Dynamic fracture of C/SiC composites under high strain-rate loading: microstructures and mechanisms

T. Li^{a,b}, D. Fan^b, L. Lu^{b,c}, J.Y. Huang^{b,c}, J.C. Eb, F. Zhao^b, M.L. Qi^d, T. Sun^e, K. Fezzaa^e, X.H. Xiao^e, X.M. Zhou^b, T. Suo^a, W. Chen^f, Y.L. Li^{a,*}, M.H. Zhu^{g,*}, S.N. Luo^{b,*}

^aFundamental Science on Aircraft Structural Mechanics and Strength Laboratory, Northwestern Polytechnical University, Xian 710072, Shaanxi, P. R. China

^bThe Peac Institute of Multiscale Sciences, Chengdu, Sichuan 610207, P. R. China

^cCAS Key Laboratory of Materials Behavior and Design, Department of Modern Mechanics, University of Science and Technology of China, Hefei, Anhui 230027, P. R. China

^dSchool of Science, Wuhan University of Technology, Wuhan, Hubei 430070, P. R. China

^eAdvanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439, USA

^fSchool of Aeronautics and Astronautics, and School of Material Science Engineering, Purdue University, West Lafayette, Indiana 47907, USA

^gKey Laboratory of Advanced Technologies of Materials, Ministry of Education, Southwest Jiaotong University, Chengdu, Sichuan 610031, P. R. China

Abstract

We investigate dynamic fracture of C/SiC composites under high strain-rate compression or tension with split Hopkinson pressure bar (SHPB) and gas gun loading. Components of the as-fabricated composites are mapped and quantified with X-ray computed tomography, including C fibers and fiber bundles, SiC matrix, and inter- and intrabundle voids. Compression loading is applied along the out-of- and in-plane directions by SPHB at strain rates of $10^2 - 10^3 \text{ s}^{-1}$ along with *in situ* X-ray phase contrast imaging. Out-of-plane direction compression and tension are examined with gas gun impact at strain rates $10^4 - 10^5 \text{ s}^{-1}$. For the out-of-plane loading, compression induces fracture via void collapse and shear damage banding, while delamination dominates fracture for the in-plane direction compression. With increasing strain rates, the compression failure modes transit from interbundle to intrabundle fracture of SiC, and then to fiber and bundle breaking. Tensile failure involves delamina-

*Corresponding author

Email addresses: liyulong@nwpu.edu.cn (Y.L. Li), zhuminhao@swjtu.cn (M.H. Zhu), sluo@pims.ac.cn (S.N. Luo)

tion, fiber pullout and fiber breaking. In contrary to normal solids, dynamic tensile or spall strength decreases with increasing impact velocities, owing to compression-induced predamage before subsequent tensile loading.

1. Introduction

Carbon fiber-reinforced SiC-matrix composites, or C/SiC composites, have low density, **high specific stiffness and specific strengths**, and high failure stress at high temperatures. Such advantages render C/SiC composites a key structure component for thermal protection in aeronautical and astronautical vehicles [1–3]. These vehicles are inevitably subject to impact loading during service. Therefore, it is important to investigate their response to high-strain rate loading, in particular, dynamic fracture upon compression and tension, as well as the underlying mechanisms and microstructural effects.

During fabrication of a C/SiC composite, a large number of voids/pores are introduced during chemical vapor infiltration (CVI) [4, 5] of SiC into C fiber bundles and fibers, to form the SiC matrix. Microstructures of different C/SiC composites have been characterized extensively with scanning electron microscope (SEM) [6–8]. For instance, Fan *et al.* [8] quantified the microstructures of two-dimensional (2D) and 3D C/SiC composites, respectively, and analyzed the effects of intrinsic voids, interfaces, and residual thermal stresses on their properties. However, SEM is a 2D technique and 3D SEM characterization has to involve serial sectioning and polishing. Compared to SEM, X-ray computed tomography (XCT) is a nondestructive 3D technique capable of resolving internal structure with micron resolutions, and does not require tedious sample preparation. With XCT, void shape and size distributions, and interface structures can be revealed [9–13]. The synchrotron micro CT has been utilized to characterize the epoxy composites [10, 12, 13], 3D C/C [9] and 3D C/SiC [11]. Related to macroscopic mechanical properties of porous ceramic composites such as 2D C/SiC. However, XCT characterization of C/SiC composites with varying microstructures is rare, despite their engineering and scientific significances.

Extensive investigations have been devoted to the fracture strength, toughness, and corresponding fracture mechanisms of C/SiC composites under quasi-static loading conditions [14–19]. Nevertheless, the mechanical properties of C/SiC composites under high strain-rate loading have not been adequately studied. Chen and Li [20] conducted tensile tests on C/SiC composites using the split Hopkinson tension bar for loading and high-speed photography for capturing surface fracturing, and proposed dynamic fracture mechanisms and a phenomenological constitutive relation with a damage model. **Studies on mechanical properties and failure mechanisms of C/SiC composites under higher strain rates ($>10^4 \text{ s}^{-1}$), as in gas-gun impact loading, are extremely rare. While SHPB loading has been widely used in dynamic experiments at strain rates of $10^2 - 10^4 \text{ s}^{-1}$, the preexisting microstructures, and their response to dynamic loading and roles in dynamic fracture of C/SiC composites have not been investigated in detail. Therefore,** it is desirable to reveal internal microstructures dynamically with penetrating techniques such as X-ray phase contrast imaging (XPCI) [21, 22]. Such measurements under dynamic compression or tension are valuable for characterizing deformation and damage dynamics and establishing constitutive models, but have not been applied to composites yet.

