Adhesion properties of novel corrosion resistant hybrid structures

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Abstract
By employing hybrid structures, the most advantageous properties of the constituent materials can be achieved in one component. Typically hybrid structures introduce high specific strength or stiffness, but also other attractive and improved functionalities, such as good damping properties and more economical manufacturing processes, are possible to obtain when using hybrid technology. In a polymer/metal hybrid, stainless steel instead of more regularly used mild steel or aluminium would be a tempting choice due to its good mechanical properties and corrosion resistance. However, stainless steel is difficult to adhere to polymeric materials and improved solutions for the manufacturing of hybrids of this kind are needed.

In this study, the idea of utilization of thin rubber layer as an adhesive between stainless steel and glass fibre reinforced epoxy composite (GFRP) is used. Both mild steel and stainless steel with different surface finishes together with GFRP laminates were used as substrates for the ethylene propylene diene (EPDM) based rubber. The adhesion and microstructure of the interfaces were characterised. Transmission electron microscopical (TEM) studies indicated that a close contact between the components can be achieved and thus high quality interfaces are created by vulcanising the rubber to the steel or GFRP surface without pre-treatments. Stainless steel/rubber/GFRP hybrid structures where the strength of the steel/GFRP interface is defined by the cohesive strength of the rubber can be manufactured, as seen from the results of the peel tests. Since the fracture locates inside the rubber instead of at the interface, the prediction of the structure's behaviour is also easier.
Keywords: *Hybrid structure, Adhesion, Floating roller peel test, FIB, TEM*

1. Introduction

Hybrid materials are increasingly employed in several fields of industry. In a hybrid structure, the advantages of different materials can be combined and properties unattainable by any existing single material can be achieved. In addition to lighter weight, attractive solutions can be achieved through improved performance and more economical manufacturing processes. Thus, hybrid structures can solve a wide range of design challenges faced today.

Typically durable interfaces within hybrid structures are challenging to manufacture due to the different physiochemical properties of the components from different material groups, such as metals, polymers or ceramics. Several different bonding methods are used within hybrids [1] and the selection of the constituent materials and the structure geometry, for example, are factors affecting the choice of the bonding method. For laminar structures of two rigid components, adhesive bonding is the most obvious choice. The advantages of adhesive bonding over mechanical fastening or welding, are, among others, the possibility to join dissimilar materials as well as to reduce internal stress concentrations [2].

In adhesive bonding, special surface treatments and adhesives are used. Both chemical and mechanical surface pre-treatments are used to prepare the surfaces to be adhered responsive to the adhesive. Chemical surface treatments, such as etching or anodizing, used for metal surfaces before polymer joining [3, 4], may include the use of hazardous chemicals and solvents, whereas mechanical surface treatments are typically time consuming. The selection of the applied adhesive is done according to the adhered materials and the mechanical and environmental requirements of the application. In addition to structural adhesives, coupling agents, like silanes, or coatings can also be used [1, 3, 4]. Thus, adhesive bonding includes numerous stages. Another drawback of the adhesive method is that it may lead to poor predictability of the joint durability and failure modes compared with mechanical fastening or welding [2].

To simplify the manufacturing of adhesive bonds and to make it more cost-effective, the number of the operation stages should be kept low. In addition, adequate strength of the joint and especially its predictability have to be ensured. To overcome these challenges, new methods and material combinations for adhesive bonding have to be studied.

Stainless steel is a tempting choice for the metal component in corrosion resistant metal/polymer hybrid structures for a load bearing application due to its good corrosion resistance and mechanical properties. However, joining polymeric materials to stainless steel sheets is difficult and requires the application of pre-treatments and adhesives [3], which complicates the manufacturing. As an attempt to simplify the manufacturing process without a loss of the adhesion properties or an increase in the manufacturing costs in relation to the gained benefits, we framed the question if it is possible to replace the conventional adhesives with a new solution.

Rubbers can be modified with additives to be adaptive for both metallic and polymeric materials [5, 6]. They are used in composite and hybrid structures together with mild steel, aluminium, plastics, fabric, and cords to increase strength, minimize internal stress concentrations, and to simplify mounting of the structure [7]. In addition, added value for the structure, such as improved energy absorption properties, can be achieved by the inclusion of rubber. However, stainless steel/rubber combinations are rather uncommon. In this study, we suggest that a stainless steel/composite hybrid structure, which would otherwise be difficult to manufacture, could be formed by vulcanising a thin rubber layer between the steel and composite sheets. The application of rubber would not only simplify the manufacturing process but also introduce improved dynamic properties to the hybrid structure.

