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ON THE CHARACTERIZATION OF MECHANICAL PROPERTIES OF ALUMINA CERAMICS FOR HIGH SPEED APPLICATION

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“ Research is what I’m doing when I don’t know what I’m doing.”

Wernher Von Braun

“ After all, the ultimate goal of all research is not objectivity, but truth.”

Helene Deutsch

Abstract

Ceramics have been widely used under impact conditions due to the notable features such as light weight and high compressive strength. The high speed applications require better understanding of mechanical properties of ceramics at various length scales. This work aims to characterize the mechanical properties of alumina ceramic from microscopic up to macroscopic scales.

Dynamic experiments and finite element simulations were developed to investigate the underlying deformation and fracture mechanisms of alumina. The dynamic response of alumina ceramic was determined in a modified split-Hopkinson pressure bar test. The analysis of the wave signals included wave dispersion correction and a direct analytical solution of elastic wave propagation in the specimen to align the measured signals. It was found that equilibrium is established after approximately five wave propagation cycles within specimens, thus supporting the assumption of stress field uniformity. Post-test scanning electron microscopic examination reveals that the failure modes of alumina are strain rate sensitive. A predominant intergranular fracture can be observed at low loading rate, while a combination of intergranular and transgranular fractures can be found in the dynamic test.

To support the observations from the quasi-static test at the grain level, micro-cantilever beams with equilateral triangle cross-section were subjected to a low rate load at the free end. A 3D FE model of polycrystalline alumina was established based on the experimental observations. The simulation results show that the localized stress distribution is affected by the material orientations. The crack

initiates at the bottom of the cantilever beam at the vicinity of the fixed end, and then propagates along the grain boundaries to the top.

Finally, an analytical model was developed to describe the penetration behavior based on quasi-static spherical cavity expansion theory. A damage evolution function was proposed in the crack zone. The analytical solution shows that the penetration resistance is influenced by both material properties and damage evolution parameters. The penetration resistance and penetration velocity can be obtained for the process of penetrating the thick ceramic material by long rod projectiles.

This work demonstrates both theoretical and practical importance on the understanding of the behavior of ceramic materials, especially at different length and time scales. The consideration of deformation and failure mechanisms over a wide range of strain rates ensures that the development of a physically realistic constitutive model is suitable for engineering application.

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Chapter 1

Introduction

1.1 Background

Ceramics are one of the most well-known and oldest classes of materials, which have been widely utilized in many industrial areas, ranging from pottery vessels to aerospace engineering nowadays. Nevertheless, their catastrophic mode of failure caused by their low toughness is the main reason; they are not replacing metals in many applications, especially in armor application. However, the enhancement of bullet resistance of material by involving the hard enamel coatings can be traced back to World War I; while glass was recognized as an cost-effective material as a skin layer on fibreglass armor since World War II [84]. Why ceramic materials were so popular is attributable to the relatively lower density and the higher hardness than metal materials [32, 105, 106]. Fine ceramic armors were originally brought into practice at the time of Vietnam War [62, 85, 138]. Meanwhile, protection of

aircraft against attack from flak and fragment was involved. Whereas, disadvantages were found from such armors due to the ineffectiveness against direct impact crash of submachine rifle on the floor [169]. Later, composite armors were quickly designed and applied since researchers found that if ceramics were attached to the surface of the metal, not only the weight of composite decreased, but the shock resistance of the composite increased dramatically. After the Vietnam War, studies of armor materials on the ballistic impact properties presented were comparatively few for over one decade (until the middle 1980s) [3]. However, research did keep on understanding the mechanical properties of rock-like material at different conditions with wide applications such as shale oil extraction, forecasting of earthquakes, mining and underground nuclear explosion [72, 107]. Therefore, with the increase of the revived interest in the use of ceramics to protect for vehicles and human beings, some knowledge was available regarding to the dynamic and fracture mechanics of brittle materials.

Ceramics are attractive due to their outstanding properties such as high strength-to-weight ratio, high hardness, low compressibility, and superior weathering, wear, abrasion and erosion resistance. Though efforts have been paid, the understanding of a combination of mechanical and physical properties of ceramic materials has not been sufficiently represented yet.

1.2 Motivation

So far the mechanical responses of metal and its composites have been understood well by both experimental and numerical approaches. Meanwhile most of the researchers [24, 45, 76, 78, 100, 111, 136, 155, 175, 187] are transferring their focus on the fabrication of ceramics and ceramic composites nowadays. However, most of them were relevant with the effect of additives on process of fabrication or microstructure. And some come to the conclusions on the statistics characterization of reinforcement phase and geometry of cellular ceramics [128]. Few researchers concentrated on the behavior of ceramics especially at high strain rate; maybe it is still under exploring right now, although some kinds of ceramics and ceramic composites have been commercialized.

Therefore, it is necessary to pay more attention to investigate the behavior of ceramic materials in detail. The topic is not only theoretically important for the performance of ceramics supported by both experimental and numerical methods, but also practically significant for the application of ceramics. However, it is certain that many challenging problems are encountered during the implementation process. For example, it is a technological challenge to accurately measure the high rate behavior of ceramics due to the small failure strain (normally < 0.02) under dynamic state.

1.3 Objectives

It is known that experimental method is the preferred one to characterize of the properties of materials. However, drawbacks are indwelling in the process while deriving the response of materials, especially at high loading rates. For example, stress-equilibrium can be examined from the obtained wave in the split-Hopkinson pressure bar testing experiment by right; unfortunately, it is not easy to achieve this directly due to the unavoidable presence of wave dispersion. In order to comprehensively understand the mechanical behavior of ceramics, the detail objectives will be achieved as follows:

- a) To accurate characterize the properties of ceramics (alumina) subjected to a uniaxial compressive loading. Both the quasi-static and high strain rate testing of ceramics will be carried out since the testing of ceramic under dynamic strain rate is a forever challenge due to the high failure strength and the low failure strain;
- b) To investigate the deformation, fracture and failure of ceramics based on the experimental observation, which can provide a unique evidence to develop a physically realistic constitutive model. One of the challenges in the implementation of experiment is to capture the dynamic crack propagation;
- c) To further explore the crack initiation and propagation in the microscopic level at low loading rates by means of experimental and numerical methods. The challenges at micro scale involve both sample preparation and exact measurement in experiments and representative structure generation in simulation. Due to the

heterogeneous properties of single grain, statistical analysis is required to achieve the intrinsic mechanisms of polycrystalline materials;

d) To study the anti-ballistic performance of ceramics in high speed application, the penetration resistance of ceramics is a key factor. During a penetration process, the degradation of Young's modulus is inevitable. Based on available quasi-static spherical cavity expansion theory (CET), the penetration resistance (R_t) has been resolved couple with a proposed damage evolution function.

1.4 Scope of the work

Pertinent review of the literature on the investigation of ceramic material revealed that the current state of the knowledge can be improved.

An analytical solution, which can be applied to accurately characterize the ceramic-like material subjected to split-Hopkinson pressure bar test, is given based on one-dimensional wave propagation equation coupled with proper boundary conditions. This solution can help to verify the assumption of stress equilibrium in a conventional split-Hopkinson pressure bar test.

A series of tests are performed to achieve the reliable experimental results by using the given analytical solution. Alumina 99.5% ceramic specimens (5 *mm* in length and 5 *mm* in diameter) are used. A high-speed camera is used for taking the images of deformation process. Meanwhile, the 3D full scale finite element model was developed. Simulation results are used to examine the stress status and failure process of the specimen during deformation.

Voronoi model for simulation of crack initiation and propagation in the grain level of alumina ceramics is presented. Experiments on the micro-cantilever beam fabricated by focus-ion beam (FIB) technique are conducted. Based on the model, simulations are performed for different combinations of material orientations and grain numbers with different grain sizes. The detailed damage evolution can be observed from simulation.

An available model in literature [157], namely quasi-static spherical cavity expansion model, for projectile impact onto monolithic ceramic target is recurred, accounting for damage evolution. Finally, the penetration resistance and velocity of the projectile can be found.

1.5 Thesis organization

There are seven chapters composed in this thesis, outlined as follows:

Chapter 1 introduces the motivation of investigating the mechanical performances of ceramics, followed by the objective and outline of the present research work.

Chapter 2 provides a literature review on experimental methodology to accurately characterize brittle material at high strain rates, deformation and fracture features of ceramic material at different loading rates, experimental and numerical methods to investigate crack initiation and propagation at microscopic level, and the constitutive behavior of ceramics.

Chapter 3 presents a method to accurately characterize dynamic stress-strain curves of ceramics in a split Hopkinson pressure bar test. An analytical solution based on one-dimensional stress wave propagation equations with proper boundary conditions was derived to align the waves at bar-specimen ends. FE model was established to make compare with experimental measurements, during which the material constants for JH-2 model were determined. Simulation results prove that the proposed method is reliable to quantify the dynamic behavior of ceramics subject to a uniaxial compression test.

Chapter 4 illustrates the failure process of ceramic materials suffering dynamic loading based on the experimental observations and FE model provided in Chapter 3. The material demonstrates the rate-dependent properties within the tested strain rates ($10^{-5} - \sim 2000 \text{ s}^{-1}$). On-line observation shows that longitudinal crack generated on either end of the specimen before peak strength achieved. Post-examination also reveals that the failure modes of ceramic material are rate dependent.

Chapter 5 introduces experimental and numerical method to study the crack initiation and propagation in the microscopic level. Micro-cantilever beams were fabricated via FIB cutting technology. A lateral load was applied on the free end in a nano-indenter. The intergranular fracture can be observed, which is similar with the experimental results obtained from post-examination of the ceramic fractures at quasi-static test in Chapter 4. Meanwhile, a method to generate the statistical models based on hardcore Voronoi tessellations was involved to detail

the experimental results. Both experimental and numerical results show a good consistence.

Chapter 6 develops a constitutive model for ceramic material based on the quasi-static spherical cavity expansion theory, during which a damage evolution function is proposed. The penetration velocity derived from the model shows a good agreement with the experimental data provided in literature [157].

Chapter 7 summarizes the current research work, and recommends the possible future work.

Chapter 2

Literature Review

2.1 Ceramics and their applications

Ceramics as a main component of layer armor composites demonstrates excellent properties. Usually, the objective of developing ceramics or ceramic composites is to achieve lightweight, structural materials combining high strength with improved toughness, damage tolerance and thermal shock resistance [15, 110, 152] due to wide application areas such as supersonic planes, high-speed civil transport planes, turbine blades, heat shields, rocket nozzles and propulsion components, re-entry thermal protection for spacecraft, rocket cone frusta, braking materials, cutting tool inserts, high-wear parts, valve seats, high-precision balls, bearing for corrosive environments and plungers for chemical pumps [67, 131]. Ceramic and its composites have been of interest to armor application since World War II due to their outstanding properties compared with other materials such as metals

(see Figure 2.1). For example, the strength of ceramics demonstrates rate dependency for both intact and fractured materials [16, 21, 64]. Whereas, the history has often shown a lag between development in the laboratory and application. Therefore, it is important to accurately characterize and well understand the mechanical behavior of materials before utility, especially for high speed application [25, 56, 61, 143].

FIGURE 2.1: A typical composite armor consisting of ceramic front face and metallic backing plate

2.2 Improvements of characterization methodology of ceramics at high strain rate

As ceramic materials are increasingly used in high speed applications due to their light weight and high compressive strength, the dynamic behavior of these materials needs to be accurately characterized in order to predict their service performance. The high rate properties of ceramics can be typically measured using

the split-Hopkinson pressure (SHPB) technique [6, 17, 42, 73, 140, 158, 170]. The SHPB technique, which was initially developed to measure dynamic behavior of metals [94, 139], has been modified to test other materials such as polymer and foams [47, 95, 96, 190]. However, to accurately characterize stress-strain curves of ceramics at high strain rates on the order of 10^3 s^{-1} using the SHPB is still a challenge because of the high failure strength and the low failure strain (typically less than 2%). This requires not only the modification of the SHPB experimental technique and the specimen design but also the subsequent reliable processing and analysis of the signals recorded in the bars.

2.2.1 Experimental improvements

In most cases, ceramics cannot yield locally since the ceramic deforms in a nearly linear elastic manner and fails at small strain value. Stress concentration can cause premature fracture of specimen in the early stage. The first one is that the specimen surfaces are not flat and parallel in the fabrication process. The second one is the misalignment of the bar system. And the last one is the indentation caused by the stiffer testing materials.

In order to minimize the stress concentration at the specimen edge, two kinds of specimen configurations were proposed previously [91, 158] as illustrated in Figure 2.2. One (Figure 2.2(a)) is dumbbell shaped and the other (Figure 2.2(b)) is a cylinder specimen sandwiched between inserts made of hard materials such as tungsten carbide [140]. The former one can overcome the stress concentration

caused by indentation effectively and the latter one is the most commonly used as it is easier and more cost effective to fabricate a cylindrical specimen.

FIGURE 2.2: Specimen configurations of brittle materials: (a) dumbbell shaped specimen, and (b) specimen sandwiched between inserts [91, 158]

For the sake of eliminating the stress concentration effectively, Sunny et al [158] designed a novel inserts made of maraging steel as shown in Figure 2.3 and validated the reliability of dynamic mechanical characterization of low strain-to-failure materials.

FIGURE 2.3: A novel design of inserts: (a) schematic of inserts, and (b) actual design [158]

In the split-Hopkinson pressure bar system, it is critically essential in the testing of brittle materials to align the striker, incident and transmitted bars owing to the sensitivity of the specimen to stress concentrations. Otherwise the measured strain will be inaccurate. Unlike static testing machine, there are no systems for self-alignment in a split-Hopkinson pressure bar instruments. A slight misalignment along the loading axis would be expected in practice. Chen et al [17] got a revelation from the quasi-static testing machine, and developed a simplified universal joint positioned between the specimen and output bar to overcome the slight misalignment as shown in Figure 2.4.

FIGURE 2.4: Universal joint in a specimen assembly [17]

However, the constant strain rate deformation is also required in the split-Hopkinson pressure bar test for those strain rate sensitive brittle materials. It means that the dynamic stress state of testing specimen should be in equilibrium and the strain rate is constant before specimen fails. It is impossible for the impact system to adjust the loading pulse in real-time from the specimen feedback in most situations. Christensen et al [21] modified the input pulse shape by varying the shape of the striker to improve the accuracy and resolution of the mechanical response of rock

materials in 1972. They employed pulse shaping skill in the compression Kolsky bar test, and made a conclusion that a ramp input pulse was more suitable to obtain the response of rock in contrast with a square pulse [21]. And this technique to modify the profile of input shape is still being used for measuring the mechanical behavior of brittle materials, though problems still do exist in designing and fabricating the specialized striker.

FIGURE 2.5: SHPB configurations with preloading bar [36]

A three-bar technique [36] was also developed as illustrated in Figure 2.5. A preloading bar, which has the same length and material with incident and transmitted bar, and a dummy specimen preferably of the same material as that of the testing specimen, were implemented for pulse shaping to the traditional split-Hopkinson pressure bar. However, the preloading bar was substituted for another bar lower in strength to reduce the effects of wave dispersion by Parry et al in 1995 [127]. And they found that the longer the preloading bar was, the more effective it was to minimize the stress wave dispersion [127]. Later, a modified split-Hopkinson pressure bar system was widely used by removing the preloading bar and attaching a dummy specimen on the incident bar directly [43], which was

considered as a simplified three-bar system. Copper is the widespread used material for dummy specimen. Frew et al [44] found that a single pulse shaper was not sufficient to produce an incident pulse with high amplitude because of the low yield strength of copper. A dual pulse shaping technique was developed as shown in Figure 2.6 for characterizing the elastic-plastic response of materials.

FIGURE 2.6: Schematic diagram of dual pulse shaping technique [43]

With the efforts of researchers reviewed above, split-Hopkinson pressure bar or Kolsky bar technique is perhaps the best experimental technique at this moment to characterize the dynamic properties of ceramic materials.

2.2.2 Data process methodology

Generally, the specimen stress-strain curve can be calculated using the force and velocity at the two bar-specimen interfaces on the assumption of one-dimensional wave propagation theory and uniaxial homogeneous deformation of specimens [31, 66, 81]. However, strain gauges are usually mounted away from the bar-specimen interface in the SHPB system, e.g., in the middle of the bars, in order to avoid the

superposition of wave signals near the interface. Recorded data thus needs to be analyzed to correct the inevitable dispersion of waves and align wave signals to the bar-specimen interfaces, which can significantly affect the accuracy of calculated stress-strain curves especially in ceramics that fail at a small strain [18, 98, 180, 191].

Based on Pochhammer-Chree's longitudinal wave analysis [20, 129], a significant amount of work has been conducted to diminish wave dispersion effects in SHPB testing [40, 53, 54, 98, 164, 182, 190]. Periodic wave pulses can be decomposed in the time domain using Fourier series to correct the dispersion [31, 40, 69]. In recent studies [53, 54, 98, 129, 182, 190, 192], a fast Fourier transform (FFT) algorithm in the frequency domain was suggested to remove dispersion effects in order to reconstruct the original stress wave history, and was found to be more effective and reliable compared to the solution in the time domain. Wave dispersion correction can significantly influence the final calculated dynamic stress-strain curve of ceramics, although it may seem to have little effect on wave signal oscillation in the range of small strains [98].

Alignment of wave signals to bar-specimen interfaces in the SHPB system has been widely investigated in the literature [98, 101, 190–192]. A small misalignment in the start of each wave can considerably affect the reconstructed shape of dynamic stress-strain curves [98, 190–192]. The time shifting between signal recording locations and bar-specimen interfaces can be calculated based on their distance and the longitudinal wave velocity in the bar. However, it is often difficult to precisely quantify this distance due to the imperfection of bar-specimen interfaces,

e.g., caused by the lubrication, thus leading to an error in alignment. Moreover, wave propagation in specimens is often overlooked. Instead, Zhao and Gary [191] predicted the exact origins of waves based on the shape of the wave, i.e., by simulating reflected and transmitted waves at the early stage of SHPB testing. However, very few studies [134, 148, 178] examined stress equilibrium in specimens during the SHPB experiment, in particular of ceramics, which motivates us to investigate since the dynamic behavior of ceramic material measured from SHPB test is under the assumption that stress/force in equilibrium in specimens.

