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Improvement of adhesive bonding of grit-blasted steel substrates by using diluted resin as a primer

Binhua Wang[a,b], Xiaozhi Hu[b,*], Pengmin Lu[a]

[a] Key Laboratory of Road Construction Technology and Equipment, MOE, Chang'an University, Xi'an 710064, PR China
[b] School of Mechanical and Chemical Engineering, University of Western Australia, Perth, WA 6009, Australia

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ABSTRACT

This study reveals the micro-structural details on the metal substrate surface prepared by grit blasting, and then proposes a simple resin pre-coating method aiming at full wetting of the substrate surface for stronger adhesive bonding. The resin pre-coating solution consisting of around 90% acetone and around 10% resin without hardener is used as a primer, which can be sprayed or blushed onto the grit-blasted metal substrate. The acetone solution can carry resin deep into micro-cavities created by grit blasting and effectively coat and wet micro-debris so that micro-voids or gaps between the adhesive joint and metal substrate can be removed. Since the resin pre-coating does not contain hardener and remains wet, the wettability of the substrate is also improved. The normal epoxy adhesive with hardener can then be applied onto the substrate surface. Despite having the primer-like function, the proposed resin pre-coating method still maintains the simplicity of one epoxy resin system. Based on the current study, a resin and acetone solution without hardener does not seem to have adverse effects on the final bonding strengths of adhesive joints, although acetone is known to have detrimental effects on resin and hardener adhesive systems. Four different surface conditions are examined, each having 14 specimens: (1) Grit-Blasted (GB) surface, (2) GB-surface with ultrasonic cleaning, (3) GB-surface with resin Pre-Coating (PC) only, and (4) GB-surface with both ultrasonic cleaning and PC. 25% improvement in the shear strength has been achieved by the resin pre-coating method, even without ultrasonic cleaning, in comparison with 8% improvement after ultrasonic cleaning. These results show GB-surface with PC is beneficial to adhesive bonding, which can be adopted for structural applications even if thorough substrate surface cleaning on site is not possible. The improved wettability of metal substrates after resin pre-coating contributes to the maximum possible utilization of the contact areas over the roughened substrate surfaces and thus leads to the enhanced adhesive bond strength.

1. Introduction

Adhesive bonding of metals and fibre composites has been widely used in various composite structures, where the interfacial bond strength is the key concern. Steel, among various metal substrates, is the most heavily used structural material in automobile industry, marine engineering, civil infrastructure, and oil and gas industries. In the past decade, carbon-fibre reinforcement of steel structures has attracted increasing attention because of its high strength and similar elastic modulus [1–4]. Adhesive joining of carbon-fibre composites to steel structures is also convenient and flexible, showing a promising future in wide applications [5–7]. However, the shear stress transfer between carbon-fibre composites and steel substrates and thus the interfacial bond strength is the limiting factor to the success of carbon-fibre reinforcement [8]. Due to the huge dissimilarities of material compositions and properties at the adhesive and steel substrate interface, adhesive bond failure along the adhesive/steel interface is known to be the dominant failure mode, which can significantly reduce the effectiveness of carbon-fibre reinforcement.

To ensure strong interfacial bonding, grit blasting or grinding has been commonly used to prepare the steel substrate to have rough, fresh and more reactive contact surface areas between the adhesive and substrate. The strong interfacial bonding on a grit-roughened substrate can also enhance micro- mechanical interlocking between the poxy adhesive and steel substrate [9–12]. Other methods such as chemical etching [13–15] and atmospheric plasma [16–20] are also used in well-equipped factories and laboratories. Furthermore, grit-blasted metal substrates can be ultrasonically cleaned in laboratories to ensure the best possible bonding conditions [21]. However, in many real engineering applications, chemical etching and ultrasonic cleaning cannot

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[*] Corresponding author.
E-mail address: xiao.zhi.hu@uwa.edu.au (X. Hu).

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be adopted on site due to the limitation of equipment and work environment [22,23]. As a result, grit-blasting preparation of steel substrates followed by air blowing remains the most common and practical method for adhesive bonding of steel structures [24,25]. The down side of this substrate surface preparation process is that the best possible adhesive joining conditions may not be achieved because micro-debris and broken grit particles, embedded in the metal substrate or trapped inside micro-cracks created by repeated plastic deformation during the grit-blasting process, cannot be completely removed by air blowing. Furthermore, it can be difficult to achieve complete wetting on the grit-roughened substrate surface due to the presence of micro-cavities generated from grit blasting.

