

Preparation and properties of Ti/CF/PMR Polyimide Laminates

Jie Tao*, Yubing Hu; Huaguan Li; Xuelong Fu, Xian Zhang, Lei Pan

College of Material Science and Technology, Nanjing University of Aeronautics and
Astronautics, 210016 Nanjing, China

* Corresponding author. Tel.: +86-25-5211-2911

E-mail addresses: taojie@nuaa.edu.cn (Jie Tao).

Abstract:

Ti/CF/PMR Polyimide (Ti/CFRP) laminates were manufactured by hot press process in this work to investigate their mechanical performance. The experimental results indicated that the Ti/CFRP exhibited excellent mechanical properties. The conservation rates of tensile and interlaminar shear strength under 300 °C were approximately 43% and 53%, respectively. Then, thermal cycling treatment was conducted on the Ti/CFRP ranged from -65 °C to 135 °C. It was found that the mechanical properties of the Ti/CFRP stayed unchanged after thermal cycling because the interface between the titanium and CFRP was continuous and intact. Moreover, the perforation resistance and impact responses of the Ti/CFRP were also evaluated. The responses of the Ti/CFRP under different impact energy were summarized.

Keywords: Hybrid; high-temperature property; mechanical; impact

1. Introduction

Fiber Metal Laminates (FMLs) are a relatively new type of aerospace materials composed of alternatively stacked thin metal sheets and fiber reinforced composite materials [1-4]. They possess unique characteristics such as manufacturing flexibility, impact behavior, corrosion resistance, fire resistance, elevated damage tolerance and superior fatigue properties. Therefore, FMLs have been selected as promising materials in manufacture of the aircraft. GLARE is one of the widely investigated FML which has been successfully used in A380. However, the next generation aircrafts are designed to fly faster and to last longer. Traditional GLARE could not bear such strict environment such as high temperature and thermal cycling. It is necessary to design and develop new type of FMLs to meet the requirements. Previous researchers have developed Ti/CF/PEEK laminates for high temperature use [5]. Nevertheless, the mechanical properties of the Ti/CF/PEEK laminates start to reduce after 143°C (T_g of the PEEK resin). In order to further improve the temperature resistance, relevant polymer need to be investigated. Polyimide resins possess high thermal stability (>300 °C), high glass transition temperature (T_g> 200 °C), high tensile strength, low creep, flexibility and excellent radiation shielding capability, which make them attractive materials for aeronautics and space structural components [6]. Thus, Ti/CF/PMR Polyimide (Ti/CFRP) laminates can be promising materials for high temperature application. In this work, Ti/CFRP laminates were prepared and investigated. Two basic properties, tensile and interlaminar shear properties, were evaluated under different test conditions. Moreover, impact responses of the Ti/CFRP were also discussed.

2. Experimental procedure

Ti/CFRP laminates were manufactured according to the process reported in our previous work [2]. Tensile and ILSS tests were conducted under different temperatures (-55 °C, 24°C, 120°C, 220°C, 300°C) according to ASTM D3039 and ASTM D2344,

respectively.

