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Short Communication

A novel stress-relaxing approach based on forming Fe–Ni invar effect interlayer for joining C/C composites

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ABSTRACT

Taking advantage of the invar effect of Fe–Ni alloy at low temperatures, a novel stress-relaxing approach based on forming Fe–Ni invar interlayer was proposed for joining carbon fiber reinforced carbon (C/C) composites in this paper. Mixed Fe + Ni62Ti38 (at %) powders were used as the filler materials. Microstructure, mechanical property, and formation mechanism of the bonded joints were investigated. The results indicate that, based on the eutectic reaction of (Fe,Ni)–C at the C/C/interlayer interface and the in-situ reaction of Ti–C in the interlayer, a (Fe,Ni)–C eutectic reaction layer was formed at the C/C side interface and the Fe–Ni invar effect interlayer reinforced by TiC particles was obtained. Owing to the low thermal expansion coefficient and good plasticity of the Fe–Ni alloy matrix at low temperatures, the high residual thermal stress of the joint was effectively reduced and the shear strength of the joint was improved. With the increase of Ni content in the Fe–Ni alloy matrix of the interlayer, the shear strength of the joint decreases gradually. When the composition of the Fe–Ni matrix was designed to be Fe–36Ni, i.e., the invar alloy with the lowest thermal expansion coefficient at 100 °C, the average shear strength of the bonded joint reached the highest value of 36 MPa. The fracture of the joint occurred in the (Fe,Ni)–C eutectic reaction layer close to the C/C composite.

1. Introduction

The demand for carbon fiber reinforced carbon (C/C) composites in aerospace field has greatly increased because of their low density, high temperature and thermal shock resistance, and low coefficient of thermal expansion (CTE). They are promising new-type high-temperature structural materials in the manufacture of aerospace parts, such as combustion chamber components, rocket nozzles and thermal protection structures of hypersonic aircraft [1,2]. However, the machinability of C/C composites is poor, so it is required by joining to fabricate large size or complex shape parts in practical applications. As the developing of C/C composites, the joining of C/C composites is becoming increasingly important for their wider use in aerospace [3,4].

As for the joining of C/C composites, numerous strategies have been proposed to the present, including adhesive

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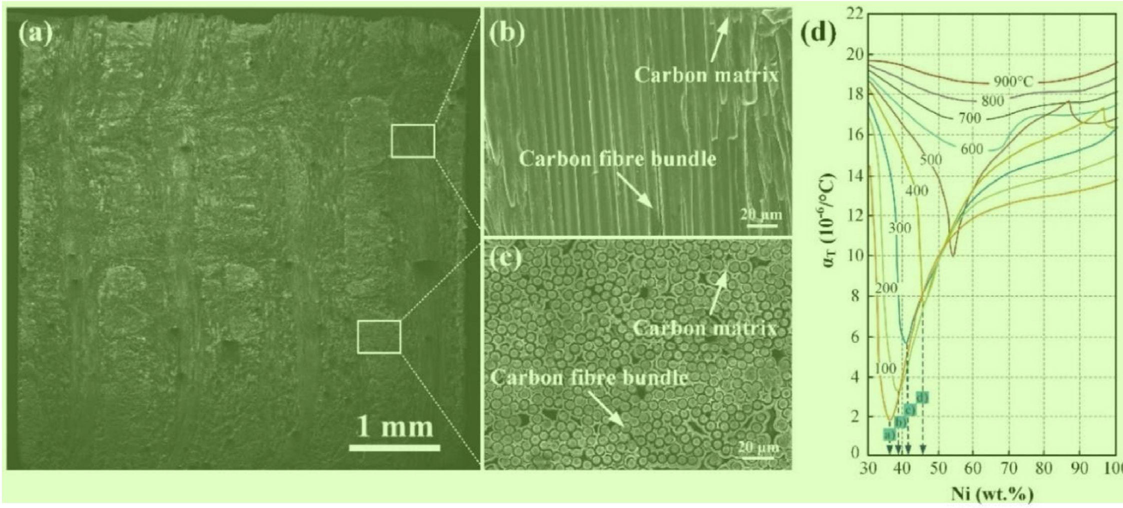

Fig. 1 – (a) Macrostructure of the C/C composite; (b,c) microstructures of the C/C composite; and (d) CTE isotherm of Fe–Ni alloy [24].

bonding [5,6], reaction bonding [7,8], active brazing [9–12], and diffusion brazing [13,14]. Due to their pressure-free, simple-process, and good-adaptability to joint structures, the brazing-type methods are considered to be the most suitable methods for joining C/C composites. However, the mechanical properties of the brazed C/C joints are generally poor, and rarely meet the requirements of practical applications.

