Chapter 1

INTRODUCTION: BREAKING APART

Fracture is a word with nearly overwhelmingly negative connotations. Its close relatives—cracking, breaking, rupturing—generally relate to objects, relationships, and the like being sundered irrevocably. Consider the sad sighs and echoed consolations that follow the sentence, “We broke up,” or the wince-inducing sound of a plate shattering on the ground, usually followed by a string of curse words. People do not like things to change, especially when that change is irreversible.

In materials science, the case is no different. The trouble is that fracture in many materials is not particularly predictable nor fully understood. Engineers using materials in applications have different criteria—the most well-known being the Tresca and von Mises criteria—for estimating when an object might fail [1]. However, even these are estimates, since real materials may have unknown flaws and may deviate from ideal models’ stress states, and the users and vendors of these parts and materials are often advised to report an even smaller load to failure than calculated, just in case. When fracture is allowed, it is usually never meant to lead all the way to failure, and a growing crack is carefully monitored as it extends further into a material.

However, in many materials, that growing crack cannot be monitored due to the fact that its speed of advancement is simply too fast to be captured before catastrophic failure occurs. Further complicating the issue is that a subset of these materials undergoes only very limited, visually undetectable, plastic deformation, and some exhibit no plasticity whatsoever. As a consequence, cracks in these materials advance quickly, often progressing to failure in the same step as crack genesis, or pop-in [2]. These materials are termed brittle materials.

The earliest mathematical basis for brittle fracture was formulated more than a century ago by Griffith [3]. If this was all there was to the story, studying these materials would be much simpler than it actually is. However, Griffith and other early researchers into brittle fracture focused on ideal, isotropic, homogeneous structures with equally idealized cracks taking on nice geometric shapes and undergoing evenly distributed loads. Real materials, however, are rarely homogeneous or isotropic, thus leading to uneven internal and external stresses and properties; real cracks are rarely straight or elliptical; and real samples have flaws both intrinsic and artificial, such as those induced by manufacturing [4]. As a result, brittle cracks are highly dependent on microstructure and rarely behave according to mathematical models for their entire lifetime, and much about how they progress is unknown due to the speed at which they break after the initial onset of failure.

As such, in order to use brittle materials in applications requiring them to perform well over a specific lifetime, many verification tests must be undertaken to determine their integrity, which is highly dependent on the size and density of flaws present in the material prior to its use, as well as how reproducible the microstructural conditions are. Testing criteria reliant on detecting the onset of plasticity or other phenomena indicating imminent or eventual fracture are limited in brittle materials due to the lack of plasticity. Instead, specific materials in specific geometries must be tested to failure many times, often hundreds or thousands, in order to create a Weibull distribution and to gain a statistical estimate of when fracture occurs in that material and geometry [5].

Of course, progress has been made in stopping the progression of cracks through materials both brittle and ductile alike. The incorporation of various inclusions of differing aspect ratios and sizes can lead to crack bridging, inducing additional constraints on a material that keep the crack from separating under a normally catastrophic load [6–8], or crack deflection, reorienting the crack into a region which might not be as conducive to further extension [9–12]. Interfaces in composites or grain boundaries can also serve as locations for crack deflection or arrest, and layered brittle materials or engineered grain boundaries are therefore attractive in some situations [13–15]. Some materials also rely on phase transformations ahead of the growing crack, which can blunt the crack's speed and hinder its progress [6,16]. However, some materials have no natural barriers to crack formation and cannot be engineered with additional fracture resistance without hampering the original intended use. Further utilization of these materials requires a better understanding of the conditions that cause failure, especially in situations where creating many thousands of parts for calculating a failure criterion is infeasible.

