.** Lower hemisphere, equal area pole figures for (a) plagioclase, (b) augite, and (c) pigeonite using a  $10^{\circ}$  half width with one point representing the mean orientation for each grain. n grains in each sample. For samples with n > 1000, dots are a representative sample of 1000 randomly selected grains. M is the fabric strength calculated using the M-index (Skemer et al., 2005).



Fig. 7. M-index of relict grains (diamonds) and recrystallized grains (circles) as a function of bulk strain for (a) plagioclase, (b) augite, and (c) pigeonite. Samples with fewer than 400 grains were excluded. Error bars represent two standard deviations.

compositional changes in natural rocks (Terry and Heidelbach, 2006).

If diffusion of calcium was responsible for the homogenization of pyroxene in our samples, we would expect to see the proportion of pigeonite increase with experiment duration and lamellae in relict grains become narrower and disappear in deformed or annealed samples. While most experiments ran for 10 - 12 days, one experiment ( $\gamma =$ 6.7) lasted for 4 days. In this experiment 39 % of pyroxene was pigeonite in composition. In contrast, after a 12 day long experiment to a lower strain ( $\gamma = 2$ ), only 17 % of pyroxene was pigeonite. In addition, lamellae (less than 2 µm in size) persist in relict pyroxene grains, even after 10 – 12 days of high temperature deformation. Thus, the change in pyroxene phase abundance is more strongly correlated with strain magnitude than experiment duration, indicating that a deformation related process, rather than diffusion alone, is responsible for this change. We propose that the migration of high angle grain boundaries enhances the homogenization of pyroxene into the low calcium clinopyroxene phase. This phase change may affect the strength of the rock if recrystallized pigeonite is weaker or stronger than recrystallized augite or enstatite. Additionally, a reduction in viscosity contrast between recrystallized pyroxene and plagioclase could enhance the rate of phase mixing (Cross et al., 2020). However, at present there is insufficient experimental data on the rheology of pigeonite and augite, so we cannot quantify this effect.

#### 4.2.3. Domain width and phase mixing

Domain width thinning and phase mixing are important elements of the formation of mylonitic textures (Linckens et al., 2015). In studies of

 Table 2

 Previous experimental studies on the rheology of mafic rocks.

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Author	Material	Apparatus	Temperature ( °C)	Pressure (MPa)	Max Strain
(Kronenberg and Shelton 1980)	Maryland Diabase	Griggs type	800 - 1000	500 – 1500	$\epsilon = 0.18$
(Mackwell et al., 1998)	Maryland Diabase	Gas medium	970 – 1070	400 – 500	$\epsilon =$ 0.084
(Dimanov et al., 2007)	Synthetic 50/50 % anorthite/ diopside aggregate	Paterson type	950 – 1180	200 - 400	$\gamma = 5$
This study	Diabase	LVT	1270 – 1350	$\begin{array}{c} 1900 \pm \\ 100 \end{array}$	$\gamma =$ 9.5

naturally deformed mafic mylonites, all assumed to be high strain, both compositional banding and well-mixed regions are observed (Allard et al., 2021; Hansen et al., 2013; Kanagawa et al., 2008; Kenkmann and Dresen, 2002; Mehl and Hirth, 2008; Raimbourg et al., 2008). Kenkmann and Dresen (2001) attribute the transition from compositional banding to phase mixing to increased strain and grain-size reduction. Mehl and Hirth (2008) observe phases progressively mixing with increasing distance from orthopyroxene porphyroclasts which they interpret to be evidence for grain boundary sliding.

In the experimental work by Kronenberg and Shelton (1980) and Mackwell et al. (1998) the authors observe no change in the shape and size of phase domains or phase mixing in their triaxial experiments. However, progressive flattening and elongation of phase domains is observed in higher strain experiments (Dimanov et al., 2007). In the most highly strained experiments by Dimanov et al. (2007),  $\gamma > 4$ , some phase domains are a few grains to a single grain thick and grain scale phase mixing is widespread. Since the initial grain size of Dimanov et al. (2007) is similar to that of a fully recrystallized material and initial phase domains are smaller, grain scale phase mixing may occur more readily than in our samples, where grain size and domain width is still evolving.

