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Friction surface structure of a C_f/C-SiC composite brake disc after bedding testing on a full-scale dynamometer

Guangyu Bian Houzheng Wu*

Department of Materials, Loughborough University, Leicestershire, LE11 3TU, UK

Abstract

We have examined friction surface structure of a carbon ceramic brake disc tested on a full-scale dynamometer with microscopy techniques. The bedded friction surface is composed of two types of regions: transferred materials (TM) and SiC. The TM regions were formed through the deposition of wear debris into surface voids, followed by compaction and crystallite refinement during braking. A thin friction layer (FL) was developed on top of TM and SiC regions with nano-sized copper/iron oxide crystallites as the primary constituent. Analysis shows that debris generated from pad is the main source of TM and FL. No evidence shows chemical diffusion bonding between TM and composite constituent. On silicon carbide surface, dislocations were activated as the sources of surface fracture.

Key words:

Carbon-ceramic brake disc, friction surface, transferred materials, friction layer
microstructure

* Corresponding author: Tel: +44 1509 223342; Fax: +44 1509 223949; E-mail: h.wu2@lboro.ac.uk (H. Wu)

1. Introduction

Almost all discs (or rotors) of brakes for road vehicles are currently made of grey cast iron.

Apart from its low cost, another driving factor is that friction performance of these brakes can be conveniently engineered to a desired level¹. Nonetheless, such an essential merit cannot be easily matched for brake discs made from other materials. The distinction of cast iron brakes ought to mostly attribute to the enriched knowledge about correlations between friction performance and friction surface structure. By now, there have been a large number of studies focusing on the structure of friction surfaces^{2,3,4,5,6,7,8}, making it even possible to predict the friction performance, as demonstrated by recent research through numerical modelling^{9,10}.

However, for an alternative or new brake disc material, the lack of enough equivalent knowledge can make it challenging to achieve the desired friction performance for a brake. The introducing of carbon fibre reinforced carbon-silicon carbide ($C_f/C-SiC$) into road vehicle brakes has demonstrated this journey, along with other candidate materials such as silicon carbide particle reinforced aluminium matrix composites (Al-MMC)^{11,12}.

$C_f/C-SiC$ brakes have achieved success in premier cars for more than a decade, but come up with an incredibly high cost. Even so, some desired friction performance has not yet achieved to a desired level, for example, fast bedding in (running in), consistent friction performance under all operating conditions, and suitability for wet environment¹³. Friction performance of a brake should be determined by the carbon ceramic composite and coupled friction material, but it is the friction surface developed during braking that is in charge to directly dictate its friction performance. Therefore, to make a brake engineer more capable and confident in

improving or optimising friction performance, it is necessary to have enough knowledge about the friction surface. *As per* such situation, a while ago, we started to understand the friction surface structure and its development on the surface of a C_f/C-SiC brake disc through testing it against an organic pad (Note, organic pad is widely called by engineers, and it actually an organic polymeric or resin bonded friction material¹⁴, commonly used in road vehicles for normal duty.) on a laboratory-scale dynamometer. These results have already been reported in previous publications^{13,15,16,17}. The study was later extended to the friction surface of a composite brake disc tested on a car^{18,19}, which is compared to that tested on laboratory-scale dynamometer. The conclusion is that, knowledge acquired from braking tests on a laboratory-scale dynamometer is significant enough in supporting the understanding of testing results on a car, although differences were seen on the friction surface features. However, there is yet no knowledge available in the literature on the friction surface of a C_f/C-SiC brake disc tested on a full-scale dynamometer, which is a widely used testing rig in industry for engineering friction materials and brakes to achieve desired friction performance. Recently, we had the opportunity to access such tested samples through a cooperative research project, and detailed investigation of friction surface structure had hence been accomplished. The key results on the friction surface microstructure of the carbon ceramic composite disc after bedding test is to be reported here.