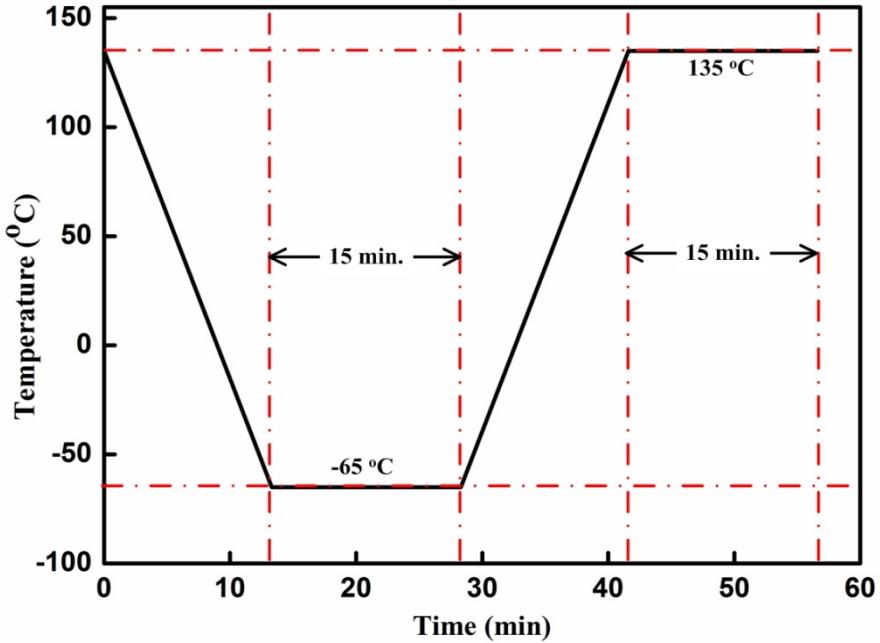


Fig. 1 Temperature-Time curve in one thermal cycling process

Quasi-static perforation tests and low velocity impact tests were conducted on the Ti/CFRP laminates. Here, square plates, with dimensions of 100×100 mm, were clamped between two square-shaped steel fixtures with a 72 mm square central aperture. The clamps were then bolted to a steel block in preparation for testing. A hemi-spherical steel indenter, with a diameter of 10 mm, was used to load the samples centrally.

3. Results and discussion

3.1 Mechanical properties under different temperatures

Figure 2 shows the tensile strength and ILSS tests results under different temperatures. There is a general tendency towards reduction in ultimate strength and ILSS with increasing temperature. When the temperature increases, the degradation in the mechanical properties of the constituent materials and the interface properties causes the decrements of the Ti/CFRP strengths. It is known that one of the roles of

the polymer matrix is to transfer mechanical loads from surface titanium to the reinforcement materials. With the temperature increasing, crack is easy to generate inside the polyimide. Thus, mechanical loads cannot be well transferred to carbon fibers. The tensile strength decreases about 57% at 300 °C comparing with that under room temperature.

Meanwhile, the ILSS also decreases with the test temperature increase as shown in Fig. 2. It is known that the interlaminar bonding between titanium and prepreg is attributed to the micromechanical interlock between the titanium and fiber-reinforced polyimide layers [2]. Temperature rising weakens the intermolecular forces between the matrix and the titanium, resulting in the degradation of the micromechanical interlock strength. Hence, applied stresses cannot be well transferred to the fibers at elevated temperature. As the strength of the resin decreases with the temperature increase, micromechanical interlock is deteriorated, leading to the decline of the ILSS. Thus the ILSS of Ti/CFRP at 300 °C is only 53% of that obtained at room temperature.

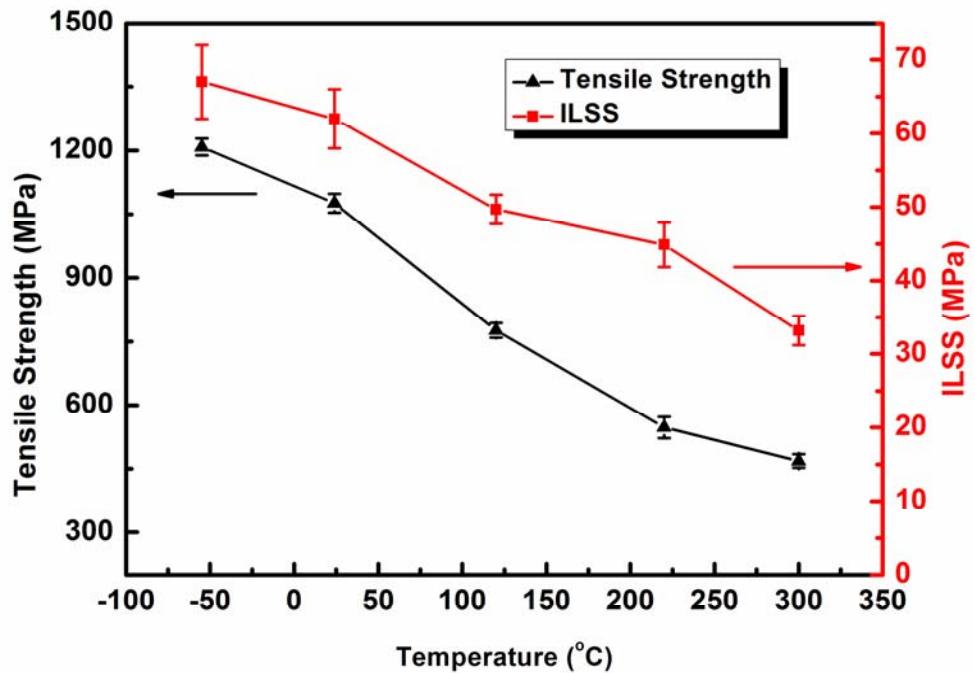


Fig. 2 Tensile and ILSS tests result evaluated at different temperature.