Past studies of brittle fracture have tended to fall into two categories: postmortem analyses and two-dimensional (2D) in-situ experiments. Types of post-mortem study include fractography, the study of already-fractured surfaces, as a way to investigate deviations along boundaries and crystallographic planes, and sequential focused-ion beam analysis to track the crack's shape change over progressively deeper layers [17–20]. However, this form of analysis requires an already failed sample and gives no insight into the shape of the crack front or location of the crack tip during arbitrary points during its growth phase. Some forms of postmortem measurements do allow for a three-dimensional (3D) crack front to be obtained without destroying the sample, such as by using tomography or acoustic methods combined with indentation, which usually does not cause ultimate failure in a material [21,22]. However, such methods are limited to the observation of a single fracture event for each indentation rather than a progressing crack front at multiple load steps. 2D experiments can more accurately measure the velocity of a crack front as it grows perpendicular to a surface, and can also detect branching and the opening of crack fronts that later closed due to the remission of stress at failure, if present and visible on the surface [23,24]. 2D methods cannot, however, visualize the crack front at other depths within the material and cannot therefore track any other interactions with interior microstructure.

One area of research addressing the unknown factors influencing brittle crack growth is modeling, which allows microstructures, stress and strain fields, and cracks to be simulated and subsequently monitored upon the application or change of environmental factors in time. An optimized model would ideally even be able to predict where and when a crack would appear and propagate in a material, given a few initial conditions. However, several limitations still apply to computational models. The first is that because brittle materials crack elastically, no plastic deformation zone exists to blunt the crack, giving it a sharp tip acting as a singularity in the stress field [25,26]. In order to accurately capture that stress field, nodes in the model mesh at which calculations occur must be made progressively denser in the volume near to the crack tip, drastically increasing computational time and complexity. Furthermore, no matter how small the voxels are that are associated with each node, that singularity can never be fully described by finite voxels, limiting understanding of how the stress field develops and impacts the path of a growing crack. Secondly, models on their own can serve only as elaborate guesswork without combining them and verifying them against experimental observations to be sure that the equations, initial conditions, and simulations are accurately capturing the realities of materials and samples.

A promising method for mitigating the meshing issue is to use a phase-field (PF) model in place of methods such as finite element analysis (FEA). FEA models have been used in many engineering and physics computations, as properties at nodes can be finely tuned to create conditions imitating real-life situations and experiments. However, cracks can be difficult to model, not only because of the singularity at the tip, but because upon separation of the bulk material, and movement of the crack tip the mesh must be recomputed [27,28]. PF models, on the other hand, use continuum mechanics to create smoothly varying fields of properties throughout a volume, changes in which affect the entirety of the studied region. A material's closeness to cracking or its having been already cracked are also properties that can be tracked. As such, PF models are increasingly being explored for use in modeling fracture, especially in brittle materials [29,30].

The work presented in this thesis addresses the need for model verification as well as the unknowns in our current understanding of brittle fracture by capturing the progression of a real crack in a real microstructure in real time. By performing tests at a synchrotron X-ray facility, the interior of a fracturing brittle material can be viewed, and many types of experiment performed with a fast acquisition rate. The high resolution of synchrotron X-rays also allows small features, including narrow cracks and small grains, to be viewed and tracked spatially, which many laboratory systems cannot achieve. Specifically, diffraction experiments can reveal aspects of the microstructure of a material before, after, and during crack propagation, while tomography experiments can capture the extent of the growing crack. By using a geometry capable of stable crack propagation, these experiments can be performed and data collected for brittle materials with a crack within them over multiple iterations of crack growth. This thesis explores the results of performing these types of experiments on several different brittle materials with the purpose of collaborating with PF modelers to enhance knowledge of where cracks propagate and grow in real microstructures.

Chapter 2 of this thesis covers the theoretical basics of brittle fracture and presents the experimental method used at the synchrotron, including the material geometry and the types of X-ray techniques along with the method of load application, before introducing the classes of materials studied in the next chapters. Chapter 3 describes experiments performed on the first material, a glass-ceramic, for the purpose of determining to what extent a second phase present in an isotropic matrix impacts crack growth. Chapter 4 presents similar experiments performed on aluminum oxynitride, an anisotropic polycrystalline material, to explore the effect of larger grains and grain boundaries on crack path. Chapter 5 extends the aluminum oxynitride experiments by using Voronoi-Laguerre tessellations to estimate microstructure in regions where it was not precisely obtained, and uses additional data not examined in Chapter 4 to examine the effects collections of many grains have upon the crack as compared to the effect of individual grains. Chapter 6 concludes the thesis, speculates on the effects of this work on the field of brittle fracture, and presents ideas for the future direction of this work and the field in general, in both designing experiments and connecting them to computational models.

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