In this paper, we will examine the friction surface structure with a focus on that of a C_f/C-SiC composite brake disc after bedding tests on a full-scale dynamometer by pairing it with organic pads. Note, the friction surface of an organic pad is equally important, but a number

of papers have been published by other reseachers, hence no detailed investigation was carried out on the friction surface of a pad in this study. The braking test scheme was modified based on AK master schedules²⁰, a widely used industry testing standard. Compared to the testing on a laboratory-scale dynamometer, braking testing used in this paper has an energy input and testing environment much closer to the braking conditions used in a road vehicle. Hence, it is expected that the knowledge on the friction surface will help researchers and engineers to understand the friction performance of a C_f/C-SiC brake for them to engineer a carbon ceramic brake with appropriate friction performamnce.

2. Experimental procedure

2.1. Brake disc and pad

The carbon ceramic brake disc and organic pads were acquired from market. The brake disc is composed of a C_f/C-SiC composite core and a surface layer with a thickness of ~2 mm. It is noted that the surface layer is the same type of composite as the core, but has significantly higher SiC content. The diameter and thickness of the carbon ceramic disc is 350 mm and 34 mm, respectively. Ventilation vanes and through thickness circular holes were engineered by the original manufacturer. A stainless steel bell was bolted on the carbon ceramic disc to bridge the brake disc with the tub of the testing rig.

The organic pads consist of a block of as-moulded friction material and a steel back-plate; both parts were bonded together by the original manufacturer. All surfaces of the friction

material blocks were finished by the manufacturer. The thickness of the friction material, including the bonding layer was 16.24 mm. The total friction area of the pads was $\sim 108 \text{ mm}^2$. The organic pads were fixed on a calliper that had four pistons, each of which had an area of 25.55 mm^2 . The cylinders were connected to a master cylinder with a diameter of 28 mm.

Due to reasons of commercial interest, it is unlikely to have all details on microstructure and chemicals provided by the manufacturers of the composite brake disc and organic pads. Therefore, such information was acquired through in-house characterisation, and will be presented in the results section as essential information for understanding the development of friction surface.

2.2. Friction test

The braking test was completed on a customised dynamometer with an inertial of 63.4 kg.m^2 at Alcan Component Ltd, UK. During each braking snub, data on friction and temperature were logged at a frequency of 100 Hz. The surface temperature of the disc was measured with a rubbing thermal couple, maintained physical contact with the friction surface of the disc across the test.

The test programme was designed to include 6 testing segments: segment-I - initial green and bedding; segment-II - wet testing; segment-III - recovery; segment-IV - high temperature fade; segment-V - the repeated wet testing; and segment-VI - motorway performance. The friction performance on each segment is to be published separately²¹. In this paper, we will focus on

the structure of friction surface after completing segment-I test only, also called initial green and bedding segment. The sub-sections in this segment were specified based on the AK-Master guideline, as summarised in Table 1. The testing in segment-I started from green, then bedding, followed by characteristics checking, pressure series, and characteristics checking again. Then braking testing was stop, and brake disc and pads were taken off from the testing rig for post-braking examination.

To examine the friction surface after the segment-I testing, samples were cored from the surface of the brake disc with a core drill of 5 mm in a diameter at the 6 and 12 o'clock positions in the middle circle of the friction track. No samples were taken from the pads in order to maintain the contact area the same between brake disc and pad in all test segments.

2.3. Microstructure and chemical composition characterization

The microstructure and chemical composition of as-polished surfaces of the brake disc and pad, and their friction surfaces after braking testing were examined with optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS).

For the as-received disc and pad, the polishing was completed on a Kemet 15 lapping grinder (Kemet, Kent, UK). All grinding and polishing steps were completed with diamond slurry, starting from a grit size of 25 μm , followed by 14 μm , 8 μm , 3 μm , and 1 μm for the final step.

For an as-braked surface, no further treatment was done before the examination with OM and SEM/EDS.