Gas gun flyer-plate impact loading is a standard, well established, method for very high strain-rate loading ($\dot{\epsilon} \sim 10^4 - 10^7 \text{ s}^{-1}$) [23], and has been widely used to study mechanical properties of various materials [24–31], including the orientation effects on shock response of a carbon fiber-epoxy composite [27], and punch damage of a woven carbon fiber reinforced-epoxy composite with carbon nanotube additions [30]. Recently, spallation of carbon nanotube/epoxy composites under various impact velocities was investigated, and dynamic fracture toughnesses were obtained at high strain rates ($\sim 10^5 \text{ s}^{-1}$). Cooper [32] and Paris [33] studied the spall strength and fracture energy of SiC ceramic, and proposed damage modeling below the HEL. C/SiC composites, commonly used in the outside-wall structures, may undergo shock loading at impact velocities on the order of 100 m/s, but their dynamic response under such conditions has not been investigated.

In the present study, we use XCT, SEM, dynamic XPCI, and optical velocimetry to characterize the microstructure and dynamic response of C/SiC composites under split Hopkinson pressure bar (SHPB) and gas gun loading. 3D microstructure of the as-fabricated C/SiC composites is constructed from XCT, and the voids and interfaces are analyzed. Dynamic compression experiments at $10^2 - 10^3 \text{ s}^{-1}$ are conducted with SHPB, along with XPCI to resolve the microstructure evolution *in situ* and in real time. Higher strain-rate compression and tension experiments ($10^4 - 10^5 \text{ s}^{-1}$) are carried out on a single-stage gas gun, and the free-surface velocity histories are measured to investigate void collapse and spallation. The samples recovered from SHPB and gas gun loading are examined with SEM. Dynamic fracture mechanisms under compression and tension are discussed.

2. Materials and characterization

The 2D C/SiC composites are fabricated by the State Key Laboratory of Solidification Processing at Northwestern Polytechnical University, People's Republic of China. The T300 carbon fiber bundles with a tow size of 1k are provided by Toray Industries, Inc. In order to increase the toughness of C/SiC composites, pyrolytic carbon, formed with the reactant gas of propene and the diluent of argon at $\sim 1200 \text{ K}$, is deposited on the carbon fibers to create 100–200 nm thick interfacial layers. Carbon fiber bundles are interlaced orthogonally into a laminate, and the average thickness of the woven fabric is 0.16 mm. C/SiC composites are prepared via CVI of SiC into stacked 2D carbon fiber laminates at 1273 K, with the reactant gas of CH_3SiCl_3 . The resulting SiC coating on carbon fibers is 30–50 μm thick, and forms the SiC matrix with embedded C fiber laminates. At ambient conditions, the density of the C/SiC composite is $\rho_0 = 2.05 \text{ g cm}^{-3}$, and the sound speed is $C_0 = 2165 \text{ m/s}$.

SEM micrographs in Fig. 1 show laminates of orthogonally woven carbon fiber bundles (*A* and *B*), and SiC coating between fiber bundles and on fibers within a single bundle serving as a bonding matrix, as well as voids enclosed by

Figure 1: SEM micrographs of as-fabricated C/SiC composites. (a) Microstructure as viewed along the in-plane direction: orthogonally woven C fiber bundles (*A* and *B*, gray), SiC (white), and voids (black, *C*). (b) SiC coating containing cracks (*E*), viewed along the out-of-plane direction, i.e., the arrow denoted by *D* in (a).

neighboring bundles (*C*; interbundle voids) which SiC fails to infiltrate. Carbon fibers, coated by SiC, are shown in Fig. 1(b). Cracks (*E*) form within the SiC matrix perpendicular to the fiber length direction, owing to residual thermal stress incurred by the mismatch in thermal expansion coefficients between C fibers and SiC coating. The thermal expansion coefficient of C fibers ranges from $-0.1 \times 10^{-6} \text{ K}^{-1}$ to $1.1 \times 10^{-6} \text{ K}^{-1}$ along the length direction, and that of the SiC matrix synthesized via CVI is considerably larger ($4.8 \times 10^{-6} \text{ K}^{-1}$).

The 3D microstructure of C/SiC composites reconstructed with XCT conducted at the 2-BM beamline of the Advanced Photon Source is shown in Fig. 2(a)), with four structural components: C fibers, SiC matrix, and inter- and intrabundle voids. (3D XCT is performed prior to dynamic loading, independent from *in situ* 2D XPCI during SHPB loading.) The intrabundle voids are also referred to as interfiber voids. Note that interfiber voids are not identified in SEM (Fig. 1). The dense SiC coating covers the bundle surfaces and fibers, preventing the oxidization of C fibers in high temperature environments. As CVI proceeds, the SiC coating on the bundle surfaces blocks the vapor paths and leads to irregularly shaped interbundle voids (inset, Fig. 2(c)). The interbundle voids are closely related to those formed during stacking the C fiber laminates, which are only partially filled via CVI. The concentration of the reactive vapor

Figure 2: XCT characterization of as-fabricated C/SiC composites. (a) 3D microstructure of C/SiC. (b) A pie-chart of component volume fractions. The legends refer to both figures. (c) Size distribution of interbundle/interfiber voids (inset) in the as-fabricated C/SiC composites. Frequency refers to the number of voids within a $1\text{-}\mu\text{m}^3$ binning width. (d) Size (diameter d) distribution of interfiber voids (inset) in the as-fabricated C/SiC composites. Frequency (n) refers to the number of voids within a $3\text{-}\mu\text{m}$ binning width. The curve denotes an exponential fit. The dimensions of the bundle in the inset is $418 \times 439 \times 159 \mu\text{m}^3$.

on the surface of each fiber bundle is higher than that inside the bundle. The SiC deposit on the bundle surface also impedes the infiltration into the bundle interior, giving rise to interfiber voids (inset, Fig. 2(d)), which develop between neighboring fibers and naturally follow the fiber shape. The volume fractions of C fibers, SiC, and interbundle and interfiber voids are 40%, 44%, 12% and 4%, respectively. The overall porosity is about 16% (Fig. 2(b)).