In this paper, we present a simple resin pre-coating method, which can be used to achieve a near perfect wetting condition on site using a single resin and hardener adhesive system without a commercial primer. Four different surface conditions are examined in this study. They are: (1) Grit-Blasted (GB) steel surface, (2) GB-surface with ultrasonic cleaning, (3) GB-surface with our proposed resin Pre-Coating (PC), but without ultrasonic cleaning, and (4) GB-surface with both ultrasonic cleaning and PC. Our preliminary results show GB-surface with PC only is sufficient, which implies thorough substrate surface cleaning on site is not necessary for adhesive bonding. This can be useful for many structural applications when chemical etching and ultrasonic cleaning cannot be used due to the limitation of equipment and work environment on site.

2. Method and surface preparation

2.1. Interfacial zone from grit-blasting & role of resin pre-coating

Adhesive failure between steel substrates or between carbon-fibre face sheet and steel-substrate, as illustrated in Fig. 1, can be described by two most likely crack growth paths. (1) Adhesive failure or cracking along the bonding interface between steel substrate and adhesive joint, and (2) cohesive failure or cracking within adhesive joint. In the case of carbon-fibre face sheet and steel substrate, the interface between carbon-fibre face sheet and epoxy adhesive joint is fairly strong as carbon fibre itself contains around 30 vol% of epoxy.

In general, the interfacial shear strength of an adhesive joint is controlled by the interface between the steel substrate and epoxy adhesive because of the huge variation in material properties. That is the reason why a steel substrate surface needs to be roughened through grit blasting, and then cleaned thoroughly by ultrasonic cleaning to achieve the best possible surface condition for adhesion [4,26]. It is noted that micro-dirt on a grit-blasted surface needs to be removed [27,28], otherwise the benefits of grit blasting will be compromised. While small test samples can be ultrasonically cleaned in laboratory, it cannot be adopted for large engineering structures on site. The most likely option in practice may be air blowing by compressed air although it is not as effective as ultrasonic cleaning.

In order to have a good understanding of the interfacial bonding and controlling mechanisms, the enclosed area along the adhesive/steel interface in Fig. 1 is enlarged and illustrated by a hypothesised interfacial zone model in Fig. 2. As a result of grit-blasting, a steel substrate surface is no longer flat, but turned into an “interfacial zone” of certain depth, as in Fig. 2(a). Sticky epoxy with hardener may not be able to penetrate deep into micro- fissures or cracks. Furthermore, micro-dirt on the grit-blasted surface is not desirable for good surface wetting, as schematically illustrated in Fig. 2(b).

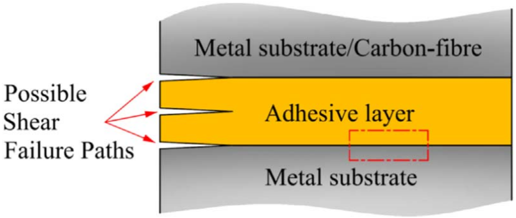


Fig. 1. Three possible crack paths or failure modes in epoxy joint between steel substrates. The adhesive failure, or interfacial cracking, can occur along both interfaces.

It would be an ideal scenario that epoxy or resin can completely wet the micro-dirt and micro-particles and penetrate deep into all micro-cracks within the interfacial zone, as shown in Fig. 2(c). Afterward, the normal well-mixed epoxy with hardener can be applied, as in Fig. 2(d). Diffusion during the curing process should lead to the final uniform curing, as in Fig. 2(e), as long as the interfacial zone is not too deep and the resin pre-coating is not too thick.

Complete wetting of the steel substrate is critical to the bonding strength of adhesive joint [9,29], which is unfortunately hard to achieve on site because of the relatively “dirty” substrate surface condition. What is shown in Fig. 2 outlines the possibility of achieving perfect substrate wetting on site without the need of thorough surface cleaning.

Acetone is widely used in laboratory as a cleaning agent because of its excellent surface wettability. Resin without hardener can be easily dissolved in acetone, which can then be taken deep into those micro-fissures of the interfacial zone by the acetone-resin solution, as illustrated in Fig. 2(c). In this study, resin pre-coating is used to achieve the complete wetting of a grit-blasted steel substrate even if micro-dirt and grit-particles exist within the interfacial zone. The fresh steel substrate surface after grit blasting is also temporarily protested by the resin pre-coating so that the on-site engineers have more time to complete the adhesive bonding.

2.2. Materials and sample preparations

Flat mild steel bars of dimensions 6000×25×3 mm^3 supplied by Midalia Company were cut into the dimensions of 40×25×3 mm^3. Commercial Selleys Araldite Super Strength bi-component composed of resin and hardener was selected for bonding the steel substrates. Grit-blasting was carried out by using GMA Premium Blast garnet with the grit size of 30–60 (as marked by the supplier) under a pressure of 5 bar for 10 s. During the process of grit-blasting, the nozzle with the inner diameter of 7 mm was almost located at an angle of approximately 90° and kept a distance of about 50 mm from the surface of steel substrate. Acetone was used to clean the steel substrates. The diluted resin/acetone solution contains 10 wt% of resin. Various wt% of resin solutions have been tested [8], and it appears the 10 wt% resin/acetone solution gives the best adhesion under the short-term test conditions adopted in this study.