For instance, Zhang et al. [15] performed the brazing of a C/C composite using five kinds of Fe-based active brazing alloys, pure iron, mild steel, 4J36 alloy, stainless steels and SS630 alloy. They reported that the joint brazed with SS630 alloy filler exhibited the lowest contact angle and the best shear strength 16 ± 1 MPa. Xu et al. [16] achieved the brazing of C/C composites successfully using $AlCoCrFeNi_{2.1}$ high-entropy alloys filler, and investigated the microstructure and shear strength of the joints brazed under different processing parameters. They pointed out that the highest shear strength of the joint reached 21.9 MPa when brazed at 1440 °C for 10 min. Furthermore, Yi et al. [17] brazed the C/C composites with Ag–Cu–Ti and Ni–Cr–P–Ti pasty brazing filler, and the highest shear strengths of the joints was measured to be 26.71 and 34.02 MPa, respectively. Their results indicated that except for interface brittle compounds, the high residual stress caused by the difference in CTE between the C/C substrate and the interlayer is the main reasons for the low joint strength.

According to the existing studies, a major factor that affects the joint performance in the brazing of C/C composites is the high residual thermal stress in the joint. To solve this problem, composite brazing is a mainstream method at present. Relevant research results show that the composite brazing reduces the CTE of the interlayer by adding some reinforcing particles with low CTE (such as TiC [18], graphene sponge [19], β-$LiAlSiO_4$ [20], CNT_s [21]) into the common brazing filler to match the difference in CTE between the interlayer and the C/C substrates, and thus alleviates the high residual stress in the joint and improve the joint strength. However, it is limited to reduce the CTE of the interlayer just by adding reinforcements. Adding too much reinforcing phase will reduce the plastic deformation ability of the interlayer, especially at low temperatures. It is not conducive to relax the joint thermal stress furtherly. Moreover, due to the restriction of low melting point of the common brazing fillers, it is difficult to raise the heat-resistance of the joint by composite brazing.

It is widely known that Fe–Ni invar alloy has extremely low CTE and good plasticity in the low temperature range from Curie temperature to room temperature (RT) [22,23]. Taking advantage of the invar effect of Fe–Ni alloy at low temperatures, in this paper, a novel stress-relaxing approach based on forming Fe–Ni invar alloy interlayer was proposed for joining C/C composites. By using mixed Fe + Ni62Ti38 (at %) powder as the filler material, a Fe–Ni invar interlayer reinforced by TiC particles would be obtained via the Ti–C reaction between the filler and C/C substrates, thus achieving the low-stress joining of C/C composites. Meanwhile, the high melting temperature of the Fe–Ni invar interlayer ensured the high heat resistance of the bonded joint. Microstructure and formation mechanism of the joints were investigated. Mechanical property and fracture behavior of the joints bonded under various process conditions were discussed.

2. Experimental procedures

The C/C composite used was of two-dimensional (2D) and three-dimensional (3D) punctured structures, which was prepared by thermal gradient chemical vapor infiltration (CVI). It was provided by National University of Defense Technology. Macrostructure and microstructure of the C/C composite are as shown in Fig. 1(a–c). The density of the C/C composite is 1.65–1.78 g/cm^3, and the CTE is 0–2 × 10^{-6} K^{-1}.