Since voids are expected to play an important role in mechanical properties, we select representative volume elements (RVE) to quantify the size distributions of interbundle and interfiber voids (Figs. 2(c) and (d)). **The sensitivities are about $1.4 \mu\text{m}$ and $2 \mu\text{m}$ for distinguishing interbundle and intrabundle/interfiber voids from XCT measurements, respectively.** The 3D void configurations are shown in the insets. In the RVE for interbundle voids ($\sim 1 \text{ mm}^3$; Fig. 2(c)),

Figure 3: (a) Schematic setup of SHPB with a high-speed X-ray imaging system. 1: striker; 2: incident bar; 3: strain gauge; 4: sample; 5: piezoelectric force transducer; 6: scintillator; 7: mirror; 8: high-speed camera. (b) Schematic setup of gas gun flyer-plate impact experiments. 1: polycarbonate sabot; 2: recess for rarefaction waves; 3: flyer plate; 4: sample; 5: sample holder; 6: lens; 7: optical fiber connected to the dual laser Doppler pin system (DPS); 8: optical fibers and detectors for the optical beam block system; 9: gun barrel; 10: vacuum/target chamber; 11: mirror; 12: soft materials; 13: soft-recovery assembly.

there are 287 voids with a volume greater than $2 \mu\text{m}^3$ and the voids with a volume size greater than $1024 \mu\text{m}^3$ accounts for 13% of the total interbundle void volume. The number of the interbundle voids decreases rapidly with increasing void size. Within a $418 \times 439 \times 159 \mu\text{m}^3$ RVE largely from a single fiber bundle, the interfiber void number frequency (n) within a $3\text{-}\mu\text{m}$ bin width decreases exponentially with increasing void diameter (d): $n(d) \propto \exp(-d/d_0)$, with the characteristic size scale $d_0 = 2.9 \mu\text{m}$ (Fig. 2(d)).

3. Dynamic loading with split Hopkinson pressure bar and gas gun

SHPB and gas gun loading are applied to achieve high strain rates in different regimes, and their schematic setups are shown in Fig. 3. The working principle and data analysis of SHPB experiments were presented in Refs. [34–36], and details of synchrotron XPCI experiments with SHPB can be found in Refs. [21, 22, 37, 38]. Our SHPB experiments are conducted at the APS beamline 32-ID-B. As shown in Fig. 3(a), a striker (1) impacts an incident bar (2) and

produces a stress pulse dynamically loading a sample (4) at a strain rate of $10^2 - 10^4 \text{ s}^{-1}$. The striker and incident bar, 12.7 mm in diameter, are made of high-strength steel. The input strain pulse is measured with a strain gage (3) on the incident bar, and also serves as the trigger to a high-speed camera (8) and oscilloscopes. A transmission bar is not used due to space limitations in the experimental hutch. Instead, a force transducer (5), i.e., a Kistler low impedance fast response quartz load cell, is used to measure the force history by virtue of piezoelectricity. Incident X-ray pulses are synchronized with the loading, and transmitted through the sample to a $\text{Lu}_3\text{Al}_5\text{O}_{12}:\text{Ce}$ scintillator, converting X-rays into visible light, which is then relayed to the high-speed camera.

In SHPB experiments, the sample length is $l = 3 \text{ mm}$, and the cross-sectional dimensions are $1.5 \times 1.5 \text{ mm}^2$. **We explore two loading directions, the in-plane and out-of-plane directions of the C/SiC composites, which are parallel and perpendicular to the laminate plane, respectively.** The high-speed camera is operated at a frame rate of 45260 frames/s. The engineering strain rate ($\dot{\epsilon}$) is estimated as

$$\dot{\epsilon} = \frac{C_L}{l}(\epsilon_I - \epsilon_R). \quad (1)$$

Here $C_L = 5000 \text{ m/s}$ is the longitudinal sound speed of the bars, strain ϵ refers to that measured on the incident bar, and subscripts I and R denote incident and reflected waves, respectively.

The methodology of gas gun plate impact experiments, as well as data interpretation and representative examples, can be found in Refs. [23–31, 39, 40]. Flyer plate impact experiments are conducted with a 14-mm bore, single-stage, gas gun to investigate dynamic compression and tension responses of the C/SiC composites. In Fig. 3(b), a flyer plate (3) is attached to polycarbonate sabot (1), with a recess (2) immediately behind it. When a solenoid valve is fired, compressed N_2 or He is released from a high-pressure gas reservoir into the gun barrel (9), accelerating the sabot–flyer plate assembly. Upon exiting the muzzle, the flyer plate impacts the target or sample under consideration (4). The

flyer plate velocity is measured with an optical beam block system (8), and the free surface velocity of the target, with a dual-laser Doppler pin system or DPS (7). DPS is essentially a displacement interferometer similar to photon Doppler velocimeter. The uncertainties in velocity measurements are $\sim 1\%$. The muzzle, target and diagnostics are located in a vacuum chamber (10). The shocked samples are “soft” recovered with foams (6) for SEM examination.