Four different surface preparation methods were considered in this study, i.e. (i) Grit-Blasting (GB), (ii) grit-blasting followed by ultrasonic cleaning in acetone at the room temperature for 30 min (GB/Cleaning), (iii) grit-blasting followed immediately by the pre-coating of 10 wt% of resin (GB/Pre-Coating (PC)), and (iv) grit-blasting followed firstly by cleaning and then PC (GB/Cleaning/PC).

Before the above surface preparations, as-received steel substrates were first ultrasonic cleaned in acetone at the room temperature for 30 min to remove any surface dust and possible oil contamination. The cleaned substrates were then grit-blasted for 30 s, followed by surface cleaning by compressed air. The steel substrates were then divided into four groups, for the four different surface preparation tests. All specimens were allowed to dry the room temperature for 10 min.

Shear strengths of the adhesive joints with different surface conditions were obtained by the common single lap shear (SLS) tests. The dimensions of assembled specimens used in SLS tests are showed in Fig. 3. The bond area was 13 mm×25 mm and was held by a small spring clamp during the curing process. The curing temperature was kept at 40 °C in an oven for 20 min for the first curing period and 60 °C for 10 h for the following curing period. Fourteen samples were prepared and tested for each substrate surface condition.

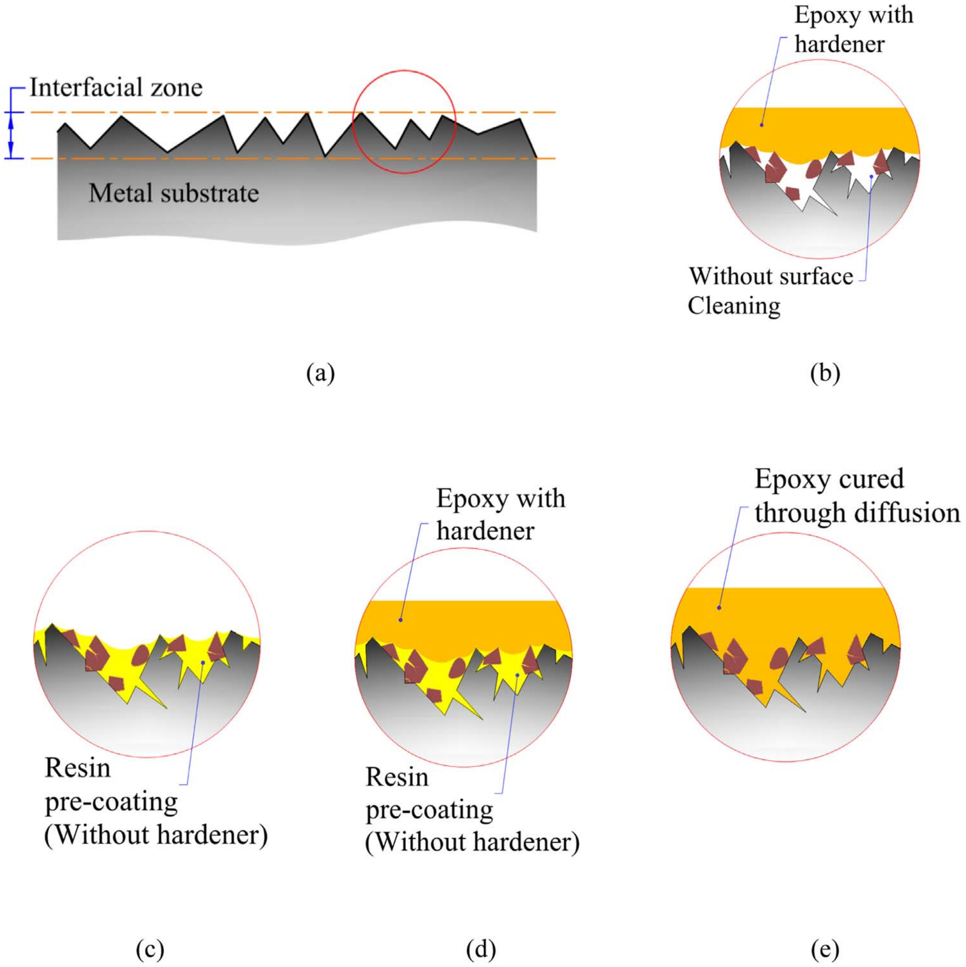


Fig. 2. (a) Cross-section view of the interfacial zone after grit blasting with depth of around 10 μm, (b) normal epoxy adhesive with hardener may not be able to penetrate deep into submicron micro-cracks and wet all micro-debris, (c) acetone with around 10 wt% of resin without hardener can penetrate deep into those sub-micron cavities and ensure the complete wetting of the interfacial zone, (d) epoxy adhesive with hardener can then be applied on the substrate surface with improved wettability from the resin pre-coating, and (e) resin pre-coating becomes part of the epoxy adhesive joint during the curing process through hardener diffusion so that the maximum possible surface contact area is utilized.

