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Stressful crystal histories recorded around melt inclusions in volcanic quartz

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Abstract (228 words)

Magma ascent and eruption are driven by a set of internally and externally generated stresses that act upon the magma. By studying the crystal lattice around melt inclusions, whose morphology has a relationship to time that is largely independent of stress state, we may characterize the distribution and magnitude of residual strain produced post inclusion entrapment. We present microstructural maps of quartz crystals from six large rhyolitic eruptions using synchrotron Laue X-ray microdiffraction to quantify elastic residual strain. We visualize plastic strain using average diffraction peak width and lattice misorientation. Locked in by lattice defects and dislocations, quartz crystals preserve similar yet relatively small magnitudes of elastic residual stress (~50-150 MPa) in comparison to the strength of quartz (~10 GPa), whereas the distribution of strain and impact of the inclusions' presence in the lattice visibly varies between samples. We hypothesize that dislocation and twin systems limit the amount of residual stress preserved, as crystals with longstanding deformation histories may have established complex networks that plastically accommodate imparted strain. Given the lack of stress-free haloes around faceted inclusions, most measured strain is imparted after inclusion faceting that occurs dominantly during storage at near-magmatic temperatures. Fragmentation may be one of the final strain events that superimposes stresses of ~100 MPa across all studied crystals. Overall, volcanic quartz crystals preserve complex, overprinted deformation textures indicating crystals may have prolonged deformation histories.

Key words: residual stress, melt inclusions, quartz, fragmentation, plastic strain, Laue diffraction

Introduction

The dynamics of magma during storage, ascent, and eruption are driven by external and internal forces (Cassidy et al., 2018). Crystals present in the magma are witnesses of those forces. Similar to how crystals capture geochemical, temperature, and pressure conditions through their composition and trapped inclusions, they can also record some of the strains produced by forces they experience (e.g., Wheeler et al., 2001; Kendrick et al., 2017; Befus et al., 2019; Barbee et al., 2020). Those strains can arise from stresses imparted by force chains between crystals in regions where magma accumulates, is stored, and then mobilized (Cates et al., 1998; Bergantz et al., 2017). Crystals are further stressed as magma fragments during eruption. With potential signatures of multiple overprinted stress events, connecting recorded stresses in crystals to specific processes and eruption chronology can be ambiguous.

Strains in volcanic crystals can be observed via various techniques. Undulatory extinction observed under cross polars is the simplest method. Strain magnitude has been quantified in some recent studies by electron backscatter diffraction (EBSD). EBSD measurements have been useful for recording the stresses from storage, e.g., in melt-rich mush piles (Vinet et al., 2015; Wieser et al., 2020), to strain localization during extrusion (Wallace et al., 2019; Lavalleé and Kendrick, 2022) and the brittle-ductile transition that accompanies fragmentation (Kendrick et al., 2017). Strains have also been measured and mapped using x-ray microdiffraction (μXRD), by comparing the lattice spacing and parameters of a stressed crystal to that of an ideal, non-stressed standard (Kunz et al., 2009; Tamura, 2014; Vinet et al., 2015).

To elucidate the relative timing of strain events experienced and recorded by crystal cargo that eventually reaches Earth's surface in a large explosive eruption, we characterized quartz crystals from six large-volume high-silica rhyolite eruptions using μXRD. Throughout the magma system, growing crystals commonly entrap small (~10s-100s μm) parcels of surrounding melt inside the crystal, that are then preserved as pressurized capsules of melt. We exploit the relationship of melt inclusion entrapment and spontaneous inclusion faceting (Gualda et al., 2012a; Pamukcu et al., 2015) to show that most strain preserved in the crystals was imparted post-faceting. Faceting only appreciably happens during storage at near-magmatic temperatures, thus constraining the timing of strain. Preserved strains may also represent the maximum elastic stress able to be recorded by these crystals during fragmentation and ascent. Because natural quartz strength is ~10-15 GPa, the residual stress magnitudes may be modulated by established dislocation networks that plastically accommodate strain after a certain threshold is met (Goldsby et al., 2004; Broz et al., 2006; Whitney et al., 2007; Strozewski et al., 2021; Ceccato et al., 2022).

Samples and Methods

Quartz crystals were manually extracted from gently crushed pumice clasts collected from pyroclastic deposits emplaced during large explosive eruptions of high-silica rhyolite (Table 1). Each crystal was examined under a petrographic microscope for melt inclusions that shared similar radii and position within the crystal. We targeted inclusions ~100 μm in diameter that were far from the edges of the host crystal. The host quartz crystals are 1-4 mm in diameter and range from anhedral and partially fractured to euhedral hexagonal bipyramids. Inclusions display varying amounts of faceting and bubbles.

In some crystals adjacent inclusions were chosen to include different inclusion textures within the same crystal, including two with cracks not produced by sample preparation (Figure 1). Crystals were mounted on glass slides with Crystalbond before being ground using progressively finer grits until the targeted inclusion was exposed on the flat polished surface.

All crystals were analyzed using synchrotron X-ray microdiffraction (μXRD) at beamline 12.3.2 of the Advanced Light Source, Lawrence Berkeley National Laboratory, USA (Tamura et al., 2009; Tamura, 2014). Diffraction patterns were collected with a DECTRIS Pilatus 1M detector. A typical experiment analyzed an area of ~400 by 400 μm, using a spot size of ~1 μm^2, a 5-μm step, and 0.5 second exposure time. All samples were scanned using polychromatic X-rays (Laue diffraction).