2.3 Failure and fracture in ceramics

Layered composites composed of materials such as ceramics, metals and adhesives has received tremendous interests due to the light weight, good shock resistance and excellent impact energy capacity [7, 64, 140]. The ballistic performance of composite armor is strongly dependent upon the dynamic behavior of its layered materials, in particular the ceramics in which the strength increases with the confining pressure [16, 21, 64]. However, unlike metallic layered materials such as steel and aluminium alloys [75, 92], the properties of ceramics subjected to dynamic loads are not well characterized and understood due to the high failure strength and low failure strain [7, 140]. Moreover, ceramics tend to be sensitive to the strain rate especially when it is above a critical value [73, 140]. To enable the use of ceramics in composite armor structures, it is necessary to investigate the dynamic deformation, failure and fracture mechanisms of the ceramics.

A portion of research has been conducted to study the failure mechanism of ceramics in the two length scales: (1) the nucleation, growth and interaction of micro-cracks, and (2) the macroscopic failure behavior including macro-cracking and fragmentation [48, 108, 132, 135, 156]. Micro-cracks nucleate at heterogeneities in ceramics such as grain boundaries and inclusions [145]. Microstructural features have a significant effect on the micro-crack propagation path; and micro-cracking plays a crucial role in determining the overall failure behavior in ceramics [1, 33, 116, 120, 121]. The fracture of grains (i.e., transgranular fracture) and the failure of grain boundaries (i.e., intergranular fracture) can be possible fracture modes in ceramics dependent on the chemical compositions, microstructure and the external loading rates. Experimental observations in various length scales have exhibited that the failure and fracture process in ceramics is significantly influenced by the loading rate. Although the simplex failure mechanism in ceramics has been observed under quasi-static rates [115], the appearance of dislocation at the tip of micro-cracks causes different complex failure processes in the ceramics under dynamic loading. It is important to directly explore the underlying mechanism for dynamic failure and fracture process in ceramics.

A number of existing experimental studies have focused on the macro-cracking process and fragmentation during the failure of various ceramics subjected to dynamic loading [172, 189]. It can be argued that the complex dynamic failure and fracture mechanisms may vary from one ceramic material to another, which are intrinsically determined by the chemical compositions and microstructural features arising in the manufacture (e.g., sintering or hot-pressing). For a specific ceramic,

characterization of the dynamic behavior is thus required for its application in composite armor.

FIGURE 2.7: Stress-time history of SiC-N with high speed camera images of the failure process under uniaxial dynamic compression. Note (i) Inset shows the dynamic loading configuration; (ii) On the right, red arrows show the WC platens and blue arrows show damage regions and crack propagation [70]

Though great efforts have been paid thus far, the dynamic deformation and failure mechanisms haven't come to an agreement. Wang and Ramesh [171] post-exam the fragmentation sizes of SiC-N under optical microscopy and scanning electron microscopy (SEM), of which lognormal distribution is followed. However, the online surveillance of dynamic deformation procedure (see Figure 2.7) shows that the longitudinal crack appears in arrears of the achievement of failure strength [70], which is different from the phenomenon (see Figure 2.8) observed by Chen et al [17]. In their experiments [17], the appearances of longitudinal cracks are prior to the failure strength for both perfect cylindrical SiC-N sample (see Figure 2.8(a)) and pre-flaw one (see Figure 2.8(b)), resulting in axial splitting as the

failure mode. Therefore, the investigation of failure process and failure modes should be necessary for the underlying nature of ceramic materials.

(a)

(b)

FIGURE 2.8: Axial stress-time history of SiC-N with dynamic fracture and failure process under uniaxial dynamic compression at different stages. The different stages are indicated in the stress versus time curves: (a) perfect specimen, and (b) pre-flaw specimen [17]

Besides experimental characterization methodologies, numerical simulation such as finite element modelling has extensively used to predict the stress distribution in ceramics subjected to dynamic loads and investigate the damage evolution that it is usually difficult to observe experimentally due to the instantaneous nature at high stress rates. For accurate numerical simulation, various constitutive models have been developed to quantify the mechanical response of ceramics under different loading rates. Taylor et al. [161] assumed that the damage in brittle rocks only accumulates in a tensile state and then proposed an ideal plastic constitutive equation to simulate the dynamic fracture behavior of the rocks. A mixed-mode cohesive law to describe the fracture process was implemented in a FE model to predict the dynamic fracture and fragmentation of various ceramics [185]. The Johnson-Holmquist material model with damage (JH-2) [74] incorporates the polynomial equation of pressure state as a function of volumetric strains, the specific strength for both the intact and fractured materials, and the damage evolution rule to determine the occurrence of fracture in ceramics. The JH-2 model has been widely accepted to simulate the mechanical behavior of damaged brittle materials subjected to high pressures, large strain and high strain rates.

As known, ceramic materials contains amount of ionic or covalent bonds, which lead to some special behavior and consequently the reliability in engineering application. The investigation of failure of ceramics is crucial. Researchers [30, 162] have found that fracture of ceramic components depends on many factors, such as the chemistry and microstructure of the material [100], which result in many modes of failure in-service, summarized as two groups of failure modes, namely, thermal

shock failure and contact loading failure. The failure mechanism is similar in the thermal shock failure, e.g., the gradual expansion of the recrystallized structure makes the intergranular fracture [90]. For the contact loading failure, the failure mechanisms vary from both materials and loading conditions [140, 145, 156, 162]. The possible fracture modes in ceramics are intergranular fracture, transgranular fracture or a combination of them dependent on the microstructure, chemical additives and rate of deformation [1, 33, 48]. Research found that: 1) microstructure had little influence on the overall mechanical response, but significant effect on the crack propagation path [144], 2) the introduce of chemical additive (CaO) in fabrication of AlN affected the failure mode changed from predominantly transgranular fracture in the undoped AlN to predominantly intergranular one [177], 3) the failure and fracture process of ceramics demonstrated strain rate sensitivity changed from simplex mechanism at quasi-static rates to complicated one at high strain rates [115, 153]. To understand the underlying mechanism of ceramics, it is necessary to study the failure and fracture process in grain level.

2.4 Characterization of ceramic materials in microscopic level

To better understand the material performances at the macroscopic level, it is important to investigate the fracture mechanisms of the material at the grain level. The differences, such as the heterogeneous properties of the single grain, in microstructure of materials result in different failure mechanisms. Usually, the

overall performances of materials are manipulated by the properties and orientation distributions of grains; and the local properties of materials depend on the morphology in the microscopic level.

2.4.1 Experimental skills

To characterize the $3D$ micro morphology, the reconstruction of microstructure is crucial. Experimentally, the skills for reconstruction of microstructure, including optical microscopy, transmission electron microscopy (TEM) or scanning electron microscopy (SEM) [59, 149, 165], can be labeled as destructive and nondestructive techniques, which must be capable of offering information on crystallographic features such as grain orientation distributions and micro-morphology [154]. Particularly, the nondestructive methodology of reconstruction refers to the investigation of crack evolution and micro-damage; while the destructive reconstructions are used to extract the statistic features of microstructure for large polycrystalline aggregates, and evaluate the artificial morphology of microstructure [11]. Both the output of destructive and nondestructive data can be further converted into FE models for the sake of numerical simulation [102].

Among destructive techniques, serial sectioning plays an important role to measure the real grain structure. The collection of morphology information is based on a sequence of stacked $2D$ sections of material followed by post-processing method to rebuild the real microstructure [150, 188]. In particular, the material needs to be micro polished (e.g., mechanical, chemical, laser-based, etc) carefully for each layer to keep the feature of microstructure since factors such as the accuracy

and precision of polishing techniques, sampling resolutions and reconfiguration methodologies have significant influence on the quality of rebuilt microstructure [151]. However, the combination of serial sectioning with other methods, such as electron back scattering diffraction (EBSD) [93] and dual-beam focused ion beam-scanning electron microscope (DB FIB-SEM) [60, 165], reveals application potential in materials science [117].

The widely used reconstructed nondestructive techniques for microstructures are X-ray techniques consisting of X-ray tomography, X-ray diffraction microscopy, and differential aperture X-ray microscopy. By introducing the X-ray tomography techniques, the chemical distribution of material can be determined. The micro morphology also can be demonstrated visually accompanied with supplementary crystallographic information to investigate the effect of microstructure on the mechanical performances. Usually, the reconstruction technique based on the diffraction of synchrotron beams can be divided into two classes: one is three-dimensional X-ray diffraction microscopy (3DXRD) [119]; the other is differential aperture X-ray microscopy (DAXM) [71, 89].

Ludwig et al [102] presented a nondestructive bulk characterization method, which provided a unique way to visualize the 3D grain structure of a polycrystalline material and was successfully applied to investigate a short fatigue crack propagation along grain boundary of a cast Al alloy [103], combined synchrotron radiation X-ray micro tomography with electron back-scattering diffraction (EBSD) to observe the diffusion of liquid gallium (contrast agent) along grain boundaries.

Larson et al [89] invented a differential-aperture X-ray microscopy (DAXM) technique with implementation of polychromatic synchrotron X-ray micro-beams. The method can be used to measure the local crystal orientation, structure and stress tensors with a spatial resolution of submicrometer to investigate the materials such as monocrystalline, polycrystalline, composite and functionally graded materials at the critical mesoscopic length scales (tenths to hundreds of microns). As a nondestructive reconstruction technique, the method allows in-situ reconstruction of microstructure unlike pre- or post-deformation measurement in destructive techniques [71].

King [79] and Ludwig [104] demonstrated a technique named X-ray diffraction contrast tomography known as DCT. The method is similar to 3DXRD, but together with X-ray diffraction imaging and image reconstruction from projections, which can offer an on-line access to 3D grain profile and crystallographic orientation. Not only can the method be used for real-time surveillance of microstructure related deformation mechanisms and damage evolution [80], but also make a comparison with simulation results based on the reconstructed structure.

In order to quantitative characterize the 3D microstructure of polycrystalline aluminum (Al) doping with stannum (Sn) aiming to improve the X-ray contrast at grain boundaries, Dobrich et al [29] introduced an absorption contrast X-ray micro-tomography to extract reliable values for the size and topology of near 5000 individual grains in polycrystalline Al-Sn, and concluded that better agreement can be achieved with the reconstruction microstructure.

2.4.2 Numerical skills

Though detailed information on microstructure can be determined via experimental techniques, post-processing procedure of reconstruction is sophisticated and time-consuming besides being quite expensive. Moreover, a trade-off between accuracy and time cost should be taken into account. On the other hand, with the development of processing capability of computer, the application of reliable computational models attracts great interests in the field of material science and engineering.

To generate the virtual morphology of microstructure, there are two resorts popularly employed: one is the regular morphology; the other is analytical random morphology. By homogenization theories, regular morphologies are prevalent since such kinds of microstructures are easy to generate and to mesh [193]. The purpose of using the regular morphology is to establish the relationship between micro- and macro-properties, rather than to investigate the localized phenomena of real polycrystalline materials. Figure 2.9 shows the example regular morphologies introduced in the literatures [11, 133, 137, 193].

The methods for generation of the analytical random morphologies of microstructure have been proposed in published studies [5, 12, 38, 168]. Among these methods, Voronoi tessellation is the most popular one to represent the polycrystalline microstructure due to the definition of grains with straight edges and curved

FIGURE 2.9: Examples of regular morphologies: (a) cubic grains, (b) rhombic grains, and (c) truncated octahedral [11, 133, 137, 193]

faces or planar faces [5, 168]. The analytical random generated Voronoi tessellation representative microstructure is widely accepted as a first order approximation of polycrystalline microstructures to investigate the behavior of materials such as metal and ceramics at the microscopic level [12, 38, 82, 137, 193]. And the variability of Voronoi algorithms, such as Hardcore-Voronoi, Poisson-Voronoi, centroidal Voronoi and Laguerre-Voronoi tessellation, promotes its application [9, 82, 83, 125, 130, 154].

With the development of the computational capability of computer, the numerical investigation on fracture behavior of materials at grain level becomes more and more prevalent. One of the widely used methods is the molecular dynamics (MD) simulation to study the intergranular and transgranular crack propagation behavior at grain boundary [183]. By using this method, crack velocity can be achieved by modelling a sharp ductile/brittle interface crack propagation at microscopic level clearly [63]. The other one is the finite element method (FEM) to simulate the fracture process [26, 58, 179, 185]. A Lagrangian finite element method is

proposed coupled with a cohesive-law fracture model enable multiple crack propagated along arbitrary paths [14]. This method can show good agreement of radial fracture histories with experiment. Another FEM method to model the crack propagation in single crystals is based on crystal plasticity finite element computations of a pre-cracked single crystal [97]. This method can simulate 3D crack propagation process, during which crack front fragmentation and bifurcation can be captured. Among FEM methods, X-FEM as a newborn one is a good candidate for the simulation of complex dynamic crack evolution [26, 58]. However, pre-crack is required to investigate the crack propagation for most of the FEM simulations [58, 97, 185] due to the research interests. To investigate the overall mechanical property of a material structure composed of numerous grains at microscopic level, both the properties of material and interface are important. The cohesive law (Abaqus) / tie-break contact (LS-Dyna) property has been successfully implemented into FEM method to represent microcrack initiation, propagation as a natural outcome of the calculated material response. The intergranular damage in polycrystalline metals was fruitfully modelled by cohesive element [147]. And the crack initiation and propagation in the microstructure have been simulated by tie-break property [39]. Therefore, the numerical simulation enables the behavior of polycrystalline materials at grain level to better understand the macroscopic mechanical property.

2.5 Constitutive behavior of ceramic material

Ceramics for protection of both human beings and vehicles, whose notable characteristic such as light weight, high strength in compression and high Hugoniot elastic limit makes them suitable for high speed applications in particular, have been widely investigated on the ballistic performance against penetration for recent years due to the significant enhancement of bulletproof property by attaching the ceramics front plate to the metal backing materials [32, 84, 85, 105, 106, 146]. Therefore both theoretical and practical value can be reflected via investigation on the dynamic response of ceramic materials.

The investigation on dynamic performance of ceramic material can be traced back to 1960s since the discovery of ceramics backed by metal or composites offering an outstanding weight-efficient armor system to stop a high velocity projectile [62, 109, 138]. In such environment, the penetration depth method (known as depth-of-penetration, DOP) began to be widely used to study the material anti-penetration capability [2, 55, 84, 85]. One of the advantages of DOP test is the tested specimen is close to the actual size. However, drawbacks do exist such as little information obtained (e.g. only residual depth on the target) [120, 181]. With the improvement of DOP test, the method has become standard and been extensively accepted (e.g. U.S. Army and Materials Research Laboratory) to evaluate the anti-penetration ability of materials.

Meanwhile the development of experimental methodologies promotes the investigation of constitutive behavior of ceramics, especially in dynamic properties. Usually, the description of constitutive behavior of ceramic material can be divided into five categories:

The first one is the simple instantaneous models based on Griffith criteria [36, 173, 174] which assumed that failure occurs instantaneously when stress, strain, energy, or any other state variable reaches a critical value. And the deformation processes are described by Hooke's law. The model can provide an approximation and quick engineering estimation of the solutions to certain dynamic problems. However, extensive experiments should be carried out to validate the predictions.

The second one is called the plasticity based model [51] where elastic-plastic constitutive models with and without dilatation were used to describe the inelastic deformations. Plasticity theory based constitutive and failure model descriptions for brittle materials provide an alternative and improved method which has been successfully employed in explosive loading analysis of armor, oil shale, fragmentation of rocks and concrete structures.

The third one is the nucleation and growth model [27] which are built on the observation of material failures because of the growth of either cracks or voids that nucleate from heterogeneities on the microscopic scale in the solids. The effect of strain rate was involved into the failure of ceramics by means of plastic constitutive equations.

The fourth one is the fragmentation model [52] which can be used to describe tensile damage in terms of crack density where the characteristic flaw size is assumed to be proportional to the radius of the average fragment size.

The fifth one is fracture mechanics based model [112, 113] which seems plausible to simulate the behavior of brittle materials at the quasi-static rate. The application of this model can be extended to the high rate testing with proper modification.

However, a practical constitutive equation may contain one or a combination of the above approaches depending on the problem itself.

Recently, progress on the constitutive models of ceramics has been made. Some of the models have been applied to the engineering design. TCK model [161] is an ideal plastic constitutive equation proposed by Taylor, Chen and Kuszmul, which simplifies the compression behavior of brittle materials, assumed that damage accumulated only under tension state. Based on TCK model, Rajendran [131, 132] successfully simulated the stress history of AD85 ceramics under plate impact test by introducing the crack growth model to describe the inelastic response under tension as shown in Figure 2.10. The inelastic deformation caused by the interaction of nucleation of crack, propagation and microcrack. JH-2 ceramic model proposed by Johnson and Holmquist [64, 65] used to simulate the dynamic behavior of brittle materials is more prevalent in engineering design as shown in Figure 2.11. The model includes a representation of the intact and fractured strength, a pressure-volume relationship that can include bulking, and a damage model that transitions the material from an intact state to a fractured state. The damage for fracture is defined by a ratio of accumulated strain and fracture strain.

The constants in the model can be obtained by SHPB testing and plate impact testing. Nowadays, the model has been employed for commercial software such as EPIC, Autodyn and CTH. Certainly, there are some other models available to simulate the mechanical response of brittle material such as crack extension sliding model [4], microplane model (see Figure 2.12) and microcracking multiple-plane model (see Figure 2.13). However, cavity expansion analysis (CEA) plays a crucial role on the investigation of the ability of a target to resist penetration.

FIGURE 2.10: A comparison between model and measured stress history in a AD85 bar-on-bar experiment [132]

FIGURE 2.11: Computational and test results for three flyer plate impact tests [65]

FIGURE 2.12: Comparison of microplane models with test data of Evans and Marathe [10]

(a)

(b)

FIGURE 2.13: Axial stress history at different impact velocity: (a) measured, and (b) computed [37]

2.6 Summary

A detailed literature review on the methodology of characterization of the ceramic material at high strain rates including experimental improvements and available data process procedures, failure and deformation mechanisms, microscopic characterization tools consisting of both experimental and numerical methodologies, and the dynamic constitutive behavior of brittle materials, has been done in this chapter. Based on the review section, the research will focus on: a) accurate characterization of the material properties by means of split-Hopkinson pressure bar technique at high strain rate; b) dynamic deformation and failure process of material; c) crack initiation and propagation at the microscopic level; and d) the macroscopic penetration behavior of ceramics.