To acquire more detailed information on the structure of friction surface of the carbon ceramic composite brake, a cross-sectional examination of the friction surface was accomplished with transmission electron microscopy (TEM) and EDS. The samples for TEM analysis were made on the friction surface with focused ion beam microscopy (FIB) (FEI, Nava 600 nanolab, Oregon, USA). The procedure is highlighted here:

- (a) First, a 1 μm thick platinum coating was deposited on the interested region of the friction surface, to prevent Ga^+ implantation and sputter erosion of the top portion of the surface.
- (b) A rectangle trench was dug adjacent to the platinum strip using a 20 nA 30 kV Ga^+ ion beam.
- (c) When the trench was large enough, the second trench adjacent to the other side of the platinum strip was dug under the same condition. Before the thickness of the wall reduced to about 2 μm , both sides were milled using a 7 nA 30 kV Ga^+ beam, including clean and smooth the surfaces of the cross section.
- (d) The membrane for TEM was then lift out and welded on a Cu grit after cutting off from the bulk using a 3 nA 30 kV Ga^+ beam.
- (e) A final thinning process was carried out using a 1 nA 5 kV Ga^+ beam at an inclined angle of 1.5° to the surface of the membrane, followed by a beam current reduced to 0.2 nA when the thickness of sample was less than 500 nm, to minimise the depth of any residual damage by the ion beam. Nonetheless, surface damage by Ga^+ beam is unavoidable if no other thinning method is used, and scale of the damage is estimated to be <20 nm.

1 The typical thickness of a TEM sample made by following this procedure was about 200 nm.

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6 Optical microscopy (MeF3, Reichert-Jung, Wien, Austria) was operated under a combined
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8 lighting condition of polarisation and differentiation interference contrast (DIC). SEM/EDS
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10 analysis was accomplished on a field emission gun SEM (FEG-SEM) (LEO 1530 VP, UK)
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12 with an X-Max 80 mm² EDS detector (Oxford Instruments, UK). The SEM was operated
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14 under a tension of 10 kV for both imaging and EDS acquisition. TEM observation and
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16 chemical element identification were carried out on JEOL 2000F (JEOL, Japan) with an EDS
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18 probe (Oxford Instrument, UK), under a high tension of 200 kV.
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30 **3 Results and discussion**

31 **3.1 Microstructure and chemicals on the as-finished surface of brake disc and pad**

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33 A representative OM image of an as-polished surface of the carbon ceramic composite disc is
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35 shown in Figure 1(a). Carbon fibre/carbon (C_f/C), silicon carbide (SiC) and silicon (Si)
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37 regions are differentiated distinctly under polarised and DIC lighting conditions, as labelled in
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39 the image. It is noted that the carbon ceramic composite is highly heterogeneous in terms of
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41 the size and distribution of SiC/Si and C_f/C regions. For a ceramic composite manufactured
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43 via silicon-melt infiltration, Si in each SiC/Si region is always surrounded by SiC that is
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45 developed through a chemical reaction between silicon and carbon; each carbon fibre in a
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47 C_f/C region is continually wrapped by pyrolytic carbon (PyC) which is derived from
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49 carbonaceous resins, or gases, depending on which manufacture route is used.²²
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Microstructure details of this type of composite are reported in literature.^{23,24,25} The hierarchy structure among the constituents is maintained similar for all composites, but their relative fractions vary from one material to another, which is entirely decided by manufacturers who may wish to tailor the microstructure to achieve pre-specified attributes through adapting their own manufacture route and processing parameters. In ref. 13, two types of C_f/C-SiC were studied on the impact of fraction of each constituent on friction performance by testing them against same organic pad. The carbon ceramic composite used for the brake disc of brake-H is a similar material used in this study. By using image software, Image-J (NIH, USA), the area fractions of SiC, Si and C_f/C regions are estimated, and summarised in Table 2.