3.2 Thermal cycling effects

During the thermal cycling process, the constituent materials behave variously according to the difference in coefficients of thermal expansion. Metal layers shrink more than prepreg layers at the cool-down process. Microcracks or delaminations may occur because of the thermal residual stresses. Tensile properties may decrease owing to the damages of the interface. However, the tensile results do not reveal any significant change.

Figure 3a shows the tensile load-displacement curve of the Ti/CFRP with different thermal cycling treatment. It is obvious that different curves display the same tendency, while the samples after thermal cycling treatment exhibit almost the same tensile strength, as indicated in Fig. 3b.

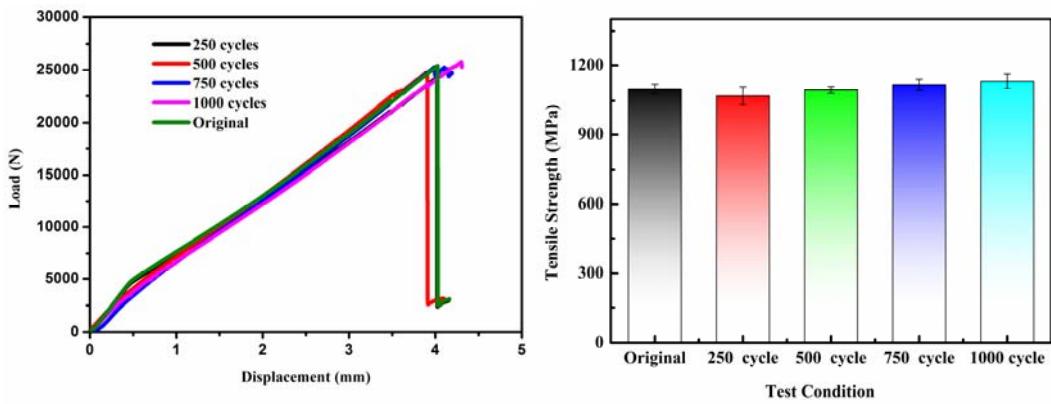


Fig. 3 (a) Load-displacement curves following tensile tests;
 (b) Comparison of tensile strength on the Ti/CFRP with different thermal cycling treatment.

As mentioned above, the interlaminar bonding between the constituent materials influences the overall tensile property. The tensile results demonstrate that the employed thermal cycling treatment does not influence the tensile properties of the Ti/CFRP. Thus it can be deduced that the interface of the Ti/CFRP stay unbroken after thermal cycling treatment. Moreover, the tensile properties of the constituent materials do not decrease after thermal cycling treatment as well.

Interlaminar characteristic of Ti/CFRP is determined by the property of the matrix polymer and the interface bonding strength. The ILSS results presented in Fig. 4

suggests that thermal cycling treatment do not cause any appreciable effect on ILSS strength, indicating the unspoilt of the polyimide matrix and the interface of fiber/polyimide and titanium/polyimide.