Chapter 3

Characterization of ceramics subjected to a uniaxial compressive loading

3.1 Introduction

The aim of this chapter was to accurately characterize dynamic stress-strain responses of ceramic materials that fail at the small strain ($< 2\%$) and high strain rate ($\sim 2000 \text{ s}^{-1}$) in a SHPB technique. Dispersion of wave signals recorded in SHPB tests was corrected based on the Pochhammer-Chree solution with an FFT algorithm in the frequency domain. A new direct analytical solution of one-dimensional stress wave propagation equations was derived with proper boundary

conditions to determine the exact time shifts and align the waves. The calculated specimen strain was compared with the strain measured using strain gauges mounted on the tested specimen. Force equilibrium of specimen ends was examined in SHPB testing. A FE model consisting of the full scale SHPB bars and the ceramic specimen was developed to further investigate the stress uniformity within the specimen in addition to the force equilibrium at the interfaces, allowing for the reliable calculation of dynamic stress-strain curves of ceramics.

3.2 Experimental methodology

3.2.1 Quasi-static compressive testing

The quasi-static experiment was carried out in the Instron 5569, which is an electromechanical test machine used for characterizing the properties of various materials in tension or compression by moving the crosshead in the direction upward or downward via screw driven system. The test specimen (5 *mm* in diameter and 12 *mm* in length) was sandwiched between surface hardened steel plates that were secured between the rigid frame base and the moving crosshead to avoid the damage of loading rams of tester as shown in Figure 3.1. The applied load was measured by a load cell in the machine. The measured strain from the tester directly is not accurate due to the high stiffness of ceramics; a pair of strain gauges was mounted on the opposite sides of the sample. In addition, specimens were lubricated with light grease to minimize the interfacial friction.

FIGURE 3.1: Quasi-static compression test of alumina: (a) the schematic, and (b) experimental setup

Other features of the tester relevant to the tests are listed below:

- 1) Crosshead velocity is 0.072 mm/min ;
- 2) Data acquisition rate is 10 Hz .

3.2.2 Split-Hopkinson pressure bar (SHPB) experiments

Uniaxial compression experiments of ceramics at dynamic strain rates of $\sim 2000 \text{ s}^{-1}$ were performed in a split-Hopkinson pressure bar (SHPB) system (Figure 3.2(a)). The SHPB system consisted of $D_0 = 20 \text{ mm}$ diameter striker, input and output bars with length of 400 mm , 1200 mm and 1200 mm , respectively. The bars were all made of YAG300 maraging steel (Young's modulus $E_0 = 184 \text{ GPa}$, Poisson's ratio $\nu = 0.30$, density $\rho_0 = 8030 \text{ kg m}^{-3}$, and thus wave velocity $c_0 = \sqrt{E_0/\rho_0} = 4786 \text{ m s}^{-1}$). Note that pulse shaping technique, despite the potential

to minimize wave dispersion, was not introduced as it could result in reduced strain rates [43, 127].

FIGURE 3.2: The split-Hopkinson pressure bar (SHPB) system: (a) the schematic of SHPB configuration, and (b) the setup of ceramic specimen and the definition of one-dimensional coordinate system

Cylindrical alumina (Al_2O_3) specimens (Chair Man Hi-Tech Co. Ltd., Taiwan) of the diameter $D = 5 \text{ mm}$ and length $l = 5 \text{ mm}$ were prepared and placed between the input and output bars during the SHPB experiments. In a SHPB test, the impact by the striker generates an elastic pulse (called incident wave, σ_i or ε_i in terms of bar stress or strain) in the input bar that propagates towards the

specimen. When the incident wave reaches the bar-specimen interface, a portion of it (reflected wave, σ_r or ε_r) is reflected back into the input bar, and the remaining portion (transmitted wave, σ_t or ε_t) is transmitted into the output bar through the specimen. Strain gauges were mounted approximately in the middle of the bars to record these waves through the Wheatstone half bridge circuits, TML (Tokyo Sokki Kenkyujo Co. Ltd., Japan) dynamic strainmeter and Yokogawa (Yokogawa Electric Corp., Japan) oscilloscope (Figure 3.2(a)).

To avoid the indentation into the steel bars caused by the harder ceramic specimens, a pair of cylindrical tungsten carbide (WC, diameter 17 mm and length 17 mm) inserts was sandwiched between the bars and specimen (see Figure 3.2(b)) [140, 158]. In the SHPB test on a WC insert being the specimen, the entire incident wave was transmitted with negligible losses but was not reflected at the bar-insert interface (Figure 3.3(a)); this is similar to the wave propagation in the dummy test without a specimen (Figure 3.3(b)). Therefore, the wave impedance of the WC inserts matches that of the maraging steel bars. The steel sleeves were used to confine and further strengthen the WC inserts such that the ceramic specimen failed before the inserts did (Figure 3.2(b)).

A pair of strain gauges, forming part of a Wheatstone half bridge circuit, was mounted on the opposite surfaces of specimens (Figure 3.2(b)) to directly measure the strain history in specimens. It is often difficult to measure the entire strain history as a result of the earlier failure of strain gauges caused by crack propagation in the specimen. However, the strain history, incompletely but directly measured, can be used to validate the calculated strain as will be discussed below.

FIGURE 3.3: Recorded wave signals in two SHPB tests to verify the impedance match between the WC inserts and bars: (a) a WC insert as the specimen, and (b) no specimen

3.3 Finite element modelling of SHPB experiment

A 3D FE model using the ABAQUS/Explicit solver (Dassault Systèmes Simulia Corp., RI, USA) was developed to simulate the strain wave propagation in the bars and the stress distribution/evolution in the alumina during an SHPB experiment

as well as the deformation and failure process. The FE model consisted of the full scale geometries of the striker, input and output bars as well as the WC inserts and alumina specimen (Figure 3.4). Hexahedral elements were used to mesh the FE model. The deletion of FE elements was applied to the corresponding part in alumina where damage occurred. The mesh density of FE elements was selected for the computational efficiency/accuracy and energy dissipation during the damage process. The alumina was meshed with the finest FE elements. The radii of the alumina, WC inserts and SHPB bars were then meshed with 25, 20 and 10 FE elements, respectively. The dynamic response of alumina in an SHPB test was simulated as a function of the FE mesh density in the radial direction of the alumina specimen. As shown in Figure 3.5, a convergence of simulation results was achieved if the radius of alumina was meshed with at least 10 FE elements. However, a higher mesh density of 25 elements in the radius was used in the alumina to better simulate the fragmentation during the SHPB test because the fragments can be as small as $100\ \mu\text{m}$ (as will be discussed in Chapter 4).

FIGURE 3.4: The full scale geometrical model of an SHPB test of a ceramic specimen and the FE meshes along the longitudinal direction

FIGURE 3.5: The relative difference between predicted and measured results as a function of the FE element density in the radius of the alumina

An initial impact velocity of 20 m s^{-1} was applied to the striker. A frictional coefficient of 0.06 was defined for all the interfacial contacts in the SHPB system, e.g., the interface between the striker and input bar. The elastic material data were specified for the striker, input and output bars and the WC inserts.

The constitutive behavior of alumina ceramics at high strain rates was quantified using the Johnson-Holmquist material model with damage (JH-2), a widely accepted model for brittle materials subjected to high loading rates, high pressure and large deformation [74]. To describe the material behavior of ceramics, the JH-2 model considers the equation of state (EOS) in a polynomial form, the specific strength model for both the intact and fractured materials and the damage evolution rule. The polynomial EOS evaluates the current pressure state by the variation of volumetric strains. The damage model determines the occurrence of fracture in ceramics. Most of the constitutive parameters for the JH-2 model of

alumina were obtained from the experiments in this study; only a few parameters were from literature [159]. The determination of JH-2 parameter will be demonstrated below.

3.4 Analysis of SHPB wave signals

The stress (σ_i , σ_r and σ_t) or strain (ε_i , ε_r and ε_t) waves in the bars were analyzed to reduce noises by filtering the high frequency oscillation, correct the wave dispersion, and subsequently align to the bar-specimen interfaces, as detailed by the flowchart in Figure 3.6. With the assumption of axial uniformity of specimen stress and strain fields:

$$\varepsilon_i + \varepsilon_r = \varepsilon_t \quad (3.1)$$

the average stress (σ) and strain (ε) history of the specimen can be calculated as follows, which will be discussed in Section 3.6.3.

$$\sigma(t) = \frac{A_0 E_0}{A} \varepsilon_t(t) \quad (3.2)$$

$$\varepsilon(t) = \frac{2c_0}{l} \int_0^t [\varepsilon_i(\tau) - \varepsilon_r(\tau)] d\tau \quad (3.3)$$

where A_0 ($= \pi D_0^2/4$) and A ($= \pi D^2/4$) are the cross section area of the bars and the specimen respectively, and t is the time.

FIGURE 3.6: Data analysis procedure of wave signals recorded in SHPB bars to calculate stress-strain curves of specimens

3.4.1 Noise reduction

Figure 3.7 illustrates the representative wave signals recorded in an SHPB test of alumina. In a conventional SHPB test, a low pass filter in hardware is an efficient way to remove the high frequency oscillation of signals, e.g., typically ≥ 30 kHz in the TML dynamic strainmeter used in this work.

However, the loading duration in an SHPB test of ceramics (failure strain $< 2\%$) is usually less than $20 \mu s$ at a strain rate on the order of $10^3 s^{-1}$, thus requiring the signal information at higher frequency (e.g., > 50 kHz) be recorded so that a sharp rising edge of the incident wave can be obtained. In this study, the low pass

FIGURE 3.7: Recorded wave signals in a SHPB test of Al_2O_3 that were subjected to noise reduction by filtering high frequency oscillation (red) and subsequent wave dispersion correction (blue): (a) a full period, and (b) zoom in views to highlight the signal process in the rising stage of incident, reflected and transmitted waves

filter function in the dynamic strainmeter was deactivated to retain all frequency components of the signal. The noise of wave signals was then reduced in the post-test FFT algorithm by filtering the frequency (≥ 100 kHz) oscillation. Figure 3.7(b) shows a good agreement between the rising edges of the wave signals before and after filtering the high frequency components by FFT.

3.4.2 Wave dispersion correction

Strain waves disperse inevitably when propagating to another location in the SH-PB bars. Correction of the wave dispersion in the frequency domain using an FFT algorithm [99, 191, 194] along with the Pochhammer-Chree solution [20, 129] was conducted to reconstruct strain waves $\varepsilon(x_0 + \Delta x, t)$ at the bar-specimen interfaces $(x_0 + \Delta x)$ from the waves $\varepsilon(x_0, t)$ measured in the strain gauge locations (x_0) , e.g., in the middle of the bars. Note that $\varepsilon(x_0, t)$ can be ε_i , ε_r and ε_t as measured in the strain gauges.

The discrete Fourier spectrum $\varepsilon(x_0, f)$ was calculated from the data in a fixed window around the original wave $\varepsilon(x_0, t)$:

$$\varepsilon(x_0, f) = F\{\varepsilon(x_0, t)\} \quad (3.4)$$

The time scale was shifted by $t_s = t - \Delta x/c_0$ to express the wave $\varepsilon(x_0 + \Delta x, t_s)$ at the bar-specimen interfaces. The i^{th} Fourier component in the spectrum of the wave $\varepsilon(x_0 + \Delta x, f)$ at the new location $x_0 + \Delta x$ was thus obtained:

$$\varepsilon(x_0 + \Delta x, f_i) = \varepsilon(x_0, f) e^{-j\Delta\varphi_i} \quad (3.5)$$

where $\Delta\varphi_i$ is the phase increment of the i^{th} Fourier component:

$$\Delta\varphi_i = 2\pi f_i \Delta x \left(\frac{1}{c_i} - \frac{1}{c_0} \right) \quad (3.6)$$

To calculate $\Delta\varphi_i$, the phase velocity c_i can be obtained from Bancroft's deduction for Pochhammer-Chree's analysis of wave propagation. In this study, Bancroft's deduction was solved directly rather than by interpolating the provided data [8, 99, 191, 194]. The difference in the phase velocities results in the dispersion of waves when travelling along the bars. The inverse FFT algorithm was finally taken to reconstruct the waves in the time domain (t_s) at the bar-specimen interfaces:

$$\varepsilon(x_0 + \Delta x, t_s) = F^{-1} \{ \varepsilon(x_0 + \Delta x, f) \} \quad (3.7)$$

Following wave dispersion correction (refer to Figure 3.7), the processed signals ε_i and ε_r represented the wave histories at the input bar-specimen interface while ε_t was reconstructed to the output bar-specimen interface. The correction improved the accuracy of the wave signals at the bar-specimen interfaces in the SHPB system. The previous work [191] validated the wave dispersion correction procedure using the zero force at the free end of an input bar without a specimen.

3.4.3 Alignment of wave signals

The three waves in SHPB bars, once the dispersion is corrected, needs to be synchronized such that the force and velocity at the bar-specimen interfaces can be calculated. To accurately align the reflected (ε_r) and transmitted(ε_t) waves to the interfaces, both waves were predicted for a known incident (ε_i) wave and a fictitious elastic specimen assumed to have a Young's modulus equal to that of

the real ceramic specimen. The predicted shapes of the reflected and transmitted waves were compared with the measured ones for alignment.

During SHPB testing of a completely elastic specimen (Young's modulus E , density ρ , and wave velocity c), the one-dimensional wave propagation equation in the specimen is governed by:

$$\frac{\partial^2 \sigma}{\partial t^2} = c^2 \frac{\partial^2 \sigma}{\partial x^2} \quad (3.8)$$

with the boundary conditions at the two specimen ends:

$$\frac{A}{A_0} \sigma(0, t) - \rho_0 c_0 v(0, t) = 2\sigma_i(t) \quad (3.9)$$

$$\frac{A}{A_0} \sigma(l, t) + \rho_0 c_0 v(l, t) = 0 \quad (3.10)$$

where $\sigma(x, t)$ and $v(x, t)$ are the stress and particle velocity histories in the specimen, respectively. The location x originating from the input end ($x = 0$) is defined in a coordinate system as shown in Figure 3.2(b). Note that the WC inserts can be considered to be part of the SHPB bars because of their impedance match (Figure 3.3).

A direct analytical solution, instead of a numerical solution as in the literature [191], of the wave propagation equations in a specimen was introduced to predict the wave shapes in this study. The direct Laplace transform was used to solve Eq.(3.8), Eq.(3.9), and Eq.(3.10), assuming stress equilibrium in the elastic specimen:

$$\rho \frac{\partial v}{\partial t} = \frac{\partial \sigma}{\partial x} \quad (3.11)$$

The specimen stresses $\sigma(0, t)$ and $\sigma(l, t)$ at the input and output bar-specimen interfaces can be calculated:

$$\sigma(0, t) = \frac{2A_0}{A} \left[\frac{1}{1-r} \sigma_i(t) u(t) - \frac{2r}{1-r} \sum_{k=1}^{\infty} \frac{(1-r)^{2k}}{(1+r)^{2k+1}} \sigma_i(t-2k\tau) u(t-2k\tau) \right] \quad (3.12)$$

$$\begin{aligned} \sigma(l, t) = \frac{A_0}{A} & \left[\frac{4r}{(1+r)^2} \sigma_i(t-\tau) u(t-\tau) \right. \\ & \left. + \frac{4r}{1+r} \sum_{k=1}^{\infty} \frac{(1-r)^{2k}}{(1+r)^{2k+1}} \sigma_i(t-(2k+1)\tau) u(t-(2k+1)\tau) \right] \end{aligned} \quad (3.13)$$

where $u(t)$ is the unit step function, $\tau = l/c$ is the wave travel time between the specimen ends (equal to half a propagation cycle 2τ), and $r = \frac{\rho_0 c_0 A_0}{\rho c A}$ is the generalized wave impedance ratio between the bars and specimen.

The $\sigma(0, t)$ and $\sigma(l, t)$ can be related to the wave propagation at the bar-specimen interfaces during SHPB testing:

$$\frac{A}{A_0} \sigma(0, t) = \sigma_i(t) + \sigma_r(t) \quad (3.14)$$

$$\frac{A}{A_0} \sigma(l, t) = \sigma_t(t) \quad (3.15)$$

Substituting Eq.(3.12) and Eq.(3.13) to Eq.(3.14) and Eq.(3.15), the reflected and transmitted waves can be derived as:

$$\sigma_r(t) = \frac{1-r}{1+r}\sigma_i(t)u(t) - \frac{4r}{1+r} \sum_{k=1}^{\infty} \frac{(1-r)^{2k}}{(1+r)^{2k+1}} \sigma_i(t-2k\tau)u(t-2k\tau) \quad (3.16)$$

$$\sigma_t(t) = \frac{4r}{(1+r)^2}\sigma_i(t-\tau)u(t-\tau) + \frac{4r}{1+r} \sum_{k=1}^{\infty} \frac{(1-r)^{2k}}{(1+r)^{2k+1}} \sigma_i(t-(2k+1)\tau)u(t-(2k+1)\tau) \quad (3.17)$$

The first terms in Eqs. (3.16) and (3.17), respectively, represent the immediate reflection at the input bar-specimen interface ($x = 0$) and the transmission at the output bar-specimen interface ($x = l$) after the incident wave propagates through the specimen. The second terms in the two equations indicate the contribution of the waves travelling within the specimen between the two interfaces.

Given the incident wave reconstructed at the input bar-specimen interface (Figure 3.8), two fictitious reflected and transmitted waves were predicted for an SHPB test of a fully elastic specimen using Eqs. (3.16) and (3.17). The profiles of the two predicted waves are only similar to the measured ones (in the SHPB test of real ceramics) in the early stage (initial part of the rising edge) but different in most of the other portions, because the wave propagation in the specimen is assumed to be totally elastic in the test. Note that the predictions, affected by $r = \frac{\rho_0 c_0 A_0}{\rho c A}$ in Eqs. (3.16) and (3.17), are sensitive to the Young's modulus of the specimen. The modulus as measured in the quasi-static compression experiments was used in the prediction to verify the accuracy.

FIGURE 3.8: Alignment of reflected and transmitted strain waves to the incident wave in SHPB testing of Al_2O_3 . Both reflected and transmitted waves were shifted based on the shapes of corresponding predicted waves (dotted lines) that were predicted by Eqs. (3.16) and (3.17)

The measured reflected and transmitted waves, following wave dispersion correction, were shifted to synchronize the incident wave so that their rising edges matched those of the corresponding predicted waves (refer to Figure 3.8). Note that in SHPB experiments, ceramic specimens (Al_2O_3) start to deform elastically in the early stage (represented by the rising edge of waves) although inelastic deformation can arise under further loading [73, 140]. The alignment of the waves was then justified because comparison of the wave shapes was only made in the rising edges of the waves. Therefore the three aligned ε_i , ε_r and ε_t (see Figure 3.8) represented the strain waves at the bar-specimen interfaces.