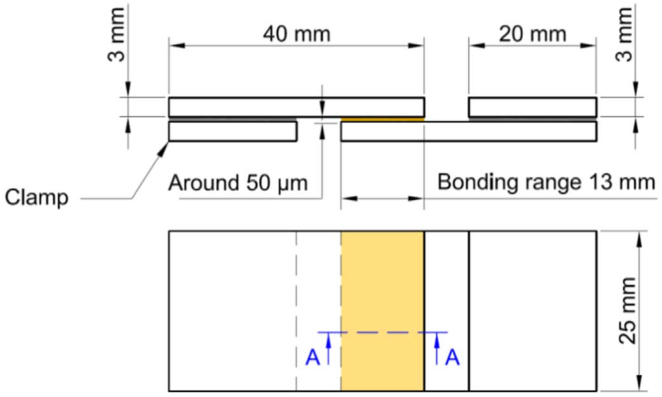


Fig. 3. Specimen dimension, as used in [30].

3. Shear strength from single lap shear tests

3.1. Testing condition and effects of substrate preparation methods

The single lap shear (SLS) tests were performed by using Instron 5982 mechanical testing machine with a 100 kN load cell. The displacement control mode with a rate of 0.5 mm/min was selected during the tensile/shear tests. The tests were stopped after the peak loads were recorded. Major test results and specimen details are listed in Table 1, and the corresponding shear strengths are displayed in Fig. 4 as well to show the trend of strength variation.

As expected, the shear strength of adhesive joints between grit-blasted (GB) steel substrates is higher than that of the smooth specimens polished by #120 SiC paper. The GB/cleaning condition (ultrasonic cleaning in acetone for 30 min) shows a further 8% improvement, indicating it is beneficial to have a thoroughly cleaned GB substrate surface. The GB/pre-coating (PC) did not include the ultrasonic cleaning step, yet its shear strength is higher than that with ultrasonic cleaning after GB. This finding is significant as it shows on-site engineers can achieve the full benefit of grit-blasting surface preparation without the need of thorough surface cleaning. It should be mentioned that despite of large scatters in Fig. 4, the lowest strength value of GB/cleaning (22.57 MPa) is still higher than the average of GB/cleaning (22.10 MPa). The benefits of resin pre-coating are thus confirmed.

The last condition, GB/cleaning/PC, gave the best result, which however cannot be adopted by on-site engineers. If the hypothesized model of the interfacial zone illustrated in Fig. 2 are correct, it is possible to narrow the gap between the GB/PC and GB/cleaning/PC conditions after further pre-coating optimisation.

The 14 results per group shown in Fig. 4 consist of three separate sample preparations and tests. It is likely each time, the grit-blasting condition may vary, e.g. potential variation in the grit conditions (size, old and new). For instance, the best result from group of 4 specimens showed that the shear strength from GB/PC was 50% higher than that of base grit-blasted specimens. Yet the combined results of 14 tests

Table 1
Specimen Details and Test Results[a].

Preparation methods	Specimen quantity	Maximum shear stress/ MPa	Minimum shear stress/ MPa	Average shear stress/ MPa	Average improvement/%
GB[b]	14	23.33	17.11	20.54	0
GB/Cleaning[c]	14	25.69	18.54	22.10	8%
GB/Pre-Coating(PC)	14	27.56	22.57	25.58	25%
GB/Cleaning[c]/PC	14	30.25	23.68	27.21	32%

[a] 6 smooth samples were also prepared using #120 SiC paper, and shown in Fig. 4.
[b] After GB, all steel substrate surfaces were cleaned by compressed air for around 15 s.
[c] Ultrasonic cleaning in acetone for 30 min.