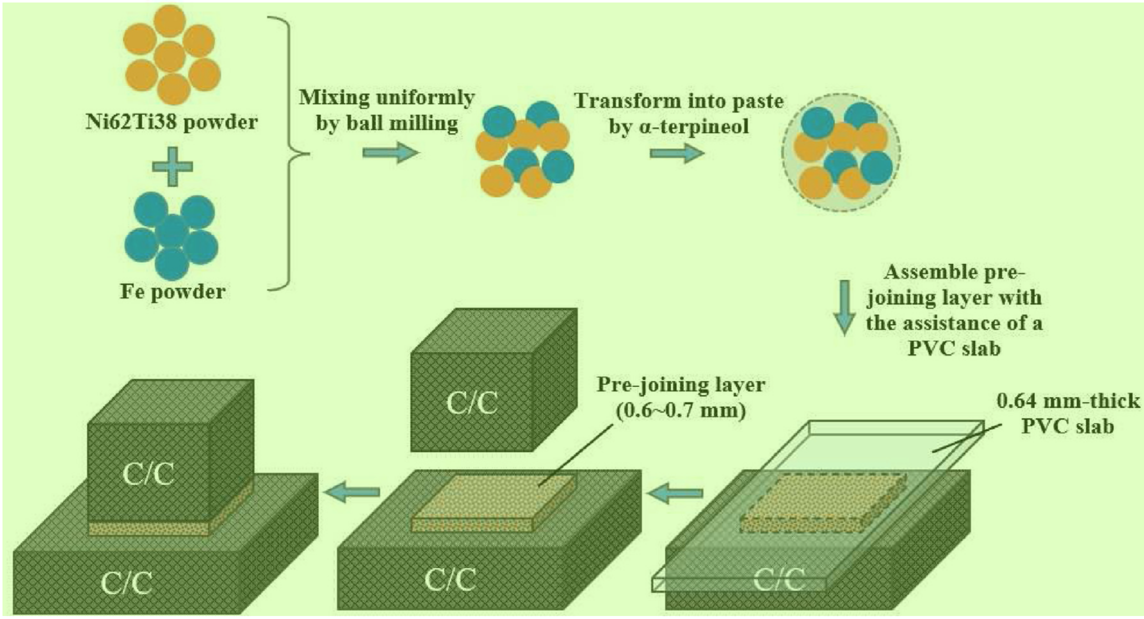

Fig. 2 – Schematic diagram of the experiment procedures.

The raw composite was cut into 5 × 5 × 4 mm^3 and 10 × 10 × 4 mm^3 pieces by wire cutting. The bonding surfaces were polished with 240-mesh abrasive papers. All the pieces were ultrasonically cleaned with ethanol for 10 min and dried below 50 °C before joining.

The filler materials were mechanically mixed by Fe powder (2–3 μm) and Ni62Ti38 (at %) alloy powder (20–30 μm). The Fe powder was provided by Hebei Yanchuang Advanced Materials Technology Co., Ltd, and the Ni62Ti38 alloy powder was prepared by Xipuman Additive Technology (Ningxia) Co., Ltd. There are two bases for choosing Fe + Ni62Ti38 as the filler material. First, by using a mixture of two different powders containing Fe and Ni elements respectively as the original materials, it is convenient to mix the Fe–Ni invar interlayers with the desired compositions. Second, Ni62Ti38 alloy powder not only has a suitable Ti content, but also has a lower melting point compared to Ni and Ti pure metal powders. By using Ni62Ti38 powder as a part of the filler material, Ni–Ti liquid would be obtained at a lower temperature, promoting the brazing of C/C composites. The two powders were weighed according to the mass ratio of Fe/Ni 36/64, 39/61, 42/58 and 46/54 to obtain the Fe–Ni invar interlayer with the Ni content of 36%, 39%, 42% and 46%, i.e., the Ni content corresponding to the Fe–Ni alloy with the lowest CTE at 100, 200, 300 and 400 °C respectively, as shown in Fig. 1 (d). For convenience, the filler materials with various Fe/Ni ratios are represented by Ni36, Ni39, Ni42, and Ni46.

As shown in Fig. 2, the mixed powders were transformed into a paste by α-terpineol and then assembled between the C/C pieces to form a pre-interlayer. The thickness of the pre-interlayer was kept at 0.6–0.7 mm with the assistance of polyvinyl chloride (PVC) slab with the fixed thickness of 0.64 mm. The function of the PVC slab was to ensure that the thickness of the interlayer consistent before joining. The joining was carried out at 1350 °C for 10–40 min in a vacuum brazing furnace. The vacuum was kept below 5 × 10^{-3} Pa. After joining, the joints were cooled to RT inside the furnace.

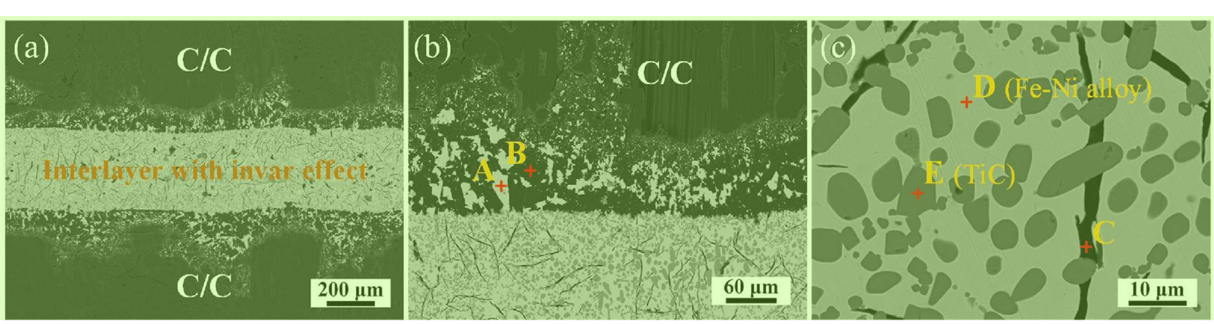

Fig. 3 – Microstructure of the joint brazed with Ni36 filler material at 1350 °C for 30min: (a) whole structure of the joint; (b) interface of C/C composite; (c) magnification image of the interlayer.