In our experiments we observe domain thinning (Fig. 5a), including the formation of monolayers, only at the highest shear strains (Fig. 2e and f). Some grain scale phase mixing occurs, but the microstructure at high strains is dominated by compositional banding (Fig. 2e and f). The measured phase boundary density in our samples has not yet reached the theoretical maximum phase boundary density at a strain of 9.5. We suggest that phase mixing in our experiments is limited primarily by strain and the initial size of phase domains.

To estimate the strain required for domains to reach a "steady state" microstructural configuration of small, well-mixed grains, we use the model developed by Cross and Skemer 2017. This model assumes that the critical strain for the formation of mylonitic textures is related to the progressive thinning of mineral domains until they are only one grain thick ("monolayers"). The critical strain required for the development of monolayers is dependent on the starting domain size, the width of a recrystallized grain perpendicular to the shear plane, and the strength contrast between phases and can be estimated using the following equation (Cross et al., 2020):

$$\frac{W_{initial}}{W_{critical}} \approx \frac{\dot{W}_{initial}}{\dot{W}_{critical}} \frac{\dot{\chi}_{w}^{\prime}}{\dot{\gamma}_{s}}$$
(4)

Where  $\gamma_{critical}$  is the strain required for monolayer development,  $w_{initial}$  is the initial domain width,  $w_{critical}$  is minor axis of a recrystallized grain, which at high strains is the width of a recrystallized grain measured

γ

perpendicular to the shear plane,  $\dot{\gamma}_w$  is the strain rate of the weak phase, and  $\dot{\gamma}_s$  is the strain rate of the strong phase.

When the viscosity contrast between phases approaches zero,  $\frac{\dot{\gamma}_w}{\dot{\chi}_c} = 1$ and  $\gamma_{critical} \cong \frac{w_{initial}}{w_{critical}}$ . As the viscosity contrast increases, phase mixing becomes less efficient and  $\gamma_{critical}$  increases. If we assume the two phases are isoviscous at our experimental conditions, we can estimate a minimum critical strain necessary for widespread monolayer development. In our experiments, the median width of recrystallized grain for plagioclase and pyroxene is 1.5 µm and 1 µm respectively. Starting domain widths for plagioclase range between 10 µm (25th percentile) and 67  $\mu$ m (75th percentile) with a median domain width of 31  $\mu$ m. Thus, we expect initially small plagioclase phase domains develop into monolayers by a strain of  $\gamma = 7$  and larger domains (> 67 µm) at strains in excess of  $\gamma = 45$ . In pyroxene, starting domains are slightly smaller, ranging between 6 µm (25th percentile) and 58 µm (75th percentile) with a median domain width of 23 µm. The width of recrystallized pyroxene grains is also smaller. Small pyroxene domains will develop into monolayers by  $\gamma = 6$ , and large domains will require strains greater than  $\gamma = 58$ . As noted earlier, due to the resolution limitations, our recrystallized grain size measurements are an upper limit. If the recrystallized grain size is smaller, the critical strain needed to create widespread monolayers could be even higher.  $\gamma = 58$  should be considered a lower limit. In our experiments, monolayers are observed in all deformed specimens; however, monolayers become common in our  $\gamma = 6.7$ experiment and are more abundant at  $\gamma = 9.5$ . At the highest strain, thicker domains remain. According to the model, we expect nearly all domains to have become monolayers by  $\gamma = 60$ , assuming there is no strength contrast between the two phases. However, if one phase is stronger than the other, phase mixing will be less efficient, and greater strains will be required for widespread monolayer development. We infer that the strength contrast between plagioclase and pyroxene in our experiments is reduced as grain size reduces. We observe similar domain widths of recrystallized grains in both phases and do not observe boudinage of recrystallized layers. Additionally, the flow laws of anorthite (Rybacki and Dresen, 2004) and diopside (Bystricky and Mackwell, 2001) indicate that strength contrast is minimized with decreasing grain size at our temperature and strain rate conditions. However, at geologic temperature and strain-rate conditions, this contrast is expected to be larger and thus  $\gamma_{critical}$  may be greater than what is predicted here.