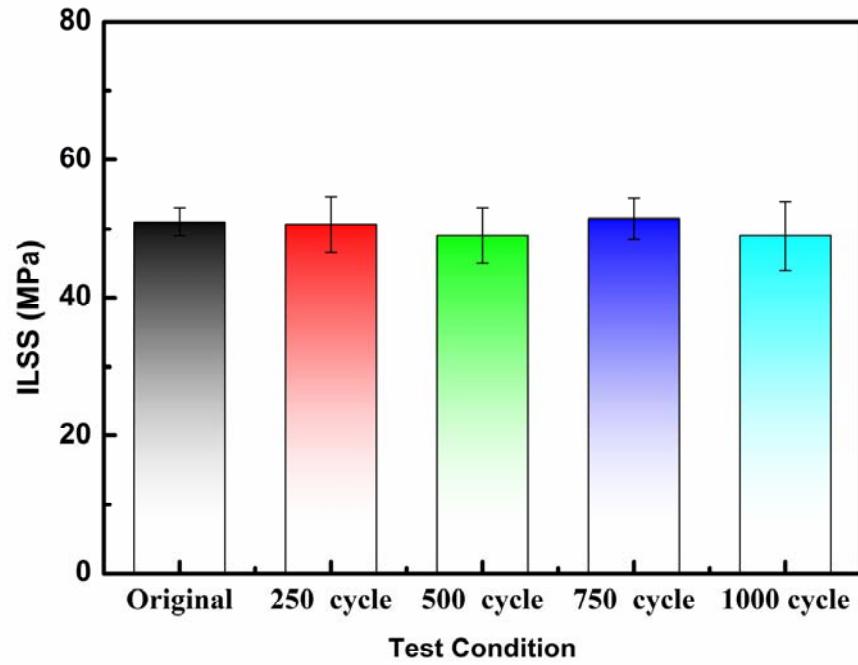


Fig. 4 Comparison of ILSS on the Ti/CFRP with different thermal cycling treatment.

SEM images of the Ti/CFRP after thermal cycling (Fig.5b to Fig. 5e) exhibit no significant variation in comparison with the original one (Fig. 5a). The titanium-prepreg interfaces stay unbroken and continuous after thermal cycling. Moreover, higher magnification image indicates that no visible micro-crack occurs after thermal fatigue for 1000 cycles as shown in Figure 5f.

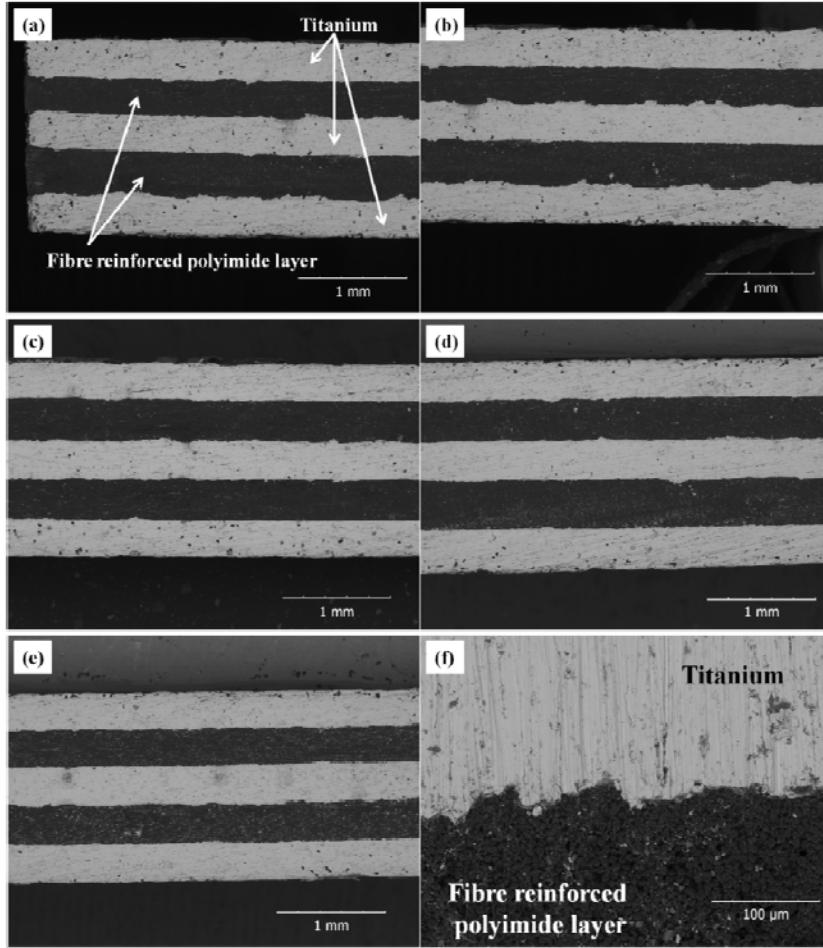


Fig. 5.Cross-sectional SEM pictures of the specimens after thermal cycling

- (a) original specimen $\times 50$ (b) thermal cycled for 250 cycles $\times 50$
- (c) thermal cycled for 500 cycles $\times 50$ (d) thermal cycled for 750 cycles $\times 50$
- (e) thermal cycled for 1000 cycles $\times 50$ (f) thermal cycled for 1000 cycles $\times 500$.

