Microstructure and mechanical properties of Al/Fe micro-laminated composites fabricated by ultrasonic consolidation

Y Wang, F M Hou, Q Yang, Z Y Fan, H Y Pan and H Xu

School of Materials Science and Engineering, North University of China, Taiyuan 030051, China

Email:xh725@263.net

Abstract. Ultrasonic consolidation technology is a new method for preparing high-performance laminate composite materials. This rapid consolidation additive manufacturing technology provides a new idea for the integration of complementary functional laminated composite structures. It has great potential for preparation of new generation of complex laminated composite parts, broadening the application field of laminated composite material, and means an advanced 3D additive manufacturing technology for materials and structures. In this paper, it is aimed at Al/carbon steel and Al/Fe laminated composites systems with low cost and multilayer structures, Al/carbon steel and Al/Fe laminated composite samples were consolidated by ultrasonic consolidation process. And the Al/carbon steel and Al/Fe interfacial bonding were studied by SEM and EDS. The hardness and tensile properties of laminated composites were tested by mechanical properties together with the fracture morphology of each interface was studied.

1. Introduction

With the improvement of anti-armor weapons and damage effect, the advanced material single high strength alloy steel for protective armor applied in the upgrading of weapons is gradually replaced by laminated composites with stronger anti-penetration capability. The main purpose is to combine the advantages of two or more materials to improve defects in some aspects of physical and mechanical properties of single material, such as improving strength and increasing toughness and so on [1]. The soft and hard gradient combination of the layered structure with large difference can improve the bonding strength of the composite, which can effectively reduce the compression shear damage and tensile failure of the impact load, and reduce the impact energy of the high intensity, reduce the impact energy of the high-intensity, short-duration secondary pulse load on the structure, in order to make laminated composite as the protective structure can effectively improve the ability to resist the secondary damage of the shock wave.

Through high frequency vibration friction, on the one hand, ultrasonic consolidation cleans up the consolidated interface of the layered metal foil, on the other hand, the atoms at the metal interface are also activated by the ultrasonic energy and pressure instantaneously, under the condition of limited temperature rise and plastic deformation, the metal bond bonding and diffusion of the consolidated workpiece is formed, and a solid phase connection is formed [2-6], the schematic diagram of the ultrasonic consolidation forming mechanism is shown in figure 1. Ultrasonic consolidation technology is not limited by the performance of materials and has wide scope of consolidation, this low
It has been studied to combine the strength of composites with hard and soft gradients of Fe and Al with large difference in yield strength, and they have good protection effect against explosion [18-19]. Rest et al [20] and Lee et al [21] prepared Fe-Al composites by friction stir welding, a micron-sized Fe-Al intermetallic compound was formed at the interface, and the fracture was found to be located on the side of the Al. Lin et al [22] studied the brazability of Fe-Al by adding Al-Cu solder under the protection of aluminum steel tungsten inert gas, and generated Fe₄Al₃ intermetallic compound with a thickness of about 3-5 nm at the interface. Sierrs et al [23] and Peyre et al [24] studied the weldability of Fe-Al dissimilar materials by laser deep penetration welding, and generated intermetallic compounds mainly composed of Fe₂Al₅ phase at the interface between the fusion zone and the steel. The hardness of Fe₂Al₅ is as high as 1200HV, but many cracks appear at the joint, which seriously affects the mechanical properties of the joint. The maximum joint strength is only 90MPa. The above preparation process of the Fe-Al laminated composites belongs to the metallurgical bonding technology, but it is easy to break at the joint, the addition of the solder process and the joint performance are relatively low. Ultrasonic consolidation technology has unique application in industrial production as a solid phase connection method, and the low temperature forming process can form a solid solid-state bonding, which also greatly reduces the formation of intermetallic compounds and improves the interface joint strength, such as Patel et al [25] used this technology to weld Pb and galvanized steel plate, It was found that there were Al-Zn eutectic reaction layer and Fe-Al intermetallic compound layer at the Al/steel interface, and the maximum joint strength reached 3.7KN. However, there are few studies on the use of ultrasonic consolidation for Al-Fe composites with large differences and good shock resistance. Therefore, in this paper, it is aimed at Al/carbon steel and Al/Fe laminated composite systems with low cost and multilayer structures, Al-Fe and Al-carbon steel laminated composites were prepared by ultrasonic consolidation, the interface forming
mechanism of Al-Fe laminated composites was analyzed, and its mechanical properties were tested. It has a broad application prospect and academic value for studying the fundamentals of multi-layered protective structural systems and their popularization and application.