As incident polychromatic X-rays impinge on the quartz sample, crystal lattice planes in a certain orientation will interact constructively with the incident beam and produce a diffracted ray, given by Bragg's Law (Bragg and Bragg, 1913). Laue μXRD measures numerous microstructural properties, including the residual elastic strains in the crystal lattice (Noyan and Cohen, 1987; Tamura et al., 2003; Robach et al., 2011). Laue diffraction spot positions can be used to measure the difference in unit cell shape between a deformed sample and undeformed standard. Each set of lattice planes that satisfies Bragg's Law produces a spot on the diffraction pattern, whose position and shape are determined by the spacing and features of the lattice. Elastic deformation should produce variations in lattice and unit cell parameters that are able to relax after the imparted stress is removed. But if the deformation causes irreversible defects such as dislocations in the crystal, these defects "lock in" the elastic strains and prevent their relaxation. These residual strains manifest as shifted diffraction spots relative to their unstrained (or reference) positions. Specifically, the change in shape of the unit cell at constant volume (deviatoric strain) produces a relative tilt of the lattice planes and results in a respective displacement of the Laue diffraction spots. The angular difference between these Laue diffraction spots are then used to quantify elastic strain preserved in the crystal lattice (Figure 2). The uncertainty in strain is impacted by the crystal lattice standard (quartz) and the number of diffraction spots automatically indexed using the X-ray Microdiffraction Analysis Software (XMAS) (Tamura, 2014). Previous studies using the same setup and instrumentation estimate an uncertainty of 0.3×10^{-3} strain using an unstrained hydrothermal quartz crystal as a standard. It should also be noted that the phase change from hexagonal β-quartz to trigonal α-quartz at 573 °C does not influence the deviatoric elastic strains measured with μXRD, as the resultant volume change is orthogonal to the crystal axes (Carpenter et al., 1998). The most prominent factors that contribute to strain uncertainty are accuracy of determining Laue reflection positions, CCD detector spatial correction, and experimental geometry calibration (Poshadel et al. 2012).

Derivation of the deviatoric elastic strain tensor from the Laue diffraction pattern comes from the homogeneity property, which relates a deformed versus undeformed unit cell via a deformation matrix (Catti, 1985; Chung and Ice, 1999). The total elastic strain tensor ε_{ij} is then computed with the knowledge of the dilatational component of the strain, estimated by measuring the energy (or wavelength) of a single reflection in addition to the deviatoric strain tensor (Ice et al., 2000). The total strain tensor is used to obtain the total stress tensor σ_{ij}, calculated using Hooke's Law and the elastic stiffness constants of quartz C_{ijkl} (Li et al. 2020). Stress maps presented in this study show equivalent stress σ_{eq}, a scalar quantity representing the magnitude of preserved multiaxial deviatoric stress at each scan spot (pixel)

$$\sigma_{eq}=\sqrt{\frac{(\sigma_{11}-\sigma_{22})^2+(\sigma_{11}-\sigma_{33})^2+(\sigma_{22}-\sigma_{33})^2+6\left(\sigma_{12}{}^2+\sigma_{13}{}^2+\sigma_{23}{}^2\right)}{2}}.$$

A detailed explanation of how strain and stress are measured and calculated using Laue microdiffraction is provided by Kunz et al. (2009), Tamura (2014), Chen et al. (2015), and Wenk et al. (2020). An overview of calculations and equations is also provided in the supplemental text of this study.

Laue diffraction patterns also record plastic deformation. Plastic deformation usually occurs through dislocation growth and migration (Chen et al., 2015). A sample with a significant amount of dislocations will produce a distribution of local lattice plane tilts that upon summation directly broadens the diffraction peaks (Tamura, 2014; Zhou et al., 2016). Here, diffraction peak width is the average of the full width at half maximum of the diffraction peaks that comprise each diffraction pattern (pixel) taken in the Region of Interest (ROI). Observed peak width variations in single crystals arise from contributions predominantly from plastic strain, but can also be influenced by the instrumental profile and more subtly temperature and compositional effects (Warren and Averbach, 1950; Warren, 1959; Zhao and Zhang et al., 2008). Because experimental conditions and instrumental contributions are constant throughout data collection, we assume that the majority of the observed variation in all peak width maps predominantly arises from variations in plastic strain (dislocation and defect density) (Figure 3). The presence of geometrically necessary dislocations (GND) will cause asymmetric broadening of the Laue peaks (Barabash et al., 2002; Barabash and Ice, 2005; Magid et al., 2009; Budiman et al., 2015). The angular length of the reflection (peak broadening) can be used to calculate the density of GND, which can also be measured via calculating misorientation between two adjacent points in the sample ROI. For single crystals, the misorientation angle is directly proportional to (as it is caused by) the GND density (Wheeler et al., 2001; Hughes et al., 2003; Magid et al., 2005, 2009; Brewer et al., 2006). Because there can be multiple Bragg angles and X-ray energies for a given diffraction spot in a Laue pattern (one per map pixel), we deem an informal qualitative approach most prudent and are interested in the relative variation in peak width as it relates to variations in magnitude of preserved plastic strain (Moffat, 2019).

Laue intensity is a measure of the strength of the diffraction signal and can be influenced by factors both inherent to the sample and to X-ray diffraction. Diffraction patterns typically have significant background from X-ray fluorescence, thermal diffuse scattering, or even equipment around the sample (Tamura, 2014). Notably, plastic deformation also leads to peak broadening and in turn lowers intensity values in those areas with higher plastic strain, exhibited by comparing the peak width and intensity maps (Figures 3, 4) (Zhou et al., 2016). In general, Laue intensity maps are a helpful overview of microstructural features present in the ROI, and visually function similar to an SEM image. The thin, very straight, cross-cutting lines seen most prominently in the background intensity maps arise from fluctuations in the overall intensity of the incoming beam during the scan. In addition, reported means and distributions of strain and stress exclude the regions where melt inclusions (glass) intersect the surface, as these do not represent the quartz crystal lattice. White patches in residual stress maps are areas of pixels that could not be indexed. There are small populations of stresses above 400 MPa, but for inter-comparison and ease of seeing stress distributions on the map, the color bar has been set from 0 to 400 MPa for all samples.