The microstructures of the bonded joints were observed by a field emission scanning electron microscopy (FE-SEM) equipped with energy dispersive X-ray spectrometry (EDS). The mechanical properties of the joints were determined by shear strength testing at room temperature. The testing process was the same as that in Wang et al. [18]. To ensure the results were reliable, at least three samples were used for mechanical characterization.

3. Results and discussions

3.1. Microstructure analyses of the bonded joints

Fig. 3 presents the typical microstructure of the joint obtained by using Ni36 filler material at 1350 °C for 30min. Fig. 3a shows the overall structure of the joint, and Fig. 3b and c presents the enlarged views of the C/C/interlayer interface and the interlayer, respectively. The EDS results on different phases in the joint are provided in Table 1.

As shown in Fig. 3a, a compact interlayer with thickness of about 340 μm was formed between the C/C substrates, and a transition layer consisting of bright white phase (A) and black phase (B) was formed between the interlayer and the C/C composite. As shown in Fig. 3b, the thickness of the transition layer is about 104 μm. According to the EDS results, the white phase (A) mainly contains Fe and Ni, and the black phase (B) is mainly composed of C element. It is inferred that the transition layer was formed by the eutectic reaction of Fe–C and Ni–C during brazing. The reaction equations are as follows:

$$Fe_s + C_s \leftrightarrow (Fe,C)_l \tag{1}$$

$$Ni_s + C_s \leftrightarrow (Ni,C)_l \tag{2}$$

It can be seen from the picture that the eutectic reaction is violent. There is no carbide layer at the C/C/interlayer interface as formed in common active brazing [25,26].

As shown in Fig. 3c, there are three phases in the interlayer which were represented by C, D and E, respectively. The fine gray particles (E) and the black banded phase (C) are uniformly distributed in the white matrix phase (D). According to the EDS results, the white matrix is FeNi36 invar alloy, which contains a small amount of Ti and C. The fine gray particles are mainly composed of Ti and C, and the Ti/C atom ratio is close to 1/1, so they are inferred to be TiC particles. The formation of TiC particles indicates that C atoms in the C/C composite diffused into the interlayer during the joining process and reacted with Ti in the brazing filler. The black banded phase mainly contains C element, which was formed by the precipitation of the C element dissolved in the Fe–Ni alloy during cooling.

Something should be pointed out is that the Ni–Ti binary system under equilibrium conditions has three stable intermetallic compounds of Ti_2Ni, TiNi, and $TiNi_3$ [27,28], and these compounds typically have poor plasticity. In the joining of high thermal-expansion mismatched C/C composites and metals, the formation of these compounds in the interlayer is detrimental to the joint strength. Although the filler material in the present work contained Ni62Ti38 (at %) alloy powder, however, almost all of the Ti atoms in the filler material reacted with the C atoms that diffused into the interlayer from the C/C substrates to form TiC. Therefore, there was no Ni–Ti compound formed in the interlayer during the joining process.

Table 1 – Chemical compositions of the phases in the joint (at %).

Position	Fe	Ni	Ti	C	Possible phase
A	49.71	27.43	–	22.87	$(Fe,Ni)_s$
B	0.08	–	–	99.92	C
C	0.27	0.11	–	99.62	C
D	49.91	27.89	0.73	21.47	$(Fe,Ni)_s$
E	0.61	–	47.23	52.17	TiC
F	–	–	–	100	C fiber
G	0.12	–	–	99.88	C
H	37.15	20.28	–	42.57	$(Fe,Ni)_s$

Fig. 4 presents the microstructures of the joints brazed with various filler materials at 1350 °C for 30 min. There are some differences in the microstructures of the joints with different Fe/Ni ratios. When using Ni36 filler material, as shown in Fig. 4 (a), the joint is uniform and compact, without obvious defects such as voids and cracks. As the Ni content increases, as shown in Fig. 4(b–d), a few voids appear in the center of the interlayer. When the proportion of Ni62Ti38 powder in the filler material increased, the flowability and filling ability of the filler decreases at joining temperature, leading to the formation of voids in the joint. It can be seen from the pictures that as the proportion of Ni increases, the thickness of the transition layer at the interface between the C/C composites and the interlayer decreases. Adequate interface reaction would be beneficial for improving joint strength. The results indicate that the matrix compositions of the obtained interlayers are basically consistent with the designed compositions.