2. Experimental

2.1. Experimental materials
Industrial purity aluminum 1060 and iron foil are placed in alternating layers in all ultrasonic consolidation experiments. The chemical composition of the materials is given in tables 1, 2 and 3. The foil Al 1060 and iron have a shear size of 800×20×0.2mm. Although ultrasonic consolidation can clean the interface, it is necessary to pretreat the foils. Because the oxides be formed can accumulate at the interface in the reaction, these oxides eventually form obvious cracks and cavities, which will result in defects in the obtained laminated composites, and reduce the interface bonding rate and mechanical properties. The surface oxide on the foil was removed by washing with 4-10 wt% NaOH aqueous solution tank for 4-5 minutes for stripping treatment and then dried with acetone. And the foil was polished by 1000# emery paper until the surface presents silvery white. The obtained Al/Fe laminated composite is selected oriented perpendicular to the direction of the ultrasonic consolidation lamination to standard grinding and polishing, the ZEISS-Image optical microscope, SU5000 scanning electron microscope (SEM) and EDS was used to study the interface microstructure, Composition distribution and fracture morphology of polished samples, the Vickers Hardness of laminated composites was characterized by microhardness, and the accuracy was guaranteed by multiple times calculating the average of the hardness of cross-section. The Instron3382 electronic stretcher was used to test the tensile properties of the obtained laminated composites.

| element | C | Si | Mn | S | P | Cr | Ni | Cu | Fe |
|---------|---|----|----|---|---|----|----|----|----|
| wt %    | 0.56 | 0.25 | 0.75 | 0.03 | 0.03 | 0.7 | 0.35 | 0.25 | Bal. |

Table 1. Chemical composition of carbon steel.

| element | C | Si | Mn | S | Al | Cr | Ni | Cu | Fe |
|---------|---|----|----|---|----|----|----|----|----|
| wt %    | 0.002 | 0.005 | 0.02 | 0.003 | 0.004 | 0.01 | 0.01 | 0.01 | Bal. |

Table 2. Chemical composition of pure iron.

| element | Si | Fe | Cu | Mg | Mn | Zn | Ti | V | Al |
|---------|----|----|----|----|----|----|----|---|----|
| wt %    | 0.25 | 0.35 | 0.05 | 0.03 | 0.03 | 0.05 | 0.03 | 0.05 | Bal. |

Table 3. Chemical composition of 1060 aluminum alloy.

2.2. Selection of experimental equipment and parameters
The experimental equipment is the ultrasonic consolidation machine of Harbin Engineering University as shown in figure 2. The maximum load of this machine is 23000N, the maximum power is 9kw, and the maximum consolidation speed is 80mm/s. The feed way of this machine is manual feeding. Firstly, the substrate is preheated to given temperature. Then consolidated foils are placed alternately on the substrate. Finally, the main parameters in the ultrasonic consolidation process are regulated to complete the consolidation, and the laminated composites are obtained.
In this experiment, the frequency of ultrasonic consolidation machine was 20KHZ, the samples were divided into two groups according to the different laminate materials: the first group was the consolidation parameters of Al and carbon steel; the second group was consolidation parameters of Al and pure iron.

| sample      | Substrate preheating temperature (°C) | amplitude (μm) | Static pressure (Kgf) | Speed (mm/s) |
|-------------|---------------------------------------|----------------|------------------------|--------------|
| Al/Carbon steel | 190                                     | 28             | 250                    | 25           |
| Al/Fe         | 200                                     | 25             | 250                    | 30           |

3. Results and discussion

3.1. Micro-interface analysis of Al/carbon steel and Al/iron
(1) Micro-interface analysis of Al/carbon steel laminated composites