Results

Our sample suite preserves a range of inclusion faceting, glass quality, and presence of bubbles and cracks (Figure 1). Inclusions from Bishop Tuff and La Primavera are the most pristine, with clear glass free of visible signs of post-entrapment crystallization (PEC) or devitrification. The La Primavera inclusion contains a ~3 vol.% bubble on the lower inclusion edge. The Bishop and La Primavera inclusions are partially faceted with rounded corners and beveled edges, with a faceting strength of 1.0 according to the Boro et al. (2021) scale. Mesa Falls and Bandelier Tuff inclusions are the most faceted with relatively straight edges and developed corners, with faceting strengths of 2.0 and 1.5, respectively. The distinction between the faceting strengths is because the Mesa Falls inclusions have sharp corners whereas the Bandelier inclusion corners are still rounded. Bandelier also has a small ~5 vol.% bubble. Both Mesa and Bandelier inclusions are slightly fuzzy and brown which indicates the presence of incipient crystallites (devitrification). The Huckleberry Ridge Tuff and Tuff of Bluff Point inclusions appear most affected by PEC and devitrification, and all contain bubbles (with the bottom Bluff Point inclusion containing two bubbles). Given the low vol.% of all bubbles and appreciable post-entrapment effects, and large crystallite touching the bubble of the main Huckleberry inclusion, these bubbles are likely present as a result of a combination of shrinkage, PEC, and devitrification. The Tuff of Bluff Point inclusions have a faceting strength of 1.5, with straight edges and rounded corners, The Bluff Point inclusions are also heavily devitrified, with relatively coarse crystallites distributed throughout the inclusion interior, and minor to no observable PEC. The Huckleberry Ridge inclusions also have appreciable devitrification given their dark brown appearance, but more so a relatively thick rim of PEC where quartz material from the inclusion has crystallized. The Huckleberry inclusions are the least faceted compared to the other samples (faceting strength = 0.5), with rounded sides and undeveloped corners. The observed 2D shape of the inclusions can be influenced by the angle that the sample is polished in relation to the inclusion geometry, but these effects overall are relatively minor and do not affect their placement on the faceting scale by more than one grading.

Maps of residual elastic strain show consistent results in all samples (Figure 5). Strain values range from approximately -1.5×10^{-3} to 1.5×10^{-3} across all samples. Specific ranges for each sample are as follows (all in units of 10^{-3} strain): Bishop -1.86 to 2.14, Bandelier -0.75 to 1.53, La Primavera -1.55 to 1.71, Huckleberry Ridge -0.60 to 1.02, Bluff Point -1.06 to 1.29, and Mesa Falls -0.60 to 0.53. There is a clear impact from the inclusions' presence mostly prominently in the Bishop map, but also in the La Primavera and Bluff Point maps. However, the impact of the inclusions' presence is not obvious in the Bandelier, Huckleberry Ridge, and Mesa Falls maps. Compressive strain is clearly heightened around the Bishop inclusion, most concentrated in the lower boundary, and decays away from the inclusion border. The Bandelier sample has strain magnitudes comparable to La Primavera, but only shows a mottled distribution of strain in the ROI. The La Primavera sample shows impact from both the inclusion and pre-existing crack, with heightened strain on the lower and right inclusion margins. The heightened strain is also of differing sense (both extensional and compressional) throughout this region. The Huckleberry Ridge inclusion shows a broad distribution of heightened extensional strain across the ROI, contributed to by the many inclusions around and deeper in the sample. The Bluff Point sample has relatively high magnitudes of preserved strain, the highest values ($\sim 1.2 \times 10^{-3}$ strain) exist along the lower border of the upper inclusion. There are notable amounts of both compressional and extensional strain preserved around the crack (Figure 5e). Interestingly, the Bluff Point inclusions both show heightened strain around their lower margins but with opposing sense – the top preserving extension, the bottom compression. The Mesa Falls ROI preserves the lowest magnitude strain and displays mostly compressional strain that does not necessarily correlate with the inclusions' locations. Notably, the samples with cracks in their ROI (La Primavera and Bluff Point), do not record the highest strain of all samples, whereas samples with larger or similar strains (Bishop, Bandelier) do not have cracks. Moreover, inclusions are all of similar size (~100-200 μm) but Bishop and La Primavera are the largest, the Primavera inclusion also having the largest aspect ratio (~2). Importantly, most crystals record a disturbance in the magnitude and orientation of their strain fields around the inclusion displayed by the variable in-plane stretching direction of the lattice (e.g., the arrows in Figure 5). The directionality of the strain field does not appear in some residual stress or strain maps themselves, but strain fields are clearly impacted proximal to the inclusions in all samples.

Residual stress maps show similar magnitudes of stress in all samples, with means ranging from ~50-150 MPa (Figure 6, Table 2). All samples possess positive skewness in their residual stress distributions, with Bishop and La Primavera having the longest tails (Figure 6a, 6c). Average residual stress values for each sample are as follows: Bishop Tuff (80 MPa), Bandelier Tuff (67 MPa), La Primavera (144 MPa), Huckleberry Ridge Tuff (90 MPa), Tuff of Bluff Point (58 MPa), Mesa Falls Tuff (56 MPa). These values align with previous residual stress studies of volcanic crystals (Befus et al., 2019). Though similar in magnitude, the spatial distribution of residual stresses varies considerably between each sample. Inclusion faceting strength does not appear to correlate with preserved residual stress or strain, nor the stress associated with the thermal volume change of the inclusions (Figure 7). The presence of bubbles in the inclusion glass does not seem to significantly contribute to the crystal stress distributions, nor the degree of PEC or glass devitrification.

Residual stresses are most commonly the highest near inclusions. Such distributions are pronounced in the Bishop Tuff, La Primavera, and Tuff of Bluff Point crystals. The Bishop Tuff sample shows a relatively homogeneous and low (<100 MPa) distribution of stress >50 μm from the inclusion, but there is a concentration of elevated residual stress (~300-400 MPa) within 50-100 μm of at least one inclusion boundary (Figure 6). Elevated stress near inclusions is also demonstrated texturally as pre-existing cracks radiate from inclusions in some samples from the Tuff of Bluff Point and La Primavera. Quartz from the Tuff of Bluff Point preserves a region of heightened residual stress along the lower margins of both inclusions (~120-300 MPa), both extending up to about 50 μm. The heightened residual stress also extends at least 100 μm further along the trend of the preexisting crack, but its total length extends past the mapped field of view (Figure 6e). Residual stresses also increase along some of the margins of the La Primavera inclusion, again extending ~150-200 μm from the inclusion boundary (right side in Figure 6c). The extent of the residual stresses correlates with the length of a crack in the La Primavera crystal. In other locations surrounding the inclusion the stress distributions are striped and muddled and spread away from the crack propagating through inclusion to the lower right. In the rightmost La Primavera ROI, there are vertically oriented structures in the peak width, intensity, strain, and stress maps that are near a short, thin crack on the right side of the inclusion, but are spatially shifted from this feature (Figures 1c, 3c, 4c, 5c, 6c). There are elevated residual stresses on the right La Primavera inclusion border that correlate with this small crack (Figure 6c). The large patch of higher residual stress (~300-500 MPa) toward the bottom of the La Primavera ROI (Figure 6c) may also be contributed to by deeper inclusions seen in its Laue intensity map (Figure 4c). The distribution of residual stresses surrounding inclusions from the Bandelier Tuff and Mesa Falls Tuff do not show an obvious relationship to the presence of the inclusions. The Bandelier background intensity map shows anomalies in the top right region that correlates with high residual stresses in the same region (Figure 4b). The distribution in the Huckleberry Ridge Tuff is less clear. The Huckleberry Ridge Tuff map has the second highest mean residual stress of all samples and the broadest spread of stress values (Figure 6d), including two inclusions below the surface that are visible in the peak width and Laue intensity maps (Figure 4d). These deeper inclusions may moderately heighten the stress measured at the surface of the sample (e.g., darker areas to the upper right of both surface inclusions in Figure 6d).