3.5 Constitutive model of alumina material

3.5.1 Johnson-Holmquist II model

Johnson-Holmquist II model [100] is useful to represent the brittle material subjected to large deformation, high loading rates and high pressures taken into account damage model and equation of state with a polynomial form.

JH-2 model is a developed one based on the yield surface evolution with damage. Both the intact and fractured strength are considered in this model. The softening of the material occurs with the increase of the plastic strain, during which the state of material transfer from intact to fracture. The normalized equivalent stress or strength can be expressed as:

$$\sigma^* = \sigma_i^* - D(\sigma_i^* - \sigma_f^*) \quad (3.18)$$

where σ_i^* is the normalized equivalent stress of intact material, σ_f^* is the normalized stress of fracture material, and D is the damage ranging from 0 to 1.

The general expression of the normalized equivalent stresses (σ^* , σ_i^* , σ_f^*) is denoted as:

$$\sigma^* = \frac{\sigma}{\sigma_{HEL}} \quad (3.19)$$

where σ is the actual equivalent stress and σ_{HEL} is the equivalent stress at the Hugoniot Elastic Limit (*HEL*).

The actual equivalent stress can be calculated from the following equation:

$$\sigma = \sqrt{\frac{1}{2} [(\sigma_x - \sigma_y)^2 + (\sigma_x - \sigma_z)^2 + (\sigma_y - \sigma_z)^2 + 6(\tau_{xy}^2 + \tau_{xz}^2 + \tau_{yz}^2)]} \quad (3.20)$$

where σ_x , σ_y and σ_z are normal stresses, τ_{xy} , τ_{xz} and τ_{yz} are the shear stresses.

The normalized strength for the intact material can be described as:

$$\sigma_i^* = A(P^* + T^*)^N (1 + C \cdot \ln \dot{\varepsilon}^*) \quad (3.21)$$

while the normalized strength for the fractured material is given by:

$$\sigma_f^* = B(P^*)^M (1 + C \cdot \ln \dot{\varepsilon}^*) \quad (3.22)$$

The normalized fracture strength can be limited by $\sigma_f^* \leq SFMAX$. This optional fracture strength parameter is included to provide more flexibility in defining the important fracture strength [74]. A , B , C , M , N , and T are the constants for a material. The actual pressure is denoted by P , the superscript (*) and subscript (*HEL*) represent the normalized pressure and the pressure at the *HEL*, respectively. The relationship is given by $P^* = P/P_{HEL}$. Similarly, the normalized maximum tensile hydrostatic pressure is denoted as $T^* = T/T_{HEL}$, where T is the maximum tensile pressure in hydrostatic state. The dimensionless strain rate can be expressed as $\dot{\varepsilon}^* = \dot{\varepsilon}/\dot{\varepsilon}_0$, where $\dot{\varepsilon}$ is the actual strain rate and $\dot{\varepsilon}_0 = 1.0 \text{ s}^{-1}$ is the reference strain rate.

The definition of damage accumulation for fracture is in a manner akin to that used in the Johnson-Cook fracture model [166]. It can be described by:

$$D = \sum \Delta \varepsilon_P / \varepsilon_P^f \quad (3.23)$$

where $\Delta \varepsilon_P$ is the plastic strain during a cycle of integration and $\varepsilon_P^f = f(P)$ is the plastic strain to fracture under a constant pressure, P . The specific expression is

$$\varepsilon_P^f = D_1 (P^* + T^*)^{D_2} \quad (3.24)$$

where D_1 and D_2 are constants. The material can't withstand any plastic strain at $P^* = -T^*$; however, ε_P^f increases with P^* .

The hydrostatic pressure for the intact material (at $D = 0$ or before fracture begins) can be expressed as below:

$$P = K_1 \mu + K_2 \mu^2 + K_3 \mu^3 \quad (3.25)$$

where K_1 (bulk modulus), K_2 and K_3 are the constants; and $\mu = \rho / \rho_0 - 1$ is the bulk strain, in which current density and initial density are given by ρ and ρ_0 , respectively. For tensile pressure ($\mu < 0$), $P = K_1 \mu$. Energy effects are supposed to be overlooked.

With the increase of damage accumulation ($D > 0$), bulking (pressure increase and / or volumetric strain increase) can take place. Now an additional incremental

pressure ΔP is added, such that

$$P = K_1\mu + K_2\mu^2 + K_3\mu^3 + \Delta P \quad (3.26)$$

The pressure increment is determined from energy considerations; it varies from $\Delta P = 0$ at $D = 0$ to $\Delta P = \Delta P_{max}$ at $D = 1$. The incremental internal elastic energy decrease (due to decreased shear and deviator stresses) is converted to potential internal energy by incrementally increasing ΔP . The decrease in the shear and deviator stresses occurs because the strength decreases as the damage increases.

The general expression from the elastic internal energy of the shear and deviator stresses is

$$U = \frac{\sigma^2}{6G} \quad (3.27)$$

where σ is the equivalent plastic flow stress and G is the shear modulus of elasticity.

The incremental energy loss is

$$\Delta U = U_{D(t)} - U_{D(t+\Delta t)} \quad (3.28)$$

where $U_{D(t)}$ and $U_{D(t+\Delta t)}$ are computed from Eq.(3.27) using $\sigma_{t+\Delta t}$ for both energies.

If the energy loss ΔU is converted to potential hydrostatic energy through ΔP , an approximate equation for this energy conservation is

$$(\Delta P_{t+\Delta t} - \Delta P_t)\mu_{t+\Delta t} + (\Delta P_{t+\Delta t}^2 - \Delta P_t^2)/2K_1 = \beta\Delta U \quad (3.29)$$

where β is the energy loss coefficient when the energy transfers from elastic to potential hydrostatic. The first term is the approximate potential hydrostatic energy for $\mu > 0$ and the second term is the corresponding potential energy for $\mu < 0$.

$$\Delta P_{t+\Delta t} = -K_1\mu_{t+\Delta t} + \sqrt{(K_1\mu_{t+\Delta t} + \Delta P_t)^2 + 2\beta K_1\Delta U} \quad (3.30)$$

As expected, when $\beta = 0$, ΔP yields 0.

3.5.2 Parameters determination of JH-2 model

The mechanical properties of Al_2O_3 material such as density, Poisson's ratio, and Young's modulus (shear modulus) or bulk modulus can be determined from quasi-static experiments (see Table 3.1).

Usually, it is difficult to determine the parameters of equation of state since the axial stresses obtained from a plate impact test produced a one-dimensional strain state contain both hydrostatic pressure and the deviator stress. The presence of deviator stress can result in a higher value in the equation of state. The fitting constants $K_1 = 250 \text{ GPa}$, $K_2 = -274 \text{ GPa}$ and $K_3 = 2934 \text{ GPa}$ are obtained based on the data provided by Chi et al [19]. The model shows a good agreement with the data (see Figure 3.9).

FIGURE 3.9: Equation of state curve of alumina ceramics

HEL is the elastic limit of material at one-dimensional strain state, and can be determined by plate impact test. Based on the experimental data provided in the literature, the HEL for Alumina ranges from 5~9 GPa [57, 120]. In the model, the HEL is taken as 7.0 GPa [159]. The HEL can be split into deviator stress and hydrostatic stress as:

$$HEL = P_{HEL} + \frac{2}{3}\sigma_{HEL} \quad (3.31)$$

Hooke's law in terms of shear modulus and bulk strain can be rewritten as:

$$\sigma = 2G \frac{\mu}{1 + \mu} \quad (3.32)$$

Therefore, the HEL can be expressed as a function of bulk strain at HEL :

TABLE 3.1: JH-2 constitutive parameters for the JH-2 model of Alumina

Parameter	Value
Density($kg\ m^{-3}$), ρ_0	3857
Young's modulus (GPa), E	390
Intact strength constant, A	0.958
Fracture strength constant, B	0.45*
Strain rate constant, C	0.0076
Intact strength exponent, N	0.243
Fracture strength exponent, M	0.6*
Hydro tensile limit (GPa), T	0.462
Hugoniot elastic limit (GPa), HEL	7*
Shear modulus (GPa), G	157.2
Pressure (EOS) constant, K_1	250
Pressure (EOS) constant, K_2	-274
Pressure (EOS) constant, K_3	2934
Damage constant, D_1	0.1
Damage constant, D_2	0.7

* Data were obtained from the literature [159].

$$HEL = K_1\mu_{HEL} + K_2\mu_{HEL}^2 + K_3\mu_{HEL}^3 + \frac{4}{3}G\frac{\mu_{HEL}}{1 + \mu_{HEL}} \quad (3.33)$$

Then $\mu_{HEL} = 0.015454$ and $P_{HEL} = 3.8089 \text{ GPa}$ can be determined.

Relationship between hydrostatic stress and failure stress of specimen can be found in Figure 3.10 at quasi-static (10^{-5} s^{-1}) and dynamic ($\sim 2000 \text{ s}^{-1}$) strain rates, respectively. In order to obtain strain rate effect parameter C , the ratio of σ_1/σ_2 yields a constant of 1.16 with the increase of hydrostatic pressure (see Figure 3.11).

FIGURE 3.10: Relationship between hydrostatic stress and failure stress of specimen at quasi-static (10^{-5} s^{-1}) and dynamic ($\sim 2000 \text{ s}^{-1}$) strain rates

Therefore, the strain rate sensitivity parameter can be calculated $C = 0.0076$.

To obtain the intact strength model parameters A and N when $D = 0$, assume the strain rate for plate impact test is 10^5 s^{-1} at the HEL. Therefore, A and N can be determined as 0.9577 and 0.2433, respectively. The fracture strength

FIGURE 3.11: Relationship between the ratio of σ_1/σ_2 and hydrostatic pressure

model parameters $B = 0.45$ and $M = 0.6$ for fracture specimen ($D = 1$) were used the data provided in literature [159]. The damage parameters $D_1 = 0.1$ and $D_2 = 0.7$ were determined by comparison of simulation and experimental data (refer to Table 3.2).

TABLE 3.2: Determination of damage constants D_1 and D_2

No. of simulation	Damage constant D_1	Damage constant D_2	Failure stress (GPa)	Relative error $(\sigma_{Exp} - \sigma_{Simul})/\sigma_{Exp} \times 100\%$
S01	0.005	0.7	1751.68	44.9%
S02	0.02	0.7	1916.54	39.7%
S03	0.04	0.7	2293.38	27.8%
S04	0.06	0.7	2602.50	18.1%
S05	0.08	0.7	2876.29	9.5%
S06	0.09	0.7	3091.20	2.8%
S07	0.1	0.7	3164.8	0.5%
S08	0.11	0.7	3385.6	6.5%
S09	0.13	0.7	3621.12	13.9%
S10	0.15	0.7	3915.50	23.1%
S11	0.1	0.5	3444.48	8.3%
S12	0.1	0.6	3297.28	3.7%
S13	0.1	0.8	3150.08	0.9%
S14	0.1	0.9	3061.76	3.7%
S15	0.1	1.0	3002.88	5.6%
S16	0.1	2.0	2973.44	6.4%

3.6 Results and discussion

3.6.1 Quasi-static compressive behavior

Both Poisson's ratios and stress-strain responses of alumina were measured under a constant strain rate of about $\sim 1.5 \times 10^{-5} \text{ s}^{-1}$. And a linear elastic response until the specimen fractured was observed. Figure 3.12 illustrates the variance of Poisson's ratio with time. The measured Poisson's ratio is 0.24 for alumina. Three representative stress-strain curves of alumina are displayed in Figure 3.13 with the

measured Young's modulus of 390 GPa . The failure strength is about 2.0 GPa and the failure strain is around 0.5% .

FIGURE 3.12: Measured Poisson's ratio of alumina materials during quasi-static compression

FIGURE 3.13: Measured stress versus strain curves of alumina materials at quasi-static compression

3.6.2 Validation and analysis of wave signals predicted in the FE model

With an initial striker velocity of 20 m s^{-1} , the propagation of elastic strain waves in the bars was simulated in the FE model of the full scale SHPB system. The predicted strain waves (ε_i , ε_r and ε_t) in the middle of the input and output bars were compared to the waves that were measured during the SHPB experiments (Figure 3.14). Note that the experimentally measured strain waves shown in Figure 3.14 were processed to reduce noise that arose due to the recording equipment; but such noise is not expected in the FE model. for the comparison purpose, the time was synchronized to that in the model. The agreement between the experimental and simulated strain waves (Figure 3.14) validated the FE model of SHPB compression as well as the use of JH-2 material model for alumina.

FIGURE 3.14: Comparison of strain waves in the SHPB system: experimental measurement versus FE prediction (Note: the noise was analytically filtered in the experimental results)

Using the analysis method of SHPB experimental data as described in Section 3.4, the predicted strain waves in the middle of the input and output bars were processed to correct the wave dispersion and align to the bar-specimen interfaces. Similarly, using Eqs.(3.16) and (3.17), the fictitious reflected and transmitted strain waves at the bar-specimen interfaces were calculated in an SHPB test of a fully elastic ceramic. As shown in Figure 3.15, these two fictitious waves are consistent with the strain waves at the bar-specimen interfaces that were directly predicted by the full scale SHPB FE model on the fully elastic alumina (without considering the inelastic behavior). The consistency further supports the developed FE model and the material constitutive formulation.

FIGURE 3.15: Comparison of reflected and transmitted strain waves in an SHPB test of a fully elastic ceramic: FE prediction versus analytical calculation using Eqs.(3.16) and (3.17)

3.6.3 Force and stress equilibrium

To verify the assumption of axial deformation uniformity in a specimen as defined by Eq. (3.1) such that the specimen stress and strain histories can be calculated, this study also examined the equilibrium of forces at the input (F_{in}) and output (F_{out}) ends of a specimen. The forces can be calculated in terms of the known ε_i , ε_r and ε_t strain waves at the bar-specimen interfaces:

$$F_{in}(t) = E_0 A_0 [\varepsilon_i(t) + \varepsilon_r(t)] \quad (3.34)$$

$$F_{out}(t) = E_0 A_0 \varepsilon_t(t) \quad (3.35)$$

To quantify the stress equilibrium, a coefficient (η) was defined by normalizing the difference between the forces at the specimen ends with their average ($(F_{in} + F_{out})/2$):

$$\eta = 2 \left| \frac{F_{in} - F_{out}}{F_{in} + F_{out}} \right| = 2 \left| \frac{\varepsilon_i + \varepsilon_r - \varepsilon_t}{\varepsilon_i + \varepsilon_r + \varepsilon_t} \right| \quad (3.36)$$

Figure 3.16 and 3.17 illustrate the evolution of equilibrium coefficient η during the SHPB compression of alumina, which was calculated based on the strain waves obtained from the experiment and the FE prediction, respectively. The forces are not in equilibrium at the very early stage of SHPB compression of alumina. The coefficient η reduces to $< 5\%$ after $5 \mu s$ that is equivalent to ~ 5 wave travel cycles within the specimens. Note that a wave propagation cycle $2\tau = 2l/c \approx 1.0 \mu s$ for alumina. Ravichandran and Subhash [134] suggested that the stress can be considered to be equilibrated if η is less than 5%. Therefore, the force equilibrium

at the two ends of the specimen is established after ~ 5 wave propagation cycles in the specimen during the SHPB compression experiments.

FIGURE 3.16: Verification of specimen stress equilibrium in SHPB testing of Al_2O_3 . Transmitted strain wave was compared to the sum of incident and reflected strain waves. Stress equilibrium was further quantified by the coefficient

$$\eta$$

The predicted stress distribution and its evolution with time (refer to Figure 3.18) were examined to verify the equilibrium of stress within the alumina during the SHPB compression. At the early stage of SHPB loading (e.g. at $124 \mu s$ in the FE model, refer to Figure 3.18(a)), the stresses are non-uniformly distributed throughout the alumina, especially at the ends due to the interfacial friction. However, a more uniform stress field can be observed at the late stage of the loading (e.g. at $133 \mu s$ in Figure 3.18(b)).

FIGURE 3.17: FE prediction of force equilibrium at specimen ends in the SHPB compression of alumina. Transmitted strain wave was compared to the sum of incident and reflected strain waves. Stress equilibrium was further quantified by the coefficient η

FIGURE 3.18: Predicted longitudinal axial stress distribution within an alumina specimen at the three stages of an SHPB compression: (a) 124 μs , (b) 133 μs

The coefficient of variation (C_v), defined as the ratio of the standard deviation (S_σ) to the average ($\bar{\sigma}$) of stresses, was used to further quantify the stress equilibrium in alumina:

$$C_v = \left| \frac{S_\sigma}{\bar{\sigma}} \right| \quad (3.37)$$

The C_v was calculated based on the predicted stress distribution in the entire volume ($C_{v,vol}$) and along the central line ($C_{v,line}$) in the specimen (see Figure 3.19). The $C_{v,vol}$ is approximately ten times greater because the significant stress concentration occurs in the specimen especially at the corners. However, as the SHPB load is applied, both the $C_{v,vol}$ and $C_{v,line}$ gradually decrease and become convergent to 0.17 and 0.02, respectively, after $5 \mu s$ of SHPB loading. the convergence of both $C_{v,vol}$ and $C_{v,line}$ implies a possible equilibrium of stress in the ceramic specimen. The examination of stress equilibrium in alumina is consistent with the analytical results in the previous work [134]. The assumption of axial uniformity of specimen stress and strain field as defined by Eq. (3.1) can be satisfied in the SHPB.

FIGURE 3.19: Verification of specimen stress equilibrium in an SHPB experiment of alumina by the coefficient of variation of stress distribution in the entire volume ($C_{v,vol}$) and along the central line ($C_{v,line}$)

3.6.4 Dynamic stress-strain curves of alumina

The stress and strain history of the specimens was calculated from the aligned ε_i , ε_r and ε_t waves using Eqs. (3.2) and (3.3) (see Figure 3.20). The specimen strain was also measured directly from the strain gauges mounted on the specimen surfaces. But only the initial portion of strain history was recorded because crack propagation on specimen surfaces induces the early failure of strain gauges prior to the fracture of ceramic specimens. The good agreement between the calculated and measured strains in specimens thus supports the alignment of strain waves and the use of two-wave analysis (Eq. (3.3)) to calculate strain. The accurate calculation of specimen strains using the developed analysis method avoids mounting strain gauges on specimens, and thus allows for the in-situ observation of failure process in ceramics using high speed photography.