Figure 1(b) shows a representative OM image of an as-polished surface of the pad used in this study. The main constituents, such as steel and copper are distinguished clearly, as labelled in the image. Other constituents, acting as fillers, abrasives, lubricants and binders cannot be resolved and recognized straightforwardly under the current surface finishing and OM imaging conditions. An EDS probe attached to a SEM was therefore used to detect the typical chemical elements. The average weight fraction of each detected chemical element is summarised in Table 3. It is noted that a significant amount of Fe, Cu, C and O exists inside the pad. Whilst the corresponding chemical compounds cannot be identified without other analytical methods involved, such chemical information should be helpful enough in retrieving the evolution of friction surface of the disc after braking. It is worth noting that, due to the highly heterogeneous nature of an organic friction material, the quantitative value for

each element is informative only.

3.2 Friction surface developed on the organic pad after bedding tests

After completing test segment-I, the surfaces of the pads are visually smooth and lustrous. Small pits and fissures are noted on the surface, but their total fraction is less than 10% of the total surface area, according to the estimation from the images. Representative morphology of friction surface is shown in Figure 2. Compared to the polished surface before testing, the friction surface was experienced severe smearing, as demonstrated by the clearly visible abrasion trails in Figure 2. SEM/EDS analysis shows that the surface is composed of Fe, Cu, C and O as the primary chemical elements, and Mo, Mg, Al, S, Ca, Ti, Sn and etc. as minor ones, indicating oxidised Cu and Fe are likely the dominant crystallites on the friction surface, similar constituents found on friction surface of different organic pads by other researchers^{7,26,27,28}. The oxides of Cu and Fe ought to be the consequence of oxidation of metallic Cu and Fe during the braking. Majority of the C detected must have originated from the graphite and/or resin included in the pad formulation.

It is worth pointing out that for a brake composed of cast iron disc and organic pad, the oxide friction layer, particularly the ferritic oxide crystallites, on pad surface might be transferred from the disc surface. In this study, there is no such a source on the disc surface available for such a way of material transfer, but whether C, SiC and Si had transferred from composite disc surface to pad surface remains unknown due to lack of detailed analysis. The observation however does suggest that, whilst the brake disc is a non-metallic material, the friction surface

chemical composition of the organic pad is very similar to that after braking against the cast iron disc. We think that such a fact should be attributed to the formulation of the pad, where a significant amount of copper and steel in the formulation make direct contribution for the development of oxidised friction surface. The development of such friction surface can be retrieved by following the friction plateau model proposed by Eriksson and Jacobson⁷, but more detailed analysis is out of the range of this paper with a focus on the friction surface of the composite brake disc.

3.3 Friction surface structure and chemical composition on the C_f/C-SiC disc after bedding tests

I. Overview

An optical microscopy image of a core, taken off from the friction surface after accomplishing testing segment-I, is presented in Figure 3(a). The low magnification OM image of the friction surface shows no distinguishable features. When the surface was imaged with SEM under increased magnification, two types of regions become distinguishable based on contrast: one in dark grey, and another in white grey, as shown in Figure 3(b). Note, the dark grey and white grey are used for the convenience of description of the observation; there is no rigorously scientific definition here, and details in these regions will be disclosed by other examinations in this paper. Without surface topographic contribution accounted, the contrast in a SEM image has a strong correlation to the atomic weight of all chemical elements inside the probed region by an electron beam: darker contrast tends to be contributed by lighter elements, and whiter one by heavier elements. SEM/EDS analysis of these two regions shows