In the present study, the structure and the adhesion properties of steel/rubber/glass fibre reinforced epoxy composite (GFRP) hybrid structures are characterised. Both mild and stainless steel sheets are used in the hybrid together with compatible ethylene propylene diene (EPDM) based rubber and GFRP sheets. In addition, five different surface treatments for the stainless steel were used to study the effect of the steel surface topography on the adhesion strength. Scanning and transmission electron microscopy are used to characterise the components and the structure. The adhesion properties between the rubbers and the steel sheets as well as GFRP sheets are investigated by floating roller peel tests.

2. Materials and methods

In the present study, the adhesion properties of steel/rubber and composite/rubber interfaces were studied. Two steel grades, cold rolled mild steel EN 10130 DC01 (Ruukki Metals Oy, Finland) and stainless steel AISI 304 (Outokumpu Stainless Oy, Finland) were used. The mild steel was passivation treated as is customary for grades used as industrially coated. The aim of the passivation treatment is to enhance the adhesion properties of the steel but the procedure is not public. Five different surface treatments for the stainless steel (Table 1) were used to investigate the effect of the steel surface topography on the adhesion properties. The ac-received surfaces 2B, 2D and 2J are also defined in the standard EN 10088-2. The industrially polished (IP) surface mentioned in the Table 1 is a stainless steel sheet with 2D surface finish which is electrolytically polished by SpecialSteelStudio (Finland). The sand blasting media used for the SB samples was aluminium oxide (grit 36, average particle size 483 µm). The thickness of the steel sheets was 0.5 mm, but a thicker metal stiffener was glued on the back side of the metal component to prevent its bending during peel testing.

Table 1: *The studied substrates and their average profile roughness parameters (R_a) measured with laser profilometer.*

Code	*Surface treatment*	R_a *[µm]*
CR	*Cold rolled, passivation treated EN 10130*	*0.43*
2B	*Cold rolled, heat treated, pickled, skin passed AISI 304*	*0.35*
2D	*Cold rolled, heat treated, pickled AISI 304*	*0.38*
2J	*Dry brushed AISI 304*	*0.31*
SB	*Sand blasted AISI 304*	*2.46*
IP	*Industrially polished AISI 304*	*0.39*
GFRP	*HexForce® T470 peel ply*	*23.51*

The glass fibre reinforced plastic (GFRP) composite was manufactured in-house by vacuum infusion from stitched 0/90 E-glass fibre fabrics (682 g/m^2, Ahlstrom Oyj, Finland) and Sicomin SR 1660 / SD 7820 epoxy. The thickness of the GFRP sheets was 13 mm and its fibre content was about 45 vol-%. A metal stiffener was glued on the back side of the GFRP sheets to prevent its bending during peel testing. The heat resistant epoxy was chosen to resist the vulcanising temperature of the rubber. From the adhered GFRP surface, a HexForce® T470 (Hexcel Co., USA) peel ply was removed prior rubber attachment.

The EPDM based rubbers adhered to the steel and composite surfaces were manufactured by Teknikum Oy, Finland (grade A) and by Kraiburg GmbH, Germany (grades B and C). The grade A has a trade name Teknikum TRA10 and its ingredients are EPDM rubber, ZnO, stearic acid, polyethylene wax, carbon black, paraffin oil, internal adhesion promoter and peroxide. The grade B is designed for stainless steel and composite but is not a commercial grade. The grade C is designed for mild steels. The compositions of the rubbers from Kraiburg GmbH are not available.

Prior to the rubber bonding, the substrates were studied with Laser profilometer (UBM-Microfocus Compact, NanoFocus AG, Germany). At least a length of 50 µm was used to measure the average surface roughness parameter R_a of the surfaces in two perpendicular directions, longitudinal and transverse to the rolling direction of the steel. In addition, the steel surfaces were investigated with Field Emission Gun Scanning Electron Microscope, FEG-SEM (Zeiss ULTRAplus, Germany).

The steel/rubber and composite/rubber samples were manufactured by vulcanising the rubber to the substrate under heat and pressure. The steel surfaces were rinsed in ethanol and acetone prior rubber bonding; otherwise they were in the as-received stage. Any pre-treatments for the composite surface were not done after the removal of the peel ply. The vulcanising conditions are listed in Table 2. The vulcanising of the rubbers was done according to the manufacturers' guidelines, except for the GFRP/rubber A samples, for which the lower vulcanising temperature was compensated with longer vulcanisation time. To ensure that a high enough degree of vulcanisation is reached during GFRP/rubber A sample vulcanisation, differential scanning calorimetry scans (DSC) (DSC 204 F1, Netzsch, Germany) were done for rubber A.