FIGURE 3.20: Stress and strain history of specimens calculated from the bar wave signals in SHPB testing. Calculated strains were compared to those measured directly from strain gauges on alumina specimen

The dynamic strain rate is $\sim 2000 \text{ s}^{-1}$ as estimated by the average slope of the approximately linear portion of strain curves immediately prior to the fracture of alumina specimens (Figure 3.20). Figure 3.21 illustrates the representative stress-strain curves of alumina at the dynamic strain rates. The dynamic stress-strain response was also calculated using the strain waves in the middle of the input and output bars as predicted by the full scale FE model. The stress-strain curve predicted by the FE model agrees well with that measured in the SHPB experiments.

FIGURE 3.21: Stress-strain curves of alumina at dynamic and quasi-static strain rates

3.6.5 Strain rate dependency in alumina

To investigate the rate dependency of both ceramics, compressive stress-strain response at quasi-static strain rates (10^{-5} s^{-1}) was measured in a screw-driven loading rig. At low loading rates, only elastic deformation occurs before the brittle

failure. However, under high rate compression, significant amount of inelastic deformation occurs in both ceramics following the initial elastic deformation. Based on the experimental results, failure strain (and toughness) increases with strain rates, e.g., 0.005 versus 0.013 in Al_2O_3 at the rates of 10^{-5} and $\sim 2000 \text{ s}^{-1}$, respectively. For the failure strength, rate sensitivity is observed in Al_2O_3 up to the rate of $\sim 2000 \text{ s}^{-1}$. The previous work [73, 86] reveals that there exists a transition strain rate above which the compressive strength of ceramics increases with strain rates. In this study, the strain rate of $\sim 2000 \text{ s}^{-1}$ achieved in the SHPB test of ceramics may be greater than the critical rate of alumina (e.g., 100 to 400 s^{-1}).

3.7 Summary

The SHPB technique with a modified specimen setup was used to characterize the dynamic behavior of alumina at a strain rate of $\sim 2000 \text{ s}^{-1}$. The recorded SHPB wave signals were processed in a data analysis code that includes noise reduction, wave dispersion correction and subsequent alignment of the waves to the bar-specimen interfaces. The developed 3D FE model of the full scale SHPB test further verified the stress uniformity in the specimen and the force equilibrium at the specimen ends. The following conclusions can be drawn.

Correction of the wave dispersion using an FFT algorithm in the frequency domain improves the reconstruction of wave signals at the bar-specimen interfaces. The direct analytical solution derived for one-dimensional wave propagation using the

Laplace transform can precisely synchronize the waves to the interfaces based on the rising edge of waves.

Both the SHPB experiment and the FE simulation reveal that the forces at the specimen ends are equilibrated after ~ 5 wave propagation cycles within the specimens. It was found that the predicted stress field in the specimen becomes more uniform after the same period. These support the assumption of uniaxial homogeneous deformation in the specimen.

The calculated specimen strain is consistent with the strain measured by strain gauges mounted on the specimen, thus verifying the presented analysis method. The data analysis method for SHPB wave signals proves to be a reliable tool to quantify the dynamic stress-strain curves of alumina.

Chapter 4

Dynamic failure and fracture mechanism in alumina ceramics

4.1 Introduction

The goal of this chapter was to investigate the mechanism of the failure and fracture behavior of alumina subjected to dynamic loads. Dynamic uniaxial compression experiments in a split-Hopkinson pressure bar (SHPB) system fitted with high speed photography were conducted to quantify the stress history of alumina and directly observe the failure process in real time. The fracture surface on alumina fragments was examined in scanning electron microscope (SEM) to further explore the fracture mechanism in the failure process. An experimentally validated FE model consisting of the full scale SHPB bars and the alumina specimen was

developed to simulated the stress distribution and the damage evolution in alumina. The FE predictions were combined with high speed photographic observations to analyze and determine the dynamic failure mechanism in alumina.

4.2 Experimental procedure

Cylindrical hot-pressed alumina specimens (Chair Man Hi-Tech Co. Ltd., Taiwan) of the diameter $d = 5 \text{ mm}$ and the length $l = 5 \text{ mm}$ and 12 mm were manufactured for uniaxial compression experiments at dynamic and quasi-static rates, respectively. The end surfaces of the specimens were polished to produce good perpendicularity from the longitudinal axis. As surface defects may lead to early failure of ceramics during compression, the specimen surfaces were examined in optical microscope prior to mechanical testing, and only the specimens without surface defects were testes. The specimen ends were lubricated with Castrol LMX grease to minimize the interfacial friction that may induce complex stress states in the specimen under uniaxial compression.

The dynamic uniaxial compression experiments of alumina specimen ($d = 5 \text{ mm}$ and $l = 5 \text{ mm}$) were conducted in a split-Hopkinson pressure bar system schematically shown in Figure 4.1(a). The SHPB procedure of eramics and the subsequent analysis of measured strain waves were detailed in the previous chapter. The striker, input and output bars of 20 mm in diameter were 400 mm , 1200 mm and 1200 mm in length, respectively. The bars were made of YAG300 maraging steel with the Young's modulus of 184 GPa , Poisson's ratio of 0.30 and yield strength of 2000

MPa. A pair of wave impedance-matched cylindrical tungsten carbide inserts (diameter 17 mm and length 17 mm) was sandwiched between the bars and specimen to prevent the indentation into the steel bars by the hard ceramic specimen (refer to Figure 4.1(b)). The inserts were further confined and strengthened by the steel sleeves such that they could maintain intact prior to the specimen failure.

A range of dynamic strain rates ($200\text{--}2000\text{ s}^{-1}$) were achieved in the SHPB experiments by varying the gas chamber pressure and thus initial striker velocity. The strain gauges mounted approximately in the middle of the input and output bars recorded the incident (ε_i), reflected (ε_r) and transmitted (ε_t) strain waves. The Photron FASTCAM SA5 (Photron Ltd., Japan) high speed camera was used to track the deformation, failure and fracture process of alumina in the SHPB tests (Figure 4.1(b)). The applied frame rate was $580\,000\text{ fps}$ (frames per second) and hence the frame interval was $\sim 1.7\text{ }\mu\text{s}$. The camera was triggered by the transmitted wave signal using the 'end' trigger mode.

To investigate the strain rate dependency, the quasi-static uniaxial compression tests of alumina specimens ($d = 5\text{ mm}$ and $l = 12\text{ mm}$) were performed in an INSTRON (INSTRON, MA, USA) screw driven loading machine with controlled displacement rates. The specimens were sandwiched between a pair of surface hardened steel inserts to avoid indentation. The strain history in the specimens was directly recorded using two strain gauges mounted on the opposite circumferential surfaces of the specimens.

FIGURE 4.1: (a) The schematic of the split-Hopkinson pressure bar (SHPB) system, and (b) the setup of the ceramic specimen and high speed camera

4.3 Finite element modelling

ABAQUS/Explicit software (DassaultSystèmesSimulia Corp., RI, USA) was employed to help analyze and determine the dynamic failure mechanism. The finite element model used in this chapter is the same with the one listed in Section 3.3. The real size model of the whole SHPB setup was meshed with hexahedral elements. The interfacial contact friction coefficient of 0.06 was assigned to the whole model. The detail material property of alumina can be found in Table 3.1 (JH-2 model). For the striker, input and output bars as well as the tungsten carbide inserts, only the elastic properties were specified since they deformed elastically

during the SHPB experiments. In order to simulate the specimen subjected to different loading rates, the initial impact velocity of striker can be varied.

4.4 Results and discussion

4.4.1 Quasi-static and dynamic response of alumina

Figure 3.13 shows the representative stress-strain curves of alumina at quasi-static (10^{-5} s^{-1}) strain rates. At the quasi-static rates, only linear elastic deformation (Young's modulus 390 GPa) is observed in alumina prior to the fracture at $\sim 2000 \text{ MPa}$.

The strain waves recorded in the middle of the input and output bars during the SHPB tests were analyzed to filter the high frequency noise, correct the wave dispersion and subsequently align to the bar-specimen interfaces as detailed in the previous chapter. The force equilibrium at specimen ends was achieved after approximately 5 wave propagation cycles within the specimens. On the assumption of axial uniformity of deformation and one-directional elastic wave propagation, the stress and strain histories of the specimen were calculated using the measured strain waves in the bars (Figure 4.2). The dynamic strain rates ($\sim 2000 \text{ s}^{-1}$) were estimated by the average slope of the strain history (strain versus time) curves prior to the fracture of alumina specimens. The compressive failure strength of alumina is approximately 3000 MPa at the strain rate of $\sim 2000 \text{ s}^{-1}$. The failure

strength (σ_f) increases with the strain rates (compare Figure 3.13 and Figure 4.2, implying an increased energy dissipation capacity of alumina at higher strain rates.

FIGURE 4.2: Stress versus time curves at the dynamic strain rate of $\sim 2000 \text{ s}^{-1}$ during the SHPB tests and simulation of alumina

4.4.2 Quantification of strain rate dependency of failure strength

Figure 4.3 and Table 4.1 illustrate the failure strength measured under uniaxial compression as a function of strain rates. The measured data demonstrates the same trend with the data provided in the published works for alumina materials [1, 73, 153]. It is now well known that the compressive failure strength of both sintered and hot-pressed as well as hot pressed and successively hot-isostatically pressed alumina ceramics, beyond the critical strain rate, demonstrates strain rate dependency. However, it can be seen from Figure 4.3 and Table 4.1 that the compressive failure strength of alumina ceramics varies from one material to another due to the different fabrication processes.

TABLE 4.1: Failure strength(MPa) of Al_2O_3 at different strain rates

	Strain rate (s^{-1})	$\sim 10^{-5}$	~ 200	~ 600	~ 800	~ 1300	~ 2000
Research work	Fabrication process						
Present work	Sintered	2015 ± 185	2016 ± 106	–	2553 ± 45	–	3060 ± 115
Acharya (2015)	Sintered	~ 3000	–	–	~ 3000	–	–
Jiao (2004)	Sintered	–	–	~ 3850	–	~ 4100	~ 3600
Staehler (1995)	Hot-pressed	~ 2800	–	–	–	~ 5000	–
Jiao (2004)	Hot-pressed	~ 2950	–	–	–	–	~ 3850

It can be seen from Figure 4.3 that there exists a transition strain rate ($\dot{\epsilon}_t$), above which a strong strain rate dependency is observed in the failure strength of alumina. However, below the transition value, the strain rate insignificantly affects the failure strength. The transition strain rate $\dot{\epsilon}_t = \sim 250 \text{ s}^{-1}$ as estimated experimentally in this study is within the range of $100\text{-}400 \text{ s}^{-1}$ for alumina as measured by other researchers [87, 88].

FIGURE 4.3: The compressive failure strength of alumina as a function of strain rates

At the low strain rates ($\dot{\epsilon} < \dot{\epsilon}_t$), the relatively insensitive relation between the failure strength and strain rate was quantitatively described by the power law formula:

$$\sigma_f \propto \dot{\epsilon}^{\frac{1}{n+1}} \quad (4.1)$$

where n is the strain rate sensitivity exponent. The fitted exponent $n = 120$ at the low rate is within the value for alumina available in the literature [87] (refer to Table 4.2).

The compressive failure strength of alumina was regressed with the high strain rates ($\dot{\epsilon} > \dot{\epsilon}_t$) using the relation:

$$\sigma_f \propto \dot{\epsilon}^n \quad (4.2)$$

The rate sensitivity exponent $n = 0.327$ at the high strain rates is close to the predicted value (1/3) of alumina [87, 88] (Table 4.2). Note that in an SHPB test it is difficult to achieve a constant strain rate in ceramics before the specimen fails due to the very small failure strain. The strain rate increases from zero to the dynamic level under the SHPB load. The dynamic strain rate is defined as the average strain rate prior to the fracture, which makes possible the determination of the relation between the failure strength of alumina and dynamic strain rates.

TABLE 4.2: Strain rate sensitivity exponent (n) of alumina

Strain rate range	n , measured in this study	n , in the literature
$\dot{\epsilon} < \dot{\epsilon}_t$	120	50-200 [87]
$\dot{\epsilon} > \dot{\epsilon}_t$	0.327	1/3 [87, 88]

4.4.3 Validation of FE model

The detailed validation procedure can be found in Section 3.6.2, from which the FE predictions for both the strain waves in the bars (Figure 3.14) and stress versus time curves of alumina (Figure 4.2) match those measured in the SHPB experiments. The FE model also simulated the evolution of stress distribution in the alumina as well as the dynamic deformation, failure and fracture process (Figure 4.4). In the SHPB experiments, the failure and fracture process of alumina was captured by the high speed camera (Figure 4.5). The predicted failure process on the specimen surface is compared to that experimentally recorded at the five SHPB loading stages as indicated in the stress versus time curves in Figure 4.4 and 4.5. Note that the input bar is located in the left of the specimen in Figure 4.4 and 4.5. During the failure, longitudinal axial cracking occurs on the surface of the alumina specimen as observed in both the high speed images and FE predictions (Figure 4.4 and 4.5). The agreement between the experimentally observed and FE predicted surface failure process further supports the FE model and the material data used.

The characterization of dynamic failure process of alumina using high speed video camera is limited by the trade-off between the frame rate and image resolution.

FIGURE 4.4: Predicted maximum principal stress distribution and failure process in alumina as five different stages during an SHPB load. The five stages (a) to (e) are indicated in the predicted stress versus time and stress versus strain curves, respectively (top). The figures (b) to (e) also show the stress distribution in the longitudinal and middle transverse cross sections

FIGURE 4.5: High speed photographs of the dynamic failure and fracture process of an alumina specimen at five different stages during an SHPB test. The five stages (a) to (e) are indicated in the stress versus time and stress versus strain curves, respectively (top)

An increased frame rate (i.e., shortened frame interval), as required for the very short loading time in the SHPB of alumina (e.g., $< 15 \mu s$ in Figure 4.4 and 4.5), results in the reduced image resolution. However, the FE simulation can predict the real time process of failure in alumina subjected to dynamic loads. Moreover, both the external (surface) and internal damage evolution can be predicted in the FE model, thus offering detailed exploration of the dynamic behavior of alumina.

4.4.4 Failure process and mechanism under dynamic loads

4.4.4.1 Relation between stress distribution and failure process in alumina

At the early stage of the SHPB compression (Figure 4.4(a)), the stress within the alumina as well as the forces at the specimen ends is not equilibrated until after approximately $5\ \mu s$ equivalent to five wave propagation cycles (refer to Chapter 3). The stress is low, thus only elastic deformation occurs in the alumina (Figure 4.4(a) and Figure 4.5(a)). The average strain rate is not constant but increases with the load.

As the average stress in alumina increases to approximately $2000\ MPa$ (close to the quasi-static failure strength), the force equilibrium at the alumina ends becomes established. The stress distribution tends to be symmetrically equilibrated throughout the specimen. However, stress concentration continues existing in the alumina; in particular, the concentrated tensile stress is localized in the center of the two specimen ends (Figure 4.4(b)). Since the alumina is a brittle material with strong resistance to compression but weak resistance to tension, the damage emerges in these tensile stress concentration areas. The high speed photographic observation reveals that very fine powders were squeezed out from the specimen ends (refer to Figure 4.5(b)). The ejected powders combined with the lubricants were generated due to the stress concentration. However, the FE prediction doesn't show the damage perhaps because the FE element size (characteristic length $100\ \mu s$) is bigger than the ejected fine powder (Figure 4.4(b)).

Further loads during the SHPB compression cause highly multiaxial stress state in alumina as well as considerable tensile stress concentration on the surface (especially near the edge) and in the diagonal of the specimen (Figure 4.4(c)), which can then comminute the ceramic material. If the damage occurred in the alumina according to the JH-2 damage criterion, the corresponding FE elements were deleted in the model. Formation of damage, as represented by the deleted FE elements, occurs in the circumferential edges of the specimen ends where tensile stresses concentrate (Figure 4.4(c)). Due to the wave travel along the specimen, the damage at the left end (i.e., the input bar side in Figure 4.4(c)) arises slightly earlier than that at the other end. In the high speed photograph at the corresponding stress level, a small visible crack indicated by the arrow occurred at the left end of the alumina specimen (see Figure 4.5(c)).

With further loading, the formed crack tends to propagate towards the high tensile stress concentration area in alumina ceramics. Figure 4.4(d) shows that the damage (i.e., the deleted FE elements) propagates along the loading direction, giving rise to longitudinal axial cracks on the surface of alumina. Internally, the damage also evolves from the edges of the end surfaces towards the center of the specimen, leaving behind 45° damage bands (refer to the longitudinal cross-sectional view in Figure 4.4(d)). During the SHPB experiments, a number of cracks were observed on the specimen surface as shown by the arrows in the high speed photograph in Figure 4.5(d). The cracks propagated longitudinally and rapidly with a rate of 1000 m s^{-1} (averaged from the photographs in Figure 4.5(c) to 4.5(d)). The rapid propagation also resulted in the emission of light in the SHPB experiment.

Before the maximum stress (i.e., failure strength σ_f) is reached, the center of the alumina specimen is subjected to substantial compressive stress (see the longitudinal and middle transverse cross-sectional views in Figure 4.4(d)). A significant amount of strain energy is thus stored in the center. When the damage (cracking) evolves to the specimen center under further SHPB loads (Figure 4.4(e)), the instantaneous release of the strain energy causes the crush of the alumina specimen, consequently producing the small pieces of fragments (Figure 4.5(e)). The stress starts to drop due to the reduced load bearing capacity of the alumina specimen, as demonstrated by the stress versus time curves in Figure 4.2 (or Figure 4.4 and 4.5). Meanwhile, the radial strain increases significantly, approximately 0.14 versus 0.30 as measured in Figure 4.5(d) and 4.5(e), respectively.