The above results show that the interlayer with invar effect was successfully obtained by using mixed Fe + Ni62Ti38 powder as the filler to join the C/C composites. During the cooling process of the joint from the joining temperature to the Curie temperature, the TiC particles and precipitated C contribute to relieve the CTE of the interlayer, relieving the thermal stress of the joint generated at this stage. During the cooling process from the Curie temperature to the RT, the Fe–Ni invar alloy matrix would help to reduce the thermal stress generated at the low-temperature stage.

3.2. Mechanical property and fracture behavior of the joints

Fig. 5 shows the average RT shear strength of the joints bonded by using the filler materials with different Ni contents. The results indicate that with the increase of Ni content in the Fe–Ni alloy matrix of the interlayer, the shear strength of the joint decreases gradually. When Ni36 filler was used, the shear strength of the joint reached the maximum value, 36 MPa. The reason might be that the CTE of the Ni36 interlayer is the lowest at low temperature (100 °C) and the effect of stress-relaxing by the invar interlayer is most obvious. With the increase of Ni content in the Fe–Ni invar matrix, the CTE of the interlayer increases. In this case, the residual thermal stress of

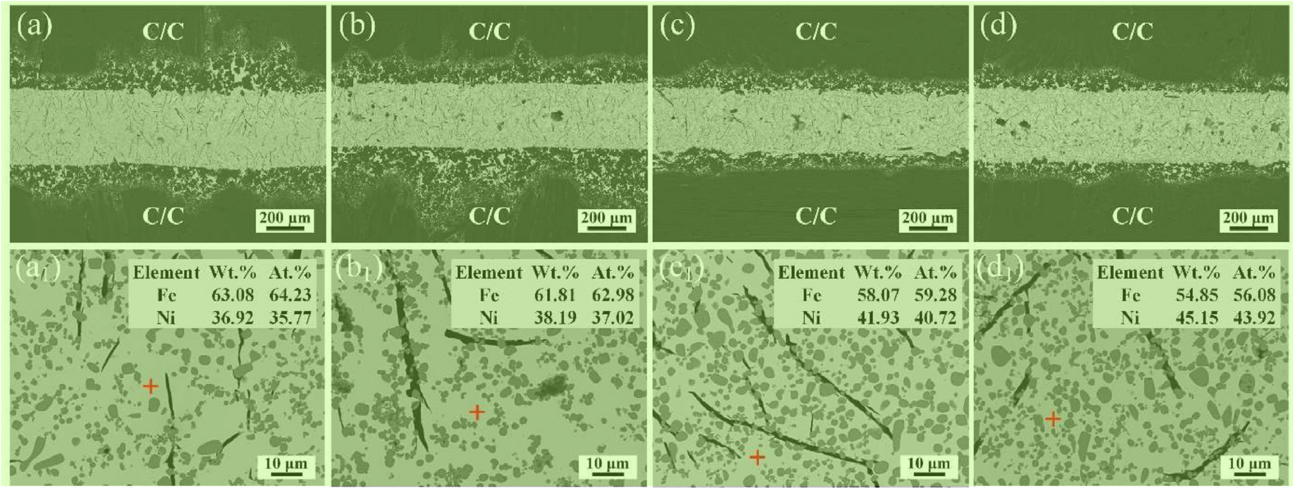

Fig. 4 – Microstructures of the joints and corresponding interlayers with different Fe/Ni ratios: (a, a_1) Ni36; (b, b_1) Ni39; (c, c_1) Ni42; (d, d_1) Ni46.

the joint is relatively high, so the joint strength becomes lower.

To further analyze the effect of Ni content in the Fe–Ni invar interlayers on the residual thermal stress, a finite element method was applied. The whole simulation was modeled using ANSYS 15.0. The mechanical properties of the Fe–36Ni, Fe–39Ni, Fe–42Ni, and Fe–46Ni alloys are calculated via JMate Pro software. According to the material composite theory [29], the elastic modulus and CTE of the TiC particle reinforced Fe–Ni matrix composite interlayers can be calculated by Eqs. (3) and (4).

$$E_C = E_M\left[1 + \frac{f(E_I - E_M)}{E_M + S(1-f)(E_I - E_M)}\right] \tag{3}$$

$$\alpha_c = \alpha_M - \frac{fE_I}{(E_M - E_I)[S - f(S-1)] - E_M}(\alpha_I - \alpha_M) \tag{4}$$