3.3Perforation resistance and impact responses

The detail information of the samples used in these tests is summarized in Table 1.

Table 1 Detail information of the Ti/CFRP

Code	Configuration	Thickness (mm)	Areal density (g/cm ²)
FML-3/2	[Ti/0/90/Ti/90/0/Ti]	1.22	4.47
FML-4/3	[Ti/0/90/Ti/90/0/Ti/90/0/Ti]	1.75	6.14
FML-5/4	[Ti/0/90/Ti/90/0/Ti/0/90/Ti/90/0/Ti]	2.35	7.95

Figure 6 shows typical load-displacement traces following quasi-static indentation on Ti/CFRP with different configurations. The sample starts to fracture when the indentor comes into contact with the sample. The load force increases with the displacement extending, before a tensile crack appears on the lower surface and the

load starts to reduce. The load gradually drops to zero as the indentor perforate the samples. It is observed that the maximum load increasing rapidly with the laminate plies increases.

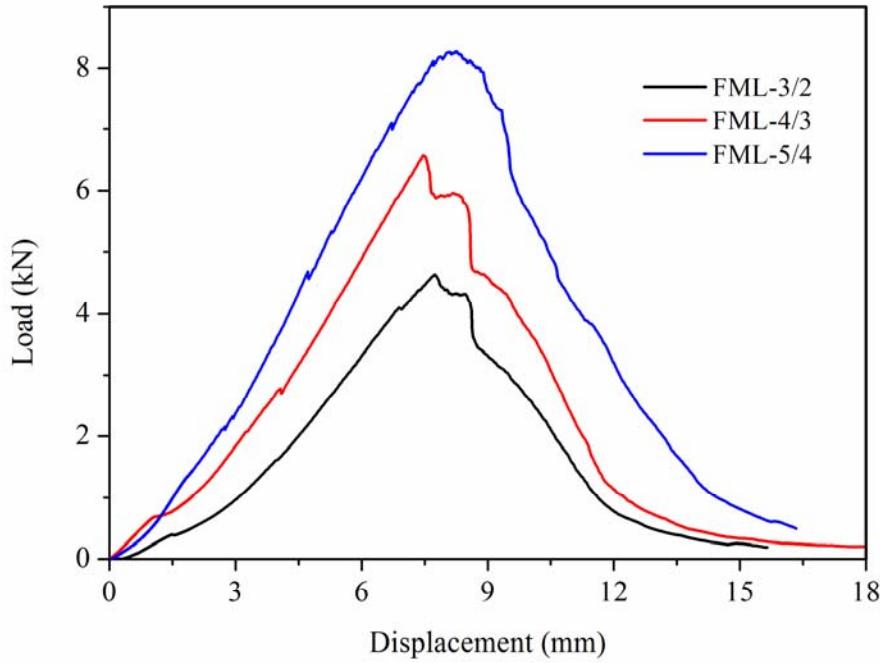


Fig. 6.Typical load-displacement curves following quasi-static tests

The energy absorbed by the Ti/CFRP during the perforation process can be determined by means of the areas under the various load-displacement curves. Figure 7 presents the relationship between the absorbed energy and the thickness of the Ti/CFRP. It is clearly that the energy absorbed by the laminates increases linearly with the thickness expended. The relationship can be concluded as equation 1, which can be used to predict the energy absorption property of the Ti/CFRP with different configuration. Here, E is the energy absorbed by the sample during the perforation process, h is the thickness of the sample.

$$E = 30.13 \times h - 10.43 \quad (\text{Eq.1})$$

The detail tests results presented in Table 2 indicate that with the increase of the total plies of the Ti/CFRP, the metal volumefraction decreases rapidly while the specific energy absorption (SEA) increases as expected.