4.4.4.2 Dynamic failure mechanism

Figure 4.6 illustrates the fragments of the alumina specimens collected after the uniaxial compression experiments. The large fragments with the average size of 2-5 mm were observed after quasi-static compression (Figure 4.6(a)). However, after dynamic loads in the SHPB tests, the fragments are small with a non-uniform size distribution ranging from 100-1500 μm for the majority (Figure 4.6(b)). Based on the FE simulation, the relatively larger fragments more likely result from the external circumferential part of the specimen, while the smaller ones from the internal portion. This implies that a number of small cracks grow to concurrently fracture the specimen under dynamic loads whilst only a few major large cracks under quasi-static loads.

FIGURE 4.6: The fragments of alumina specimens after uniaxial compression at: (a) quasi-static, and (b) & (c) dynamic loading rates

Wing crack model has been widely used to understand the failure process of brittle materials subjected to uniaxial compression [140, 172]. Tensile stresses are localized near grain boundaries and inclusions in the alumina subjected to uniaxial compression during the SHPB test. The localized tensile stresses drive axially-orientated micro-cracks to nucleate and grow to a subcritical state before the coalescence of the micro-cracks. Macroscopic failure process (i.e., micro-cracking)

of alumina then results from the coalescence of a large number of compression-induced, interacting and tensile micro-cracks. The propagation of macro-cracks is then dominated by the tensile stress concentration in the specimen that is mainly determined by the loading condition and specimen geometry (Figure 4.4). The macro-crack propagation in preferred orientations causes the longitudinal axial splitting failure on the specimen surface (Figure 4.4(b) to (d) and 4.5(b) to (d)) as well as the diagonal damage inside the alumina (Figure 4.4(b) to (d)).

4.4.5 Fracture mechanism

Figure 4.7 and 4.8 show the SEM examination of the fracture surfaces on alumina fragments after the compression experiments at quasi-static and dynamic strain rates, respectively. As typically observed in the alumina subjected to quasi-static compression (Figure 4.7(a)), the fracture surface is uneven and full of sharp edges, suggesting that the crack propagates along the grain boundaries due to the relatively weak interfacial strength (see the red solid line in Figure 4.7(b)). Therefore, the intergranular fracture dominates the failure process at low loading rates. The final fracture occurs at the peak stress as a result of the growth and coalescence of a few major cracks along the grain boundaries of alumina, thus producing large fragments (Figure 4.6(a)).

The SEM examination of typical alumina fragments obtained from the SHPB tests reveals a combination of uneven and smooth features in the fracture surface (Figure 4.8(a)). The strain rate increases from zero to the dynamic level during

FIGURE 4.7: Fracture of alumina subjected to quasi-static compression: (a) SEM image of the fracture surface, and (b) the schematic of the intergranular (red solid line) crack propagation process

FIGURE 4.8: Fracture of alumina subjected to dynamic compression: (a) SEM image of the fracture surface, and (b) the schematic of the combined intergranular (red solid line) and transgranular (red dashed line) crack propagation process

the SHPB compression. When the strain rate is less than the transition value, the subcritical crack nucleation and growth are suppressed due to the crack kinetics criteria; micro-cracks propagate along grain boundaries (refer to the red solid line in Figure 4.8(b)), resulting in the uneven fracture surface (intergranular fracture). When the dynamic strain rate increases above the transition value, the strain energy released near the crack tip is significantly high to cause the split of an individual grain in front of it (the red dashed line in Figure 4.8(b)) and the smooth

fracture surface (transgranular fracture). Therefore, the failure process of alumina is governed by both intergranular and transgranular fracture mechanisms under dynamic loading.

4.5 Summary

The mechanism of dynamic failure and fracture process in alumina ceramics was investigated in the SHPB experiments with high speed photography and the developed full scale 3D FE model. The predicted stress distribution and damage evolution in alumina were related to the experimental observations to further analyze the dynamic failure mechanism. The conclusions were drawn as follows.

The compressive failure strength of alumina is significantly sensitive to the high strain rates (above the transition rate, $\sim 250 \text{ s}^{-1}$) but almost insensitive to the low rates. A power law formula can quantify the strain rate dependency of failure strength.

Under dynamic uniaxial compression, highly multiaxial stress state occurs in alumina; and substantial tensile stresses concentrate on the surface and along the diagonal of the cylindrical specimen. The damage (cracking) tends to evolve from and propagate towards the high tensile stress concentration areas.

Both the high speed photographic observation and FE predications reveal that cracks form in the circumferential edges of the specimen ends and propagate longitudinally and rapidly with a rate of $\sim 1000 \text{ m s}^{-1}$, causing the axial splitting

failure on the alumina surface. The cracks also propagate diagonally within the alumina and give rise to the internal $\sim 45^\circ$ damage bands.

The failure process of alumina subjected to dynamic loads is governed by both the intergranular and transgranular fracture mechanisms, while intergranular fracture dominates the quasi-static failure process.

Chapter 5

Intergranular fracture in polycrystalline alumina ceramics

5.1 Introduction

As discussed in Chapter 4, the intergranular fracture dominates the failure of alumina ceramics at quasi-static strain rates. The aim of this chapter is to investigate the crack initiation and propagation of an alumina ceramic material in a micro-cantilever beam. The beams in the experiments were fabricated by the focused ion beam machining technique. The applied load exerted on the free end of the beam was measured in a nano-indenter with controlled displacement. Due to the limited information obtained from the experiments, finite element models of statistically representative polycrystalline structures were generated based on hardcore-Voronoi tessellation. Cohesive law was involved to model the grain

boundary failure (intergranular failure). This intergranular fracture can be observed from both the macroscopic quasi-static experiments in Chapter 4 and the micromechanical experiments. Finally, numerical results are compared with the experimental measurements, from which agreement with acceptable differences can be observed, based on the serial systematically parametric study on grain number, orientation distribution, and grain size.

5.2 Experimental procedure and observations

5.2.1 Materials and microstructure

The alumina specimens (Chair Man Hi-Tech Co. Ltd. Taiwan) were polished and then thermally etched (annealing in the air atmosphere at $1200\text{ }^{\circ}\text{C}$). Figure 5.1 shows the representative polycrystalline microstructure of alumina. Further analysis reveals that the average grain size is approximately $1.2\text{ }\mu\text{m}$.

5.2.2 Microcantilever bending experiments

The alumina microcantilever beams were prepared on a polished alumina specimen in an FEI Helios Nanolab 450S (FEI Co., Oregon, USA) using the focus ion beam (FIB) milling technique. A gallium ion beam operating at 30 kV with beam currents of 2.5 nA to 47 nA was applied to cut the microcantilever beam specimens (Figure 5.2). The microcantilever beam was approximately $20\text{ }\mu\text{m}$ in length. The transverse cross section was an equilateral triangle with the side length of $5\text{ }\mu\text{m}$.

FIGURE 5.1: Microstructure of an alumina specimen which was thermally etched

Note that it is difficult to prepare other types of cross sections such as a rectangle using the FIB technique.

Microcantilever bending experiments were conducted in an MTS nanoindenter XP (MTS Systems Corp., Minnesota, USA). The berkovich indenter was applied to the top surface near the free end of the microcantilever beam at a low strain rate until the beam failed (Figure 5.2). The displacement of the nanoindenter tip as well as the reaction force was recorded during the bending experiments. The final fracture feature in the alumina was examined in the scanning electron microscope (SEM).

FIGURE 5.2: (a) The schematic of a microcantilever beam for bending experiments, and (b) a typical alumina microcantilever specimen prepared by focused ion beam technique

5.2.3 Observations on deformation and intergranular fracture in alumina

Figure 5.3(a) shows the typical history of the load (P) applied near the free end of the microcantilever beams. At the initial stage of the bending load, the indentation depth into the alumina surface can be a considerable portion of total displacement of the nanoindenter tip; thus the load varies nonlinearly with the measured displacement (Figure 5.3(b)). Under further loads, the increased deflection of the microcantilever beam (ΔL) dominates the total displacement of the tip. Therefore, the applied bending load increases approximately linearly with the measured displacement (Figure 5.3(b)), representing the elastic deformation of the beam. To characterize the deflection of the microcantilever beam, the measured load versus displacement curve is shifted such that the linear portion passes the origin. The elastic modulus ($E = 390 \text{ GPa}$) of the bulk alumina can be quantified based on the slope of the curve ($P/\Delta L$):

$$E = \frac{P}{\Delta L} \cdot \frac{L_A^3}{3I} \quad (5.1)$$

where L_A is the distance between the nanoindenter tip and the fixed end of the microcantilever, and I is the moment of inertia of the transverse cross section.

The alumina microcantilever beam fails when the measured load reaches the peak

FIGURE 5.3: (a) The applied load history at the free end of an alumina microcantilever beam, and (b) the load versus displacement curve. Note that given the initial indentation into the specimen surface, the measured displacement of the nanoindenter tip (black) is corrected to calculate the deflection of the microcantilever beam at the free end (red)

(~ 1.18 mN) and subsequently drops abruptly in the bending experiment (Figure 5.3). The SEM examination reveals that the fracture of alumina occurs near the fixed end of the micro cantilever where the internal bending moment is the maximum (see Figure 5.4). The fracture surface is uneven and full of sharp edges, implying that the intergranular fracture dominates the failure process of the alumina in the bending test at low loading rates.

FIGURE 5.4: Typical fracture feature of two alumina microcantilever beams subjected to bending loads. Both the beams failed near the fixed end

5.3 Finite element modelling

A full scale 3D finite element model of the polycrystalline alumina microcantilever beam was developed in the ABAQUS/Explicit software (Dassault Systèmes Simulia Corp., RI, USA) to simulate the localized stress evolution and failure process of the beam subjected to bending loads (Figure 5.5). The FE simulation was rerun

with six sets of random grain orientations assigned to the grains in the beam.

FIGURE 5.5: The finite element model of an alumina microcantilever beam for bending tests: (a) the polycrystalline geometry generated by hardcore Voronoi tessellation, (b) the FE meshes, and (c) the cohesive elements for grain boundaries

A FE model of the cubic representative elementary volume (REV) of alumina was established to further investigate the effect of polycrystalline characteristics on FE

predictions (Figure 5.6). The response of the REV alumina subjected to quasi-static uniaxial tension was simulated as a function of the number of grains (baseline value 60) and grain size (baseline value $1.2\ \mu\text{m}$) (Table 5.1). Other variables included the random grain orientations (10 sets) and arbitrary polycrystalline morphologies (10 sets).

FIGURE 5.6: The cubic representative elementary volume of polycrystalline alumina: (a) the geometries with different grain numbers (30, 60 and 90) generated by hardcore Voronoi tessellation, (b) the FE meshes of the REV alumina with the grain number 60, and (c) the cohesive elements for the grain boundaries in the REV in (b)

This section describes in details the geometrical generation of the polycrystalline microstructure using Vroonoi tessellation, and the constitutive formulation for the individual grains and grain boundaries.

TABLE 5.1: The variation of parameters (number of grains, grain size, grain orientation and polycrystalline morphology) in the finite element model of the cubic representative elementary volume of polycrystalline alumina. Note that the length and the volume of cubic REV are estimated according to the number of grains and grain size.

Case	Number of grains	Grain size (μm)	Length of REV (μm)	Volume of REV (μm^3)	Number of grain orientations	Number of polycrystalline morphologies
1	30	1.2	3.0	27	10	10
2	60	1.2	3.78	54	10	10
3	90	1.2	4.33	81	10	10
4	60	0.3	0.95	0.85	10	5
5	60	0.6	1.90	6.79	10	5
6	60	2.4	7.57	434.3	10	5
7	60	4.8	15.1	3474.4	10	5

5.3.1 Voronoi tessellation and FE meshes of polycrystalline alumina

The grains in the 3D domain of polycrystalline alumina microcantilever beam were generated using the hardcore Voronoi tessellation technique in the open source software Neper [130] (refer to Figure 5.5(a)). The number of grains (N) in the domain was estimated in terms of the average grain size ($1.2 \mu m$) and the volume of the domain. The N nuclei (seeds) were then randomly distributed in the beam domain (i.e., seeding domain). The hardcore Voronoi regulation further specified a minimum repulsion distance (ρ) between any two nuclei (P_i and P_j) as follows:

$$|P_i - P_j| > \rho \quad \forall i, j = 1, 2, \dots, N : i \neq j \quad (5.2)$$

It can be assumed that: (1) all the nuclei remain in the fixed position and grow simultaneously in all directions at a constant rate; (2) a grain boundary is generated where the grains meet; and (3) there are no voids or overlaps in the Voronoi cells. All the points R_P in the i^{th} Voronoi cell are closer to the associated nucleus P_i than any other nuclei P_j in the microcantilever beam domain:

$$\{R_P\} = \{x \in X \mid d(P_i, x) \leq d(P_j, x), i, j = 1, 2, \dots, N : i \neq j\} \quad (5.3)$$

where P is the nuclei distribution, x is any point in the seeding domain X . The hardcore Voronoi tessellation can more regularize the cells (grains) than other regulation methods (e.g., Poisson's) and qualitatively simulate the first order properties of polycrystalline materials [38]. Similarly, the tessellation algorithm was also used to generate the grains in the cubic REV of polycrystalline alumina as listed in Table 5.1 (refer to Figure 5.6(a)).

The Voronoi cells in the microcantilever beam and the cubic REV were meshed with tetrahedral FE elements (Figures 5.5(b) and 5.6(b)) using the bottom-top algorithm in the open source meshing software *Gmsh* [49]. The discretisation was performed on the tessellation vertices ($0D$), edges ($1D$), faces ($2D$) and cells ($3D$) in sequence. The $n - D$ meshes were generated based on the $(n - 1) - D$ meshes.

Each $0D$ element contains only one node attributing a characteristic length, based on which each edge is meshed into $1D$ element whose lengths are derived from the characteristic length of the two vertices. Afterwards the generated new nodes are assigned a characteristic length to mesh each face into $2D$ triangular elements.

Similar to generation of 2D meshing, each polyhedra can be discretized into classic (4 nodes) or quadratic (10 nodes) tetrahedral elements. The successfully meshed micro-cantilever beam can be seen in Figure 5.4. The meshed structure is the same with the one used in the nano-indentation test, whose dimension is 20 μm in length with equilateral triangular cross-section of 5 μm in edge.

5.3.2 Anisotropic elastic properties of individual alumina grains

The constitutive behavior of individual grains in the polycrystalline alumina (alpha alumina in this study) was quantified by the linear anisotropic elastic formation:

$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl} \quad (5.4)$$

where C_{ijkl} are the components of the stiffness (elasticity) tensor, σ_{ij} and ε_{kl} are the stress and strain tensor, respectively. Owing to the trigonal structure of class $3\bar{m}$ of alumina crystal, only six independent elastic constants are required for the stiffness matrix C (three-fold symmetry but not orthotropic) in the stress-strain relation of alumina grains as follows [167].

$$\begin{Bmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \sigma_4 \\ \sigma_5 \\ \sigma_6 \end{Bmatrix} = \begin{bmatrix} C_{11} & C_{12} & C_{13} & C_{14} & 0 & 0 \\ C_{12} & C_{11} & C_{13} & -C_{14} & 0 & 0 \\ C_{13} & C_{13} & C_{33} & 0 & 0 & 0 \\ C_{14} & -C_{14} & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & C_{14} \\ 0 & 0 & 0 & 0 & C_{14} & \frac{1}{2}(C_{11} - C_{12}) \end{bmatrix} \times \begin{Bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \\ \varepsilon_4 \\ \varepsilon_5 \\ \varepsilon_6 \end{Bmatrix} \quad (5.5)$$

Numerous studies in the literature [50, 68, 114, 176] have reported the consistent elastic constants for alpha alumina. The anisotropic elastic constants used in the present FE models as listed in Table 5.2 were obtained from the literature [68].

TABLE 5.2: Anisotropic elastic constants stiffness of polycrystalline alumina [68]

Parameter	C_{11}	C_{12}	C_{13}	C_{14}	C_{33}	C_{44}
Value (GPa)	497	163	116	22	501	147

5.3.3 Cohesive layer for grain boundary

The failure of alumina microcantilever beams is dominated by the intergranular fracture (the failure of the interface between grains) as detailed in Section 5.2.3. Mechanical properties of the interface (grain boundary) can significantly affect the macroscopic behavior of alumina. The cohesive layer in ABAQUS was used to model the grain boundary within the alumina. The cohesive elements were

produced using the nodes that are shared by any two Voronoi tessellation entities (edges, faces or cells). Figures 5.5(c) and 5.6(c) illustrate the cohesive elements for the grain boundaries in the microcantilever beam and the cubic REV alumina, respectively.

The constitutive response of cohesive elements was defined by a traction-separation law that assumes the initially linear elastic behavior followed by the initiation and evolution of damage [35, 118] (refer to Figure 5.7). The linear traction-separation relation (line oa in Figure 5.7) defines the elastic behavior prior to damage. The damage as characterized by the stiffness degradation initiates at the separation δ_0 (i.e., traction T_0 at point a). The damage evolves along the line ab until the final fracture occurs at the separation δ_f . The damage evolution process can be quantitatively described by the fracture energy as determined by the area under the traction-separation curve or by the ratio of initial to final separations (δ_0/δ_f).

FIGURE 5.7: The traction-separation relation to characterize the damage initiation (point a) and evolution (line ab) of cohesive layers for grain boundaries

5.4 Results and discussion

5.4.1 Effect of grain characteristics on bulk elastic behavior

Parametric finite element modelling of cubic representative elementary volume of polycrystalline alumina subjected to tension at a strain rate of 0.001 s^{-1} was performed to simulate the bulk elastic behavior as a function of various microstructural features (Table 5.1). Three REVs consisting of 30, 60 and 90 grains (grain size $1.2\text{ }\mu\text{m}$) were generated to explore the influence of grain number on bulk elastic properties. Another four REVs with grain size 0.3, 0.6, 2.4 and $4.8\text{ }\mu\text{m}$ (60 grains) were simulated to investigate the grain size effect. A combination of ten different polycrystalline morphologies and ten groups of randomly generated grain orientations were created for each REV alumina. Therefore, a total number of 100 simulations were performed for each REV with a specific number of grains and grain size, making statistically valuable results. The effective bulk Young's modulus was calculated from the elastic deformation of each cubic REV alumina (Figures 5.8 and 5.9).

Figure 5.8 shows the predicted Young's modulus of the REV alumina as a function of the number of grains. For each grain number, the average modulus as well as the standard deviation ($< 2.5\%$) was calculated from the 100 FE simulations. The small standard deviation implies that the grain orientation and polycrystalline morphology have little influence on the bulk behavior of alumina. Theoretically, the response of an REV polycrystalline material well represents the bulk behavior

if the number of grains approaches infinity [126]. For the material with crystals of limited anisotropic elastic properties, an REV composed of more than 20 grains can approximate the bulk material with a small acceptable error (e.g., less than 10%) [12].