1 that the primary chemical elements are C and O, followed by Fe, Cu and Si. The whiter region
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3 should have more Cu, Fe and O, and the darker one more C. Nonetheless, it is unlikely to
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5 establish a clear picture on the structure and chemical composition of the friction surface by
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7 using the SEM/EDS data alone, primarily due to the probe depth of an electronic beam is
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9 hardly quantified. To have a better view of the friction surface in chemical composition and
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11 structure, we had cross-section samples prepared on the friction surface with FIB for high
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13 magnification examination with TEM/EDS.
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22 Figure 3 (c) shows the cross-section of the friction surface after digging two trenches on both
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24 sides of a chosen region. The cross-section was imaged with secondary electrons, and shows
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26 contrast pattern that can be linked to that of in-plane SEM image shown in Figure 3(b).
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28 TEM/EDS analysis confirms that the dark grey corresponded to a SiC region and the white
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30 grey to a TM region, as shown in Figure 3(d). The TM is deposited in a crevice surrounded by
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32 PyC and SiC, and the boundary with SiC and PyC is outlined by a dashed red line. In Figure
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34 3(d), the friction surface is visible in the top right corner. The TM, showing dark grey
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36 contrast under the current TEM imaging conditions, occupies most of the upper part of the
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38 image. The SiC crystal and C_f/PyC, showing dark grey contrast, is in the bottom right- and
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40 left-hand side, respective. The white spots/regions showing no chemical element in EDS
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42 spectrum are simply holes, or cracks.
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52 The observation reveals the following facts.
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- 56 (a) No carbon fibre and pyrolytic carbon regions are exposed to the surface of the
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1 brake disc after bedding; they are found underneath the surface only, and fully
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3 covered by the transferred materials.
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6 (b) SiC regions in the carbon ceramic composites are exposed as part of the friction
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8 surface with an estimated area fraction of ~25%, approximately half of the area
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10 fraction of SiC/Si region estimated from the as-polished surface before testing. In
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12 addition, a thin friction layer exists on top of SiC regions.
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17 (c) No Si regions are seen on or near the friction surface.
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20 Based on the observation, the structure of the friction surface after braking test is
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22 schematically shown in Figure 4, illustrating the changes before and after braking. A
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24 step-by-step observation of friction surface development in previous research¹⁵ showed that
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26 C_f and PyC on the friction surface are susceptible to fracture during braking. Hence, we
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28 believe that such fracture during braking leaves the surface with voids that are subsequently
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30 filled with TM, making the friction surface no carbon region left. SiC is a much stronger
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32 ceramic phase than C_f and PyC, but surface fracture can be developed by an abrasion
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34 process^{29,30}. Most recent research^{13,17} shows that a repeated abrasion during braking promotes
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36 the development of lattice defects such as dislocations and twins on the contact surface of SiC,
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38 and the accumulation of dislocations/twins on the surface leads to a stress concentration to
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40 initiate microcracking inside SiC crystallites. The liaison of these microcracks on the surface
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42 eventually leads to chipping off, or dislodging of a SiC region from the surface. This surface
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44 fracture was further evidenced in a SiC ceramic disc when it was tested against a mild steel
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46 pad on a laboratory-scale dynamometer.³¹ Therefore, surface fracture should be the primary
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48 reason that why not all SiC regions are shown on the friction surface. Later in this section,
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dislocation slips will be shown in SiC crystallites.

Si regions are always surrounded by SiC crystallites in the virgin composite. The loss of SiC may lead to the loss of Si. There may be other reasons, nonetheless. For instance, high temperature on the friction surface might make the Si regions melted down, and then mixed with moving debris to become part of the transferred materials.

To reveal more details on the composition of the TM and SiC regions, we had region A and B in Figure 3(c) further examined with TEM/EDS. The results are presented below.