Table 2: *The vulcanisation conditions of the different sample types.*

Sample type	*Temperature [°C]*	*Time [min]*	*Pressure [MPa]*
Stainless steel and rubber A	*160*	*15*	*1.2*
GFRP and rubber A	*130*	*18*	*1.2*
Stainless steel and rubber B	*130*	*20*	*1.2*
GFRP and rubber B	*130*	*20*	*1.2*
Cold rolled steel and rubber C	*130*	*30*	*1.2*
GFRP and rubber C	*130*	*30*	*1.2*

Methods to study the adhesion of rubber to rigid substrates are standardised [8]. The ASTM D429 standard introduces six different test methods, one of which (method B) is intended for determining the adhesive strength of rubber/metal bonding. In this test method, the rubber is peeled from the substrate at an angle of 90°. However, Cook *et al*. [9] have found that a peel angle of 45° is optimum for testing rubber/steel adhesion since it leads the fracture into the interface and the cohesive fracture of rubber is minimised. Thus a floating roller test configuration (Fig. 1) which provides a constant peel angle of 45° was used. The peel tests were performed with a universal mechanical testing machine with 1 kN load cell (Messphysik, Austria) and with a cross head rate of 100 mm/min according to the EN 1464 standard [10]. The peel test samples (size 100 x 12 mm) were cut from the bigger steel/rubber and GFRP/rubber laminates by water jetting. A pre-crack was created by inserting a 30 μm thick polymer layer between the rubber and the substrate before vulcanisation. The rubber thickness in the samples was 2 mm. For each sample type, 5-7 specimens were tested.

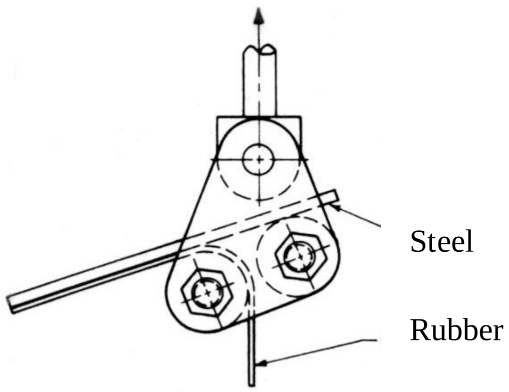


Figure 1: *The floating roller peel test configuration. Modified from* [11].

Cross-sections of the steel/rubber and composite/rubber interfaces were studied by FEG-SEM and by Transmission Electron Microscope, TEM (Jeol JEM 2010, Jeol, Japan). The FEG-SEM samples were prepared by conventional sample preparation methods including mounting, grinding and polishing. Different TEM sample preparation methods were used for different sample types. The sample preparation methods are described below.

The cross-sectional TEM samples of composite/rubber interfaces were prepared by a microtome in room temperature due to the sensitivity of the sample materials to ion beam. The sample preparation was made at the Institute of Biotechnology, University of Helsinki, Finland. The glass fibre in the composite prevented the cut with the thin sectioning method. Thus, we prepared epoxy/rubber samples by using a similar epoxy resin system as in the original GFRP sheets, similar peel ply and similar curing and vulcanisation procedures to attain suitable samples for the thin sectioning. This should not alter the interfacial conditions, since the outer layer in a well-wetted glass fibre epoxy composite is epoxy.

The cross-sectional TEM samples from the steel/rubber interface were prepared by Focused Ion Beam (FIB) method. The sample preparation was started by a traditional cross sectional sample preparation method: two pieces of steel were cut to the size of approximately 1.5 x 1 x 0.5 mm and were placed face-to-face in a titanium grid and attached with an epoxy adhesive (Fluka 45359 Epoxy - Embedding Kit) and the epoxy was cured at 65 °C for 16 hours. The EPDM rubber should endure these environments without degradation [12]. Then the samples were pre-thinned by hand to thickness of ~100 μm. The final TEM samples were made using a FEI Helios Nanolab 600 dualbeam FIB system at the University of Oulu, Finland. A layer of platinum (~ 1 µm) was first deposited on the steel/rubber interface as a protection layer. The rough TEM sample was then cut out from the bulk material and lifted off to the TEM grid with an Omniprobe needle manipulator. After that, the bulk sample was attached to the grid (FIB platinum deposition) as shown in Fig. 2. The grid was then turned 90° and the sample was thinned from both sides to the final thickness. During the final thinning, the platinum layer was removed.