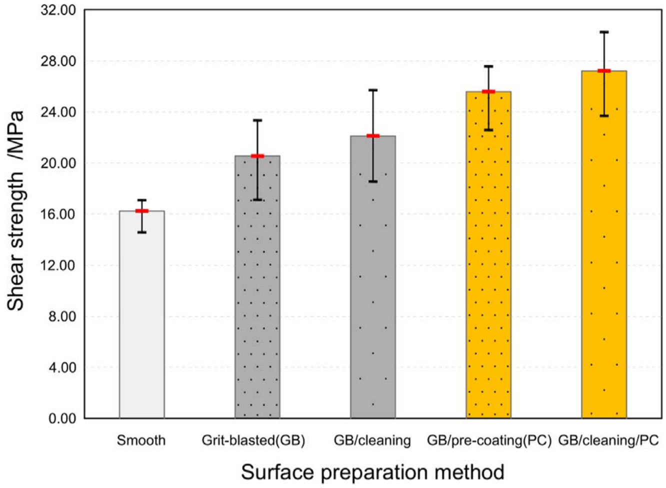


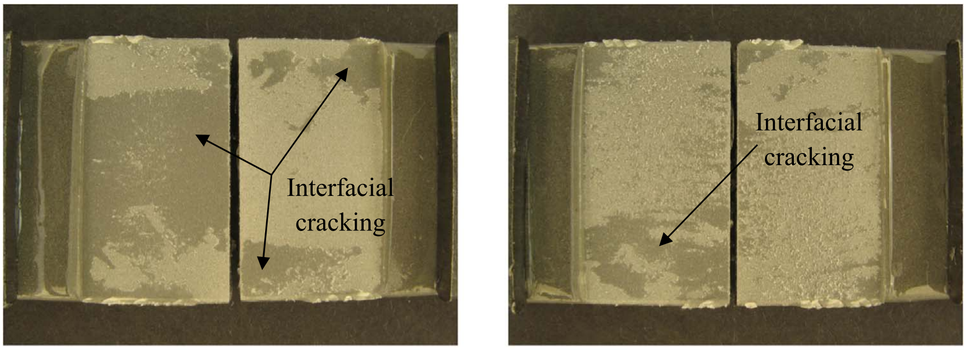
Fig. 4. Shear strength (minimum, maximum and average) measured by SLS tests on the grit-blasted steel samples with four different surface conditions. Smooth specimens polished by #120 SiC paper are included for comparison purpose.

summarised in Fig. 4 should have confirmed the benefits of resin pre-coating and the hypothesized model of the interfacial zone for grit-blasted metal substrates.

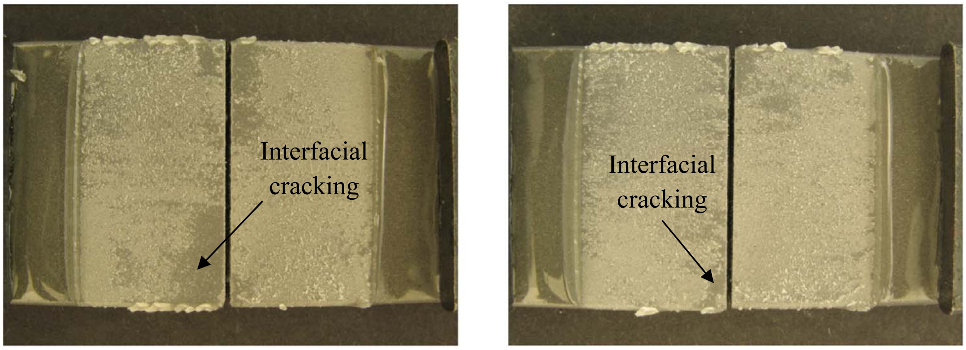
Typical fracture surfaces and failure modes of the four groups of specimens with different surface conditions are shown in Fig. 5. The base grit-blasted sample, Fig. 5(a), displays a large area of apparent interfacial failure because of the relatively weak interfacial adhesion possibly resulting from the presence of micro-voids, micro-cracks and grit debris trapped at the uneven substrate surface as explained by the interfacial zone model illustrated in Fig. 2(b). Ultrasonic cleaning after grit blasting, Fig. 5(b), has effectively reduced the interfacial fracture area leading to an enhanced interfacial shear strength. Resin pre-coating, Fig. 5(c), further reduced the interfacial fracture area, and the pre-coating with ultrasonic cleaning, Fig. 5(d), has virtually removed all interfacial failure area.

3.2. SEM observation of interfacial zone and shear failure surfaces

The scanning electron microscopy (SEM) were undertaken using “Zeiss 1555 VPSEM” microscope to observe the substrate surfaces with the detectors of Secondary electrons (SE2) or back-scatter electrons (BSD). All samples were coated with a thin layer of gold to get high