Table 2Detail results of the impact tests

Code	Thickness (mm)	Metal Volume Fraction(%)	Energy(J)	SEA($\text{J cm}^2/\text{g}$)
FML-3/2	1.22	73	27.1	6.06
FML-4/3	1.75	68	40.86	6.65
FML-5/4	2.35	64	61.06	7.67

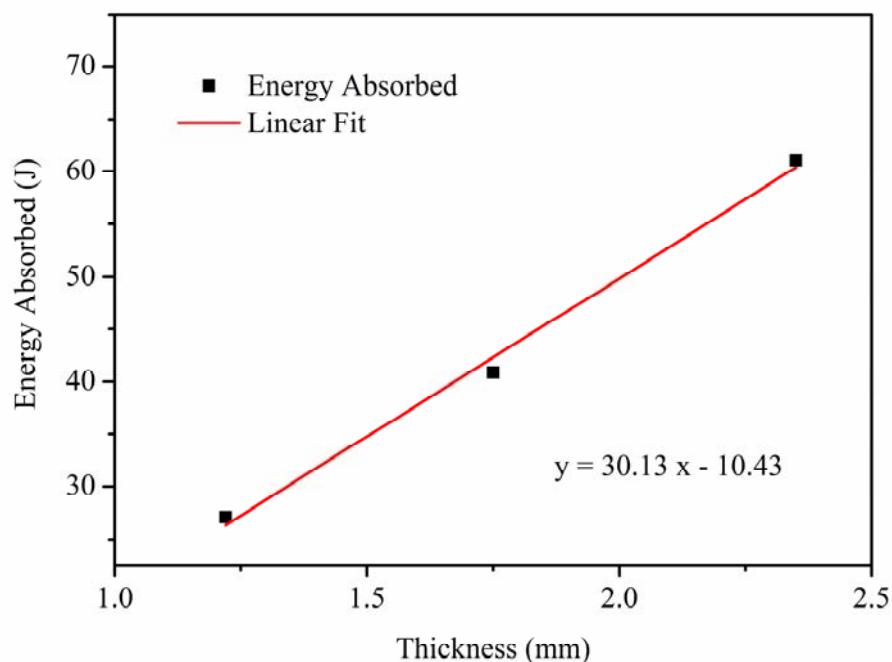


Fig. 7.Energy absorbed during quasi-static tests as a function of the thickness

The impact tests were performed on 3/2 configurationTi/CFRP under various impact energy. The load vs time curves presented in Fig.8 indicate that the load increases when the impactor gets in touch with the samples and then decreases after reaching the peak. It is also observed that the peak load of the impact test is higher when using higher impact energy. The peak load of the impact process reaches a platform when the impact energy is higher than 25J. The samples exhibit various failure modes after impact with different energy, as shown in Fig.9.Only local deformation can be found on the surface of the Ti/CFRP when suffering 5J and 15J impact. With the increase of the energy, obvious failure can be observed on the

Ti/CFRP, including interlaminar debonding and metal breakage. The Ti/CFRP can absorb energy during the impact process with these failures.