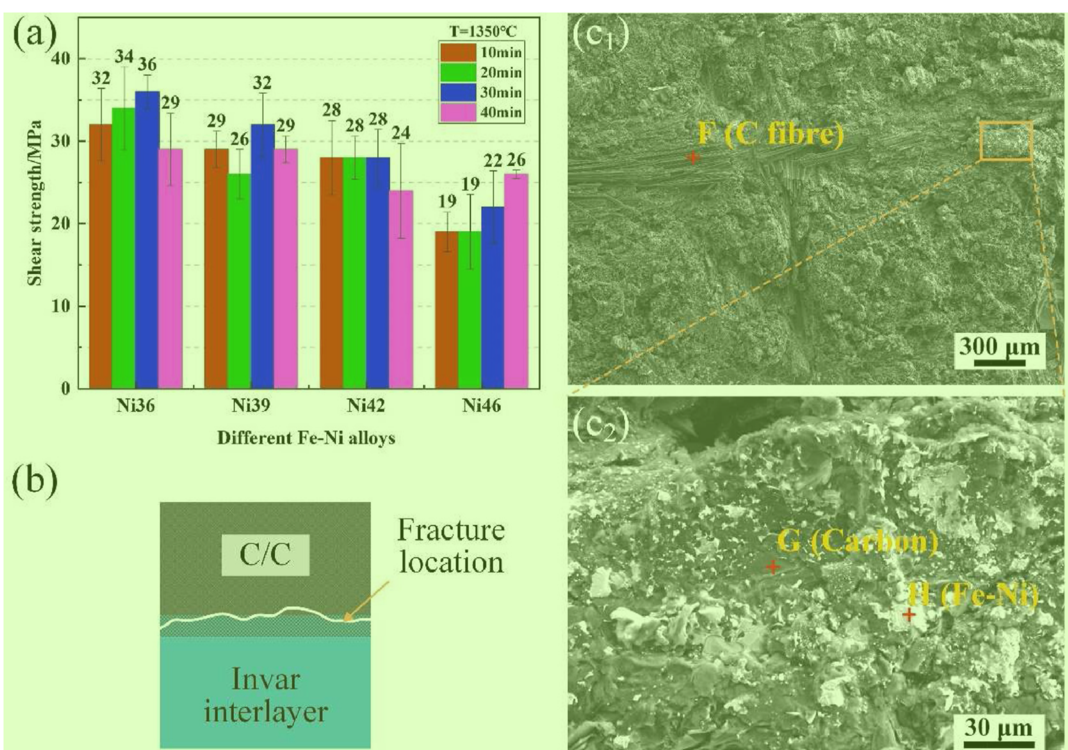

Fig. 5 – (a) shear strength of the joint with various invar interlayer; (b) fracture location of the joint; (c_{1-2}) fracture surface of the interlayer side.