The difference of the average Young's modulus among the three grain numbers is very small ($< 1\%$) as shown in Figure 5.8. This suggests that the effect of grain number on macroscopic properties of alumina is insignificant if there are at least 2-3 grains in the characteristic edge. The transverse cross sectional area of the alumina microcantilever beam is similar to the cross sectional area of the cubic REV with 30 grains (Figure 5.5 and 5.6); thus the microcantilever beam can represent a bulk alumina beam.

The Young's modulus of the REV alumina under tension also varies with the grain size as demonstrated in Figure 5.9. For each grain size, the modulus varies with a small deviation ($< 5\%$) due to the negligible effect of the grain orientation and polycrystalline morphology. However, such effect is not constant but becomes even more trivial for the smaller grain size. the average modulus increases with the grain size (Figure 5.9). This is consistent with the findings on the fracture toughness of alumina ceramics; the toughness increases with the grain size when the size is less than $4\ \mu m$ [163].

FIGURE 5.8: Effect of the number of grains on Young's modulus of the cubic REV alumina. The average modulus for each grain number is calculated from the predictions for the combination of 10 polycrystalline morphologies and 10 grain orientations

FIGURE 5.9: Effect of grain size on Young's modulus of the cubic REV alumina. The average modulus for each grain size is calculated from the predictions for the combination of 5 (10 for grain size 1.2 μm polycrystalline morphologies and 10 grain orientations. Note that the number of grains is 60 in all the REV's

5.4.2 Validation of FE model on alumina microcantilever beam

The localized stress evolution as well as the failure and fracture process in the polycrystalline alumina microcantilever beam was simulated in the full-scale FE model with the controlled displacement applied at the free end at a low loading rate. Figure 5.10 shows the predicted load versus displacement curves at the free end of the beam. The six curves are the results of the beam in which all the grains were assigned with six different sets of random crystalline orientations. Note that unlike the microcantilever bending tests by the nanoindenter tip, the initial portion of the predicted curves is almost linear because no indentation is expected in the model in which the properties of alumina grains is elastic.

Based on the six predicted load versus displacement curves (Figure 5.10), the bulk elastic modulus as calculated using Eq.(5.1) is $E = 395 \text{ GPa}$ with a very small deviation $< 2\%$; additionally the load at failure (average 1.207 mN) varies within $< 3\%$. As shown in Figure 5.11, the predicted longitudinal stress (σ_z) is tensile on the top but compressive on the bottom in the microcantilever beam. The global stress magnitude is comparable among the simulations using different grain orientations. Therefore, the FE predictions (Figure 5.10 and 5.11) reveal that the grain orientation has negligible influence on the overall deformation and failure of the beam, but it affects the localized stress distribution.

The predicted load versus displacement curves are compared to the curves that

FIGURE 5.10: Comparison between the FE predicted and experimentally measured load versus displacement curves at the free end of an alumina microcantilever beam subjected to bending loads

FIGURE 5.11: Predicted longitudinal stress distribution in the alumina microcantilever beam at the elastic stage (approximately at the displacement 200 nm in Figure 5.10 during a bending load). The figures (a) and (b) compare the predictions using two different grain orientations in the model

FIGURE 5.12: Predicted longitudinal stress distribution in the alumina microcantilever beam at five stages during a bending load. The five stages (a-e) are indicated in the predicted load versus displacement curve (one of the predicted curves in Figure 5.10)

were measured in the microcantilever bending tests (Figure 5.10). The FE predictions match the experimental measurements in both the elastic modulus (395 versus 390 *MPa*) and the failure load (1.207 versus 1.18 *mN*). The failure and fracture process of alumina microcantilever was simulated in the FE model (refer to Figure 5.12). The prediction exhibits that the intergranular fracture occurs near the fixed end of the beam as observed in the post-test SEM examinations (compare Figure 5.4 and 5.12). Note that all the six simulations demonstrate the similar failure and fracture process. The agreement between the FE predictions

and the experiments validates the developed FE model of the alumina microcantilever beam as well as the use of the material properties (such as anisotropic elasticity of grains and cohesive properties for grain boundaries).

The post-test examination can only provide limited information on the fracture of the microcantilever beam. However, the FE simulation can predict the real time internal and external process of failure in the alumina, thus offering more detailed information of the fracture behavior of alumina.

5.4.3 Intergranular fracture process in alumina

5.4.3.1 Localized stress distribution and evolution

The localized longitudinal stress (σ_z) distribution in the grains of the microcantilever beam (Figure 5.12) is tracked in one of the FE simulations to investigate the intergranular fracture process in alumina under bending loads. The characteristic lines for grain boundaries are set to be invisible in Figure 5.12 to better reveal the fracture features in the late stage of loading. Nevertheless, the stress state in each grain can be differentiated due to the anisotropic elastic properties. Figure 5.13 demonstrate the evolution of maximum principal stresses in the grain boundaries as well as the crack initiation and propagation during the fracture process. The stresses in both the grains and grain boundaries increase with the applied bending load in the early stage (refer to Figures 5.12(a)-(d) and 5.13(a)-(d)). After the peak load is reached, the crack initiates and propagates along the grain boundaries (Figures 5.12(c)-(e) and 5.13(c)-(e)), resulting in the intergranular fracture of the

alumina. The release of strain energy causes an overall abrupt drop of the load at the free end.

The top portion of the microcantilever beam near the fixed end is subjected to tension whilst the bottom part under compression. The maximum stress occurs on the top and bottom surfaces where the crack is more likely to form. However, the grain orientation (anisotropy of elasticity) causes non-uniform stress distribution at the same distance to the neutral axis. The interaction of the adjacent grains results in the tensile stress concentration in the vicinity of the bottom where grain boundaries meet the bottom edge of the beam, as shown in Figures 5.13(b). Therefore, in the present beam with an upside down triangular transverse cross section, the crack tends to initiate from the bottom (refer to Figures 5.12(c) and 5.13(c)) and to propagate along the grain boundaries (Figures 5.12(d)-(e) and 5.13(d)-(e)). However, it should be noted that the crack may initiate from either the top or the bottom near the fixed end, depending on the geometry of the cross section.

5.4.3.2 Mechanisms of intergranular fracture: shear sliding and tensile opening

Both the experiments and FE simulations reveal the intergranular fracture of alumina under the low loading rates. The crystalline morphology and orientation of grains play an important role in the occurrence of the localized stresses, which consequently determine the cracking modes during the fracture process: tensile

FIGURE 5.13: Predicted maximum principal stress distribution in the grain boundaries (cohesive elements) in the alumina microcantilever beam at five different stages during a bending load. The five stages (a-e) are indicated in the predicted load versus displacement curve (one of the predicted curves in Figure 5.10)

opening (mode I) and shear sliding (mode II). Analysis of the fracture features in the Voronoi tessellation (Figures 5.12(e)) reveals the presence of both the tensile opening and shear sliding modes in the grain boundaries.

Figures 5.14 further schematically demonstrates the cracking processes in the grains near the fixed end of a microcantilever beam with random grain morphologies and orientations. The applied load at the free end causes the shear force and maximum bending moment at the fixed end. Microscopically, the interaction of grains as well as the disparity of their orientations determines the actual tensile or shear stress state in the grain boundaries. The formation and propagation of

cracks are then dominated by tensile opening or shear sliding. In the intergranular fracture of brittle materials such as alumina, the failure modes in the grain boundaries are dictated by the localized stress state which is affected by the external loading condition and the grain morphology and orientation.

FIGURE 5.14: Schematic of micro-mechanisms for bending-induced tensile stress at the contacts between grains. Note that the lateral load exerted on the beam can be resolved into tensile and shear stresses at grain boundaries)

5.4.4 Effect of cohesive properties on intergranular fracture

The use of cohesive layers enables modelling of intergranular fracture in alumina subjected to low strain rate loads. The interfacial behavior (Figures 5.7) is quantified by the traction-separation law using parameters such as the initial stiffness, damage initiation (including initial separation δ_0 and cohesive strength T_0) and damage evolution (final separation δ_f). To investigate the effect of cohesive

properties on the failure behavior of alumina ceramics, the FE model of the microcantilever beam with the same grain morphologies and orientations was rerun as a function of the cohesive strength (T_0 for baseline) and the ratio of initial to final separations ($\delta_0/\delta_f = 1$ for baseline). All the FE simulations reveal the similar localized stress state in the beam before the degradation of cohesive layers, thus the same elastic behavior of the beam. In each simulation, the intergranular fracture process in the beam is observed and dominated by shear sliding and tensile opening modes.

FIGURE 5.15: Predicted load versus displacement curves of an alumina microcantilever beam as a function of the damage initiation ratio (δ_0/δ_f) of cohesive layers defined for grain boundaries

Figures 5.15 compares the predicted load versus displacement curves at the free end. Quantitatively, both the load and displacement at failure increases with the cohesive strength and the damage evolution (i.e., the ratio of final to initial separations) of grain boundaries. The variation of beam behavior with interfacial grain boundary properties is relatively smaller when the damage initiation ratio

δ_0/δ_f is between 0.5 and 1.0. The further reduction of the damage ratio δ_0/δ_f (e.g., between 0.25 and 0.5) suggests the increased total fracture energy of grain boundaries and slow degradation of alumina properties, thus resulting in the increased load at failure of the microcantilever beam (Figures 5.15). Considering the brittle failure nature of alumina, the damage initiation ratio δ_0/δ_f was selected to be between 0.5 and 1.0 in the present study, as suggested in the literature [184].

5.5 Summary

In this chapter, micro-cantilever beams with equilateral triangular cross-section were fabricated by focused ion beam cutting technology. An external load exerted on the free end of the micro-cantilever beam was applied in the nano-indentation tester. The experimental results show an intergranular fracture at the peak load of 1.18 mN.

Based on the experimental observation, the representative 3D finite element model of polycrystalline structure with hardcore Voronoi tessellation was developed. The mesh strategy of the generated geometry is to use *Gmsh* software package. To simulate the intergranular failure of material, the cohesive layer in ABAQUS involving Traction-Separation law was introduced. The validation of the model has been checked via prediction of Young's modulus when the anisotropic material was randomly assigned grain orientation. Parametric study shows a small deviation, revealing that the given representative elementary volume can be used to simulate the behavior of ceramic material at the microscopic level.

Finally, the real-size micro-cantilever beams with different groups of grain orientation were created to model the localized phenomena such as the crack initiation and propagation in the microstructure. By comparison of the predicted and experimental load-displacement curves, the apparent Young's modulus in simulations shows an acceptable agreement with experimental measurement. The crack initiates at the bottom due to stress concentration at the vicinity of the clamped end of micro-cantilever beam, and propagates along grain boundary. The formation and propagation of cracks are dominated by tensile opening or shear sliding. The failure modes of alumina ceramics in grain boundaries are dictated by the localized stress state which is affected by the external loading condition and the grain morphology and orientation.

Chapter 6

Penetration behavior of a ceramic material using a modified quasi-static spherical cavity expansion model

6.1 Introduction

To estimate the penetration resistance of a metal target in DOP test, cavity expansion theory has been widely employed since 1960s [160]. With the increase of the implementation of ceramic material in aerospace engineering, the investigation of penetration resistance of ceramic material attract great attention. Recent

experimental researches [124, 157] on the ceramic target have shown that the measured penetration resistance is about 2.5 times of its compressive strength [13, 41]. The purpose of this chapter is to derive the target resistance of ceramic materials and to identify important material parameters that influence the penetration resistance based on the quasi-static spherical cavity expansion theory taken into account of damage evolution.

6.2 A modified quasi-static spherical cavity expansion model

In a steady state penetration process, the stress at the target - penetrator interface leading to erosion and deceleration of penetrator can be divided into two parts: 1) the inertial stress required to accelerate target material out of the path of the penetrator; and 2) the stress required to deform the target around the cavity denoting as target resistance. Research has found that the resistance of materials whose strength depends on pressure, may be dependent on velocity due to the higher pressure in high velocity penetration. However, the coupling of target resistance and penetration velocity is neglected in the derivation.

Figure 6.1 demonstrates a high speed projectile penetration process through an infinite ceramic target. The initial velocity and residual velocity of the projectile are labeled as v_p and v_r , respectively. The penetration velocity (u) is defined as the projectile-target interfacial velocity at which the target is fractured and the projectile starts to penetrate into the target. For an infinite target, the effect

of the thickness of the target on the penetration velocity has been overlooked. The penetration velocity of the projectile is considered to be the residual velocity. Therefore, the given model can be used to describe whether the projectile penetrates through the target or not.

FIGURE 6.1: Sequence of a projectile impacting on an infinite ceramic target

Considering the spherical symmetry of the stress field at the target-penetrator interface, the response regions can be depicted as shown in the Figure 6.2. They are cavity zone, comminuted zone, cracked zone, elastic zone and undisturbed zone from center to periphery.

FIGURE 6.2: Response regions in the ceramic target

6.2.1 Elastic region ($c < r < b$)

The elastic zone ranges from $r = c$ to $r = b$, where b is the undisturbed region (stress-free zone). At the boundary of the cracked and elastic region, the hoop stress is equal to the tensile strength of material. In the derivation, the inertial effect has been overlooked.

Take tension as positive direction. The equilibrium equation can be described in the elastic region as below:

$$\frac{d\sigma_r}{dr} + 2\frac{\sigma_r - \sigma_\theta}{r} = 0 \quad (6.1)$$

The strain-displacement relation is given as:

$$\varepsilon_r = \frac{du_r}{dr}, \quad \text{and} \quad \varepsilon_\theta = \frac{u_r}{r} \quad (6.2)$$

The constitutive equations are in the form of

$$\varepsilon_r = \frac{1}{E} (\sigma_r - 2\nu\sigma_\theta) \quad (6.3)$$

$$\varepsilon_\theta = \frac{1}{E} [(1 - \nu)\sigma_\theta - \nu\sigma_r] \quad (6.4)$$

where ν is the Poisson's ratio, E is the initial elastic modulus, ε_r and ε_θ are the radial and hoop strains, respectively. u_r is the radial displacement.

From Eqs.(6.2), (6.3) and (6.4), the radial and hoop stresses can be expressed as follows:

$$\sigma_r = \frac{E}{(1+\nu)(1-2\nu)} \left[2\nu \frac{u_r}{r} + (1-\nu) \frac{du_r}{dr} \right] \quad (6.5)$$

$$\sigma_\theta = \frac{E}{(1+\nu)(1-2\nu)} \left(\frac{u_r}{r} + \nu \frac{du_r}{dr} \right) \quad (6.6)$$

Substituting Eqs.(6.5) and (6.6) into Eq.(6.1) yields

$$\frac{d^2 u_r}{dr^2} + \frac{2}{r} \frac{du_r}{dr} - 2 \frac{u_r}{r^2} = 0 \quad (6.7)$$

As a result, the general solution of Eq.(6.7) is

$$u_r = c_1 r + \frac{c_2}{r^2} \quad (6.8)$$

where c_1 and c_2 are constants that can be determined via boundary conditions at $r = b$ and $r = c$, given by [142]:

$$\sigma_r|_{r=b} = 0 \quad (6.9)$$

$$\sigma_\theta|_{r=c} = \sigma_f \quad (6.10)$$

By substituting Eq.(6.8) back into Eqs.(6.5) and (6.6) coupled with Eqs.(6.9) and (6.10), the displacement and stress in this zone can be determined as:

$$u_r = \left(\frac{\sigma_f}{2E} \right) \left[2(1-2\nu)r + (1+\nu) \frac{b^3}{r^2} \right] / \left(1 + \frac{b^3}{2c^3} \right) \quad (6.11)$$

$$\sigma_r = \sigma_f \left(1 - \frac{b^3}{r^3} \right) / \left(1 + \frac{b^3}{2c^3} \right) \quad (6.12)$$

$$\sigma_\theta = \sigma_f \left(1 + \frac{b^3}{2r^3} \right) / \left(1 + \frac{b^3}{2c^3} \right) \quad (6.13)$$

Specially, hydro static pressure and shear stress in elastic region can be expressed as:

$$P = \frac{1}{3} (\sigma_r + 2\sigma_\theta) = \frac{\sigma_f}{1 + b^3/2c^3} \quad (6.14)$$

$$\tau = \frac{1}{2} |\sigma_r - \sigma_\theta| = \frac{3\sigma_f b^3}{4r^3(1 + b^3/2c^3)} \quad (6.15)$$

6.2.2 Cracked region ($a < r < c$)

As known, the crack appears when the hoop stress reaches the tensile strength. Therefore, the cracked region will be next to the elastic region inwardly. In such region, the hoop stress is assumed to be zero since the radial cracks are considered to distribute spatially in different sizes. Therefore, the stress state is uniaxial in the cracked region. The boundary condition can be described at the cracked-comminuted interface as below with the consideration of occurring of pulverization of material, when the radial stress exceeds the uniaxial compressive strength.

$$\sigma_r|_{r=a} = -Y \quad (6.16)$$

Since $\sigma_\theta = 0$, Eq.(6.1) can be simplified as:

$$\frac{d\sigma_r}{dr} + 2\frac{\sigma_r}{r} = 0 \quad (6.17)$$

Since the general form of Eq.(6.17) is

$$\sigma_r = -\frac{c_3}{r^2} \quad (6.18)$$

By substituted Eq.(6.16) into Eq.(6.18), it yields

$$\sigma_r = -\frac{Y a^2}{r^2} \quad (6.19)$$

As required by the stress continuity at elastic-cracked boundary ($r = c$), we have

$$\left(\frac{a}{c}\right)^2 = -\frac{\sigma_f}{Y} \left(1 - \frac{b^3}{2c^3}\right) / \left(1 + \frac{b^3}{2c^3}\right) \quad (6.20)$$

FIGURE 6.3: Recovered sample from plate impact test [23]

From the recovered sample (see Figure 6.3), it can be seen that radial cracks are the major part in the cracked region. However, the hoop cracks can be observed in the cracked region next to the comminuted zone. The nearer the distance next

to comminuted region, the more serious the damage is. Both the radial and hoop cracks render the material anisotropic. Therefore, the nonlinear material response is better to involve characterizing the displacement in this region.