II. Transferred materials (TM) regions

The structure of TM included in region A is shown in Figure 5. Figure 5(a) shows the cross sectional view of the TM region under high magnification. There are two distinguishable layers inside the TM: a top layer with a thickness of ~100 nm and an underneath region. The former one is called *friction layer (FL)* in this paper, the same layer as found before^{13,17}, and the later one called *underneath layer (UL)*. By applying an aperture with a size of 200 nm, a representative selected area diffraction pattern for the FL and UL is shown in Figure 5 (b) and (c), respectively. Both regions are mostly composed of crystallites, because of clear diffraction spots. However, the sizes of these crystallites seem to be quite different. In UL, most of the diffraction spots are sitting on rings with different radii, indicating that multiple crystallites of the same material but different orientations existed inside a region of ~200 nm in diameter. Some diffraction spots have much stronger intensity than others, indicating that

some crystallites are much bigger than others, which can be recognised in the diffraction contrast image shown in Figure 5(a). In FL, the diffraction spots are also sitting on rings, but there is no spot showing significantly stronger intensity, implying that the crystallites in FL are much smaller with more homogeneous size in general, comparing to those in UL. By indexing the rings, we can confirm that the radius ratios of the arrowed rings match the ratios of lattice plane distance of (110), (111), (211) and (220) of Cu_2O (XRD JCPDS 65-3288), showing cuprite Cu_2O is the main crystal phase inside transferred materials regions. It is worth pointing out that even for the FL, diffraction patterns alone cannot exclude the existence of other crystals, but their quantity must be much smaller if they do exist, comparing to the Cu_2O . For the UL, we do notice some diffraction spots off the diffraction rings of Cu_2O , indicating that there are other crystallites in the transferred materials. However, it is difficult to index these diffraction spots without ambiguity. When the examination was moved to a deeper position, however, large size FeO crystallites are identified with full confidence, as labelled in Figure 5(d), where the FeO crystallites have a size from ~ 0.5 to $5\ \mu\text{m}$.

To validate the conclusions drawn from the diffraction patterns and uncover more information about the TM, the EDS spectra acquired from UL is shown in Figure 5(e). Inside UL, a range of chemical elements are identified, including Ca, Ti, O, Fe, Cu, Zn, Sn, P, Al, Si, etc. which approximately match those probed on the as-finished brake pad. Judging from the intensity of the energy dispersive peaks, it is obvious that Cu, Fe and O are the primary chemical elements, supporting a conclusion that the primary crystallites inside transferred materials are Cu_2O and

1 FeO. The only source for the Cu_2O and FeO is the pad, likely generated through an oxidation
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3 of debris of copper and steel during friction.⁷ Note crystallites composed of some of the minor
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5 elements should not be excluded, though their quantity should be relatively small. The types
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7 of crystallites should be largely defined by the formulation of the pad used. The above
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9 analysis demonstrates that the transferred materials deposited on the surface of the composite
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11 disc should be predominantly from the organic pad. The significant amount of Si might come
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13 from the Si embedded in SiC regions on surface of the composite disc, as discussed in
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15 previous section.
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25 Figure 5(f) show the EDS spectrum acquired in FL, and the main chemical elements include
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27 only Cu, O, and Si. The significant amount of Cu and O supports the conclusion that Cu_2O
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29 should be the main crystallites for the FL. However, the strong Si peak is hardly associated
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31 with any crystallites due to the fact that (i) no extra diffraction spots were seen and (ii) there
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33 are no chemical elements with detectable amount to form silicates. Therefore, we suspect that
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35 Si might exist in the friction layer as amorphous phase. Such amorphous Si, or SiO_x ($x \leq 2$)
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37 was clearly identified on the friction surface of a carbon ceramics disc after braking testing¹⁷.
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39 The source of amorphous Si or SiO_x might be the carbon ceramic disc where significant
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41 amount of residual Si is seen. Note, among the 5 cross section TEM samples examined in this
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43 investigation, there is no single Si region found on the friction surface. We think the
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45 temperature at the contact surface might be high enough to have all Si regions melted, and
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47 probably oxidised partially if not fully, during braking. This vitreous phase can wet the debris
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49 to form part of the transferred materials. In the early study, there was detailed analysis on the
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role of Si inside the transferred materials¹⁷.