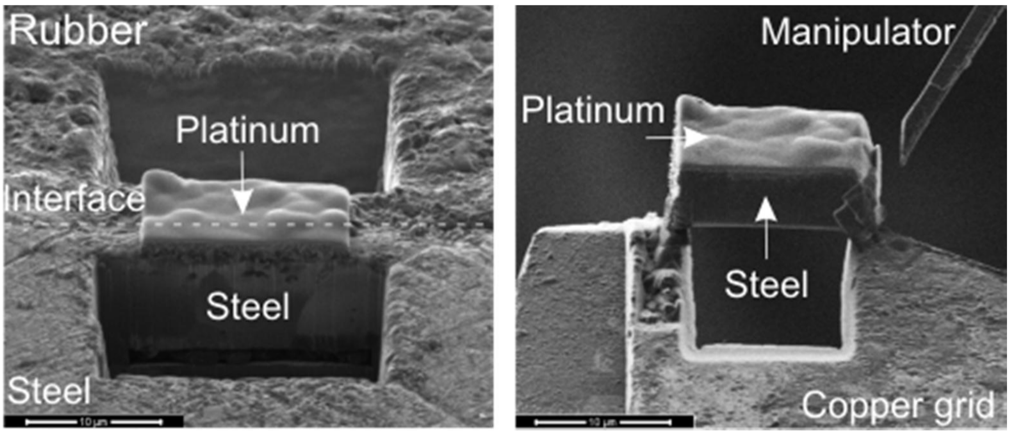


Figure 2: *a) A steel-rubber bulk sample from which the interfacial TEM sample is cut by Focused Ion Beam (FIB), b) the sample in the FIB grid. Before examination with TEM, the sample was turned so that the interface is parallel to the TEM electron beam.*

3. Results and discussion

3.1 Surface topography of the substrates

The SEM images of the different steel surfaces are shown in Fig. 3. The cold rolled mild steel surface is rather even without distinctive grain boundaries. The stainless 2B and 2D surfaces exhibit clearly distinctive grain boundaries, whereas the sand blasted stainless steel surface shows a substantially rougher surface contour where the grain boundaries are not visible. The brushed 2J surface shows oriented brush marks and the industrially polished IP surface is very smooth with some corrosion pits. The measured R_a values for the different surface finishes are presented in the Table 1. The measured R_a values fitted to the roughness range reported by the manufacturers well [13, 14]. Only the measured R_a value of the mild steel was slightly lower than the one reported by the manufacturer (0.43 µm vs. 0.6-1.9 µm), but this is supposed to be due to the passivation treatment. The roughness value reported by the manufacturer is for a grade without passivation treatment.

According to the R_a values and the SEM images, it was expected that within the stainless steel surfaces, the sand blasted surface would exhibit the highest adhesion strength as a consequence of increased surface area and mechanical interlocking, whereas the industrially polished surface would exhibit the lowest strength. It was also assumed that the as-received 2B, 2D and 2J surfaces would have intermediate adhesion strengths.

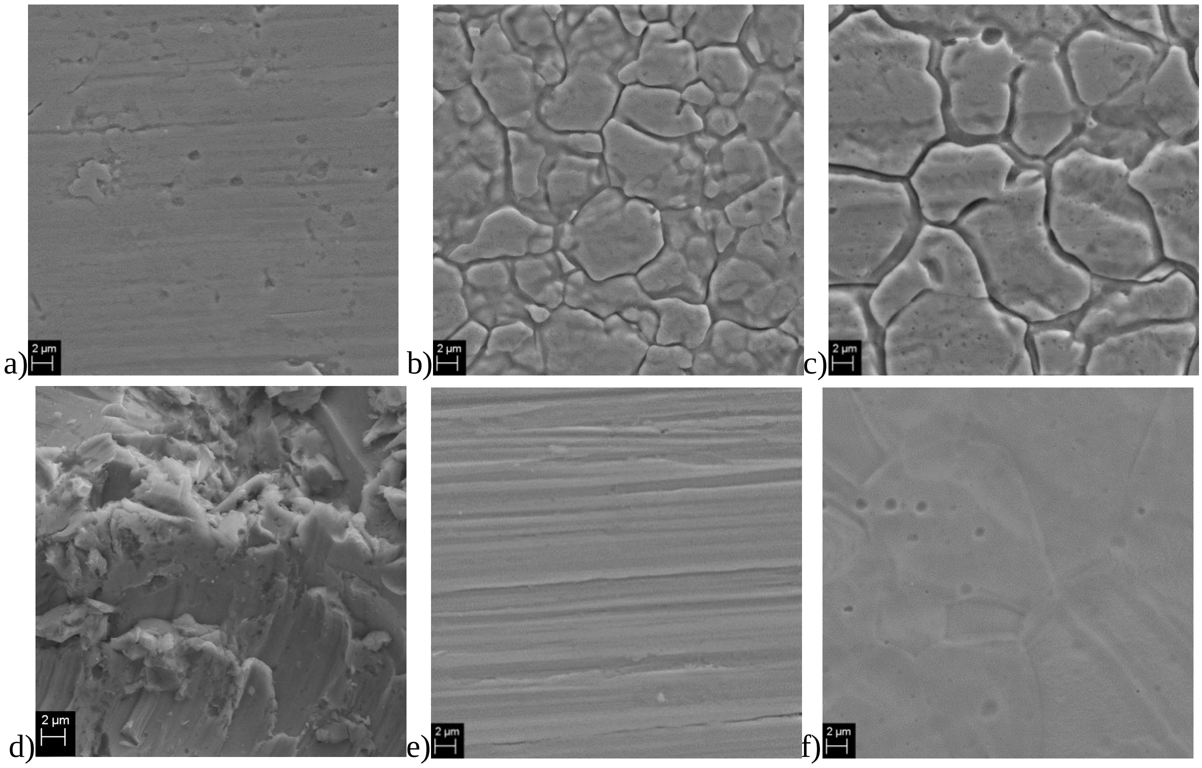


Figure 3: *Different steel surfaces: a) mild steel surface. The different stainless steel surfaces with the surface finish of b) 2B, c) 2D, d) SB, e) 2J and f) IP.*