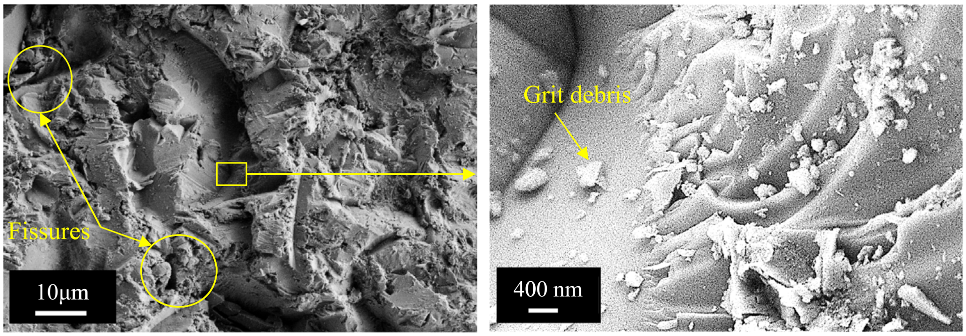


(a) Grit-blasted (GB) (b) GB/cleaning



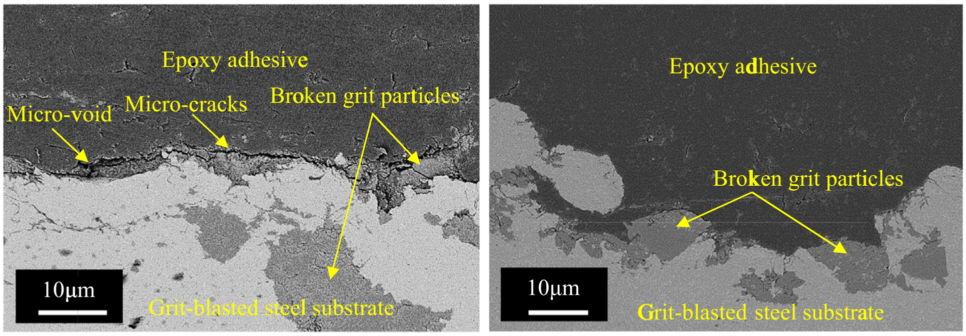
(c)GB/pre-coating (PC) (d)GB/cleaning/PC

Fig. 5. Shear failure along the interface or within the adhesive joint as indicated in Fig. 1. (a) GB has highest % of interfacial failure, and (d) GB/cleaning/PC has smallest % of interfacial failure. Sample width = 25 mm.



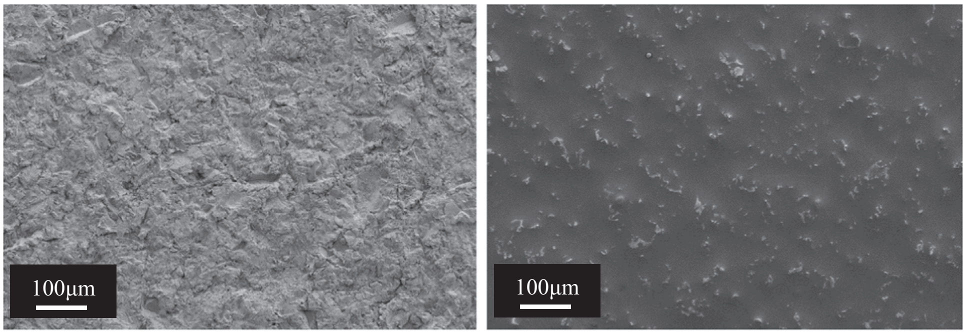
(a) (b)

Fig. 6. (a) Grit-blasted steel substrate surface after cleaning by compressed air, (b) inside the deep valley there are still many sub-micron or nano-scaled loose particles. (Secondary electron SEM images).



(a) GB (b) GB/PC

Fig. 7. Side view (A-A section as in Fig. 3) of GB and GB/PC sample before SLS test: (a) GB: micro-debris and broken grits prevented complete wettingand resulted in the growth of micro-voids and micro-cracks, leading to weak interface adhesion; (b) GB/PC: resin pre-coating improved wetting and penetrated deap into broken grits and fine gaps created by grit-blasting. (Back-scattered electrons BSD images).



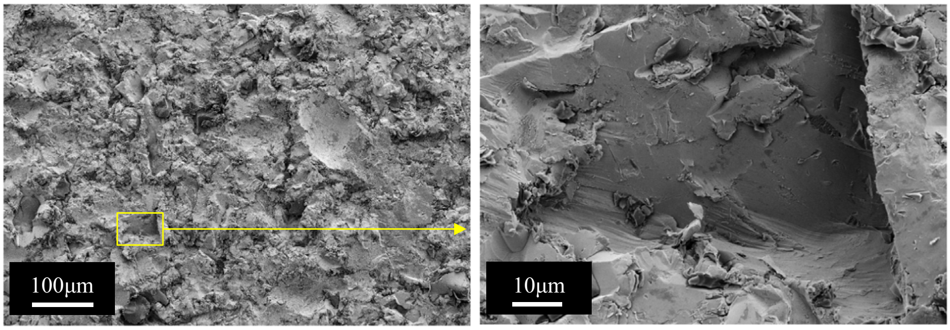
(a) (b)

Fig. 8. (a) A grit-blasted steel surface, (b) grit-blasted steel surface after pre-coating with 10 wt% resin solution [8] (Secondary electrons SEM images). The surface remains wet until application of a fully formulated resin and hardener system.