Figure 3 (a) shows the microscopic microstructure of Al/carbon steel laminated composites prepared by ultrasonic consolidation, there are two kinds of interfaces between the Al and carbon steel, the interface ① has obvious "convexity" and "concave" and the interface ② are straight. This seem to be the result of large difference in mechanical properties between Al and carbon steel during the process, cause a large difference in interface between the aluminum layer is consolidated to the carbon steel layer and the carbon steel layer is consolidated to the aluminum layer, in the figure 3 (a), the aluminum layer has a severe necking phenomenon, and the "convexity" of the interface has two morphologies of "big peak" and "small peak", the reason for this appearance is that during the ultrasonic consolidation rolling process, the consolidation force transmitted to the aluminum layer is relatively large, and the aluminum layer has serious necking phenomenon. As the roller moves forward, the continuous consolidation force near the interface pushes the grains in metal forward along the interface, finally, grain accumulation occurs in the near-end region of the consolidation interface, forming the “big peak” morphology, in the “big peak” has large amount of stress concentration, then by releasing energy, a "small peak" phenomenon occurs at the interface. The cracks in the aluminum layer, with severe necking phenomenon, are shown in figure 3 (b), and the obvious defects at the uneven interface of the undulations are shown in figure 3 (c). It is known that with the advance of ultrasonic roller, the vibration and friction generated by the same frequency ultrasonic waves are different, which leads to poor interfacial coordination of different materials. it is easy to cause the interface defect when the discordance appears in the convex or concave part of the interface.
Figure 3. (a) Microscopic image of Al/carbon steel laminated composites (b) Obvious crack of aluminum layer (c) Interface defects of Al/carbon steel laminated composites (d) Microscopic image of Al/Fe consolidation samples (e) Microstructure analysis of Al/Fe consolidated samples (f) "Toothed" morphology of Al/Fe consolidated samples.

(2) Micro-interface analysis of Al/Fe laminated composites

Figure 3 (d) shows the microscopic microstructure of Al/Fe laminated composites, the Fe and Al foils are successfully consolidated into laminated composites by ultrasonic consolidation process, the materials are well combined, with relatively straight and concave-convex interface. The reason for this phenomenon is that the different plastic deformation capacity of iron and aluminum leads to the inconsistency of interfacial synergistic deformation when materials interact with each other. The consolidated interface in figure 3 (d) ① does not have the large necking deformation like Al/carbon steel consolidation, and the interface is relatively straight, when the aluminum layer is consolidated, the uneven stress acting on the aluminum layer results in the morphology of interdigitated microstructure, when the iron layer is consolidated, because the yield strength of iron layer is higher than aluminum layer, the force transmitted by the iron layer to the aluminum layer is relatively uniform under the same consolidated force, this results in better plastic coordination at the interface
and relatively straight interface morphology. The consolidation interface in the figure 3(d) ② has "concave and convex" uneven interface phenomenon, when the rolling force is transferred from the aluminum layer to the iron layer, the corresponding synergistic deformation of the upper and lower metals is relatively poor, and it is easy to cause bulge. The toothed phenomenon occurring in figure 3(e) and figure 3(f) is similar to the rolling biting model shown in figure 4(a), when the bite angle θ is smaller than the friction angle β, the plates can be bitten, and the calculation methods of θ and β are as shown in the formula (1) and (2).

$$\theta = \arccos \left( \frac{R + h - H}{R} \right) \quad (1)$$

$$\beta = \arctan(\mu) \quad (2)$$

where R is the radius of the roller, h is half of the total thickness of the laminated composites, H is half of the original total thickness of the plate, μ is the friction coefficient between the roller and aluminum.

In this experiment, half of the measured thickness (h) is 1.08 mm, the radius of roller (R) is 40 mm, half of the original thickness of laminated composites (H) is 1.2 mm, and θ is calculated as 4.4° by substituting it into equation (1). The coefficient of friction μ between the aluminum alloy and the roll is 0.61. Substituting μ into equation (2) calculates that β is 31.3°. β>0, that is to say, the bite condition of single layer is satisfied: the friction angle is larger than the bite angle.

![Figure 4](image.png)

Figure 4. (a) straight plate (b) force of aluminum alloy in contact between roller and aluminum alloy.