The SEM images of the composite surface are shown in Fig. 4. It can be seen from the images, that the surface finish provided by the peel ply has regular and rough surface, high surface area and enables mechanical interlocking for the rubber. Thus it was expected that the composite surface would exhibit high adhesion strength.

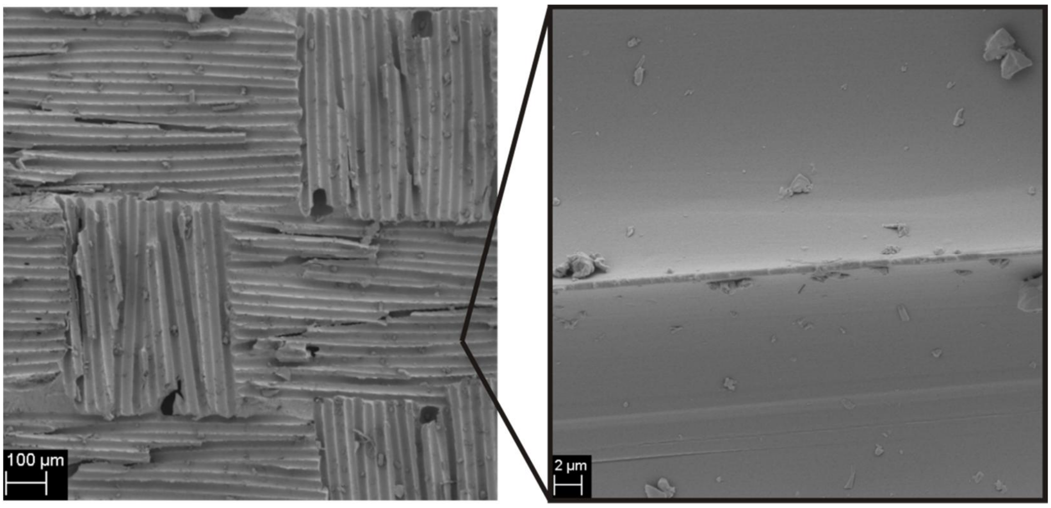


Figure 4: *The composite surface.*

3.2 Peel strength of the interfaces

The study was begun with the mild steel/rubber C interface. During the peel tests, the rubber broke cohesively before the peeling was started and thus the peel strength for the interface could not be determined. This clearly indicates that the cohesive strength of rubber C is less than its peel strength to the mild steel. Since the mild steel/rubber combinations are widely used in different applications, such as in vibration or sound damping applications [15], we set the cohesive fracture strength of rubber C as a point of comparison to the other interfaces.

For the stainless steel surfaces, the rubber B was applied first. For the surfaces 2J (brushed) and IP (industrially polished) the rubber B showed negligible adhesion. Thus the peel tests could not be performed and these interfaces were not studied at all with the rubber grade A. In the case of surface finishes 2B, 2D, and SB, the cohesive strength of rubber B exceeded its peel strength and the results are shown in Fig. 5. During the peel tests it was observed that rather than following the peel test roller and a peel angle of 45°, the rubber strip tended to show a higher peel angle, as described to be typical for floating roller peel tests in the review article written by Baldan [16]. As expected, the sand blasted surface exhibits superior peel strength when compared with the industrial surface finishes. Within the industrial surface finishes, the 2D surface exhibits slightly better adhesion which can be explained by the rougher surface of 2D when compared with 2B (higher R_a value, Table 1).