Two parallel surfaces of a flyer plate or target disk are polished to micron level or mirror finish. The flyer plates are made of Al2024; **at ambient conditions, its density $\rho_0 = 2.77 \text{ g/cm}^{-3}$, and sound speed $C_0 = 6365 \text{ m/s}$.** The thicknesses of the flyer plates and targets are 3 mm. The shock loading is applied along the out-of-plane direction of the C/SiC composites.

4. Results and discussion

4.1. SHPB loading

C/SiC composites are brittle materials with a fracture strain of about 5% under quasi-static loading [41]. In SHPB tests, it is demanded that the stresses at two ends of a sample be in equilibrium. If the strain rate is excessively high, the sample fractures before the stress equilibrium is achieved. An upper limit of strain rate normally exists for a brittle material subjected to SHPB loading. However, strain rates in real applications are usually higher than this limit. Here SHPB is merely used as a loading apparatus to investigate dynamic fracture mechanisms of C/SiC composites, along with the measured force-time histories (Figs. 4(a) and (b)).

The fracture process of C/SiC composites under out-of-plane compression with SHPB is displayed in the inset of Fig. 4(a). Initially at moment t_0 , a large number of “randomly” distributed interbundle voids exist in the sample. At t_1 , big voids are compressed to smaller volumes (e.g., $A \rightarrow B$), while small ones are closed. With increasing loading (t_2), the voids are mostly collapsed and the sample is nearly fully compacted; the laminates largely remain “intact”, but local damage around the collapsed voids can be observed. At t_5 , a pronounced

Figure 4: Typical force-time history of C/SiC composite under (a) out-of-plane and (b) in-plane compression loading with SHPB at a strain rate of $\sim 10^3 \text{ s}^{-1}$. The inset in figure (a) shows X-ray images at $t_0 = 0$, $t_1 = 22 \text{ } \mu\text{s}$, $t_2 = 44 \text{ } \mu\text{s}$, and $t_5 = 66 \text{ } \mu\text{s}$. The inset in figure (b) shows X-ray images at $t_0 = 3 \text{ } \mu\text{s}$, $t_1 = 25 \text{ } \mu\text{s}$, $t_2 = 47 \text{ } \mu\text{s}$, and $t_4 = 69 \text{ } \mu\text{s}$.

damage band (C) forms approximately at a 45° angle to the loading direction, leading to sample failure.

The corresponding force-time history is shown in Fig. 4(a). From time t_0 to t_1 , the curve shows a few fluctuations in the force, and each force drop denotes random collapse of small voids or fracture. Between t_1 and t_2 (t_3), void closure is achieved “elastically”, so the loading is sustained and the force increases smoothly with time or displacement, approximately in a linear manner. At t_3 , local damage around voids and the onset of shear damage banding (the formation of a band consisting of a large number of micro shear cracks), lead to a slight drop in force. Between t_3 and t_4 , increasing damage weakens the material and reduces the effective modulus, as indicated by the slope change. Between t_4 and t_5 , the force begins to drop continuously, indicating failure. This is consistent with the pronounced shear damage band along the maximum shear stress direction at t_5 , which is approximately at 45° relative to the loading direction (Fig. 4). Void collapse and the formation of shear damage band induce fracture and failure for the out-of-plane compression.

Different from the out-of-plane loading, dynamic fracture under in-plane compression with SHPB is mainly induced by delamination (Fig. 4(b) inset).

Figure 5: Schematics of the distance–time diagram for spallation experiments, and the corresponding free-surface velocity history. T refers to the spall thickness.

Penny-shaped interbundle voids extend along the SiC bonding layers (t_0). With increasing loading, the voids are deformed, and stress concentration leads to crack opening due to the Poisson’s effect (A ; t_1). Given the voids in the bonding layers (Fig. 2) and the brittle nature of SiC, the interlayer strength is low, and cracks are prone to nucleate there (C ; t_4), and propagate along the bonding layer (B ; t_2 and t_4). Void distributions in different layers are different, and the interface strengths vary. As a result, delamination occurs at different time at different locations. Eventually, the sample disintegrates via segmented delamination.

The corresponding force–time history is shown in Fig. 4(b). Since the long axis of the cracks is parallel to the loading direction, crack nucleation and propagation do not lead to observable stress drop along the loading direction before failure, so the force–time curve is smooth. The lateral development of main cracks (B) allows cave-in along the loading direction, resulting in the irreversible load drop at t_3 , and failure ensues catastrophically.

4.2. Gas gun loading

For plate impact experiments, shock loading is applied along the out-of-plane direction of the laminates in the C/SiC composites, for exploring spallation

Figure 6: Typical free surface velocity profiles or histories for gas gun loading at different impact velocities. The time zero is shifted to the instant of impact. Inset: spall pullback.

(dynamic fracture under tension) and compression-induced compaction.

4.2.1. Spallation

Spallation experiments are conducted at impact velocities of 45–385 m/s. The corresponding distance–time diagram of wave propagation is shown in Fig. 5. The impact of a flyer plate on a sample (O) induces shock waves propagating into the sample (OA) and the flyer plate (OB), which are reflected as release fans from the respective free surfaces (BCD and AD). **The interactions of the release fans lead to tension in region D [23, 40, 42].** Spallation occurs when the tensile stress exceeds the spall strength (σ_{sp}), and compression waves are then initiated from **the spall plane located at D** and propagate both ways. The schematic free surface velocity history (u_{fs} ; Fig. 5) shows the shock arrival (A) at the sample free surface, release from the free surface of the flyer plate (E), and pullback due to the arrival of the spall-induced compression wave (F).