All samples preserve plastic strain, as recorded by peak widths (Figure 3). In most samples the quartz host shows a rather uniform distribution of peak widths ranging from 0.01° to 0.19°. The Bishop Tuff map echoes the residual strain and stress maps, with heightened peak widths that tightly wrap the inclusion borders and taper off to background values of approximately 0.05-0.08°. The Bandelier map does not show high peak widths around the inclusion, but instead shows a division of high and low peak widths across the map, uniform in each region and divided with a curved border that cuts across the inclusion and aligns diagonally with two inclusion apices. The La Primavera ROI shows low peak widths near the cross-cutting crack, yet higher values along the lower and right inclusion borders, similar to the residual strain and stress maps. With relatively high peak widths that fan out away from the right and bottom sides of the inclusion, there is also a quasilinear patch of very low (≤ 0.03°) peak widths spanning the rightmost portion of the ROI. This region of lower peak widths correlates with marked deviations in Laue and background intensity compared to the rest of the map (Figure 4). The Huckleberry Ridge map shows heightened peak widths of 0.10-0.14° across the entire ROI except for the locations of the inclusions, even those deeper in the sample. The peak widths in the Bluff Point sample are heightened (at least +0.1° compared to other areas in the ROI) in the same areas of higher signal in the strain and stress maps, except the higher peak widths do not extend across the crack on the left of the lower inclusion (and are limited to the lower inclusion margin) unlike the other maps. The elevated peak widths on the upper inclusion display an arcuate trend that follows the lower border of the inclusion, whereas the bottom inclusion peak widths are straight and extend only to the lower right where the crack also continues. In this way, the La Primavera and lower Bluff Point inclusion demonstrate opposite trends for peak width magnitudes relative to background along each crack: The La Primavera inclusion shows lower preserved plastic strains than "background" over the length of the crack whereas that of the Bluff Point inclusion are higher than background. Similar to the Huckleberry map, the Mesa Falls peak width map shows heightened peak widths of ~0.12-0.17° distributed rather uniformly across the map, with an unstructured patch of lower values on the right side (which includes one of four inclusions). Areas of heightened proximal stresses for two inclusions (Bishop Tuff and Tuff of Bluff Point) appear to minorly spatially correlate with elevated peak widths, but throughout both ROIs, and more broadly all studied samples, there is no significant relationship between residual stress and peak width (Figure 8). Depth averaging of the X-ray signal is most evident in the Huckleberry Ridge map where inclusions ~50-200 μm below the surface modify the peak width signal (Figures 1d, 3d).

Calculating misorientation provides an additional assessment of plastic deformation given its direct relationship to dislocation (GND) density. Misorientation is a measure of the degree of crystal lattice orientation at a single location in the crystal relative to the neighboring lattice. Quartz samples in this study all demonstrate relatively low background misorientations of near zero to 0.1° (Figure 9). The magnitude of misorientation is similar between maps, and misorientation distributions echo distributions of stress and strain in their corresponding peak width, strain, and stress maps (Figures 3, 5, 6). But unlike the microstructural maps discussed thus far, most misorientation maps show patterns that can be clearly related to the inclusions. The quartz lattice bordering the inclusions (most notably Bishop, La Primavera, and Bluff Point) shows markedly high misorientations from background that wrap or radiate away from the inclusion locations. The Huckleberry Ridge and Mesa Falls maps show subtle elevated misorientations around the inclusion boundary, mostly on the lower borders. The Bishop ROI shows a large horizontal stripe of misorientation around 0.015°, compared to the darker blue region above around 0.004° and thin patch in the lower ROI of 0.01° (Figure 9a). The magnitudes of elevated misorientations bordering the inclusion are generally 0.04°-0.14°, but as high as 0.3°. The Bandelier ROI shows higher background misorientation in the top right (0.03°-0.46°), similar to distributions observed in the strain and stress maps (Figures 5, 6). There are three small regions where misorientation is at or slightly above 0.5°, qualifying these zones as low-angle grain boundaries (LAGB) (Menegon et al., 2011; Li et al., 2015). The Bandelier map is the only map that does not show any impact of the inclusion on "background" misorientation (Figure 9b). The "background" misorientation values are highest overall in the La Primavera sample, increasing from 0.01° to 0.03° towards the upper ROI (Figure 9c). The highest values on the lower inclusion border and right side of the ROI range from 0.2° to 4°. The crack in the La Primavera ROI is highlighted by the heightened misorientation along its length, both above and below the inclusion, similar to its peak width, strain, and stress maps (Figures 3c, 5c, 6c). The signatures on the right side of the La Primavera peak width, Laue and background intensity, strain, and stress maps are also reflected in the misorientation map (Figures 3c, 4c, 5c, 6c, 9c). This area (rightmost ROI) demonstrates both low-angle and high-angle grain boundaries, with misorientations between 0.5°-10° and ≥ 10°, respectively (and Dauphiné twins, discussed below) (Figure 9c). Interestingly, the Huckleberry Ridge ROI only displays very low misorientations between 0.001° and 0.027°, but no evidence of intragrain boundaries (Figure 9d). The Tuff of Bluff Point ROI has "background" values of 0.001° to 0.06°, with elevated values of 0.08° to 0.11° near the leftmost ROI around the crack, but also propagates below it (comparing Figure 1e to 9e). Mesa Falls "background" misorientation values are approximately 0° to 0.045°, near the top right of the map. There are slightly elevated misorientations (+0.015°) around Mesa Falls inclusion borders (Figure 9f). Neither Bluff Point nor Mesa Falls display low-angle and high-angle grain boundaries.