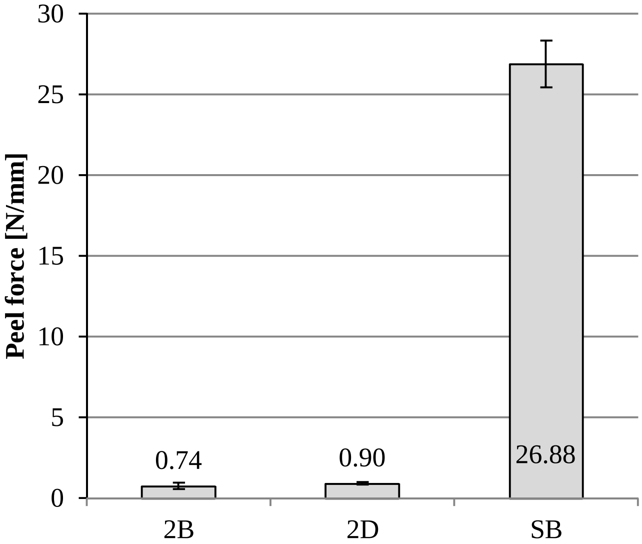
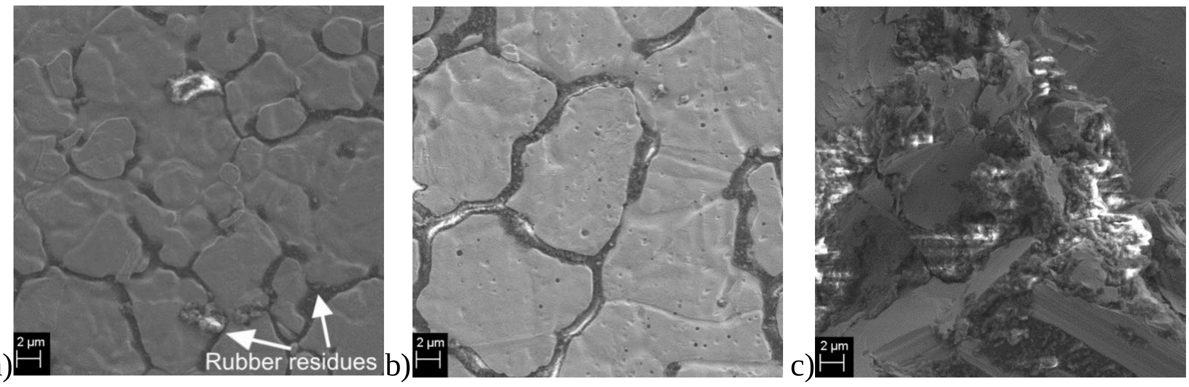
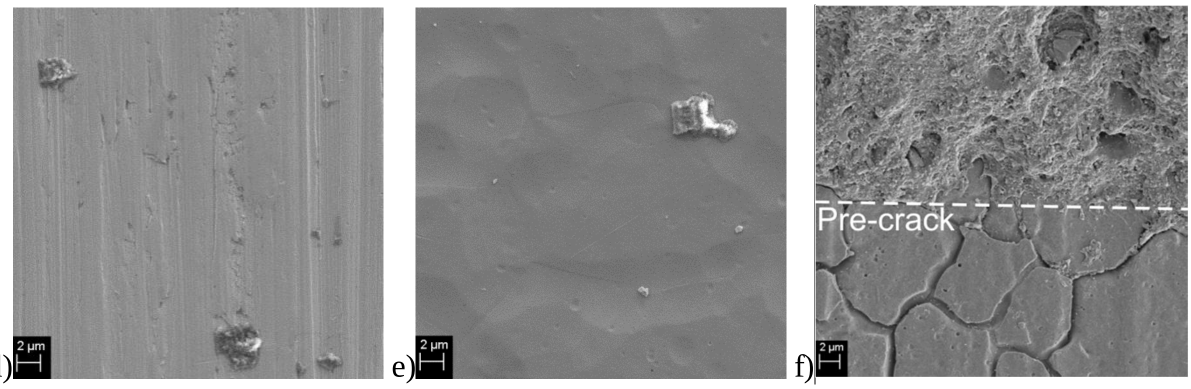


Figure 5: *Peel test results of the interfaces between rubber B and different stainless steel surfaces 2B (R_a 0.35 μm), 2D (R_a 0.38 μm) and SB (R_a 2.46 μm).*

The adhesion level of the interface is determined by the micro and macro nature of the substrate surface and the mechanical and physical properties of the adherend [17]. The test set-up parameters affecting the measured peel strength are peel angle, film thickness, peeling rate, the material properties of the substrate and the film, and the environment where the peel test is performed [18]. Thus it is difficult to compare the results of this study with the results of other researchers. However, it was found that the adhesion level gained with 2B and 2D surfaces with rubber B are about a tenth of the adhesion strength of mild steel and natural rubber [9] or stainless steel and thermoplastic urethane (TPU) [19].