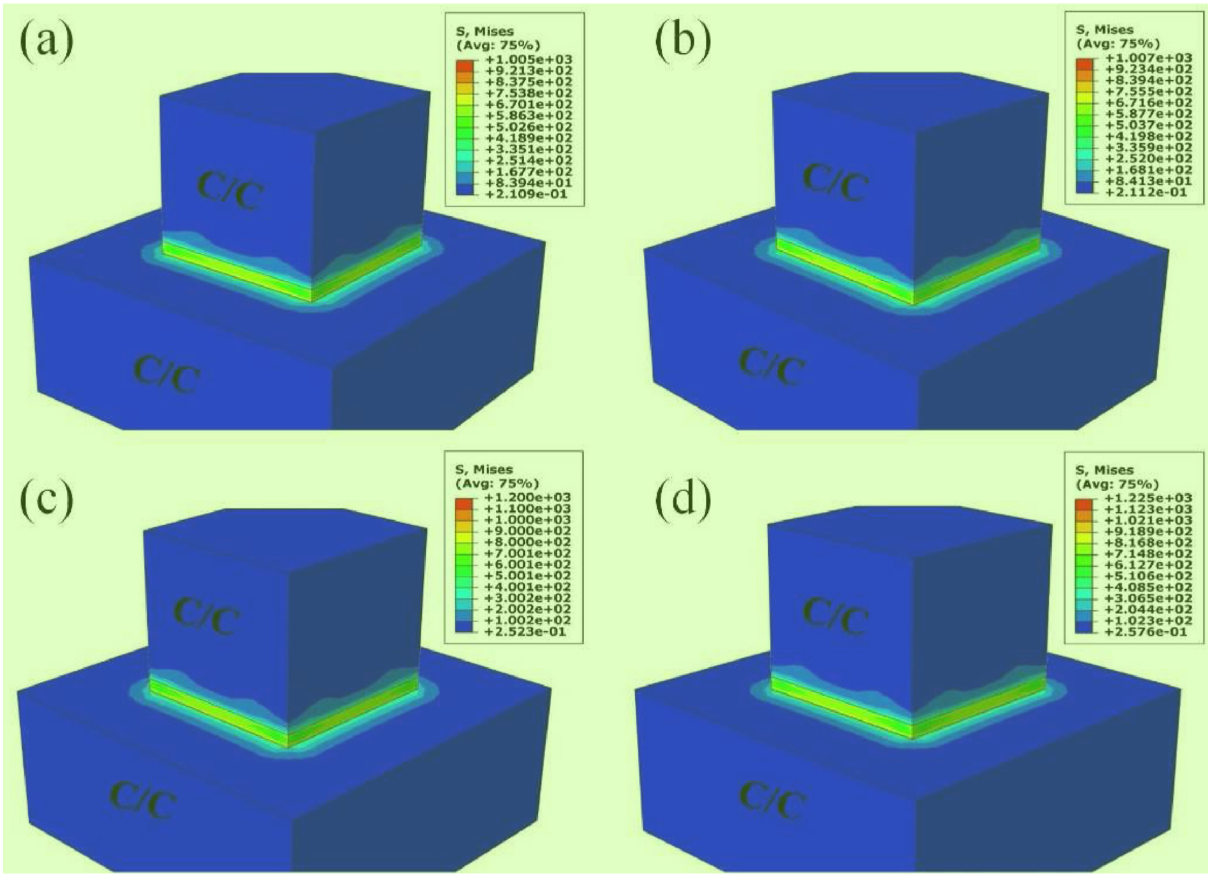

Fig. 6 – Contour plots of the computed von Mises stress in the joints with different invar interlayers: (a) Ni36; (b) Ni39; (c) Ni42; (d) Ni46.

in which, E_C, E_M, and E_1 are the elastic modulus of the composite interlayer, interlayer matrix, and TiC reinforcements; α_C, α_M and α_1 are the CTE of the composite interlayer, interlayer matrix, and TiC reinforcements; f is the volume fraction of the TiC reinforcements, and $S=(8-10\nu)/15(1-\nu)$, ν is the elastic modulus of the interlayer matrix.

Fig. 6 presents the computed contour plots of von Mises stress in the joints with different invar interlayers. The von Mises stress is a comprehensive manifestation of the residual thermal stress in all directions. The greater the von Mises stress, the higher the residual thermal stress in the joints. It is obvious that with the decrease of Ni content in the invar interlayer from 46% to 36%, the peak value of von Mises stress in the joint decreases from 1225 to 1005 MPa, a decreased of 18%. Therefore, when the matrix composition of the Fe–Ni invar interlayer was designed to be Fe–36Ni, the residual thermal stress was lowest and the shear strength of the obtained joint reached the highest.

The fracture characteristics of the joint after shear strength testing are shown in Fig. 5b and c. Elemental analysis at the positions marked in Fig. 5c_1 and 5c_2 are provided in Table 1. The results show that the fracture is uneven. Both the bright white phase (as marked by H in Fig. 5c_2) and some C fibers (as marked by F in Fig. 5c_1) can be observed from the fracture surface. It can be concluded that the fracture of the joint mainly occurred in the transient layer, i.e. the (Fe,Ni)–C eutectic reaction layer, and sometimes extended to the interior of the C/C composite. Under loading condition, the crack propagation in the transient layer could be hindered by the (Fe,Ni) metal phase and the fracture energy would be increased. This is beneficial to improve the joint strength.

3.3. *Formation mechanism of the joint*

The above results uncover that the joining process of C/C composite based on forming Fe–Ni invar interlayer to reduce the high residual stress can be sub-divided into the following four stages as shown in Fig. 7.

Stage I During the heating process, the filler material is subjected to the vacuum and high temperature, the plasticity of the Fe and Ni62Ti38 powders enhance, and the activity of elements increase. The powders are in close contact with each other, and interdiffusion take place. Because this stage is performed in solid state, the diffusion of atoms is slight.