All samples except Huckleberry Ridge have twin boundaries (Figure 9). Bishop and La Primavera show dots or clusters of twin boundaries (Figure 9a, 9c) and Bandelier, Bluff Point, and Mesa Falls display meandering, continuous twin boundaries (Figure 9b, 9e, 9f). The vast majority of twin boundaries in all maps are characterized by misorientation values of 60° about the -quartz c-axis ([0001]), establishing these features as Dauphiné twins (Frondel, 1962; Li et al. 2015). The Bishop twin boundaries are scattered throughout the ROI and are not spatially coincident with other observed microstructures, strain, or stresses. The Bandelier twin boundaries do not show a clear correlation with the distribution of peak widths, strains, or stresses, except for a short twin boundary towards the top middle that overlaps with the only area of heightened stresses in the ROI (Figures 6b, 9b). The La Primavera twin boundaries do show clustering in the lower ROI that overlaps with heightened strain and stress (Figures, 5c, 6c, 9c). The Bluff Point twin boundaries show clear overlap with the elevated strains and stresses present in the ROI (particularly along the crack on both sides of the lower inclusion), with more twin boundaries in the upper left ROI where there is no obvious elevated peak width, strain, or stress (Figures 5e, 6e, 9e). The Mesa Falls ROI shows clear Dauphiné twin boundaries that closely align with its peak width distribution (Figures 3f, 9f), and to a lesser extent the strain and stress distributions (Figures 5f, 6f). The Mesa Falls misorientation panel includes a linear misorientation distribution indicated by the orange line to show the 60° misorientations that establish the Dauphiné twin (Figure 9f).

In summary, we observed limited elastic stress magnitudes of 50-150 MPa, modest values compared to the nominal strength of natural quartz at 10-15 GPa (Figure 7) (Ceccato et al., 2022; Strozewski et al., 2021; Whitney et al., 2007; Broz et al., 2006; Goldsby et al., 2004). There is no clear relationship between elastic and plastic strain in all samples except Bishop minorly, with $R^2 \approx 0.6$ (Figure 8). Compared to background levels, heightened strains, stresses, and misorientations are observed around inclusions and cracks. Importantly, stress-free haloes do not exist around faceted inclusions.

Discussion

One challenge in petrology is the issue of scaling micro-scale observations to large-scale magmatic processes. The challenge is sometimes magnified by small sample sizes, an unavoidable issue in our synchrotron study that we acknowledge. Despite the small number of samples, we produced first order observations about the strain histories of six caldera-forming rhyolitic eruptions using a typical quartz crystal from each eruption. Although each crystal is unique in its experience of strain throughout its residence time in the magma system and journey to the surface, it is notable that crystals from all studied eruptions preserve similar magnitudes of strain (elastic and plastic) and average residual stress, which may indicate that the variance between maps in a given eruption may be minimal.

It is difficult to assess the precise physical impact of the faceted melt inclusion shape on crystal strain and stress distributions. Several models have been published calculating the residual strain present in isotropic and anisotropic hosts imparted by an anisotropic crystalline inclusion (e.g., Mazzucchelli et al., 2018; Gonzalez et al., 2021; Zhong et al., 2021). Although melt inclusions are isotropic, the challenge is that the inclusions are faceted from intermediate to strong degrees and thus the correction for glassy semi-faceted inclusions with variable devitrification, bubbles, etc. is not clear or may not be useful with the number and variability of parameters. Nevertheless, to obtain an order of magnitude estimate of the induced strain (and stress) on the surrounding crystal during inclusion-host differential thermal contraction, we calculate the relative volume change of rhyolitic melt inclusions and quartz host from the inclusion trapping temperature (Table 4) to room temperature (25 °C). We compiled compositional data for each melt inclusion from the literature (Table 3). The volumetric data used for quartz were from Kozu and Takane (1929) and accounts for the α-β phase transition at 573 °C. The equations and thermal expansion data for major oxides in rhyolitic melt were obtained from Lange and Carmichael (1990). The calculations were performed in the Excel macro document from Moore et al. (2015).

The average relative volume change from trapping to surface temperatures of the melt inclusion is $V/V_{o\ MI} = 0.9503$, and that of the quartz host $V/V_{o\ qtz} = 0.9598$ (Figure 10). The strain associated with each of these volume changes is thus 0.0497 and 0.0402 strain, respectively, with the differential strain is 9.5×10^{-3} (and the strain is -2.1×10^{-3} to the average glass transition temperature 420 °C). Using Hooke's Law and a Young's modulus of quartz $E \cong 94$ GPa (Ceccato et al., 2022), absolute maxima of inclusion-induced stress for this cooling range is ~893 MPa. Although possible to impart, this is clearly not the average magnitude of residual stress elastically preserved (both proximal and distal to the inclusion-host boundaries in all maps). However, residual elastic stresses of this magnitude are present in some quartz ROIs. Stress histograms all demonstrate some degree of positive skewness but the vast majority of stress values are under 200 MPa for all samples (Figure 6). But several well-indexed diffraction patterns (one pattern per map pixel) do demonstrate values on the order of 700-900 MPa (Bishop Tuff) and 800-1000 MPa (La Primavera). Other maximum stress values, from only a few out of several thousand pixels per sample, are <990 MPa (Bandelier Tuff), <530 MPa (Tuff of Bluff Point), <250 MPa (Huckleberry Ridge Tuff), and <435 MPa (Mesa Falls Tuff).