The FEG-SEM images from the stainless steel surfaces after rubber B peeling are shown in Fig. 6. All stainless steel surfaces show a combination of cohesive and adhesive fracture: some rubber residues can be found in all these samples. However, the amount of rubber residues correlates with the peel force by increasing with increasing peel strength. Figs. 6.a and 6.b imply that especially the grain boundaries on the as-received stainless steel surfaces offer mechanical interlocking. According to the Fig. 6.c it is not clear whether increased surface area or improved mechanical interlocking is the main reason for the increased adhesion strength at the sand blasted surface.

a)




d) e) f)

Figure 6: *Different stainless steel surfaces after rubber B peeling from a surface finish a) 2B, b) 2D, c) SB, d) 2J and e) IP. Figure f) shows the cohesive fracture of rubber A initiated from the pre-crack edge.*

Rubber A was applied to the two stainless steel surfaces 2D and SB, both of which exhibited the highest peel strengths with the rubber B. In order to verify that the interface really is stronger than the rubber, a flexible carbon fibre mat was added to the rubber's outer surface before vulcanisation to prevent the fracture of rubber. However, the failure locus was still inside the rubber in all samples. Both 2D/rubber A and SB/rubber A interfaces showed cohesive failure of the rubber (Fig. 6.f). The peel force at which the cohesive fracture of rubber A occurred exceeded the peel force of the mild steel/rubber C from this study as well as the peel strengths measured for the mild steel and natural rubber [9] and stainless steel and TPU [19]. This indicates that proper rubber compounding enables the manufacturing of stainless steel/rubber joints without additional substrate surface treatments and that the joint strength is determined by the rubber's cohesive strength.

The peel test results for GFRP/rubber interfaces showed cohesive fracture of rubber for all three rubber grades and peel strength values could not be determined. Table 3 summarises the peel test results of the present study.

Table 3: *Summary of the peel test results of the present study.*

Substrate	***Surface finish***	***Rubber***	***Interfacial peel strength [N/mm]***
Stainless steel AISI 304	*2D*	*A*	*Exceeds rubber's cohesive strength**
	SB	*A*	*Exceeds rubber's cohesive strength**
Stainless steel AISI 304	*2B*	*B*	*0.74*
	2D	*B*	*0.9*
	SB	*B*	*26.88*
	2J	*B*	*No measurable adhesion*
	IP	*B*	*No measurable adhesion*
Mild steel EN 10130	-	*C*	*Exceeds rubber's cohesive strength**
GFRP	*Peel ply*	*A*	*Exceeds rubber's cohesive strength**
	Peel ply	*B*	*Exceeds rubber's cohesive strength**
	Peel ply	*C*	*Exceeds rubber's cohesive strength**

**The cohesive fracture appeared at a peel force of 382 N, 268 N and 70 N for rubbers A, B and C, respectively.*

3.3 Characterization of the interfaces

The steel/rubber and GFRP/rubber interfaces were first characterised with FEG-SEM. However, it was difficult to deduce the true nature of the interface from these samples, since the rubber tended to smear over the interface. Thus we decided use TEM to study the interface despite the much more laborious fabrication of the TEM samples. For the TEM studies, we chose only the 2D surface finish of stainless steel. The acceleration voltage used to study the samples was 120 kV.

The steel/rubber interfaces are shown in Fig. 7 and the GFRP/rubber interfaces in Fig. 8. The rubber grades have good adhesion to the steel surfaces (Fig. 7). A close contact between the steels and the rubbers was observed and no discontinuities between the steel and rubber can be seen, meaning that a high quality interface is gained by this manufacturing method. Similarly, Fig. 8 shows a good quality interface between epoxy and rubber for all rubber grades. Especially from the Fig. 8, the reinforcing particles of the rubber can be seen as spherical darker areas. To conclude, all the studied rubber/substrate interfaces showed a close contact between the components so high quality interfaces are achieved by the manufacturing process used in this investigation.

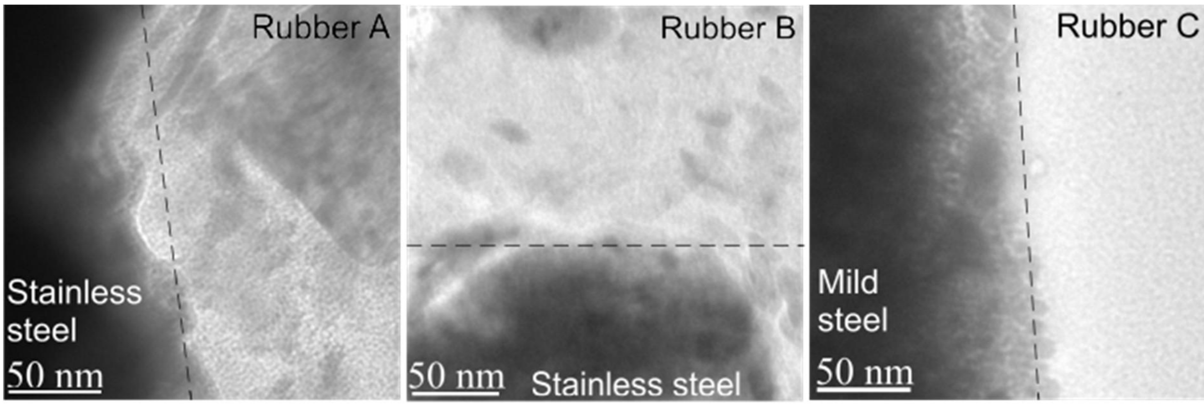


Figure 7: *Cross sectional TEM images from the steel/rubber interfaces: a) stainless steel/rubber A, b) stainless steel/rubber B and c) mild steel /rubber C. The interface is indicated by the dash line.*