Stage II When the temperature rises to melting temperature of the Ni62Ti38 alloy (1120 °C), the Ni62Ti38 powder melts and fills the interspace of the joint by capillarity. As the temperature continues to rise to the

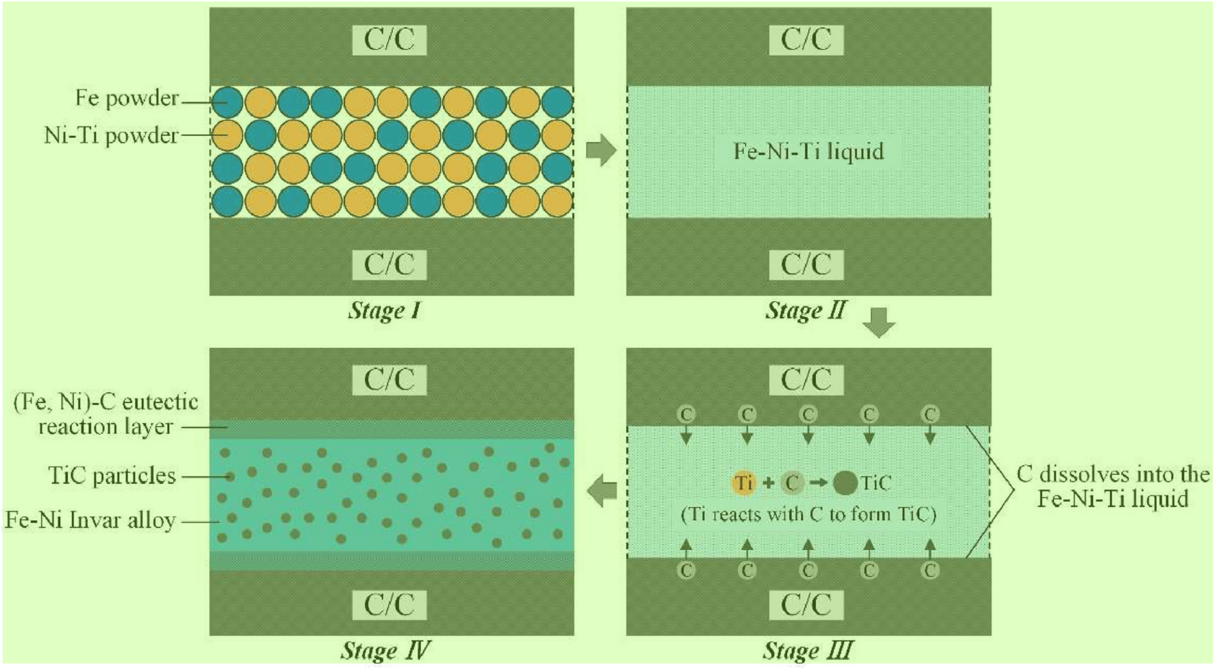

Fig. 7 – Conceptual model of the joining process of C/C composite by forming Fe–Ni invar interlayer to reduce the high residual stress.

joining temperature (1350 °C), the Ni–Ti liquid dissolves the Fe particles and forms the Fe–Ni–Ti liquid.

Stage III The main characteristics of this stage are (Fe,Ni)/C eutectic reaction and Ti/C in-situ reaction. During the course of heat preservation at the joining temperature, eutectic reaction between (Fe,Ni) and C happened at the C/C/interlayer interface, meanwhile in-situ reaction between Ti and C atoms occurred in the interlayer. The C atoms in the Fe–Ni–Ti liquid come from the C/C composite.

Stage IV As the reactions proceed, the (Fe,Ni)–C eutectic reaction layer (i.e., the transient layer) is formed at the interface between the C/C composite and the interlayer, and the Ti in the Fe–Ni–Ti liquid would be completely reacted with C to form TiC reinforcing particles. Lastly, the Fe–Ni invar interlayer is formed. The joining process is dominated by the (Fe,Ni)/C eutectic reaction and the Ti/C in-situ reaction.

4. Conclusions

(1) A novel stress-relaxing strategy based on forming Fe–Ni invar effect interlayer was proposed for joining carbon fiber reinforced carbon (C/C) composites.
(2) By using mixed Fe + Ni62Ti38 (at %) powder as filler material, the novel joining of C/C composites has been realized. A Fe–Ni invar interlayer reinforced by TiC particles and a (Fe,Ni)–C eutectic reaction layer was formed at the interface were obtained.
(3) Due to the Fe–Ni invar interlayer has very low coefficient of thermal expansion (CTE) and good plasticity at low temperatures, the residual thermal stress in the joint was effectively reduced and the joint strength was improved. The shear strength of the joint decreases with the increase of Ni content in the matrix of the Fe–Ni invar interlayer.
(4) When the matrix composition of the invar interlayer was designed to be Fe–36Ni, i.e., the invar alloy with the lowest CTE at low temperature (100 °C), the highest average shear strength of the joint was measured to be 36 MPa. The fracture occurred in the (Fe,Ni)–C eutectic reaction layer at the C/C side interface.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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