resolution scanning electron images under the accelerating voltage of 10 kV. A grit-blasted steel substrate surface after cleaning by compressed air (no epoxy was applied and no ultrasonic cleaning) is shown in Fig. 6. It is clear from Fig. 6(a) that the interfacial zone model hypothesised in Fig. 2 captures the key characteristics of the grit-blasted steel substrate surface. The section of a deep valley at the centre of Fig. 6(a) is enlarged and shown in Fig. 6(b). Indeed, many broken grit pieces measured around 200 nm were observed deep inside the valley, despite of cleaning by the compressed air. It can also be envisaged that it would be difficult for sticky epoxy to penetrate into the narrow fissures encircled in Fig. 6(a). As a result, the situation postulated in Fig. 2(b) is most likely the situation of epoxy adhesion after only GB.

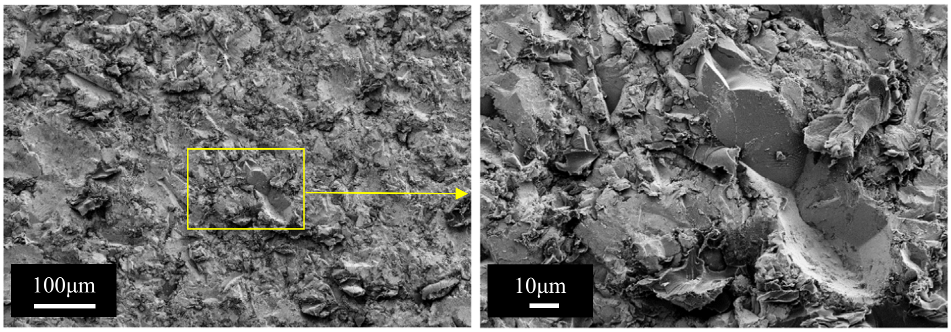
Cross-section views of two interfaces between epoxy adhesive joints and metal substrates are shown in Fig. 7: (a) grit-blasted steel substrate (GB), and (b) grit-blasted steel substrate with resin pre-coating (GB/PC). It is clear in the microscopic scale that broken grit particles partially embedded in the metal substrate hindered strong adhesive bonding because of the incomplete wetting. As a result, micro-cracks are clearly visible in Fig. 7(a) for the normal GB and adhesive-bonded sample. The benefits of resin pre-coating are clearly demonstrated in Fig. 7(b) for the GB/PC sample. The resin pre-coating process proposed in this study is able to penetrate fine cracks within broken grit particles and to achieve complete wetting of the complex interfacial zone (as illustrated in Fig. 2) created by grit blasting. The interfacial features shown in Fig. 7(a) and (b) are reflected by the shear strength measurements from GB and SB/PC samples shown in Fig. 4.

The common method used to deal with those sub-micron loose particles is ultrasonic cleaning although there is no guarantee that all those sub-micron particles will be removed. Furthermore, ultrasonic cleaning most likely cannot be adopted for on-site applications. The simple and practical method proposed in this study is to use resin pre-



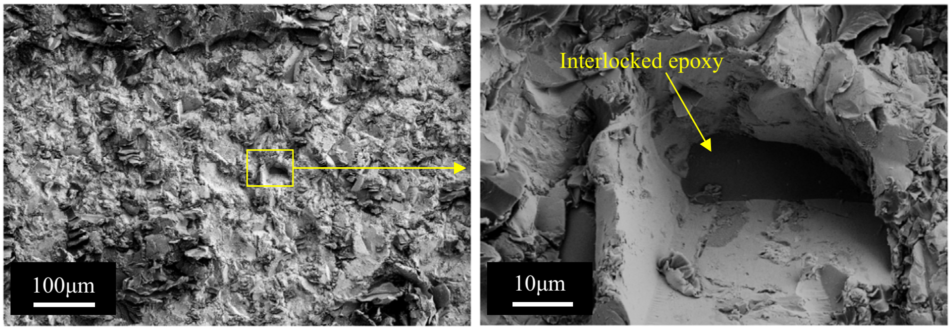
(a) (b)

Typical failure surface ofgrit-blasting (GB) substrate



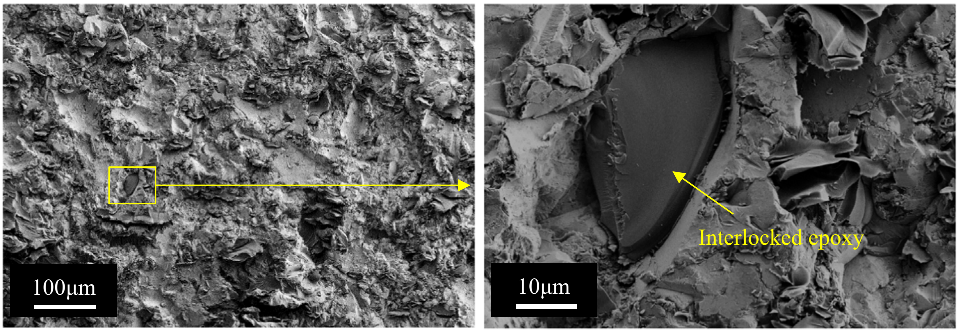
(c) (d)