Christophe Denoual [34] proposed a form of the evolution of the damage parameter as below:

$$D = 1 - \exp \left[-\frac{(\bar{\sigma})^{m+n}}{\binom{m+n}{m}} \right] \quad (6.21)$$

where m and n are material constants, and $\bar{\sigma}$ is the normalized stress. Following this form, the damage parameter is defined by the equation below, in which the damage decreases away from the center.

$$D = 1 - \exp \left[k \left(1 - \frac{c}{r} \right) \right] \quad (6.22)$$

where k is the damage evolution parameter. The given form (Eq.(6.22)) satisfied

$$\begin{cases} D = 0 & r = c \\ 0 < D < 1 & r = a \end{cases} \quad (6.23)$$

Hence, the stress-strain relation can be expressed as:

$$\sigma_r = E_D \frac{du_r}{dr} = E(1 - D) \frac{du_r}{dr} = E \cdot \exp \left[k \left(1 - \frac{c}{r} \right) \right] \frac{du_r}{dr} \quad (6.24)$$

Therefore, the displacement u_r can be expressed by taking Eq.(6.19) into Eq.(6.24) as:

$$u_r = \frac{Y a^2}{E c k} \exp \left[-k \left(1 - \frac{c}{r} \right) \right] + C_{unknown} \quad (6.25)$$

Due to the continuity of displacement at elastic-cracked interface, $C_{unknown}$ is determined by Eq.(6.11) and Eq.(6.25) as:

$$C_{unknown} = \frac{\sigma_f}{2E} \left[2(1 - 2\nu)c + (1 + \nu)\frac{b^3}{c^2} \right] / \left(1 + \frac{b^3}{2c^3} \right) - \frac{Y a^2}{E c k} \quad (6.26)$$

Therefore, the displacement u_r can be determined as:

$$u_r = \frac{\sigma_f}{2E} \left[2(1 - 2\nu)c + (1 + \nu)\frac{b^3}{c^2} \right] / \left(1 + \frac{b^3}{2c^3} \right) + \frac{Y a^2}{E c k} \left\{ \exp \left[-k \left(1 - \frac{c}{r} \right) \right] - 1 \right\} \quad (6.27)$$

Consequently, the pressure and shear stress in this region can be written as:

$$P = \frac{Y a^2}{3r^2} \quad (6.28)$$

$$\tau = \frac{Y a^2}{2r^2} \quad (6.29)$$

6.2.3 Comminuted region ($h < r < a$)

Since the brittle material will crack and pulverize subjected high pressure, Johnson-Holmquist constitutive behavior is widely used to describe its failure process, which means both intact and fragmentized materials followed different failure routine as illustrated in Figure 6.4. The upper curve is to describe the pressure-dependent

FIGURE 6.4: Qualitative constitutive description of brittle material [141]

shear strength of the intact material, whereas the lower curve is to describe the behavior of failed material. The comminuted material can't withstand any shear stress without confinements. The lower curve can be expressed as [141]:

$$\tau = \begin{cases} \lambda_l P & P < \tau_l / \lambda_l \quad (\text{unsaturated}) \\ \tau_l & P \geq \tau_l / \lambda_l \quad (\text{saturated}) \end{cases} \quad (6.30)$$

where P denotes the hydrostatic pressure; τ represents the maximum shear stress; λ_l is the pressure-shear slope (segment OF), and τ_l is the saturation level of the lower curve (segment FG).

Since $\tau_l = \lambda_l P$, (namely $\frac{\sigma_r - \sigma_\theta}{2} = \lambda_l \frac{\sigma_r + 2\sigma_\theta}{3}$), the relationship between radial and hoop stresses can be expressed as:

$$\sigma_\theta = \frac{3 - 2\lambda_l}{3 + 4\lambda_l} \sigma_r \quad (6.31)$$

Therefore, the equilibrium Eq.(6.1) can be simplified into

$$\frac{d\sigma_r}{dr} + 2\alpha \frac{\sigma_r}{r} = 0 \quad (6.32)$$

where $\alpha = \frac{6\lambda_l}{3+4\lambda_l}$

Applying the boundary condition Eq.(6.16), we have

$$\sigma_r = -Y \left(\frac{a}{r} \right)^{2\alpha} \quad (6.33)$$

The target resistance R_t , the negative of the radial stress at the cavity boundary (at $r = h$), is thus given as:

$$R_r = Y \left(\frac{a}{r} \right)^{2\alpha} \quad (6.34)$$

6.2.4 Derivation of h/a

From the mass conservation equation:

$$\frac{d}{dr} (r - u_r)^3 = 3r^2 \frac{\rho}{\rho_0} \quad (6.35)$$

Assuming that the volume is constant in the comminuted region, e.g. $\rho/\rho_0 = 1$, and that the higher order terms in $u(a)$ are neglected,

$$\frac{1}{3} \left(\frac{h}{a} \right)^3 = \frac{u_r(a)}{a} \quad (6.36)$$

Due to the continuity of displacement at $r = a$, taking Eq.(6.27) into Eq.(6.36) yields

$$\frac{1}{3} \left(\frac{h}{a} \right)^3 = \frac{\sigma_f}{2Ea} \left[2(1 - 2\nu)c + (1 + \nu) \frac{b^3}{c^2} \right] / \left(1 + \frac{b^3}{2c^3} \right) + \frac{Ya}{Eck} \left\{ \exp \left[k \left(\frac{c}{a} - 1 \right) \right] - 1 \right\} \quad (6.37)$$

There are two unknowns in Eq.(6.37), a and c . For a given geometry in the plate impact test, h and b are known, Eq.(6.20) and Eq.(6.37) can be used to determine a . Thus the penetration resistance R_t can be calculated.

6.2.5 Infinite target

For an infinite target, $b \rightarrow \infty$, substituting Eq.(6.20) into Eq.(6.37) yields

$$\left(\frac{a}{c} \right)^2 = \frac{2\sigma_f}{Y} \quad (6.38)$$

$$\frac{a}{h} = \left\{ \frac{E/(3Y)}{(1 + \nu)\sqrt{\sigma_f/(2Y)} + 1/k \cdot \sqrt{2\sigma_f/Y} \left\{ \exp \left[k \left(\sqrt{\frac{Y}{2\sigma_f}} - 1 \right) \right] - 1 \right\}} \right\}^{\frac{1}{3}} \quad (6.39)$$

and

$$R_t = Y \left\{ \frac{E/(3Y)}{(1 + \nu)\sqrt{\sigma_f/(2Y)} + 1/k \cdot \sqrt{2\sigma_f/Y} \left\{ \exp \left[k \left(\sqrt{\frac{Y}{2\sigma_f}} - 1 \right) \right] - 1 \right\}} \right\}^{\frac{2\alpha}{3}} \quad (6.40)$$

From Eq.(6.40), it can be seen that the penetration resistance is a function of material properties and damage evolution parameter k , which is different from the expression without consideration of the degradation of material property during

penetration process. If the damage evolution is not implemented, the penetration resistance only depends on the material properties [142].

6.2.6 Stationary incompressible hydrodynamic theory

The penetration of a projectile at high velocity into a target [157] may be described by the Bernoulli equation, given by:

$$\frac{1}{2}\rho_P(v_P - u)^2 + Y_P = \frac{1}{2}\rho_t u^2 + R_t \quad (6.41)$$

where density is denoted by ρ , the subscripts P and t represent projectile and target, respectively; v_p and u are the projectile velocity and penetration velocity; Y_P is the dynamic strength of the penetrator and R_t is the target resistance.

Therefore,

$$u = \frac{v_P}{1 - \rho_t/\rho_P} \left(1 - \sqrt{\rho_t/\rho_P + (1 - \rho_t/\rho_P) \frac{2(R_t - Y_P)}{\rho_P v_P^2}} \right) \quad (6.42)$$

Consequently, the penetration velocity can be determined via Eq.(6.42) when the material properties of both target and projectile are given.

6.3 Validation of theoretical model

As known, the process of penetrating the thick armor material by long rod projectiles is considered to be steady, during which a nearly constant penetration

TABLE 6.1: Material properties of projectile and target

	Target	Projectile
Density (g/cm^3)	3.89	19.3
Compressive strength (GPa)	3.0	2.0
Tensile strength (MPa)	462	-

velocity can be observed. The determination of penetration velocity u as a function of impact velocity v_i for material materials (e.g., AlN and Steel) had been experimentally measured for the past decades. Therefore, in order to validate the theoretical model, comparison has been made between the published experimental data (from Subramanian et al [157]) and the calculated results.

The penetration experiment was conducted on a 38 *mm* launch tube two-stage light-gas gun system [157]. The materials of projectile and target are tungsten and alumina, respectively. The diameter of the target is about 30 times than that of the projectile. The impact velocities of projectile are ranging from 1.5 *km/s* to 3.5 *km/s*. The radiographs were obtained during the experiment to derive the distance-time data, thus the penetration velocity. The material properties of both projectile and ceramic target are listed in Table 6.1.

As a model validation, the penetration velocities of a long tungsten projectile penetrating into an infinite ceramic target at different impact velocities have been calculated (see Figure 6.5), and then compare with the results of Subramanian et al [157]. The experimental data show a little lower than the calculated result due to the involvement of a thin aluminum cover plate in Subramanian's experiment.

The presence of the thin aluminum cover plate may slightly decrease the impact velocity, thus leading to a drop of the penetration velocity. The relative error (see Figure 6.6) between experimental data and calculated values demonstrate a trend that the higher the impact velocity is, the smaller relative error is. However, these slight differences are acceptable. As a result, the implementation of damage evolution of target material subjected to high velocity penetration by a long rod projectile is feasible.

FIGURE 6.5: Comparison between theoretical model and experimental data

The pressure-shear coefficient was first determined via experiment by Curran et al [28]. The graph of penetration resistance R_t against the pressure-shear coefficient λ_l can be seen in Figure 6.7 with the assumption that the damage evolution parameter k keeps a constant value of 1.5. Figure 6.7 also demonstrates that the increase of the compressive strength of target material results in the improvement of the penetration resistance. Based on the experimental data, the penetration resistance ranges from 7 *GPa* to 9 *GPa* [157]. Meanwhile, the measured value

FIGURE 6.6: Relative error between theoretical model and experimental data

of normal stress-shear stress coefficient μ for *AD995* alumina is 0.2 [22]. The relationship between μ and λ_l can be expressed as below:

$$\lambda_l = \frac{3\mu}{3 - 4\mu} = 0.273 \quad (6.43)$$

To let the penetration resistance fall in the range of 7 *GPa* to 9 *GPa*, the selected normal stress-shear stress coefficient $\mu = 0.23$ for the *AD995* alumina material.

Following the experimental data provided, the measured penetration resistance dropped from 9 *GPa* to 7 *GPa* with the increase of the impact velocity from 1.5 km/s to 3.5 km/s [157]. As the implementation of the damage evolution function, degradation of material properties will take place, thus the penetration resistance reduces. Figure 6.8 shows that the penetration resistance decreases with damage evolution parameter k increase. According to Eq.(6.22), the higher damage evolution parameter, the more severe degradation of material property

FIGURE 6.7: Variation of penetration resistance with pressure-shear coefficient for alumina

is. Meanwhile, the range of damage evolution parameter k should be falling in between 0.5 and 4 for the given form (see Eq.(6.22)) based on the experimental data. Thus, further proof that the evaluation of the penetration velocity with a damage evolution parameter $k = 1.5$ is reasonable.

FIGURE 6.8: Variation of penetration resistance with damage evolution parameter, k

6.4 Summary

Based on the traditional quasi-static spherical cavity expansion theory, a damage evolution function is proposed to describe the degradation of material property of ceramics at the radial direction in the crack region. The cavity expansion pressures have been derived at different regions. From the comparison between calculated values and experimental data, some remarkable conclusions can be drawn:

First, it is necessary to take into account the damage evolution in the crack region since the calculated values are close to the experimental results during a penetration process of a ceramic target subjected to a long rod projectile with high impact velocity.

Second, the penetration resistance is not only dependent on the material properties, but also on how the damage evolves. The implementation of damage evolution parameter can help to estimate the penetration resistance. The fast crack propagation in the crack zone results in the decrease of the effective Young's modulus, thus leading to a drop of penetration resistance.

Finally, the model indicates that the property of fractured material (degradation of Young's modulus) has significant influence on penetration resistance (refer to Figure 6.8).

Chapter 7

Conclusions and recommendations

In this chapter, the main conclusions are drawn first, and then several recommendations are given for the future research work.

7.1 Conclusions for the present work

7.1.1 Material characterization

Due to their high failure strength and low failure strain, accurate characterization of dynamic behavior of ceramics requires the reliable split-Hopkinson pressure bar (SHPB) technique and subsequent data analysis of recorded wave signals. In this study, an improved data analysis method is proposed for the SHPB with a modified specimen setup to calculate stress-strain curves of alumina. The wave dispersion

is corrected based on the Pochhammer-Chree solution with an FFT algorithm in the frequency domain. A direct analytical solution of wave propagation in the specimens is derived to predict the wave shapes and thereby align the wave signals to the bar-specimen interfaces. It was found that the analysis method is able to reliably reconstruct strain wave histories at the bar-specimen interfaces. It reveals that the stress equilibrium is established after ~ 5 wave propagation cycles within specimens, thus supporting the assumption of specimen stress field uniformity. The calculated specimen strain agrees well with that directly measured using strain gauges mounted on the specimen, thus validating the proposed analysis method. The 3D full-scale FE analysis can help to support the assumption of uniaxial homogeneous deformation in the specimen, because the predicted stress field in the specimen becomes more and more uniform after ~ 5 wave propagation cycles within the specimen. Thus it proves the reliability of the improved data analysis method. Using this analysis of the wave signals, the SHPB technique allows stress-strain curves of ceramics at dynamic strain rates of $\sim 2000 \text{ s}^{-1}$ to be accurately quantified.

7.1.2 Fracture and failure mechanism in alumina

The real-time observation and microscopic post-examination were introduced to investigate the underlying deformation and fracture mechanisms of ceramic materials. The studied strain rates range from 10^{-5} to $\sim 2000 \text{ s}^{-1}$. The failure strength of Al_2O_3 under impact loading demonstrates rate sensitivity above the critical strain rate $\sim 250 \text{ s}^{-1}$. The measured stress intensity exponent is 0.327 which is

quite close to the theoretical value of $1/3$. Post-observation under SEM reveals that the failure modes are dependent on the strain rates as well. The predominant intergranular fracture can be observed from quasi-static test, while a combination of intergranular and transgranular fractures can be seen on the fractured surfaces in the dynamic test. All these observations reveal that the development of a physical realistic constitutive model of ceramic materials should take into account the deformation and failure mechanisms over a wide range of strain rates.

7.1.3 Crack initiation and propagation

A microscopic experiment was carried out on the micro-cantilever beam to investigate the crack initiation and propagation. Based on the experimental results, a method to generate the 3D finite element model of polycrystalline structure was proposed. The model was validated by predicting the apparent Young's modulus when the material is anisotropic. The simulated results shows an acceptable agreement with the experiments. From the simulated results, the crack initiates at the bottom of the cantilever beam at the vicinity of the clamped end, and propagates along grain boundary. Tensile opening or shear sliding dominates the cracks formation and propagation. The failure modes of alumina ceramics in grain boundaries are dictated by the localized stress state which is affected by the external loading condition and the grain morphology and orientation.

7.1.4 Penetration behavior of ceramics

A constitutive model based on the quasi-static spherical cavity expansion theory, together with the consideration of damage evolution in the crack zone was developed. An analytical solution for the penetration resistance was obtained for an infinite target. The penetration resistance was not only a function of material properties, but also of a damage evolution parameter. The penetration resistance decreases with damage evolution parameter increase. The calculated results from the model come to an acceptable agreement with the experimental data provided in the literature [157].

7.2 Recommendations for the future work

The present research work has provided a better understanding of mechanical performances of ceramic materials. Several future research directions are suggested.

For layered composites, the investigation of its component material is important. Since metal material is widely studied, the exploration of property of soft material, as an adhesive layer present between the ceramic front plate and the metal backing plate, is necessary. Challenge does exist to characterize the low stress and low impedance material at the dynamic strain rates.

As known, ceramic materials are strain rate sensitive materials. Though the damage evolution is considered in the present model, the introduction of strain rate effect may improve the accuracy of the model. Meanwhile, as a high velocity

penetration process, the inertia effect should be considered. Furthermore, the presented model can be extended to a layered composite (e.g., bi-layer composite including a ceramic front plate and a metallic backing plate). Since the quasi-static spherical cavity expansion theory has been successfully applied into metal material, the introduction of the continuous conditions (e.g., displacement and forces) at the ceramic-metal interface can integrate constitutive equations of ceramic and metal. Thus the penetration behavior of layer composite can be determined.

Since the study of anti-ballistic performance of layered armor composite is essential, both experimental and simulate methodologies will be used. The experimental tests (e.g., depth of penetration (DOP) test) will be conducted on both layered composite and its component materials (e.g., ceramic, metal and adhesive) to obtain the extra high strain rate data to support numerical simulations. The numerical simulation can help to optimize the experimental design. Moreover, it provides an evidence for the application of layered structure.

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Publication list

Journal papers

1. **Z. Wang** and P. Li. Dynamic failure and fracture mechanism in alumina ceramics: Experimental observations and finite element modelling. *Ceramics International*. 2015. 40(10): 12763-12772
2. P. Li, **Z. Wang**, N. Petrinic and C.R. Siviour. Deformation behavior of stainless steel microlattice structures by selective laser melting. *Material Science and Engineering A*. 2014: 116-121.
3. **Z. Wang** and P. Li. Characterisation of dynamic behaviour of alumina ceramics: evaluation of stress uniformity. Under review.
4. P. Li and **Z. Wang**. A modified constitutive model incorporating strain rate effect for the compressive behaviour of adhesives. Under review.
5. R. Huang, P. Li, **Z. Wang** and T. Liu, X-ray microtomographic characterisation of strain rate dependent failure mechanism in cenosphere epoxy syntactic foams. Under review.

6. **Z. Wang** and P. Li. Intergranular fracture in polycrystalline alumina ceramics: micromechanical experimental and Voronoi cell finite element modelling. In preparation.

7. **Z. Wang** and P. Li. The penetration behavior of a ceramic target subjected to the long projectile impact. In preparation.

Conference paper

8. P. Li and **Z. Wang**, Characterising dynamic behaviour of ceramics using split-Hopkinson pressure bar techniques. The 4th International Conference on Impact of Lightweight Structures, South Africa, 2014.