When roller is in contact with aluminum alloy, the force of the aluminum alloy is shown in figure 4 (b). The roll acts on the aluminum alloy with a radial positive pressure N and a tangential friction force f. The direction of the friction force is perpendicular to the radial positive pressure, pointing in the direction of the roll rotation, and f = μ×N. The frictional force f and the positive pressure N relative to the front end point A of the composite plate aluminum alloy and ferroalloy interface are respectively:

$$M_t = f \times AB \times \cos \theta \quad (3)$$

$$M_n = N \times AB \times \sin \theta \quad (4)$$

Then:

$$\frac{M_t}{M_n} = \frac{f}{N \times \tan \theta} = \frac{\mu}{\tan \theta} > 1 \quad (5)$$

So:

$$M_t > M_n \quad (6)$$

Therefore, when the roll contacts with the aluminum alloy, the end of the aluminum alloy plate will be lifted under the frictional force of the roll, which can’t drive the foil forward. So the aluminum alloy plate is bitten finally.
In order to study the interfacial bonding, EDS scanning analysis was carried out for Al/carbon steel and Al/Fe laminated composites. The result is shown in figure 5.

![Figure 5](image)

**Figure 5.** (a) interface surface scan area of Al/carbon steel laminated composites (b) interface line scan of Al/carbon steel laminated composites (c) interface surface scan (d) toothed interface surface scan of Al/Fe laminated composites (e) straight interface surface scan of Al/Fe laminated composites (f) toothed interface line scan of Al/Fe laminated composites.

The EDS surface scan of the Al/carbon steel laminated composites in figure 5(c) shows that the interface does not undergo serious oxidation during the consolidation process, which indicates that the oxygen content is very low at the interface, there is no obvious atomic diffusion at the interface through line scan of figure 5(b), and no intermetallic compounds are formed, it can be concluded that the interface of Al/carbon steel laminated composites is mechanical bonding.

It can be seen from figure 5(d) that intermetallic compounds are not generated at the Al/Fe interface. The red area on the left side of the interface is Al, and the blue area on the right side is Fe. There are
two reasons for the absence of intermetallic compounds at the interface: On the one hand, the rapid rolling speed, strong plastic deformation and poor interface coordination, the concave and convex interfaces are not conducive to the formation of intermetallic compounds in the aluminum layer interface. On the other hand, due to the rapid rolling speed, brittle intermetallic compounds cannot be formed at the interface in short time. figure (e) shows the EDS surface scan of the Al/Fe straight interface. The red area on the left side of the interface is Fe, the blue area on the right side is Al, and atomic diffusion layer of about 1 micron is formed at the interface, the yellow region at the interface of Al/Fe is mainly Fe-Al solid solution, due to the lower temperature, solid solution with Fe-based body-centered cubic lattice is formed at the interface. The only possible is Fe3Al phase, and the formation of atomic diffusion layer improves the binding rate. It can be seen from figure (f) that the content of each element of the straight interface in the left and right sides of the atomic diffusion layer between iron and aluminum is close to 100%, and the oxygen content is generally stable in the entire interface, this shows that the oxide content of the Fe/Al bonded interface obtained by ultrasonic consolidation under non-vacuum conditions is less, and the interface during consolidated process is less affected by oxygen content. But there are always defects in the interface, the reason is tangential stress generated by the laminated composite at the dynamic interface causes the plastic deformation at the rough interface and rupture of the oxide layer, which result in the bonding of the exposed dissimilar metal layer. However, this traditional linear density of ultrasonic consolidation can only reach about 90%, and there are always defects in the interface, the broken oxides eventually are squeezed into the cavities, finally, the cavities cannot be closed, and form defects.

By analyzing the interface topography of the two groups experiments, it is found that there are a lot of defects in the interface bonding region of Al/carbon steel consolidated samples, and the "cavity" defects exist, which destroys the interface continuity of materials, the metallurgical bonding region of the laminate materials is not formed in the material interface layer by EDS. But in the laminated composite samples obtained by Al/Fe consolidation, there are “toothed” interface and “straight” interface topography, and Al/Fe consolidated samples generate trace of intermetallic compounds at the straight interface, an elemental diffusion region with a thickness of about 1 micron appears, it is an ideal combination of the laminated materials. It can be clearly found that the concave and convex defects are greatly reduced at the macroscopic interface of the Al/Fe consolidated samples, there are only few micro-defects such as "cavities", and combination effect is better.