Typical failure surface for GB & ultrasonically cleaned substrate



(e) (f)

GB/pre-coating (PC) without ultrasonic cleaning



(g) (h)

GB/cleaning/PC

Fig. 9. Shear fracture surfaces of substrates with the four different surface conditions as illustrated in Fig. 5. (Secondary electrons SEM images).

coating, which has the ability to penetrate deep into any surface fissures and completely wet all those sub-micron particles, effectively creating a "particle-reinforced" resin matrix.

The surface overviews of a GB steel substrate and a GB steel substrate with resin pre-coating are shown in Fig. 8. The resin pre-coating in Fig. 8(b) was formed by applying 10 wt% resin-acetone solution as optimized in our previous study [8]. It is clear from the comparison of Fig.8(a) and (b) that all surface fissures from grit blasting have been filled, as illustrated in Fig. 2(c). It should be emphasized again that the substrate surface with resin pre-coating remains "wet" until a fully formulated resin and hardener system is applied, which implies the wettability the substrate surface is improved during the final adhesive bonding process.

The optical observations of fracture surface features for the four surface treatment conditions shown in Fig. 5 are also observed under SEM, and their typical microscopic details are shown in Fig. 9. In Fig. 9(a), there is little epoxy (darker grey phase) left on the fracture surface for the GB-only surface condition. It seems there has not been good deep penetration of epoxy in some deep valleys, e.g. Fig. 9(b). Under the GB/cleaning surface condition in Fig. 9(c) there are darker epoxy areas, indicating better surface adhesion. More dark epoxy spots are also visible in the enlarged area in Fig. 9(d). More epoxy residuals can be observed over the GB/pre-coating surface as in Fig. 9(e), and the enlarged picture in Fig. 9(f) shows epoxy still embedded deep inside a valley, indicating good penetration of epoxy. The last condition of GB/cleaning/PC in Fig. 9(g) shows more dark epoxy residuals, and the enlarged section in Fig. 9(h) shows a large interlocked epoxy still connecting to the steel substrate.

4. Concluding remarks

In this study, we have shown the benefits of resin pre-coating for strong adhesive bonding of grit-blasted steel substrates. The simple technique has the primer-like function, yet maintains the simplicity of a single epoxy adhesive system. The resin pre-coating process can achieve complete wetting of a microscopically complex metal substrate surface containing sub-micron micro-cracks and trapped micro-particles. The pre-coated substrate surface remains wet until the final adhesive bonding, which thus improves wettability of the substrate. The main function of the proposed method is mechanical not chemical, i.e. aiming at complete wetting and promoting the mechanical interlocking between the resin adhesive and fresh and reactive contact area of the grit-roughened substrate, as illustrated in Fig. 2.

The resin pre-coating surface treatment can be applied on-site easily through blushing or spraying, which effectively removes the need of thorough surface cleaning of grit-blasted steel surfaces. It achieves stronger adhesive bonding by fully utilizing the zig-zag shaped substrate surface created by grit-blasting, and by wetting micro-debris and broken grit particles left within the interfacial zone, as illustrated in Fig. 2.

The importance of grit-blasting is well recognized for metal adhesive bonding, which represents around 25% improvement as shown in Fig. 4 through the comparison of smooth and GB surfaces. The GB/pre-coating gives the further 25% improvement from the GB condition even without thorough surface cleaning. It should be reiterated that GB plus ultrasonic cleaning in acetone (thorough surface cleaning) has only achieved around 8% improvement over the pure GB surface condition.

The promising results reported in this study show that the hypothesised interfacial zone model illustrated in Fig. 2 has captured the major features and the key toughening and strengthening mechanisms of adhesive bonding for grit-blasted metal substrates. The findings in the current study are equally useful to adhesive bonding of carbon-fibre composites to metal substrates [31] because shear failure along the CFRP/metal-substrate interface is the dominant failure mode. Therefore, potentially the resin pre-coating surface treatment can be extended to aluminium alloy substrates for aircraft aerospace applications [32].

Finally, the fact that the most elaborate surface treatment condition, GB/cleaning/PC, with 32% improvement as in Fig. 4 shows that GB/PC can still be further optimized. In principle, if all micro-particles and micro-fissures on a GB metal substrate are fully covered by resin pre-coating, there should be hardly any difference between the GB/PC and GB/cleaning/PC surface conditions.

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