Elasticity

In general, magnitudes of strain and stress across eruptions are of similar magnitude (on the orders of 1×10^{-3} strain and 100 MPa). This suggests that these magnitudes are caused by forces that are ubiquitous between these studied eruptions, such as similar amounts of thermal contraction, conduit shearing, and ascent rates, constrained by the ability of the quartz host to record these processes. Studied deposits have similar storage temperatures (~700-800 °C) (Table 4). Available magma ascent rates calculated from volatile diffusion modeling are also similar (Bishop Tuff 0.6-13 m s^{-1}, Bandelier Tuff 0.026 MPa s^{-1} (~1 m s^{-1}), Huckleberry Ridge Tuff 0.3-4.0 m s^{-1}) (Myers et al., 2018; Saalfeld et al., 2022). Residence times estimated by the difference between zircon ages and eruption ages share similar magnitudes of 10s to 100s of thousands of years (Table 2 and references therein). It is generally accepted that silicic magmas require these timescales to mature into an eruptible magma in the upper crust (e.g. Seropian et al., 2018). Over these protracted periods, volcanic crystals may dissolve, recrystallize, or otherwise experience unstable temperature conditions during periods of magma recharge and tectonic activity. Those signals could be influenced by force chains between touching crystals in the reservoir, though lower crystallinities in these eruptions may limit this effect (Table 1) (Bergantz et al., 2017; Qin and Suckale, 2020). Multiple fragmentation events are possible in all studied eruptions, causing strains that likely do not fully recover (especially those associated with ultimate eruption). These events may impart strains that vary in magnitude and orientation, and therefore may be compounded or canceled out in the crystal lattice. Elastic strains imparted by multiple fragmentations may not be preserved because of increasing accommodation by dislocations (plastic strain). This may be one reason for the lack of correlation between residual stress and both peak width and misorientation (Figures 9, 11). Moreover, though elastic strain maps seem most impacted by presence of cracks and inclusions, misorientation maps display a network of grain boundaries that do not conform to inclusion locations, highlighting the deformation history of each crystal.

All inclusions are faceted to some degree. Melt inclusion faceting would form residual stress-free quartz at the moment of reprecipitation and should thus initially produce a stress-free halo around the inclusion-host boundary (Figure 6). Areas around all studied inclusions show stresses similar to or above values found away from inclusions in the same crystal. Heightened stress is observed proximal to the inclusion-host boundary for three inclusion sets (Bishop, Primavera, Bluff Point), and the others (Bandelier, Huckleberry Ridge, Mesa Falls) show stresses of similar magnitude and distribution to the rest of their respective crystals. The lack of low-stress or stress-free halos shows that a significant portion of the observed stresses are imparted post-faceting. Larger stresses around certain inclusion sets (Bishop, La Primavera, Bluff Point) may arise from inclusion-host differential thermal contraction upon cooling. The distribution of these thermal stresses is modulated by the faceted inclusion shape, previous crystal strain history, and cooling rate. All previous crystal strain histories are overprinted by appreciable recent strain sometime after faceting (Figure 12). Faceting occurs at near-magmatic temperatures, and rapidly decreases approaching the glass transition. As all but one set (Mesa Falls) of inclusions are partially faceted (Figures 1, 7), this may imply that these crystals did not spend much more time at near-magmatic temperatures prior to eruption (10s to 100s of years, Pamukcu et al., 2015). It is possible that the observed rounded-polyhedron shapes of melt inclusions are indeed their equilibrium shape, given the energy balances involved in surface free energy minimization (Wortis, 1988). Given some inclusions have not achieved straight edges (e.g. Bishop Tuff and La Primavera), we argue that the "background" stress that overprints the initially stress-free zones around inclusions was imparted shortly before or during fragmentation, as the magma rapidly approaches T_{glass}.

Plasticity

Previous work studying residual stresses in volcanic quartz did not comment on preserved plastic strains in volcanic crystals as the modes of plastic deformation in volcanic systems are not well constrained (Befus et al., 2019). Magma mushes in recent years have been regarded and treated as viscoelastic bodies (Webb and Dingwell, 1990; Jellinek and DePaolo, 2003; Karlstrom et al., 2010; Degruyter and Huber, 2014; Liao et al., 2021), and it has been shown that crystals in experimentally deformed lavas can accommodate strain by plastic deformation (e.g., Kendrick et al., 2017). Additionally, crystals that experience significant shear and cracking are also subject to plastic strain via crack tip propagation (Broek, 1982). Natural crystals are subject to incorporating impurities and defects in their growing crystal structure, which itself may manifest complexities via resorption and inclusion of other crystal phases, melt, or fluids, all which may encourage dislocation growth.

During deformation, crystal plasticity evolves by the growth, motion, and interaction of dislocations, the density of which in a given area increases during deformation and develops heterogeneous structures that ultimately determine the response of the crystal to further applied stress (Kubin et al., 1993; Larson et al., 2007). The heterogeneity of dislocation distributions in deformed and deforming crystals gives rise to irregular lattice curvature, causing variations in lattice orientation that are quantified as misorientation. All misorientation maps show the outlines or general shape of the inclusions (Figure 9). This implies that plastic deformation distributed around inclusions is caused by their very presence and underscores our hypothesis that deformation in these crystals is significantly accommodated by plastic strain, and in turn may limit the amount accommodated and recorded as residual elastic strain. Moreover, the varying magnitude and distribution of misorientation throughout and between samples imply that preserved plastic strain bears mostly on sample strain history and lattice features (e.g., cracks and inclusions), rather than being dictated purely by the nature of the quartz host. Overall, these maps appear to represent overprinted textures from repeated or continual stress events in the magma system, further altered by inclusion stresses and both visible and annealed cracks.