3.2. Mechanical properties

Through the analysis of the microstructure of the above two groups of samples, it can be seen that the Al/Fe laminated composites prepared by the consolidation have better bonding effect at the interface, and the interface of Al/Fe laminated composites is straighter than that of Al/carbon steel laminated composites. At the same time, a small number of intermetallic compounds are formed at the interface of the Al/Fe laminated composites, which effectively increases the interfacial bonding effect of the laminated material. Due to the large amplitude of the concave-convex interface, the Al/carbon steel laminated composites have severe necking of the aluminum layer, and the fracture of the aluminum layer occurs, which reduces the properties of the materials. The large differences in interface micro-morphology lead to different mechanical properties of the laminated composites.

3.2.1. Hardness analysis. It can be seen from the figures (a) and (b) that the hardness indentation of each layer of the Al/Fe laminated composites show a significant linear contrast with the material self-property. The hardness of the aluminum layer of each laminated material is about 61HV in figure 6 (c), and the average hardness of carbon steel and iron layer is 209HV and 151HV respectively. The design of soft-hard bonded micro laminated material realizes the preparation of functionally graded composites, which provides a new way of reinforced protective performance of new composites. The hardness at the interface of Al/carbon steel and Al/Fe is 153HV and 124HV respectively, and the hardness attached to the edge of the interface is increased slightly. Because the interface is affected by consolidated energy, so there is work hardening at the edge of the interface, and the temperature
attached to the edge of the interface also will increased slightly, then the plastic deformation capacity increases. The hardness graded distribution obtained by this method can help reduce the stress concentration at the interface.

![Image](image1.png)

**Figure 6.** (a) hardness indentation of Al/Fe laminated composites (b) hardness indentation of Al/carbon steel laminated composites (c) hardness comparison of Al/Fe laminated composites.

### 3.2.2. Tensile Fracture Behavior

![Image](image2.png)

**Figure 7.** (a) stress-strain curves of Al/Fe laminated composites (b) stress-strain curves of Al/carbon steel laminated composites.

Al/Fe laminated composites in the stage of elastic deformation in figure (a): the slope of the Al/Fe laminated composites is smaller than pure iron, which indicates that the Al/Fe laminates have better ability of elastic deformation and low degree of deformation hardening. It can be seen from the Al/Fe material in the red curve in the figure that there is no stage of yield deformation at the interface. In the
strengthened stage: tensile strain occupies the majority of the total strain, and it is a critical stage in determining the tensile properties of the materials. At this stage, the strain of Al/Fe laminates is obviously higher than that of pure Fe, and the load required for Al/Fe laminates is larger, this is because the interfacial coordination is relatively good, which result in the interface having greater plastic deformation resistance during work hardening. In the fractured stage: there is no necking phenomenon in Al/Fe laminates, showing brittle fracture. It can be seen from the figure that the maximum tensile stress of Al/Fe and pure iron is 263.13 MPa and 245.97 MPa, respectively, which indicates that the Al/Fe laminates are more likely to brittle fracture than the pure iron after strengthening.

Al/carbon steel laminated composites in the stage of elastic deformation in figure (b): the slope of the Al/carbon steel laminated composites is smaller than that of carbon steel, which indicates that the Al/carbon steel materials have better ability of elastic deformation and low degree of deformation hardening. It can be seen from the Al/carbon steel material in the red curve in the figure that there is no stage of yield deformation at the interface. In the strengthened stage: the strain of Al/carbon steel laminates is obviously higher than that of carbon steel and also higher than that of Al/Fe, and the load required for Al/carbon steel laminates is larger, due to the performance of carbon steel itself, which leads to the large plastic deformation resistance of the interface in the case of work hardening, but the interfacial coordination is not better than Al/Fe. In the fractured stage: there is no necking phenomenon in Al/carbon steel laminates, showing brittle fracture. It can be seen from the figure that the maximum tensile stress of Al/carbon steel and carbon steel is 356.81 MPa and 323.35 MPa respectively, which is bigger than the maximum tensile stress of the Al/Fe laminates under the process, this indicates that Al/carbon steel laminates are more likely to brittle fracture than the Al/Fe laminates after strengthening.