Between the FL and UL, the observed difference in crystallite size and chemical composition implies that transferred materials might develop through the direct deposition of friction debris. Once the enough debris is filled in a void, friction smearing is followed. The smearing process likely brings high pressure and high temperature, leading to densification and crystal refinement. The densification process is also called tribo-sintering³², and both high pressure and temperature promote this process. For crystal refinement, pressure dictate the crystal size by following a relationship as:

$$D = K\sigma^m$$

where σ is the flow stress, and K and m are material- and mechanism-specific constants^{33,34,35}, and typical m value is about 0.7-1.6. When the compact is a continuum linear elastic body, the contact pressure declines as the position goes deeper by following a relationship inversely proportional to the squared distance from the contact surface,³⁶. Hence the crystal refinement is significantly strengthened in FL because the flow stress there is about several to tens times larger than in tUL depending on the exact position.

Why are there no other chemical elements in FL, as shown in the observation? This may be related to a “bleaching process” at higher temperatures, or other chemicals may exist in the amorphous state with a content level out of the resolution of the EDS. In general, the flash temperature on the contact surface should be the highest. Underneath the surface, temperature reduced significantly as a consequence of heat sink effect. However, more research is needed

in order to have convincing understanding of this phenomenon.

III. SiC regions

The SiC region, labelled as B in Figure 3(c), on the friction surface was examined with TEM/EDS. The representative results are summarised in Figure 6. On the friction surface, there is a layer with a thickness of ~100 nm different from the SiC. EDS spectrum shows that the main chemicals are Cu and Si. Due to oxygen peak is not particularly strong, we therefore infer the layer might include alloy of Cu and Si. Deposition of such alloy on the surface of SiC was observed on the surface of C_f/C-SiC disk tested on a laboratory-scale dynamometer¹⁷. This is fundamentally related to the low wetting angle of Cu-Si alloy on the surface of SiC. Underneath the friction transfer layer, dark contrast appears under the chosen diffraction contrast imaging condition. Such contrast is caused by the plastic slips that are activated by friction shear stress for accommodating mechanical deformation during braking. Again, similar slips were observed on friction surface of brake discs tested on laboratory-scale dynamometer^{13,17}. These slips likely take part of the responsibility for surface fracture in SiC region, as discussed under sub-section (a) of section 3.3.

It is worth pointing out that the friction surface in SiC region observed here is very similar to that tested on laboratory-scale dynamometer. However, there was no comparable features appeared in the C_f/C regions between the friction surfaces of brake discs tested on laboratory dynamometer and full scale one¹⁵. We think the degree of damage in the C_f/C regions, through either oxidation or surface fracture, is much larger when the brake disc is tested on full scale

dynamometer than that on a laboratory-scale one.

As shown by SEM and TEM examination, almost all friction transferred materials on friction surface of the brake disc are originated from the pad, and locate among SiC and C_f/C regions. Therefore, it is necessary to reveal more details on the interfaces between the TM and the carbon ceramic composite.

IV. Interface between friction transfer materials and carbon ceramic composites

i. Interface between TM and C_f/C region.

Whilst C_f/C region is not found on the friction surface, it is widely seen underneath the TM. Figure 7(a) shows an example that the deposited TM in the lower part of the image interfaced with C_f and PyC in the upper part of the image. EDS spectra acquired from sites A, B and C, as labelled around the interface region in the TEM image, show clear difference in chemical elements, making carbon region and transferred materials differentiated clearly. The structure of the interface includes the intercalation between carbon and the TM with the integration of low melting temperature constituents, such as Sn based compounds. Whilst we cannot conclude if any chemical bonding is developed between carbon and transferred materials, this intercalated structure looks very robust, making the TM difficult wiped off from the interface completely.