Figure 6 presents six representative free surface velocity histories measured with DPS. Given the considerable porosity, the $u_{fs}(t)$ curves show a pseudo yield point and pseudo plastic regime on shock rise, followed by a shock plateau and

spall pullback (inset). The tensile strain rate can be estimated with [28, 29]

$$\dot{\epsilon} \approx \frac{1}{2C_0} \left. \frac{du_{fs}(t)}{dt} \right|_{\text{release}}. \quad (2)$$

Here C_0 refers to the sound velocity at zero pressure (~ 2165 m/s). The strain rates in the present study are about $0.8\text{--}2.1 \times 10^4$ s $^{-1}$. The spall strength can be calculated as [30, 40]

$$\sigma_{sp} = \frac{1}{2} \rho_0 C_0 \Delta u_{fs}, \quad (3)$$

where ρ_0 is the initial density of the material, and Δu_{fs} is the velocity pullback, i.e., the difference in u_{fs} between E and F (Figs. 5 and 6).

Fifteen spallation experiments are conducted, and the spall strengths are summarized in Fig. 7 as a function of impact velocity. The scatter in the data is caused by microstructure variations from sample to sample. The C/SiC composites show an exponential decrease in spall strength with **increasing impact velocity**, in sharp contrast to normal solids such as **full-density** metals [42]. The spall strength of C fiber-reinforced epoxy composites is about 100 MPa [26, 27]. However, due to the existence of a large number of voids and the brittle nature of SiC matrix, the maximum spall strength of C/SiC composites is ~ 60 MPa. At the high impact velocity end (385 m/s), the spall signal disappears and the spall strength decrease to zero. Compression-induced fracture of the SiC matrix is initiated by stress concentrations around the voids, as indicated by the SHPB loading with XPCI (Fig. 4); disintegration under compression leads to the reduction in spall strength under tension. The increased compression damage with increasing shock strength gives rise to such rapid reduction in spall strength.

Two release fans encounter at point D from different paths. On the basis of the geometric relationship indicated in the distance–time diagram (Fig. 5), we obtain

$$\frac{L_f}{U_f} + \frac{L_f}{C_f} + \frac{L_s - T}{C_s} = \frac{L_s}{U_s} + \frac{T}{C_s}, \quad (4)$$

where L , T , U , and C denote sample (subscript s) or flyer plate (**subscript f**) thickness, spall thickness, **shock wave velocity**, and sound speed, respectively.

Figure 7: Spall strength of C/SiC composites as a function of impact velocity. The dashed curve is an exponential fit.

It follows that

$$T = \frac{1}{2}L_s \left(1 - \frac{C_s}{U_s}\right) + \frac{1}{2}L_f \left(\frac{C_s}{U_f} + \frac{C_s}{C_f}\right). \quad (5)$$

In our experiments, $L_f = L_s$. To the first order approximation, $C_f \approx U_f$ for the full density flyer plate, and then

$$T \approx \frac{1}{2}L_f \left[1 - \left(\frac{1}{U_s} - \frac{1}{C_f/2}\right)C_s\right]. \quad (6)$$

For a porous C/SiC sample and full-density Al2024 flyer plate, $U_s < \frac{1}{2}C_f$ (approximately, 2165 m/s vs. 6365 m/s), so T decreases as C_s increases. **The void collapse in C/SiC composites under impact leads to considerable densification, which results in an increase in C_s .** With increasing impact velocity (u_{imp}), the degree of compaction increases and thus C_s increases, giving rise to a decrease in T . Indeed, the experiments show that T decreases rapidly with increasing impact velocity (Fig. 8).

SEM micrographs of shock-recovered samples are presented in Fig. 9. At $u_{\text{imp}} = 45$ m/s, we observe incipient spallation manifested as interbundle microcracks between two laminates, e.g., *A* and *B* in Figs. 9(a) and 9(b), since the interlaminar interfaces are the weakest links. However, C fibers remain intact (Fig. 9(b)). For $u_{\text{imp}} = 88$ m/s, complete spallation occurs largely due to delamination, accompanied by pullout and breaking of a small number of fibers

Figure 8: Spall thickness versus impact velocity for gas gun loading. The inset shows images of spalled layers at corresponding impact velocities. The dashed line is a negative logarithmic fit.

(Figs. 9(c) and 9(d)). With a further increase in u_{imp} to 118 m/s, complete fracture of fiber bundles are observed at different interlaminar interfaces (F in Fig. 9(e)). A large number of fibers are pulled out and the fracture plane is rough (G in Fig. 9(f)). The interfaces between the C fibers and SiC matrix allow sliding of the fibers and their pullout gives rise to certain pseudoplasticity. At even higher u_{imp} (170 m/s), a large number of fiber bundles are fractured on the spallation plane. The fibers bundles at $I - H$ belong to different laminates, indicating a larger extent of damage at multiple layers along the shock direction. Both fibers and SiC coating are fragmented and separated. Pullout is minor due to the high loading rate, so the fracture plane is smooth (Fig. 9(h)).

4.2.2. Compression-induced fracture under gas gun loading

The compression-induced damage may lead to reduction in the spall strengths of C/SiC composites (Fig. 7). Therefore, we discuss in more detail the compression-induced deformation, as regards to shock rise in $u_{\text{fs}}(t)$ curves, i.e., the region delimited by the rectangle in Fig. 6 which is enlarged in Fig. 10(a). The shock rise is broadened, a manifestation of the averaged scattering effect of voids and interfaces. The shock rise time is much longer than that of full

Figure 9: Spall fracture: SEM micrographs of spall fracture surface of shock-recovered C/SiC composites at different impact velocities. Left panels: low magnification; right panels: high magnification. Shock loading is applied along the out-of-plane direction.

density metals (100 ns vs. 10 ns). The shock rise shows two stages separated by an inflection point (the pseudo yield point): the pseudoelastic regime I followed by the pseudoplastic regime II. The slope in regime II is steeper than in regime I, but both steepen with increasing shock strength.