The key feature of the misorientation maps is that they not only show varying degrees of plastic strain around the inclusions, but highlight more microstructural features such as zones of relatively high plastic strain (dislocation regions) and subgrain boundaries that are not identifiable in other maps. Background misorientations with values below 0.5° are regarded as a sign of low deformation or straining. Dauphiné twinning is the most striking feature in misorientation maps (Figure 9). Dauphiné twinning in quartz can arise from various mechanisms, including high stress events, plastic strain and accumulation of dislocations (mechanical twinning), and can also occur at the - quartz phase transition (transformational twinning) (e.g., Straumanis, 1949; Van Tendeloo et al., 1976; Heaney and Veblen, 1991a; Piazolo et al., 2005; Wenk et al., 2009; Li et al., 2015). Under constant differential stress and strain, the driving force of Dauphiné twinning is the minimization of stored strain energy (and Gibbs free energy) in pursuit of thermodynamic equilibrium (Tullis and Tullis, 1972; McLellan, 1978). Dauphiné twinning increases crystal deformability by decreasing its stiffness (Tullis, 1970). Specifically, twinning acts to align the direction of greater elastic compliance within the crystal to the compression axis, and thus depends on crystal orientation in reference to the applied stress. This requirement explains why Dauphiné twinning is not distributed homogeneously across sample ROIs or is entirely absent from the chosen ROI (but may be present elsewhere in the crystal). Up to a critical value of stored strain energy, a deformed material behaves elastically and can recover applied strain. Above this value, plastic flow occurs and imparts permanent deformation. In previous studies, it was found that intracrystalline plasticity was the dominant deformation process of the overall quartz microstructure, indicated by pervasive subgrain and twin boundaries (Wenk et al., 2009; Menegon et al., 2011). Furthermore, it was shown that Dauphiné twinning exerts a strong influence on plastic strain partitioning and localization within quartz grains via its relationship to stored elastic strain energy (Menegon et al., 2011). There is clear overlap of elastic strain (and stress) and peak width with Dauphiné twin boundaries only in the La Primavera, Bluff Point, and Mesa Falls quartz ROIs (Figures 3, 5, 6, 9). Bandelier has pervasive Dauphiné twin boundaries, but minimal residual stress and strain (elastic and plastic). This may indicate that the dislocation network or Dauphiné twins were developed early on in the lifetime of the Bandelier quartz sample, minimizing the amount of elastic strain that was recorded.

Experiments by Wenk et al. (2007) established that mechanical Dauphiné twinning depends on both differential stress and temperature, and showed that Dauphiné twins occur most pervasively near 100 MPa and 300-400 °C. The value of 100 MPa (differential) is similar to estimated storage pressures (mean) for all studied deposits (Table 4) and is slightly higher than average residual stress (mean deviatoric) for all samples except La Primavera (Table 2). Though these are different types of stresses, they overlap in range and have similar orders of magnitude. The temperature range of 300-400 °C is similar to estimated glass transition temperatures for deposits in this study (Table 4). Transformational Dauphiné twinning has been shown to occur at the high to low quartz phase transition at 573 °C, which is 100 °C more than the highest calculated glass transition temperature for melts in this study (Table 4). Near the phase boundary, Dauphiné twins are small and twin boundaries are very mobile (Barber and Wenk, 1991). But at lower temperatures, twins begin to coarsen and boundary mobility decreases. These twin boundaries tend to disappear and leave behind a crystal with homogeneous orientation (Wenk et al., 2009).

We hypothesize that Bishop Tuff and La Primavera quartz experienced transformational Dauphiné twinning only, because of their small and somewhat clustered distribution (Figure 9a, 9c). Bandelier Tuff, Tuff of Bluff Point, and Mesa Falls Tuff quartz may have experienced both transformational and mechanical Dauphiné twinning, the latter perhaps occurring around the time of fragmentation. This may explain why the Dauphiné twin boundaries in these samples are larger and continuous, as they did not have enough time or thermal energy to recover and disappear. The low-angle grain boundaries (LAGB) are likely associated with progressive plastic deformation, and high-angle grain boundaries (HAGB) may have once been Dauphiné twin boundaries that have lost their crystallographic relationship or further overprinted by varying directions and magnitudes of strain.

We argue that once crystals establish dislocation networks or otherwise reach their plastic yield limit, they may preferentially deform via that mechanism and in turn limit the amount of elastic deformation that both occurs and is preserved, especially at low strain rates. This may provide a first-order explanation for the lack of spatial correlation between elastic residual stress and plastic strain (Figures 8, 11), as well as relatively low magnitudes of preserved elastic stress compared to the elastic strength of quartz (Figure 7). Low strain rates also promote recoverable elastic deformation, which may relax to some degree and also modulate the magnitude of preserved elastic stress and strain, whereas relatively higher strain rates promote brittle failure. The strain rate threshold between ductile and brittle deformation for silicic melts can be as low as 10^{-2} s^{-1}, depending on the structural relaxation timescale of the melt, and in turn the viscosity and shear modulus (Cordonnier et al., 2012; Jones et al., 2022). Because all studied crystals are visibly intact (some with small cracks fully contained within the crystal), it is clear that they did not catastrophically fail in a brittle manner. The shear strain energy supplied to the ascending magma may be accommodated predominantly by the melt (e.g., via fragmentation), while crystals (especially those long-lived for ~10^3-10^5 years) experience this elastic stress brought on by high strain rates, but (1) accommodate deformation by migration of dislocation slip systems and developing twin boundaries, and (2) consequently preserve only a marginal amount of the total strain they experience from storage to surface (Vernon, 2000).

Crystal residence times

In cases where there is enough data, magma systems (and their constituent crystals) are thought to have been established 10s to 100s of thousands of years before eruption (Table 2). Thus, quartz has been in the magma system for long timescales compared to the timescales typically accepted for mobilization (Rayleigh Taylor instability growth) and assemblage of a shallow eruptible magma chamber in the upper crust, which is thought to be on the order of decades, centuries, or millennia (Seropian et al., 2018; van Zalinge et al., 2022). In this way, the crystal cargo in magma may experience a protracted and varied strain history even before migrating to the shallow crust, accruing strains from cycles of magma injection and thermal rejuvenation, volume changes and compaction, and crystal force chains. These events begin introducing and establishing dislocations that lock in elastic strains and provide avenues for plastic deformation, which will later influence how the crystal accommodates and preserves strain in the shallow chamber and subsequent eruption.

Another relevant concept to consider is that of warm versus cold storage. There continues to be evidence and sound cases for both of these storage modalities, which may ultimately vary both from system to system and over time, as dictated by their tectonic setting, magmatic history, and structure of the crust (Barboni et al., 2016; Rubin et al., 2017; Cooper, 2019). Notably, crystals from all storage systems preserve similar magnitudes of elastic stress, regardless of storage times, temperatures, eruption type, and volume (Table 1). This implies that the preserved residual stress magnitudes are inherent to, or heavily influenced by, the identity of the host phase, quartz. Most previous studies on residual stress in geologic or natural samples have focused on quartz, because of its simple crystal lattice structure and unit cell formula. It would be advantageous to extend studies to other common volcanic phases such as alkali and plagioclase feldspar, to examine if the residual stress signal found in quartz (~100 MPa), is universal or truly inherent to the host phase.