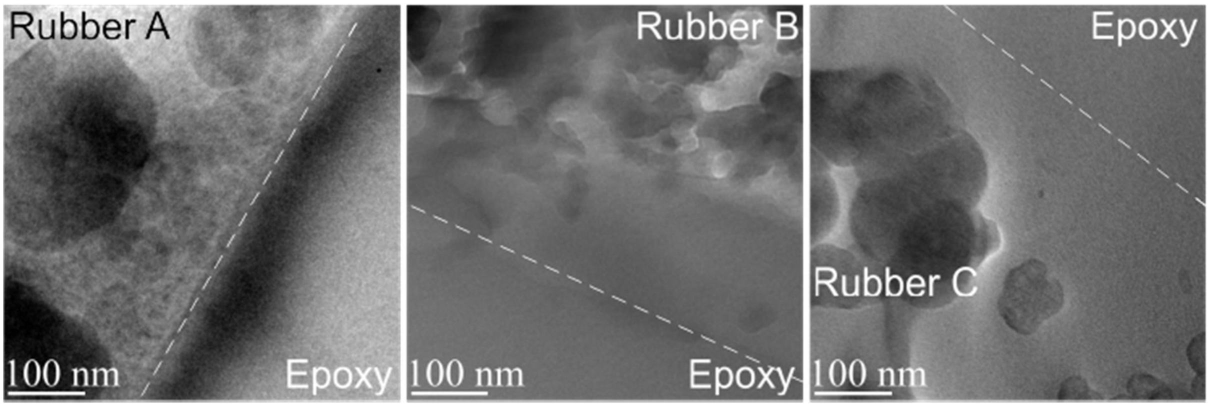


Figure 8: *Cross sectional TEM images from the epoxy/rubber interfaces: a) GFRP/rubber A, b) GFRP/rubber B and c) GFRP/rubber C. The interface is indicated by the dash line.*

Together the peel test results of the stainless steel/rubber and GFRP/rubber samples and the cross-sectional TEM images confirm that complex stainless steel/rubber/composite hybrid structures can be manufactured by using the rubber as an adhesive component. By choosing suitable rubber additives, neither time-consuming pre-treatments, nor adhesives are needed and the substrates can be used in the as-received stage. In addition, it can be concluded that for the studied steel/rubber A/composite system, the strengths of the structure's interfaces exceed the strength of the rubber. Thus, when estimating the behaviour of the steel/rubber/composite hybrid structure, rubber's cohesive data can be used instead of the steel/rubber and the composite/rubber interfacial data. This simplifies the prediction process due to the easier accessibility of bulk data when compared with the accessibility of interfacial data.

Despite the result that our structure exhibits cohesive fracture in ambient laboratory testing, the environmental resistance of it should also be tested. It has been shown that although the rubber/metal interfaces show cohesive failure in laboratory tests, the structures tend to break from the rubber/bonding agent interface in service [20]. Also other metal/polymer interfaces are prone to moist environments and introduction of high humidity leads generally to a significant decrease in interfacial strength [1]. Thus, the suitability of the structures studied in the present study for real life applications must be verified by environmental testing.

4. Conclusions

In the present study, the adhesion properties of stainless steel/EPDM rubber and glass fibre reinforced epoxy composite (GFRP)/EPDM rubber interfaces were characterised by peel test and electron microscopy. The results were compared with the results of a similar structure, where the metal component was mild steel and the EPDM rubber was compatible with that steel. In addition, different stainless steel surface finishes were used to study the effect of the surface topography on the steel/rubber adhesion strength.

Based on the peel test results, it is possible to manufacture stainless steel/EPDM rubber joints by vulcanising the rubber sheet directly to the as-received stainless steel surface. In this study, not even sand blasting was necessary to create well bonded stainless steel/rubber joints. The adhesive strength of these joints is defined by the cohesive strength of the rubber. Similar results were observed for GFRP/EPDM rubber joints. The transmission electron microscopy studies revealed that the different components are in close contact and thus the manufacturing method is suitable to create high quality joints. Since one of the material combinations lead to a structure in which the fracture located inside the rubber, we conclude that reliable stainless steel/GFRP hybrid structures manufactured with the aid of rubber are attainable.

However, before utilising the structure in real life applications in which it is exposed to different environments, the durability of its interfacial properties must be confirmed. Although stainless steel, EPDM rubber and GFRP are quite resistant to different environments by themselves, the interfacial properties may deteriorate clearly due to temperature or humidity.

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