The pseudoelastic regime shows a finite slope, because voids collapse and compaction are the predominant processes which depend on rate and history. For $u_{\text{imp}} < 88$ m/s, plateaus or “pseudoelastic” shocks form first, and void collapse ensues when the critical stress is exceeded, leading to a slight drop in u_{fs} due to stress relaxation. The material is partially compacted at the inflection point, and subsequently undergoes compression (the pseudoplastic regime). The collapse of voids leads to a decrease in pressure and thus particle velocity, while

Figure 10: Void collapse characteristics. (a) Free surface velocity profiles upon shock rise, corresponding to the region enclosed by dashed lines in Fig. 6. The arrows indicate the “pseudo yield points.” (b) Void collapse duration (red) and void collapse strength (blue) as a function of impact velocity. Shock loading is applied along the out-of-plane direction.

the subsequent densification leads to an increase in the particle velocity. However, at higher impact velocities (118–385 m/s), the processes of collapse and densification are accelerated and they may even occur approximately simultaneously, so the inflection point and velocity drop become less obvious. We define the void collapse time as the time between impact and the inflection point, and the maximum stress before void collapse is the void strength, $\sigma_{\text{void}} \approx \frac{1}{2}\rho_0 C_0 u_{\text{fs}}$, evaluated at the inflection point. The void collapse time and void strength are plotted in Fig. 10(b) as a function of impact velocity. With increasing impact velocity, void strength increases while the collapse time decreases. The increase in the void strength is attributed to the positive strain-rate dependence of porous materials [43–45] as well as the inertial effects under impact loading.

To better understand compression-induced fracture/damage, we conduct shock-recovery experiments on C/SiC samples. The setup is similar to the spall experiments except that a fused quartz window is added to diminish tensile loading. The impact velocity ranges from 61 m/s to 180 m/s. All the samples are successfully recovered, except the shot at 180 m/s. Sectional SEM micrographs of shock-recovered samples are shown in Fig. 11.

At $u_{\text{imp}} \leq 61$ m/s, the C fiber bundles are intact (Fig. 11(a)). The stress

Figure 11: Compression fracture: SEM micrographs of C/SiC composites recovered from gas gun loading at different impact velocities. Left panels: low magnification; right panels: high magnification. Shock loading is applied along the out-of-plane direction.

concentrations around the interbundle voids lead to fracture of interbundle SiC (*A* in Fig. 11(b)), and crack propagation is arrested at the C fibers. With increasing impact velocity (126 m/s), damage around interbundle voids, for instance, the asymmetric damage indicated by *B* in Fig. 11(c), and cracks penetrate a fiber bundle into its interior, fracturing the interfiber SiC or voids (*C*), while the C fibers undergo minimum damage (breaking of a few fibers, *D*). Impact at even higher velocities (180 m/s) leads to breaking of multiple fiber bundles (*E–G*, Fig. 11(f)), with multiple cracks developing into the bundles in multiple layers, and fragmentation of a large number of fibers inside a bundle. The fracture surfaces are rough, and C fibers may separate completely from the SiC coating (Fig. 11(g)). Therefore, compression-induced fracture increases with impact velocity, and gives rise to decreasing spall strength observed in Fig. 7.

4.3. Fracture mechanisms

As a structural component, C/SiC composites normally undergo out-of-plane compression. Our integrated experiments show that compression-induced fracture is rate-dependent. We summarize the modes of compression-induced fracture under different strain rates in Fig. 12. The compression direction is denoted with arrow *A*. For quasi-static loading ($\dot{\epsilon} \sim 10^{-3} \text{ s}^{-1}$) [41, 46, 47], a crack nucleates from a weak spot (e.g., around a void), and develops into a main crack *B* following the path of least resistance; it induces stress relaxation and suppresses nucleation of new cracks, and is meandering in shape (Fig. 12(a)). With increasing strain rate ($\dot{\epsilon} \sim 10^2 \text{ s}^{-1}$) [46, 47], local strain concentrations become more “homogeneous,” cracks nucleate at multiple localities, and their shapes are less curved (cracks *C* and *D* Fig. 12(b)). At $\dot{\epsilon} \sim 10^3 \text{ s}^{-1}$, the fracture mode transits from crack formation to void collapse (*F*, Fig. 12(c)), accompanied by crack formation (*E*). At $\dot{\epsilon} \sim 10^4 \text{ s}^{-1}$, the fracture modes include void collapse (*H*, Fig. 12(d)), cracking via fiber bundle splitting (*I*), and bundle breaking at the void edges (*J*). Note that the strain rates cited here are only crude estimates. However, for the in-plane compression loading, the fracture mode is primarily delamination. However, tensile failure involves interface crack, fiber pullout and fiber fracture, which occur successively with increasing impact velocity.

5. Conclusions

The microstructure of as-fabricated C/SiC composites are characterized with XCT and the structural components are mapped and quantified, including C fibers and bundles, SiC matrix, interbundle and interfiber voids.

Dynamic fracture under compression or tension is examined with SHPB and gas gun loading along the out-of-plane and in-plane directions, and the strain rates range from 10^3 s^{-1} to 10^5 s^{-1} . For the out-of-plane loading, compression induces fracture via void collapse and shear damage banding, while delamination dominates fracture for the in-plane direction compression. Tensile failure involves delamination, fiber pullout and fiber breaking.

