Chapter 6

CONCLUSION: BREAKING THROUGH

Through experiments on both the TGC system and AlON, this thesis has shown that 3D brittle cracks can be tracked and visualized *in situ* and related to phenomena and properties of the anisotropic, inhomogeneous microstructures through which they propagate. Using μ-CT to visualize the crack and second-phase particles, FF-HEDM to obtain information on the mechanical states and orientations of grains encountered by the crack, and NF-HEDM to retrieve grain boundaries and relate them to crack location, comprehensive datasets were created and analyzed.

In the TGC system, the crack in the alkali-aluminosilicate matrix was primarily deflected around the ~15 μm-diameter trevorite particles as a result of residual thermal stresses. However, when the crack would have to deflect significantly (more than 0.45 radians) to continue moving intergranularly, it did not do so. Instead, it cracked through particles transgranularly, or stopped upon reaching the grain and continued after, creating a bridge in the system. In particular, grains with a higher aspect ratio were successful in bridging the crack, and transgranular grains were often larger than the mean, accounting for the greater angle the crack would need to deflect to fracture around them. Orientation of the grain relative to the crack was not a factor in the eventual manner of crack propagation, nor were transgranular or bridging grains observed to have higher or lower stresses in any direction than intergranular ones.

Crack growth in the TGC system was also related to the average stresses experienced by the collective population of grains in the volume ahead of the crack front. When the grains ahead of the crack experienced less tensile stress in the crack opening direction than other volumes at the same load, crack extension occurred in the latter volume first, at lower loads. The volume with depressed stress, meanwhile, reached a higher load before propagating, resulting in an increase in observed fracture toughness.

In AlON, a primary question was whether cracks tended to propagate intergranularly or transgranularly. The cracks actually propagated in both manners, with growth along grain boundaries not linked to any particular crack orientation. As in the TGC system, geometric considerations were most representative of the way the crack grew rather than orientation or mechanical load. When grain boundaries were close to the crack's profile before the crack reached them, it could gradually shift orientation toward those locations. However, the cracks did not display significant deviation in order to intersect a more distant grain boundary, instead cracking closer grains transgranularly.

AlON also displayed differing fracture toughness between regions and samples. Additionally, it extended irregularly, sometimes through a few grains near the existing crack front, and sometimes by hundreds of microns. It also often did not experience any extension even as the load was increased by hundreds of N. Thus, regions of high fracture toughness existed which could arrest the grain's motions, while other regions experienced greater propagation at smaller load increments and had a smaller fracture toughness.

FF-HEDM grain data was also used to create tessellations for AlON, informed by physical properties including grain radius and centroid, and verified against NF-HEDM maps. The resultant tessellations demonstrated reproducibility when comparing the same region from different sub-volumes and different tessellations. Analyzing tessellations in regions without ground truth NF-HEDM, which was limited in the total volume able to be scanned, allowed larger volumes of grains to be considered as an ensemble and further illuminated aspects of crack growth. Evaluations of specific grains had some uncertainty due to the grain boundaries only being estimates, but when considered as a collective, interesting observations emerge. For example, many grains with lattice plane normals close to the <111> direction oriented along the direction of crack growth were present ahead of the crack front in a location where it stopped extending for 200 N. The {111} planes are preferential crack planes for some spinel materials such as AlON, and their presence may have contributed to those volumes having larger fracture toughness. Additionally, in a possibly related observation, the crack eventually grew on more intergranular planes in that region.

These results together indicate that the entirety of the sample plays a role in eventual crack path and propagation. Individual grains in both AlON and the TGC system which cracked in different manners did not demonstrate noticeable, differentiable trends in orientation or mechanical state. However, ensemble data reveals that collections of grains, the vector and tensor field properties of which impact each other, can be related to crack motion and arrest. When considering one goal of these experiments, to use them as verification for phase-field models, this reliance on broader volumetric data makes that approach even more promising due to PF models' use of vector and tensor fields.

Beyond elucidating microstructural effects on crack path, a key benefit of these experiments is that by using the DCDC geometry, unstable crack propagation can be avoided. Not only does this setup allow the crack front to be reconstructed at a stable position, but it also allows that process to be performed multiple times. As a result, information about specific volumes' impact on the crack path can be used to calculate values such as fracture toughness; in unstable configurations, these intermediate properties cannot be considered. Capturing the variance in the stress field and fracture toughness throughout the volume, as demonstrated in this thesis, is critical for modeling the crack path and for gaining new insights into the properties affecting crack deflection and inter- versus transgranularity.

This type of multimodal experimental data provides a wealth of information which will aid in developing and ensuring accuracy in anisotropic 3D model systems, including phase-field models. In turn, the vector and tensor fields utilized by models can be used in a variety of operations and calculations, such as the J-integral, which could reveal further relationships between the crack path and the surrounding microstructure and help guide the focus of future experiments. Eventually, given sufficient information about the starting state of a material, including flaws and other heterogeneous aspects, the goal is to be able to produce a fully or partially predictive model using both computation and experimentation. Such models could help determine the best use for or lifetime of a brittle material in a real application.

Even without predictive models, these experiments represent a significant step forward in understanding brittle fracture in real materials. Obtaining and analyzing combined μ-CT and HEDM datasets for many more materials is possible, and encouraged, in order to more fully understand crack paths in brittle materials representing a range of complexity. Sensible next steps would be to perform similar experiments for materials with more anisotropy, a smaller or larger grain size, or a less equiaxed aspect ratio than either TGC or AlON.

Additionally, as synchrotron technologies are continually being upgraded, including the shift to third-generation sources as at the APS and ever-better detectors, further opportunities will continue to arise. More brilliant sources can improve the quality of the data, and with increased resolution, narrower crack widths may be detected in μ-CT and smaller particles may produce sufficient signal for HEDM analysis, broadening the classes of materials that can be analyzed using these techniques. New sophisticated measurement techniques are also being created, such as point-focused HEDM, which can reveal intragranular and intergranular stresses in a way FF-HEDM cannot. Such stress data would be very useful for examining the local environment around a growing crack front. Additionally, the technique of digital image correlation, already implemented in some workflows at the synchrotron beamlines used in these experiments, could be added to the modalities used in these experiments. This technique tracks deformation and strain on sample surfaces, potentially providing more detail as to how cracks affect brittle materials beyond simply opening them up.

In conclusion, performing multimodal synchrotron techniques leveraging the stable crack propagation of the DCDC geometry has been presented as a novel and viable way to obtain visualizations of a brittle crack front and crack path at multiple stages of growth. Anisotropy and heterogeneity in real-world materials creates more complex crack fronts than idealized models, necessitating a full 3D understanding of their nature, and two such materials are considered here successfully. This work opens up a broad swath of new and exciting directions for both computational and experimental research into brittle fracture with implications for improving reliability and predictability of brittle materials in real-world applications.