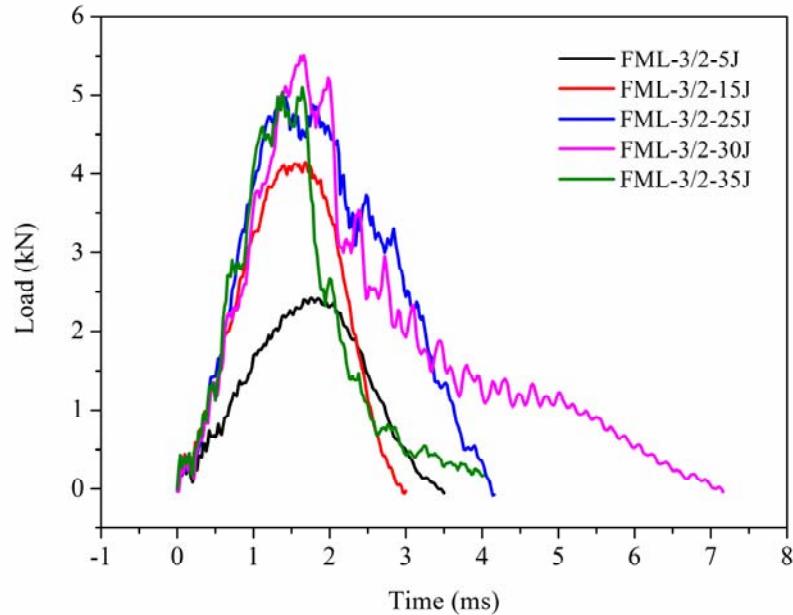


Fig. 8.Load vs time curves of the Ti/CFRP during the impact tests

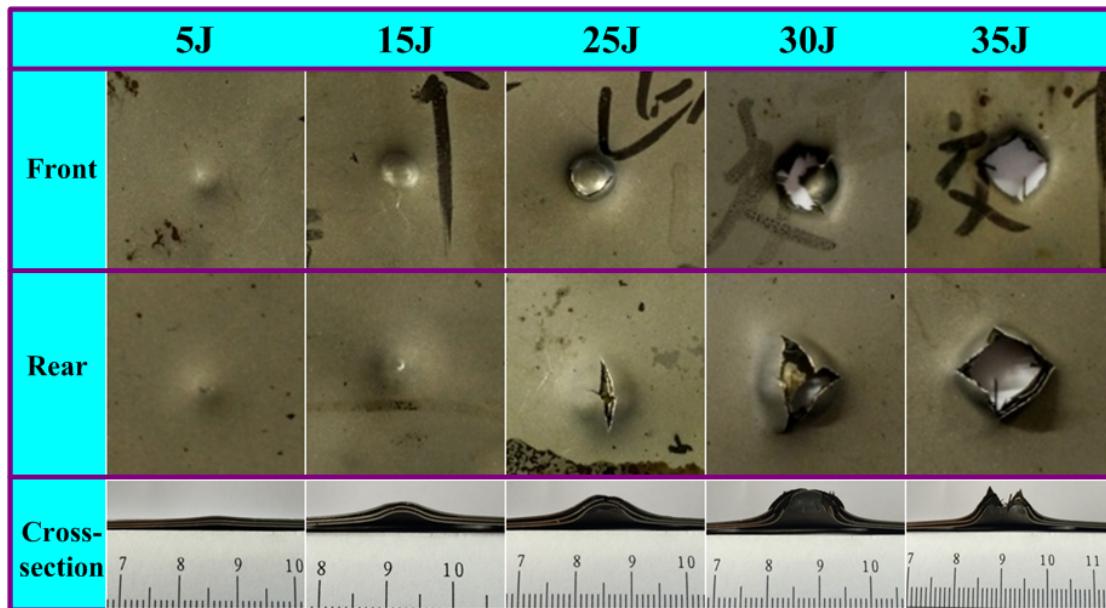


Fig. 9.Failure mode of the Ti/CFRP after impact

4. Conclusions

Within the limitations of this study, the following conclusions were drawn:

1. Tensile and ILSS properties of the Ti/CFRP were decreased with the increasing temperature. The conservation rates of tensile and ILSS under 300 °C were approximately 43% and 53% comparing with room temperature.
2. Tensile and ILSS properties were not reduced after the thermal cycling treatment. The mechanical characterizes of the constitute material of the Ti/CFRP, as well as the interface bonding, were not affected by the thermal cycling treatment.
3. The relationship between the absorbed energy and the thickness of the Ti/CFRP during the perforation process was expressed as: $E = 30.13 \times h - 10.43$. With the increase of the total plies of the Ti/CFRP, the metal volume fraction decreased rapidly and the specific energy absorption (SEA) increased.

References

- [1] Rans C D, Alderliesten R C, Benedictus R. Predicting the influence of temperature on fatigue crack propagation in Fibre Metal Laminates[J]. Engineering Fracture Mechanics, 2011, 78(10): 2193-2201.
- [2] Hu Y B, Li H G, Cai L, et al. Preparation and properties of Fibre-Metal Laminates based on carbon fibre reinforced PMR polyimide[J]. Composites Part B: Engineering, 2015, 69: 587-591.
- [3] Abouhamzeh M, Sinke J, Benedictus R. Investigation of curing effects on distortion of fibre metal laminates[J]. Composite Structures, 2015, 122: 546-552.
- [4] Asaee Z, Shadlou S, Taheri F. Low-velocity impact response of fiberglass/magnesium FMLS with a new 3D fiberglass fabric[J]. Composite Structures, 2015, 122: 155-165.
- [5] Cortes P, Cantwell W J. The tensile and fatigue properties of carbon fiber-reinforced PEEK-titanium fiber-metal laminates[J]. Journal of reinforced plastics and composites, 2004,

23(15): 1615-1623.

- [6] Yoonessi M, Shi Y, Scheiman D A, et al. Graphene polyimide nanocomposites; thermal, mechanical, and high-temperature shape memory effect. ACS Nano2012, 6(9): 7644-7655.