Figure 12: Schematics of compression-induced fracture modes in C/SiC composites under different strain rates. (a) $\dot{\epsilon} \sim 10^{-3} \text{ s}^{-1}$. (b) $\dot{\epsilon} \sim 10^2 \text{ s}^{-1}$. (c) $\dot{\epsilon} \sim 10^3 \text{ s}^{-1}$. (d) $\dot{\epsilon} \sim 10^4 \text{ s}^{-1}$; G represents the failure wave front. The red curves denote cracks.

With increasing strain rates, the compression failure modes transit from interbundle to interfiber fracture of SiC, and then to fiber and bundle breaking. In contrary to normal solids, dynamic tensile or spall strength decreases with increasing impact velocities, owing to compression-induced predamage before subsequent tensile loading.

Acknowledgements

We thank Prof. X.W. Yin at NWPU for supplying the C/SiC composite samples. Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. This work is partially supported by the 973 Project (No. 2014CB845904), National Natural Science Foundation of China (No. 11102168, No. 11272267, No. 11372256, and No. 11472227), the 111 Project of P.R. China (No. B07050), Fundamental Research Funds for the Central Universities (No. 310201401JCQ01001), and NSAF (No. U1230202).

- [1] Lamouroux F, Bourrat X, Nasalain R, Sevely J. Structure/oxidation behav-

- ior relationship in the carbonaceous constituents of 2D-C/PyC/SiC composites. *Carbon* 1993;31(8):1273–88.
- [2] Xu YD, Zhang LT. Three-dimensional carbon/silicon carbide composites prepared by chemical vapor infiltration. *J Am Ceram Soc* 1997;80(7):1897–900.
- [3] Odeshi A, Mucha H, Wielage B. Manufacture and characterisation of a low cost carbon fibre reinforced C/SiC dual matrix composite. *Carbon* 2006;44(10):1994–2001.
- [4] Mei H, Cheng LF. Stress-dependence and time-dependence of the post-fatigue tensile behavior of carbon fiber reinforced SiC matrix composites. *Compos Sci Technol* 2011;71(11):1404–9.
- [5] Pei BB, Zhu YZ, Yuan M, Huang ZR, Li YS. Effect of in situ grown sic nanowires on microstructure and mechanical properties of C/SiC composites. *Ceram Int* 2014;40(4):5191–5.
- [6] Tong YG, Bai SX, Chen K. C/C–ZrC composite prepared by chemical vapor infiltration combined with alloyed reactive melt infiltration. *Ceram Int* 2012;38(7):5723–30.
- [7] Xu YL, Zhang WH. Numerical modelling of oxidized microstructure and degraded properties of 2D C/SiC composites in air oxidizing environments below 800 °C. *Mater Sci Eng A* 2011;528(27):7974–82.
- [8] Fan SW, Zhang LT, Xu YD, Cheng LF, Lou JJ, Zhang JZ, et al. Microstructure and properties of 3D needle-punched carbon/silicon carbide brake materials. *Compos Sci Technol* 2007;67(11):2390–8.
- [9] Sharma R, Deshpande VV, Bhagat AR, Mahajan P, Mittal RK. X-ray tomographical observations of cracks and voids in 3D carbon/carbon composites. *Carbon* 2013;60:335–45.

- [10] Hernández S, Sket F, González C, LLorca J. Optimization of curing cycle in carbon fiber-reinforced laminates: void distribution and mechanical properties. *Compos Sci Technol* 2013;85:73–82.
- [11] Feng YJ, Feng ZD, Li SW, Zhang WH, Luan XA, Liu YS, et al. Micro-CT characterization on porosity structure of 3D C f/SiC m composite. *Compos A* 2011;42(11):1645–50.
- [12] Sket F, Seltzer R, Molina-Aldareguía J, Gonzalez C, LLorca J. Determination of damage micromechanisms and fracture resistance of glass fiber/epoxy cross-ply laminate by means of X-ray computed microtomography. *Compos Sci Technol* 2012;72(2):350–9.
- [13] Hu XF, Wang LB, Xu F, Xiao TQ, Zhang Z. In situ observations of fractures in short carbon fiber/epoxy composites. *Carbon* 2014;67:368–76.
- [14] Mei H, Cheng LF, Zhang LT, Luan XG, Zhang J. Behavior of two-dimensional C/SiC composites subjected to thermal cycling in controlled environments. *Carbon* 2006;44(1):121–7.
- [15] Mei H, Bai QL, Li HQ, Yu CK, Cheng LF. Effect of loading rate and temperature on monotonic tensile behavior in two-dimensional C/SiC composites. *Ceram Int* 2014;40(10):16635–40.
- [16] Mei H, Cheng LF, Ke QQ, Zhang LT. High-temperature tensile properties and oxidation behavior of carbon fiber reinforced silicon carbide bolts in a simulated re-entry environment. *Carbon* 2010;48(11):3007–13.
- [17] Mei H, Li HQ, Bai QL, Zhang Q, Cheng LF. Increasing the strength and toughness of a carbon fiber/silicon carbide composite by heat treatment. *Carbon* 2013;54:42–7.
- [18] Cai YZ, Fan SW, Liu HY, Zhang LT, Cheng LF, Dong BX, et al. Microstructures and improved wear resistance of 3D needled C/SiC composites with graphite filler. *Compos Sci Technol* 2009;69(14):2447–53.