3.3. Fracture morphology

![Figure 8](image.png)

Figure 8. (a) macroscopic morphology of fracture surface of Al/Fe laminated composites (b) "toothed" morphology of Al/Fe interface (c) macroscopic morphology of fracture surface of Al/carbon steel laminated composites (d) "toothed" topography of Al/carbon steel interface.
Figure 8 (a) shows the fracture morphology of Al/Fe laminates. From the top to the bottom is the Al-Al-Fe-Al-Fe-Al-Fe. The overall fracture morphology shows that the Al/Fe interface of the consolidation has completely cracked, however, there is still “tooth” phenomenon at the interface, and the “toothed” interface is the consolidation from aluminum to iron. The main reason is that the slow speed of consolidation, the average strain rate of Al/Fe interface small, and the deformation synergy between upper and lower metal layers is good, so it is not easy to cause local metal to turn up. Although the fracture surface of the “toothed” interface has been separated in figure 8 (b), there are still some bonding interfaces inside. From the red area, not only has layered steps but also a number of dimples at the interface of the iron layer. While the interface of the aluminum layer only has a little steps and dimples, this indicates that the fracture of aluminum layer is prior to the fracture of iron layer. And the interface expression is also different. The fracture interface of aluminum rolling on iron is the toothed surface, while the fracture interface of iron rolling on aluminum is straight and cracked. Because of the former is non-uniform plastic deformation, but the latter undergoes uniform plastic deformation.

Figure 8 (c) shows the fracture morphology of Al/carbon steel laminates. It can be seen from the fracture morphology that the Al/carbon steel interface has completely cracked under the consolidated process, and the “toothed” phenomenon at the interface is not obvious as that of Al/Fe. Although the material difference is larger than Al/Fe, because of the plastic deformation of carbon steel is not obvious as that of pure iron, which can be reflected from the dimpled size, that is to say, the compatibility of Al/carbon steel laminates is not as good as Al/Fe laminates, and there are partial aluminum tearing at the carbon steel interface. figure 8(d) shows that the fracture surface of the toothed interface has been separated. A large number of typical dimples appear at the interface of carbon steel layer, and the aluminum layer has a deeper layered river-like morphology than the nearby area, this morphology shows that the plastic deformation of the fracture at the aluminum layer toothed is larger than that in the nearby area.

4. Conclusion

(1) In this paper, two different iron-based laminated composites of Al/carbon steel and Al/Fe were successfully prepared by ultrasonic consolidation process. The interface analysis of the two groups of experiments shows that the Al/Fe laminated composites prepared by the ultrasonic consolidation have better bonding effect, and the interface of the Al/Fe laminated composites is straighter than the interface of the Al/carbon steel laminated composites. At the same time, a small amount of intermetallic compound is formed at the interface of the Al/Fe laminated composites, which effectively increases the interfacial bonding effect of the laminated materials. This is an ideal laminated material prepared in this experiment. However, the Al/carbon steel laminated composites have severe necking, and the fracture in the aluminum layer occurs due to the large amplitude of the concave-convex interface, there are residual oxides and impurities at the interface, which reduces properties of the materials.

(2) By analyzing of the mechanical properties of the two groups of samples, the results show that the hardness indentation at the interface of Al/carbon steel and Al/Fe is 153 HV and 124 HV, respectively. The hardness distribution gradient increases with the change of interface bonding distance. From the tensile test, the maximum tensile stresses of pure Fe and pure Al are 245.97 MPa and 120.91 MPa, respectively, and the maximum tensile stress of Fe/Al laminated composites increases to 263.13 MPa after ultrasonic consolidation. The maximum tensile stress of carbon steel is 323.35 MPa, and the maximum tensile stress of Al/carbon steel laminated composite is 356.81 MPa after ultrasonic consolidation. It is indicated that the mechanical properties of materials after lamination consolidation can be greatly improved, and the effect of Al/Fe laminated composites is better than the Al/carbon steel laminated composites.

(3) By analyzing the fracture morphology of the two groups, it is found that the coordination of Al/carbon steel consolidated samples is obviously inferior to that of Al/Fe consolidated samples. Moreover, the difference in mechanical properties between Al/carbon steel is larger than that of Al/Fe,
which results in poor interfacial bonding. The Al/Fe consolidated sample has good partial metallurgical bonding interface and good bonding interface due to the good coordination between the upper and lower metal layers.

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