Fracture (cracks)

As magma and crystal cargo rises to the surface (undergoing rapid changes in pressure □P and temperature □T), thermal strain arises from changes in temperature and can initiate crack generation and propagation (Grilli et al., 2021, 2022). Cracks in crystals relieve stress but can also leave behind strain shadows on annealed surfaces that may influence how crystals accommodate and preserve future strain, particularly around melt inclusions. This is evidenced by comparing the stress, strain, and peak width maps of the Bishop Tuff and La Primavera inclusions (Figures 3, 5, 6). Both inclusions are pristine and of similar size and faceting strength, with La Primavera having a small bubble. The outstanding difference between these maps is the presence of a large crack running through the inclusion, which was visibly identified inside the crystal before sample preparation.

Compared to the Bishop Tuff inclusion (aspect ratio ~1.1), the relatively high aspect ratio of the La Primavera inclusion (aspect ratio ~2) may contribute to higher differential stress and thus the tendency of the host crystal to form a proximal crack, as they have similar glassiness, shape, and volumes (La Primavera ~7×10^6 μm^3, Bishop Tuff ~12×10^6 μm^3). Cracks form during brittle failure where there are fewer dislocations to plastically accommodate strain, or when the strain rate is higher than the plastic flow's ability to accommodate deformation. There is elevated elastic strain and stress, as well as plastic strain and presence of Dauphiné twins around the La Primavera and Tuff of Bluff Point quartz cracks (Figures 3, 5, 6, 9). These regions may have reached their elastic and plastic limits, allowing for no other deformation mechanism besides failure. This failure was likely enhanced or even caused by the sudden volume change of the inclusions upon eruption, and influenced by previously accrued deformation in the nearby lattice.

The strain maps record a strain history that is integrated over time and likely includes several events of overprinting as crystals grow and compact in the chamber and migrate towards the surface prior to and during fragmentation and eruption. Although crystals can break during magma fragmentation (e.g., Cordonnier et al., 2012; van Zalinge et al., 2018; Taddeucci et al., 2021), with broken crystals preserved by melt sintering tightly around phenocryst fragments (Best and Christiansen, 1997; Wadsworth et al., 2020; Taddeucci et al., 2021), the quartz crystals studied here did not break during magma fragmentation. Fragmentation is a process hypothesized to occur many times prior to eruption of material that reaches Earth's surface (Gonnermann and Manga, 2003; Tuffen et al., 2003). This may allow for several cycles of strain and repeated possibility of crystal failure and cracking. Annealing may occur between these failure cycles, which could produce microstructural shadows. For example, the Tuff of Bluff Point Laue intensity map (Figure 4a) shows evidence of the bottom crack through the lower inclusion, and also shows a feature similar in morphology and intensity to the right of the upper inclusion, extending to the border of the ROI. Although there is nothing optically visible that would explain this signature (Figure 1), nor anything in other stress and strain maps that match this distribution, it is possible that a fracture once existed here that has since been annealed or grown over, which may explain this intensity feature that closely matches that of the lower crack. Similarly, quasilinear structures in the background intensity map for La Primavera may also indicate previous fracture and annealing events that are not optically visible (Figure 4b).

In summary, each crystal is understood to be a time-integrated, strain-compounded sample that offers a limited window into the true magnitude and extent of experienced stress and strain on their journey to the surface. However, the lack of stress-free haloes around all inclusions implies that significant stress (at or greater than background values around 100 MPa) was imparted after appreciable inclusion faceting (Figure 12), which may be during fragmentation as ~100 MPa is similar to the failure strength of magma (Cordonnier et al., 2012; Wadsworth et al., 2018). We hypothesize that networks of dislocations, twins, and subgrain boundaries that have developed over the course of each crystal's lifetime increasingly accommodate imparted strain and thus modulate the amount of elastic strain and stress recorded in volcanic quartz. As a developing dislocation network becomes more connected, a crystal's tendency to deform elastically decreases, as does its capacity to preserve elastic strain. This capacity may change most dramatically during fragmentation, when crystals that did not shatter upon eruption plastically accommodate some of the imparted strain.

Conclusion

We used natural time markers included in growing crystals from six large rhyolitic eruptions to reconstruct the chronology of strain events experienced by each quartz sample. Volcanic quartz crystals preserve complex, overprinted deformation textures that are sometimes detectably influenced by the presence of melt inclusions. Given the lack of stress-free haloes around faceted inclusions that initially precipitate stress-free quartz, the majority of recorded strain was imparted after inclusions were faceted (Figure 12). As faceting most appreciably occurs at magmatic temperatures (higher silica diffusivity), recorded strain events occurred after crystals spent several thousands of years in storage, or during slow ascent, recorded by the shape of faceted inclusions.

Compared to the elastic strength of quartz (~10 GPa), all stress maps show relatively low average residual stress of similar magnitude (~50-150 MPa), and about half of the maps show a rather uniform distribution of stress throughout the ROI (Figure 6, 12). This is interpreted as the signature of the last major strain event experienced by the crystal in the magma parcel, overprinting any previous strain signatures, which in turn are heavily influenced by established dislocation networks and the crystal's ability to plastically accommodate strain via dislocation slip. These strain signatures, regardless of their origin, are likely to be overprinted several times during a crystal's journey to the surface, and the repeated fragmentation likely en route. We hypothesize that these volcanic quartz crystals preserve only a small portion of the elastic strain and stress that they experience throughout their lifetime. The preservation capacity of most magmatic quartz is ~100 MPa (Figure 7), and subsequent deformation is predominantly accommodated by plastic strain via dislocation and twin growth and migration. We further postulate that this developed network of dislocations (crystal plasticity) may efficiently accommodate or recover the elastic stress induced by melt inclusion entrapment and may prevent crystal fracture during eruption. If true then quartz preserves an incomplete record of the magmatic and volcanic strains the crystals experienced during their journey to Earth's surface.

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