- [19] Yin XW, Cheng LF, Zhang LT, Xu YD. Thermal shock behavior of 3-dimensional C/SiC composite. *Carbon* 2002;40(6):905–10.
- [20] Chen X, Li YL. Dynamic tensile behavior of two-dimensional carbon fiber reinforced silicon carbide matrix composites. *Mater Sci Eng A* 2011;528(22):6998–7004.
- [21] Luo SN, Jensen BJ, Hooks DE, Fezzaa K, Ramos KJ, Yeager JD, et al. Gas gun shock experiments with single-pulse x-ray phase contrast imaging and diffraction at the advanced photon source. *Rev Sci Instrum* 2012;83:073903.
- [22] Hudspeth M, Claus B, Dubelman S, Black J, Mondal A, Parab N, et al. High speed synchrotron x-ray phase contrast imaging of dynamic material response to split hopkinson bar loading. *Rev Sci Instrum* 2013;84:025102.
- [23] Meyers MA. *Dynamic Behavior of Materials*. Wiley, New York; 1994.
- [24] Dandekar D, Hall C, Chhabildas L, Reinhart W. Shock response of a glass-fiber-reinforced polymer composite. *Compos Struct* 2003;61:51–9.
- [25] Yuan F, Tsai L, Prakash V, Dandekar DP, Rajendran AM. Dynamic shear strength of S2 glass fiber reinforced polymer composites under shock compression. *J Appl Phys* 2008;103:103537.
- [26] Lukyanov AA. An equation of state of a carbon-fibre epoxy composite under shock loading. *Eur Phys J B* 2010;74:35–45.
- [27] Millett J, Bourne N, Meziere Y, Vignjevic R, Lukyanov A. The effect of orientation on the shock response of a carbon fibre-epoxy composite. *Compos Sci Technol* 2007;67(15-16):3253–60.
- [28] Zaretsky E, deBotton G. The response of a glass fibers reinforced epoxy composite to an impact loading. *Int J Solids Struct* 2004;41(2):569–84.
- [29] Katz S, Zaretsky E, Grossman E, Wagner H. Dynamic tensile strength of organic fiber-reinforced epoxy micro-composites. *Compos Sci Technol* 2009;69(7-8):1250–5.

- [30] Tehrani M, Boroujeni A, Hartman T, Haugh T, Case S, Al-Haik M. Mechanical characterization and impact damage assessment of a woven carbon fiber reinforced carbon nanotube-epoxy composite. *Compos Sci Technol* 2013;75:42–8.
- [31] Bie BX, Han JH, Lu L, Zhou XM, Qi ML, Zhang Z, et al. Fracture mechanisms of carbon nanotube/epoxy composites under high strain-rate loading. *Compos Part A* 2015;68:282–8.
- [32] Cooper IZ, Rubin MB. Modeling damage in silicon carbide due to an impact stress below the HEL. *Int J Impact Eng* 2014;65:174–84.
- [33] Paris V, Frage N, Dariel MP, Zaretsky E. The spall strength of silicon carbide and boron carbide ceramics processed by spark plasma sintering. *Int J Impact Eng* 2010;37(11):1092–9.
- [34] Kolsky H. An investigation of the mechanical properties of materials at very high rates of loading. *Proc Phys Soc B* 1949;62(11):676–700.
- [35] Li YL, Ramesh KT. An optical technique for measurement of material properties in the tension kolsky bar. *Int J Impact Eng* 2007;34(4):784–94.
- [36] Chen WL, Song B. Split Hopkinson (Kolsky) Bar: Design, Testing and Applications. Springer; 2010.
- [37] Lu L, Fan D, Bie BX, Ran XX, Qi ML, Parab N, et al. Note: Dynamic strain field mapping with synchrotron X-ray digital image correlation. *Rev Sci Instrum* 2014;85:076101.
- [38] Fan D, Lu L, Li B, Qi ML, Zhao F, Sun T, et al. Transient x-ray diffraction with simultaneous imaging under high strain-rate loading. *Rev Sci Instrum* 2014;85:113902.
- [39] Millett JCF, Bourne NK. Shock response of the elastomer, polychloroprene. *J Appl Phys* 2001;89:2576–9.

- [40] Luo SN, An Q, Germann TC, Han LB. Shock-induced spall in solid and liquid cu at extreme strain rates. *J Appl Phys* 2009;106(1):13–502.
- [41] Li YL, Suo T, Liu MS. Influence of the strain rate on the mechanical behavior of the 3D needle-punched C/SiC composite. *Mater Sci Eng A* 2009;507(1):6–12.
- [42] Antoun T, Curran DR, Razorenov SV, Seaman L, Kanel GI, Utkin AV. Spall fracture. Springer; 2003.
- [43] Wang B, Zhang J, Lu G. Taylor impact test for ductile porous materials—part 2: experiments. *Int J Impact Eng* 2003;28(5):499–511.
- [44] Lopatnikov SL, Gama BA, Haque MJ, Krauthauser C, Gillespie JW, Guden M, et al. Dynamics of metal foam deformation during taylor cylinder–hopkinson bar impact experiment. *Compos Struct* 2003;61(1):61–71.
- [45] Han F, Zhu Z. The mechanical behavior of foamed aluminum. *J Mater Sci* 1999;34(2):291–9.
- [46] Liu MS, Li YL, Xu F, Xu ZJ, Cheng LF. Dynamic compressive mechanical properties and a new constitutive model of 2D-C/SiC composites. *Mater Sci Eng A* 2008;489(1):120–6.
- [47] Chen X, Li YL, Zhi Z, Guo YZ, Ouyang N. The compressive and tensile behavior of a 0/90 C fiber woven composite at high strain rates. *Carbon* 2013;61:97–104.