# 4.2.4. CPO and deformation mechanisms

Plagioclase CPO strengthens with increasing strain in both relict and recrystallized grains (Fig. 7a). At all strains, this texture is weaker in recrystallized grains than in relict grains (Fig. 7a). This observation suggests that some fraction of strain is accommodated by dislocation creep in both relict and recrystallized domains (e.g., Tullis and Yund, 1987). However, it is also possible that the CPO observed in recrystallized grains may be inherited from relict grains. Randomization of a CPO has been attributed to a transition from the dislocation creep to the diffusion creep regime (e.g., Skemer and Karato, 2008; Warren and Hirth, 2006; Zhang et al., 1994) or deformation in the dislocation accommodated grain boundary sliding (disGBS) regime (e.g., Hirth and Kohlstedt, 1995; Precigout et al., 2007; Warren and Hirth, 2006). The persistence of a CPO in recrystallized grains inherited from relict grains can occur if recrystallized grains are unable to accumulate sufficient strain as a result of being poorly interconnected (Skemer et al., 2011; Skemer and Karato, 2008).

The CPO observed in plagioclase grains in our experiments is similar to that observed in natural samples of Mehl and Hirth 2008 and in Allard et al., 2021, suggesting that similar slip systems are being activated in experiments and in nature. Mehl and Hirth 2008 observed a stronger texture (M = 0.18) in larger plagioclase grains than in smaller grains (M = 0.04). Allard et al., 2021 observed a strengthening (up to M = 0.06 in protomylonitic samples) and then reduction in texture strength (to M = 0.01 in ultramylonitic samples) with strain. These

observations are inferred to result from a complete or partial change in deformation mechanism.

In pyroxene grains in our experiments, the CPO remains generally weak (Fig. 7b and c). The slight strengthening of the texture of relict grains in augite, along with microstructural observations of the formation of dynamically recrystallized grains in all pyroxene phases, suggests that relict pyroxene grains deform at least in part via dislocation creep. Although the rheology of augite and pigeonite is not well-characterized, flow laws developed for diopside (Bystricky and Mackwell, 2001), another high-calcium clinopyroxene similar to augite, demonstrate that high-calcium clinopyroxene is expected to be stronger than plagioclase at the starting grain size at both experimental and natural conditions. If only a small fraction of the total strain is partitioned into augite due to the strength contrast between mineral phases, the development of a CPO would be limited in relict grains. The lack of texture development in recrystallized pyroxene grains could be the result of limited strain; however, the diopside flow laws suggest that grain-size reduction in clinopyroxene results in a transition to the diffusion creep regime at our experimental conditions. The development of flow laws for augite and pigeonite would allow for a more robust interpretation of our results.

We infer that recrystallized plagioclase and pyroxene grains are weaker than relict grains as a result of a transition to the GSS diffusion creep regime (Bystricky and Mackwell, 2001; Rybacki and Dresen, 2000). If phase mixing progresses further at higher strains, grain boundary pinning will suppress grain growth, keeping plagioclase and pyroxene weaker than in the starting material. Additionally, a transition to the diffusion creep regime may facilitate mixing through grain boundary sliding (GBS) (Ashby and Verrall, 1973; Dimanov et al., 2007). GBS could reduce the amount of strain necessary to develop widespread grain-scale phase mixing.

# 4.2.5. Transient microstructure and rheology during shear zone formation Our experiments demonstrate that grain size, domain width, and phase mixing evolve with increasing strain in mafic rocks. This microstructural evolution is inferred to result in a change in deformation mechanism and an associated reduction in strength. None of the microstructures characterized in our experiments have achieved steady state in our highest strain experiment ( $\gamma = 9.5$ ). This means that across this range of strains, mafic rocks exhibit a transient microstructure and

therefore transient rheology.

Linking our experiments to previous experimental work and studies of natural shear zones allows us to describe the microstructural evolution of mafic rocks across large strain scales. At low strains ( $\epsilon < 0.2$ ), the microstructure is in a quasi-steady state until the initiation of dynamic recrystallization (Kronenberg and Shelton, 1980; Mackwell et al., 1998). With increasing strain, the microstructure evolves from a random igneous texture characterized by large phase domains comprised of large grains to a microstructure characterized by narrow sub-horizontal phase domains primarily comprised of small dynamically recrystallized grains at a strain of  $\gamma = 9.5$  (this study). Roughly an order of magnitude more strain would be needed to achieve the well-mixed microstructure observed in both previous experimental work (Dimanov et al., 2007) and in studies of natural shear zones (e.g., Allard et al., 2021; Mehl and Hirth, 2008).