Because friction-induced flash temperature on a friction surface can be well above the oxidation temperature of carbon, which is around 550 °C,³⁷ the existence of a sustainable TM

on top of the C_f/C region can be very beneficial in preventing or delaying oxidation of carbon materials during braking. On the other hand, the longevity of TM on friction surface is one of the key factors that influence the stability of friction performance. Therefore, it is essential for constituents in a pad formulation to be able to promote the bonding between the TM and C_f/C, which can improve the sustainability of TM, hence the bedding speed and friction stability.

ii. Interface between TM and SiC

Figure 8(a) shows the structure around the interface between TM and Si/SiC region. The interface can be distinguished in a diffraction contrast TEM image. EDS spectra acquired at positions across the interface provide further information on chemical composition. Position A and B are on the TM side, and the EDS spectra show the primary chemical elements in TM as O, Fe, Cu, Mg and Si. Position C and D are on the Si/SiC side, and the primary chemical elements are C and Si only. It seems there is no inter-diffusion layer between Si/SiC and the TM. We may therefore conclude that the TM and the Si/SiC have no strong chemical bonding. However, without the support of high resolution TEM and electron energy loss spectroscopy (EELS), this conclusion cannot be robust enough.

Along the contour of the interface, there are no clear voids or gaps. We believe that when the TM is deposited in the voids on the friction surface of the brake disc, the normal braking pressure has the TM filled in the voids with a tight fitting to the inner surface configuration of a void. Therefore, the mechanical opposition provided by the SiC regions, remained as part of friction surface, and the wavy interface between TM and the carbon ceramic composite

should be the principle resistance to the friction force that tends to strip off the transferred materials from the friction surface.

4. Summary

TEM cross-section, along with OM and SEM in-plane examination of friction surfaces of carbon ceramic brake disc and organic pad after completing segment-I braking test on a full-scale dynamometer has shown that:

- (a) The friction surface structure of C_f/C-SiC brake disc is composed of two characteristic regions: friction transferred materials (TM) and SiC. On top of them, a thin friction layer (FL) is developed.
- (b) The friction surface of organic pad is fully bedded with ~90% area covered by a smooth and lustrous layer including Fe, Cu, C and O as main chemical elements, and Mo, Mg, Al, Si, S, Ca, Ti, Sn as minor ones.
- (c) The chemicals of TM on carbon ceramic brake disc surface include O, Fe, Cu, Mg, Si as the main ones, close to those on the pad, suggesting that wear debris generated from the pad are the main source of the TM.
- (d) The TM regions on carbon ceramic brake disc surface are largely composed of crystallites and compacted densely in craters on brake disc surface. The crystals are subject to refinement under the braking pressure and temperature, and the refinement is enhanced as the cross-section position moves closer to top friction surface.
- (e) The interfaces between the TM and C_f/C, and SiC regions on the brake disc surface are tightly in contact, but no clear evidence shows that any chemical diffusion bond exists to enhance its sustainability in all braking environmental conditions.
- (f) The main chemical elements in the FL on the brake disc surface are Fe, Cu, O and Si, and CuO₂ and FeO crystallites in a size around tens nanometers are the main constituents that are densely compacted.

(g) In the SiC regions, the plastic deformation slips are developed immediately below the FL, likely responsible for the surface fracture in SiC that leads to only about half of SiC remained on the friction surface.

The findings have shown that the composition of friction surface of carbon ceramic disc tested on full-scale dynamometer is different from that on laboratory-scale dynamometer. The interface between TM and carbon ceramic composite is not proved to be chemically bonded on either testing rig, hence fast bedding, high stable friction performance under all braking conditions (including wet) cannot be achieved. However, details on the bond need further research with high resolution TEM and EELS.

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(b)

Figure 1. Representative optical microscopy images showing the microstructure of the polished surface of the (a) brake disc composite, and (b) pad friction material. Key constituents on the surface are labelled.

Figure 2. Representative morphology of friction surface of a pad after completing braking test of the 6 sub-sections.

Figure(s)

(a)

(b)

(c)

(d)

Figure 3 Overview of friction surface of C_f/C-SiC brake disc after completing braking test of 6 sub-sections. (a) