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From fundamental understanding to innovative developments of high-performance composites for aerospace application

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Abstract: In framework of the National Key Basic Research Program (973 Program) we focus our attention on R&D of (1) continuous enhancement of composites performance both for new generation and existing materials, (2) cost-effective manufacturing technologies, particularly resin transfer molding (RTM) in conjunction with textile technology, and (3) crashworthy composite structures in design, and manufacturing and simulation methodology for aircrafts composites. Many successful examples stories such as ex-situ concept have been demonstrated that the performance potential of composites could be enhanced and maximized by basically understanding the complicated multi-scale and multi-dimensional structural characteristics in relation to properties. Based on the concept, graphite composites system with generally high impact damage resistance and balanced processing conditions has being developed consisting of base resins, modifiers and binders/tackifiers.

Key words: *ex-situ* concept; *in-situ* concept; impact damage resistance; epoxy; bismaleimide; polyimide; rtm; crashworthiness

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1 Introduction

Composite materials and structures have proven their potential for use in high performance aerospace applications over the last fifty years. High mass specific stiffness, strength and energy absorption, high functionality (e.g. through tailored anisotropy), and optimized structural concepts (e.g. due to high levels of design integration) are the main reasons for specifying composites. The materials share today for composites has reached 15% in civil aircraft and more than 50% in military aircraft and helicopters. Furthermore, there is considerable potential for further increases in these figures through applications such as the composite wing and composite fuselage, both of which are being investigated by Boeing and Airbus.

Though the success story, however, the challenge nowadays for all developments in aerospace is obviously to find a good compromise between performance and cost. For aircrafts composites, there are three major challenges recognized: (1) continuous enhancement of performance for mass production, (2) devel-

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opment of cost-effective manufacturing technologies in conjunction with textile technology and liquid molding methods, particularly resin transfer molding (RTM), (3) research on function-integrated design, and manufacturing and simulation methodology of composite components.

Having recognized these challenges and their importance for aerospace industry, the National Key Laboratory of Advanced Composites (LAC) at Beijing Institute of Aeronautical Materials (BIAM) is carrying out numerous R & D projects, especially the National Key Basic Research Program (973-Program) to fundamentally study the multi-scale and multi-dimensional nature of composite materials, in order to find a cost-effective way to further develop aircraft composite. This paper reports some examples of its current R & D activities.

2 "Ex-situ" concept to increase to the impact damage resistance of composites

Graphite laminates are widely used for primary aircraft structure. To develop high-performance aircraft composites of different chemistries for a wide range of temperatures, the technological challenge is how to increase the impact damage tolerance significantly higher than the existing materials. And in regard to existing materials, the challenge is how to cost-effectively enhance their performance without changing the general composition and processing conditions. With these concerns in mind and in spite of reinforcement materials, it is well-known that both the impact damage tolerance and temperature resistance of laminated systems are determined by resin systems, they are typically the resin-dominant properties.

A traditional method to increase the impact damage resistance of thermosets matrix composites used for aerospace is to toughen base resin. For high-performance graphite laminates, high-temperature resistant thermoplastics (TP) are usually applied as toughening agents to obtain a two phase morphology. The two-phase morphology is formed by a reaction induced spinodal phase decomposition and the subsequent coarsening mechanism (Fig. 1(a)). The phase separation and inversion are basically in an overall cooperation and *in-situ*, i. e. they occur everywhere in the composite simultaneously together with the resin impregnation^[1,2]. The resulting composites with higher delamination resistances measured, for instance, in compression strength after impact (CAI) of about 250 MPa to 300MPa have been categorized to the second or even third generation of graphite laminates. Before long, it was recognized that these CAI values set approximately the upper limit.



Fig. 1 (a) Phase-inversed granular morphology of resin cast resulting from spinodal decomposition (b) Interlaminar morphology of interleaved graphite laminate denoted as Case 1 in Table 1 indicating again the granular structure. In both cases, the TP continuous phase was chemically etched away

Inspired by natural laminates e.g. shells and woods with typically periodic layered structures, we developed a

concept^[3,4] to periodically interleave solid thermoplastic thin layers into each graphite plies. Thus, the traditional *in-situ* phase separation procedure was altered from an initially homogeneous solution of the two components to such a manner that the respective thermosets and thermoplastic components were first separated. Graphite plies were wet-impregnated as usual with the thermosets, and the TP thin films were used as the interleaves. An interaction between them occurs at elevated temperatures typically during the curing reaction. Thus, the entire procedure can be regarded as *ex-situ*. It was expected that the delamination resistance of the structure could exceeds the upper limit above mentioned by the *ex-situ* concept, and it will be an all-purposed concept for toughening any laminates independent on the chemistry of matrix resins.

Let's first demonstrate an epoxy (EP) based graphite laminate (Fig. 1(b)). The TP toughening agent used was an amorphous polyetherketone (PEK) of Chinese development. There were two ways to make the *exsitu* toughened structure; either inserting the TP films periodically into the EP/graphite prepreg plies or spraying the TP solution directly onto the prepreg surfaces. The prepared laminates were finally cured at a constant condition for all the samples studied.

For the 16-ply *ex-situ* laminates EP-based with 15 TP interleaves (Case 1 in Table 1), the CAI value was by far the highest among all samples tested, where 5288 (Case 2) stands for the *in-situ* sample as controlled with exactly the same composition as Case 1. The result was also revealed by the Boeing specification (Case 3). Moreover, the *ex-situ* specimen indicates even higher CAI-value than that of the neat PEEK laminates (Case 4). It was also proven that the in-plane mechanical properties of the *ex-situ* laminates were mostly remained and the material exhibited a rather balanced profile of strength, stiffness and impact resistance^[4,6].

In the table, AG-80/E-54/DDS (Case 5) stands for non-toughened EP-based laminates. No wonder its CAI-value is the lowest. The chemistry of the TP components seems to less affect the CAI. An example was PES (Case 6) instead of PEK as typical toughening agent for the study.

By symmetrically interleaving only 10 TP layers into the central plies and thus reducing the total volume fraction of TP, the impact resistance decreases proportionally (Case 7). The similar tendency was also found on the laminates periodically spray-coated with PEK (Case 8) since only very thin coatings were experimentally available. HST-7 (Case 9) was reported highly resistant against the impact damage due to a thick and tough interleaf. Its CAI of about 350 MPa was superiorly high. However, as also reported, the weight penalty for the material was a disadvantage. The reduced stiffness and creep tendency required additional graphite plies to maintain design properties^[6].

Case	Specimen	CAI/MPa
1	ex situ laminates with PEK films periodically interleaved	3451)
2	Laminate of 5288 (in-situ toughened) as control	267
3	ex-situ laminates with PEK spray-coated (Boeing specimens)	3451)
4	PEEK/graphite laminates (APC-2)	331
5	Laminate of AG —80/E=54/DDS system (non-toughened) as control	150
6	ex-situ laminates with PES films	298
7	PEK film ex-situ interleaved only in the central 10 plies	271
8	exsitu laminates with PEK spray-coated	308
9	Laminate of HST-7 of American Cyanamid (reported)	~350

Table 1 CAI data for different EP based graphite laminates in comparison with each other and also with PEEK

Note: 1) The value similarity between No (1) and (3) was only coincidental.

It is interesting to study the interlaminar morphology for the sample denoted as Case 1 in Table 1 (Fig. 1(b)).

It is, at the first look, nothing new except for the phase-inversed granular structure usually observed for the reaction-induced decomposition reaction: the EP-rich particles are coated by continuous TP thin films, which were previously chemically etched away for the SEM study. Furthermore, the granular structure was slightly penetrated into the graphite plies, establishing an inter-anchored boundary between the co-continuous granular structure and graphite ply impregnated with the pure EP (Fig. 2). Because of the inter-anchoring effect, crack propagation resistant was remarkably increased with a complicated fiber pull-out process (Fig. 3).



Fig. 2 Boundary region between the two-phase granular and the pure EP structure within single graphite ply (a) and the high magnification of the sample in (b)



Fig. 3 Representative of fiber pull-out after GIC testing

The particle size decreases slightly from the symmetric interply centre into a few ten microns under the ply surface. This periodically modulated concentration fluctuation of the TP component accompanied with the particle size distribution does become the characteristic for the *ex-situ* structure on the contrary to the *in-situ* structural formation. In the later case (i. e. Case 2 in Table 1), the phase-inversion structure is found overall, its concentration in interplay region perhaps does not reach the critical level. Thus, the toughening effect of the structure should be averaged. We believe that this is the reason why the *ex-situ* laminates exhibited significantly high impact damage resistance, even higher than that of the PEEK laminates (i. e. Case 4 in Table 1).

Fig. 4 shows the phase morphology for the sample spray-coated (Case 8 in Table 1). The bottom horizontal graphite fibers were mostly coated by the EP (Fig. 4(a)); suggesting that the competitive phase inversion and coarsening development ran much quickly on the surfaces than in the interply region. This is thought due to the fact that the sprayed solution reacted already with EP during the wet spraying because the main kinds of graphite fibers are sized for EP impregnation. They possess a higher affinity to EP than to TP components. This preferential richness of EP is advantageous for guaranteeing the interface bonding. Therefore, we are able to generate a concentration gradient structure for each fiber in the normal direction to the fiber surface; EP-rich on the surface and TP-rich

in the interply layer (Fig. 4(b)). For practical application it is highly desired.



Fig. 4 Phase morphology for the spray-coated samples (Case 8 in Table 1) (a) The bottom horizontal graphite fibers were mostly coated by EP, implying that the EP-rich particles grown much quickly and larger on the fiber surface than in the interply layer; (b) The concentration gradient structure for each fiber; EP-rich on the fiber surface and TP-rich in-betweens of two fibers. The separation of the fibers from the matrix was caused by the chemical etching

It is noteworthy that *ex-situ* concept demonstrated independent on resin chemistry, thus opening a door for wide application in aircraft composites. Next example is bismaleimide (BMI) based laminates^[6]. As matrix resins there were four variants (Table 2) for comparison: pure BMI (BMI 6421 of own development at LAC/BIAM) as control (Case 1); BMI /TP blend with a TP concentration of about 17.5 % (mass fraction) (Case 2). This was basically the *in-situ* concept. Laminates based on *ex-situ* concept (Case 3), in principle, it is a "selective toughening concept" compared with the Case 2, being categorized to "overall toughening concept". And the pure PEK films interleaved for Case 3 were replaced with those of PEK/BMI two-component films (Case 4). As PEK:BMI = 0:100 (0% PEK), it reduced to Case 1 as control; and for PEK:BMI 6421 = 100:0 (100% PEK), it returned to Case 3.

asc	Matrix Resin	CAI/MPa
1	BMI (Control)	180
2	BMI/TP blended (in-situ concept)	199
3	BMI/TP-films interleaved (ex-situ concept)	254
4	BMI/(BMI/TP) of two-component films interleaved (exsitu concept)	290
3 1 	BMI/(BMI/TP) of two-component films interleaved (ex-situ concept)	

Table 2 CAI-values for different BMI based graphite laminates in comparison⁷

As indicated in Table 2, the control (Case 1) shows a CAI datum of about 180 MPa, whereas for the *in-situ* concept (Case 2) CAI is increased to about 199 MPa. The enhancement in toughness is relative. However, the *ex-situ* concept resulted in a much significant CAI enhancement of about 254 MPa. The highest improvement of impact resistance was achieved by interleaving the two-component films into the plies (Case 4), resulting in a CAI of about 290 MPa, even when the quantity of PEK added into the system was comparable with that in the Case 2 and in Case 3. This comparison demonstrates obviously the efficiency of *ex-situ* concept in enhancement of impact damage resistance of graphite laminates even with intrinsically brittle matrix.

Fig. 5 shows the glass transition behavior of neat BMI resin (a) and BMI graphite laminate (b) interleaved with the two-component films of a ratio of 60,40 (Case 4 in Table 2) by means of DMTA. High-temperature peak at about 285°C in Fig. 5(a) is characteristic for Tg of the BMI, which is, however, lowered down to about 271°C in Fig. 5(b) for the laminate. The lowering effect of glass transition temperature was not only found for BMI, but also for the toughness modifier PEK which, as shown in Fig. 5(b), is slightly reduced from originally 240°C to about 234°C for the laminate. The exact cause for the lowering effect is at the moment unknown. Alone there were two characteristic temperatures recorded in Fig. 5(b) implies the existence of a two-phase structure for the *ex-situ* composites.



Fig. 5 Glass transition behavior of neat BMI (a) and the graphite laminate (b) made by ex-situ method

Correspondingly, morphological investigation revealed that a typical decomposition process occurred in the interlaminar regions (Fig. 6). One can easily find the horizon (0°) plies and the 45° plies of the laminate interleaved with the PEK/BMI two-component films (Case 4 in Table 2). The PEK component was prior the SEM study chemically etched-off. The granular structure remained was BMI component cured. As shown in the figures, the granular structure is very fine, with an average diameter of or even lower than about 1 μ m.



Fig. 6 Representatives^[8] of interply morphology of BMI laminate toughened with PEK/BMI twocomponent film (60,40) by *exsitu* concept

However, the situation changed for the laminate interleaved with pure PEK films in Fig. 7 for Case 3 in Table 2. At the same chemical etching conditions, an overlapped nodular structure of remained BMI component appeared. The average diameter of the nodular structure seems much larger than the correlation length so that the particles were strongly interconnected to form a rough morphology. As known, there are many factors affecting the spinodal decomposition and coarsening mechanism^[2], for instance, the diffusion behavior, the dissolubility behavior, the speed competition behavior, the viscosity behavior and the surface tension behavior, etc.

By taken CAI in conjunction with the morphological characteristic into consideration, it is conceivable that the completely grown and co-continuous, but not overlapped granular structure must be a good sign for high toughness and thus for the high impact damage resistance. It is also believed that the two-component films help to facilitate the spinodal decomposition and coarsening process, leading to the granular structure.

The third example was made with polyimide (PI) based graphite laminates^[9]. For this study according to *ex*situ concept, there were two options in application methods for laminated systems; (1) the graphite prepregs wetimpregnated with pure PI (Polyimide LP-15 of own development at LAC/BIAM) were additionally interleaved with PEK thin films, or (2) spray-coated with PEK powders. CAI results of the laminates made by the two ways



Fig. 7 (a) representative⁽³⁾ of interlayer morphology of BMI laminate toughened with pure PEK film by *ex-situ* method; (b) A high magnification of (a)

are compared in Table 3. C-scan results are also attached.

Specimen	CAI/MPa	C-scan results		
PI pure (Control)	212	٥	Ø	,
Pl/TP blend (in situ concept)	276	0	0	2
PI/TP-films interleaved (ex-situ concept)	309	0	1	R
PI/TP-powder coated (ex-situ concept)	327	1	P	Ø

Table 3 CAI-values for different PI based graphite laminates in comparison

It can be found that the toughness improvement is first remarkable for the *in situ* concept. However, the *ex-situ* concept was superior. Particularly the powder-coated laminates show the highest CAI of 327 MPa among all specimens studied. The C-scan results show the impact damage. It is obvious that the damage area is reduced with increased CAI.

Upon these investigations, a structural model can be proposed (Fig. 8). In the *in situ* concept, the homogeneous solution is developed under curing conditions to an overall phase-inversed granular structure. The concentration distribution of the components is even in the entire composites, hence, only the portion in the interply layers may function as the toughening agents. In the *ex situ* concept, the periodicity can be remained throughout the curing reaction at all. The solid-solid interphase interaction includes the diffusion, dissolution and phase separation within the interply layers, also leading to the phase-inversed granular structure. However, there is still a TP-rich interlayer of above 50% (vol., fraction) whereas each graphite ply is still largely impregnated with base matrix resin to

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guarantee the in-plane properties. The periodicity serves probably as the major structural contributor to the resistance improvement. It is believed that the functionality of the periodic structure can be further explored, optimized and maximized by altering the amplitude (concentration) and the period (thickness), and even for many other composites pairs.



Fig. 8 Structural model proposed for the phase development in comparison^[6]

In conclusion, a generic and innovative *ex-situ* concept has been developed for prepreg technology. It has been demonstrated that the composites with compression after impact (CAI) values generally higher than 300 MPa can be cost-effectively fabricated for prepreg/autoclave technique. The concept is additionally independent on resin chemistry. The preliminary investigation has been conducted on EP-, BMI- and PI-based laminates with successful results. New investigation is going on for other composite systems.

3 "Ex-situ" concept for resin transfer molding of composites

Liquid molding techniques are, when used in combination with textile preforming, an interesting alternative to prepreg technology for aerospace. Many different approaches to liquid molding have been developed. However, this well-established technology is still challenged by resin-related technological problems. (1) Increased resin viscosity with time and flow distance leads to problem in wetting, infusion and void formation because the cure reaction of resin begins and continues during the whole filling period; (2) Process difficulty like narrow process window requires precisely controlled temperature and limited pot time again due to the reactivity of resin. Both problems call for solution to cost-effectively increase the product quality and reduce the process sensibility.

For this end, our intention was to explore the *ex-situ* concept for resin transfer molding (RTM), because for conventional RTM technique very low-viscosity and robust resin systems with extended pot life, long out times or no requirement for storage at sub-ambient temperatures must be developed. These materials should also possess a high tolerance for impact without degradation of properties. However, it has been difficult to toughen RTMable resin systems without sacrificing the flow ability and the overall mechanical properties.

Technologically, there are two simultaneous processes critical for a successful RTM manufacturing, namely a physical process of mold filling with low-viscosity resin and a chemical process of cross-linking reaction, respective-

ly. During the mold filling, resin will flow, wet, infiltrate and impregnate the reinforcement pre-placed with a high volume fraction in the mold, thus, determining already at this stage the end-properties and -quality. In process control point of view the flow, wetting, infiltration and impregnation are always accompanied with increased resin viscosity with temperature, time and flow distance caused by the chemical reaction. It does be this interaction making the process control difficult, particularly for manufacturing of high-quality primary aerospace composite structures. To solve the problem, chemorheology, for example, was developed to predict the change of viscosity with temperature, time and 2D or 3D pattern. However, it is obvious that chemorheology neither reduces the viscosity nor prolongs the time for low viscosity.

Encouraged by success of ex-situ concept for prepreg technology, a new and generic concept of ex-situ liquid molding was developed^[10,7,11] for RTMable or RFIable two-part resin systems. The basic idea was to change the one-step process in RTM and RFI to two-step one, namely to change the simultancous, interacted and in time-scale parallel reactive and flow processes in the traditional way to a separate, in time-scale serial one. For instance, the base component and the hardener are traditionally mixed before injection. It should be immediately injected into the mold due to the curing reaction. The ex-situ concept overleaps the mixing step by separating the two reactive components, the solid one being pre-coated on the graphite reinforcement and the liquid one being injected into mold as flow medium.

Fig. 9 shows the phase change of a two-part BMI resin system for RTM with time^[7]. As the liquid component was injected into the mold, meeting the solid component at the beginning stage, the solid component was visible (in Fig. 9(a)). At the end of process (in Fig. 9(c)) there was only a homogeneous solution. Both components were completely dissolved with each other. Fig. 9(b) exhibits the mixing stage in-between. For this study, vacuum assisted RTM technique was applied.



Fig.9 Representatives⁽⁷⁾ of phase change of a two-part BMI resin system at the beginning (a), in-between (b) and at the end (c)

In this study, after the pre-coated reinforcement with the solid component was completely wetted and impregnated by the liquid component and the mold was fully filled out at a relative low temperature, heat was introduced into the system to initiate the curing reaction. As shown in Fig. 9, the two components dissolved completely at this temperature condition. Composite samples made in accordance with this concept demonstrated comparable mechanical properties listed and compared in Table 4. As shown, ex-situ technique produces almost equivalent laminate properties as traditional one, however, the process control for ex-situ RTM was much easier due to the fact that the reactive component was not mixed before injection, so that the liquid resin was still low viscose and the mold was injected at low temperature for almost non-limited time. The mold filling conditions were hence much tolerant, and the process controls will be dramatically simplified. Ex-situ RTM did not exhibit any processing difficulty at all.

	BMI-ES(ex-situ concept)	BMI(Traditional RTM)
Flexural strength/MPa	1740	1730
Flexural modulus/GPa	115	125
ILSS/MPa	98	92
Fiber volume fraction/%	55	55

Table 4 Typical mechanical properties of BMI based laminates made by ex-situ and traditional method^[7]

Technological advantage of *ex-situ* RTM concept is obvious. Separation of flow/filling and reaction process leads to better flow characteristic, better wetting, infiltration and impregnation conditions, longer pot time, all of these guarantees better product quality with equivalent mechanical properties. Moreover, *ex-situ* concept provides also an innovative way to enable toughening the composite system resin transfer molded. Table 5 reports an example of epoxy based laminates^[7].

Table 5 Comparison of CAI of comparable EP systems made by different me

Case	Specimens	CAI/MPa
1	Base EP system ¹⁾ as control	170
2	in-situ toughened EP prepreg system2) as control	267
3	EP laminate ³¹ made by ex-situ RTM method	294

Note: 1) Epoxy resin system for RTM, EP 3266 developed at LAC/BIAM;

2) Toughened epoxy prepreg system, EP 5288 developed at LAC/BIAM;

3) EP 3266-ES developed at LAC/BIAM.

It is obvious that a toughening with PEK improves significantly the laminate toughness either by *in-si-tu* method (Case 2) or by *ex-situ* RTM method (Case 3). However, the *ex-situ* laminate prevails significantly in impact damage resistance compared to the controls. It is particularly interesting to note that the need of low viscosity resin systems to allow low-temperature infusion in traditional RTM process limits considerably the use of any toughening techniques associated with prepreg resins, whereas the EP system 3266-ES reported in the table was just made by RTM method and it was successfully toughened. In correspondence with the higher CAI, Fig. 10 shows and compares the damage size of the laminates impact-damaged. It is apparent that reduced damage region for the *ex-situ* sample (Case 3 in Table 5) was verified compared to that of the control (Case 1 in Table 5).

Other important properties were also compared between *ex-situ* laminate and the control in Table 6. Though this was only a preliminary evaluation, almost no noticeable difference in the properties could be identified. The in-plane mechanical properties were still comparable; however, the CAI strength is almost 30% - 40% higher than that of the traditional one.

Table 6 Comparison of some typical properties of the two systems^[7]

	Ex-situ laminate	Control
0° flexural strength/MPa	1540	1580
0° flexural modulus/GPa	105	103
Interlaminar shear strength/MPa	89	85
Fiber volume fraction $/\%$	55	55
PEK weight percentage / %	10	0

For the EP based laminates made by ex-situ RTM technique, the two-phase granular structure could



Fig. 10 Comparison of damage size between the control (a) and ex-situ (b) by C-scan

also be found in the interply region (Fig. 11). However, the extension of the interlayer structure was very limited; the layer thickness was only about $5-8 \ \mu m$.

In conclusion, it was demonstrated that the *ex-situ* concept is applicable not only for prepreg materials but also for liquid molding like RTM technique. For RTM technique, *ex-situ* concept affects hardly the traditional process characteristic, does not sacrifice the flow ability and the overall physical properties, but makes the process conditions more tolerant. Impact damage resistance of the laminates can additionally be improved without noticeable reduction of other mechanical properties.



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Fig. 11 Interlayer morphology of EP based graphite laminate manufactured by ex-situ RTM method^[12]

With development and technological maturation of the innovative *ex-situ* concept, and in order to take the full advantage of its opportunity, two investigations are going on at LAC/BIAM. First, composite materials system is being developed both for prepregs and RTMable system with a universal target of high impact damage resistance (Fig. 12). The system consists of 2 and/or 3 sub-systems of the base resin system like epoxies, bismaleimides and polyimides, the *ex-situ* modifier system and binder or tackifier system. The modifier, binder/tackifier system should be designed to match the base resin system. Second, the concept is in principle universal, e.g. it can also be applied to existing composites and structures with target to significantly improve the impact damage characteristic. Successful story has been achieved in cooperation with aircraft industry for the both directions.

It is also envisaged that by combining the *ex-situ* concept with advanced tackification technology and preforming technology, complicated preform can be made near-shaped for RTM and the composite manufactured can be made impact damage resistant as well. Based on this idea, an advanced manufacturing system for RTM technology was proposed (Fig. 13). Many investigations have being conduced on this technological platform. An example of sine wave beam RTM-made is also shown in Fig. 13.

4 Crashworthiness study on composite structures

Integrated composite structure has been recognized as path to affordability for aircraft components; crashworthiness structure is one of the typical function-integrated examples. With the assumed wider use of composites (e. g. CFRP fuselages), crashworthiness is becoming an increasingly important topic for aero-space applications. This is because of concerns over passenger safety and the associated legal requirements.

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Fig. 12 Resin-dominant properties for graphite composites and the development target at LAC/ BIAM



Fig. 13 Manufacturing system for RTM under development at LAC/BIAM and sine wave beam manufactured on this platform

Composite materials are of particular interest for use in energy absorbing structures because of their high mass specific energy absorption capability. Energy absorption capacities of more than a 100 kJ/kg can be a-chieved with careful design^[18].

In context of function-integration and particularly the design and manufacturing issue, it is important to make the structural performance and functional response compatible in one composite structure. The compatibility can be realized simultaneously or in a serial manner. For crashworthy composite structure, the energy absorption response is initiated by a special-design trigger system, which is responsible for switching from a stable state in structures to a progressive fragmentation response, i. e. it is a serial mode. The technical challenge is how to maximize both the structural performance such as stiffness, strength and impact resistance, and the crashworthiness requirement such as adjustable crash-rate dependent failure strength (or deformation) and specific energy absorption in one structure. This is again a problem of multiscale and multi-dimensional structural design ranging from microns to engineering scale, involving fiber breakage, matrix cracking, fiber-matrix interface cracking and careful design of the fiber reinforcement and the structural geometry.

Previous investigation on chamfered end composite tubes as usual indicated that the failure mode in crush test was generally affected by textile reinforcement such as fiber type and architecture (Fig. 14). The

tube reinforced with T300 was fragmentized in the crush test (Fig. 14(a)), which resulted among all the "prepreg" samples in the highest specific energy absorption (SPEA), whereas the tube reinforced with T700 with about 10% lower SPEA showed a splaying failure mode (Fig. 14(b)). Braided tube exhibited an excellent capability on energy absorption, even in combination with a more brittle resin system. The tube was failed in a mixed mode of splaying and local buckling without fragmentation due to more densely interacted textile structure Fig. 14(c)).



Fig. 14 Representatives of 3 tubes crush-tested with different failure modes¹¹³

In Table 7, EP1 was an *in-situ* toughened and EP2 non-toughened epoxy system. The interaction between resin toughness and fiber type was complicated if we only take SPEA as a measure for crashworthy performance evaluation. On the other hand, it is also found that in design point of view, it was easier to control the architecture rather than the resin and fiber type for higher energy absorption (refer the failure modes in Fig 14).

Specimens and processing	σ _{peak} /MPa	σ_{mean}/MPa	$SPEA/(J \cdot g^{-1})$	Failure Mode
EP1/T300, Prepreg/Autoclave	154.2	119, 3	76.0	Fragmentation
EP1/T700, Prepreg/Autoclave	141.0	109.8	70.0	Splaying
EP2/T300, Prepreg/Autoclave	147.9	104.3	66.4	Fragmentation
EP2/T700, Prepreg/Autoclave	145,9	111.3	70.9	01
	183, 8	125.3	81,4	Splaying
EP (3266), RTM, triaxially braided	181.7	110.3	71.7	Splaying+local buckle

Table 7 Data of energy absorption behavior in crush test in comparison^[13]

The most widely used trigger mechanism is to chamfer one end of the composite tube. It reduces effectively the initial load peak that accompanies failure initiation followed by stable collapse. However, the concept like the chamfered end modification is not realistic for real structure like sine wave beam. Therefore, new concept should be developed to study the relationship among laminate panels, composite tubes and real structures with regard to a compatible mechanical behavior of the structural stability and energy absorption response. For this end, model sine wave beams were designed with different trigger configuration^[14]. All of the models were designed to contain discontinuity in graphite plies within the sine web as illustrated in figures in Table 8.

It was demonstrated that a stable progressive crush process was established for all the model structures, resulting generally in a fragmentation mode as designed (Fig. 15). However, depending on the trigger configuration, there was a difference in mechanical response found (Table 8 and Fig. 16). In general, the new designed trigger mechanism could fulfill the requirement by a balanced structural performance and crashworthy function. The failure initiation could also be designed and varied in a large range of critical stresses or strains. Investigation is still going on. Moreover, material damage models and approaches to the

Table 8 Model sine wave beams and energy absorption results in comparison						
Specimen	SWB-4	SWB-5	SWB-6	SWB-7		
Materials	5224/G803	5224/G803	5224/G803	5222/HT300		
Ply orientation	$[\pm 45]_3/7$ plies	$[\pm 45]_3/7$ plies	$[\pm 45]_3/7$ plies	[45/-45/0/0/90/0],		
Trigger configuration						
Peak load/kN	114,8	77.0	75.6	94.1		
$SPEA/(J \cdot g^{-1})$	31, 83	11.88	25.74	18.71		

simulation of structural failure need to be developed to facilitate design optimization,



Fig. 15 Representatives of SWB-4(a), -6(b) and -7(c) crush-tested in comparison

5 Conclusion remarks and outlook

Many materials and technological opportunities provided by ex-situ concept has been demonstrated with a set of composite examples. The core philosophy of ex-situ concept is based on the multi-scale and multi-dimensional structural nature of any composite materials which as a system can be designed, tailored and controlled by processing and manufacturing. Unique idea of ex-situ concept is nothing than placing the proper resin component to there where the potential of the component can be maximized in the system by a synergic effort. For this end, it should be known what and why the



Fig. 16 Mechanical response of different composite beams in crush test

component, how much the component and where it should go within the composite system. This is in turn an issue of understanding the function principle of composites as an integrated system and developing design methodology. Additionally, in-service behavior of the composite must also be understood.

The unique feature of composites is that the material and structure are produced in one step, and that there are many more factors influencing performance. On the one hand this is the reason for the success of composites. On the other, it is a challenge for future developments. Integrated approaches are required that take the material, process, structure and in-service conditions into account. Continuous improvement is always necessary in the fields of graphite fibers, binders/tackifiers, and matrix systems (e. g. fast curing, high temperature capability, high toughness).

Unfortunately, today wide gaps exist between research and industrial applications. The technology needs addressed herein must be pursued in a cooperative spirit with all of the parties working together. A coordinated effort is essential to develop, validate and transition processes, systems, data and methods directly to application programs and industrial users.

The Chinese National Key Basic Research Program (973-Program) aims at solving the national-strategic scientific and technological problems by fundamental researches and technological developments. With its support, a thematic COMPOSITE consortium has been set-up in form of partnership of industrial research institutes, Chinese Academy of Sciences and universities. It is expected that more successful accomplishments will be achieved after the initial successes.

The National Key laboratory of Advanced Composites (LAC) welcomes any international cooperation initiatives.

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