The development of a microstructure characterized by well-mixed, dynamically recrystallized grains deforming via GSS creep is likely an important mechanism for creating and maintaining weakness at plate boundaries (Bercovici and Ricard, 2012). In order to achieve this microstructure, we suggest that shear localization must first be initiated by some other mechanism, such as a stress concentration at the down-dip extent of a fault (Ellis and Stöckhert, 2004; Montési and Zuber, 2002), shear heating (Thielmann and Kaus, 2012), compositional gradients (Burg et al., 2005; Skemer et al., 2013), or syntectonic alteration of mineral phases (Stünitz and Tullis, 2001). As grain size is reduced via dynamic recrystallization, a transition to the GSS creep regime results in weakening that further localizes strain. With sufficient strain, the material can become fully recrystallized and phases will intermix. Microstructural evolution, and the stabilization of deformation in a weakened GSS regime, is the consequence rather than the sole cause of the localization. Strain rates of  $10^{-11} - 10^{-12} \text{ s}^{-1}$  have been estimated for natural mafic shear zones (Mehl and Hirth, 2008). To achieve a fully mixed microstructure ( $\gamma > 60$ ) would therefore take on the order of 100s of thousands to millions of years.

Although our experiments are representative of mafic rocks in general, they are best applied to dry conditions and lithologies. On other planets, such as Venus, whose crust is anticipated to be dry and mafic in composition (Basilevsky et al., 1985; Mackwell et al., 1998; Surkov et al., 1983), the large strains necessary for phase mixing may serve as a barrier to the development of long-lasting plate-boundary shear zones. Likewise, the long interval of transient microstructural evolution (and presumably rheological evolution) should be considered when modeling the rheology of mafic planetary lithospheres in our solar system and beyond. Previous numerical studies have considered the effect of grain size reduction and phase mixing (e.g., Bercovici and Ricard, 2012; Mulyukova and Bercovici, 2019, 2018; Schierjott et al., 2020); however, they often assume that grain scale phase mixing occurs concurrently with grain size reduction. Our results show that both processes can require large amounts of strain before reaching steady state and that grain scale phase mixing might not be complete even in fully recrystallized regions. We suggest that models that incorporate grain-damage (Bercovici and Ricard, 2012) should also explicitly include the strain and time needed to achieve an equilibrated microstructure using, for example, the models of Cross and Skemer, 2019 and Cross et al., 2020 (Eq. (4)). Including this incubation period may also help to explain the lag observed in the geologic record between proto-subduction and the development of plate tectonics (Bercovici and Ricard, 2014). The temperature and stress conditions play a key role in controlling the efficiency of mixing, since the strength contrast between feldspar and pyroxene varies. The microstructure of mafic rocks with initially small phase domains and grains, such as basalts, will evolve more rapidly than those with initially large phase domains and large grains, such as gabbros. As such, the rate of cooling or the persistence of static annealing of a planetary surface may play a role in its ability to localize deformation.

#### 5. Conclusion

High temperature and pressure torsional experiments on diabase demonstrate that the microstructure of mafic rocks, and hence mafic rock rheology, continues to evolve over a wide range of strain magnitudes. At strains approaching  $\gamma = 10$ , we observe the formation of narrow phase domains, including monolayers that are only a single recrystallized grain thick. Grains within monolayers have a greater proportion of their grain boundary that is in contact with another mineral phase (i.e., is a phase boundary). Pinning forces between grains of dissimilar composition will suppress grain boundary migration and growth. Once all mineral domains are thinned to monolayers, phase boundary density will approach its maximum value and mylonitization is assumed to be complete. However, while grain size, domain width, and phase mixing all evolve with strain, none of these microstructures achieved steady state in our highest strain experiment ( $\gamma = 9.5$ ). We infer that deformation microstructures in coarse-grained mafic rocks continue to evolve at strains beyond 10 and suggest that strains greater than 60 may be required to develop widespread grain scale phase mixing and to induce a complete transition from dislocation creep to grain-size sensitive creep. We conclude that mafic rocks experience a long period of transient deformation during localized deformation and that the strainweakening thought to be essential to the formation of ductile shear zones requires large strain magnitudes to attain.

#### CRediT authorship contribution statement

**Katherine Billings:** Writing – review & editing, Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Philip Skemer:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data is available through the WashU Research Data (WURD) repository https://doi.org/10.7936/6RXS-103652.

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#### Supplementary materials

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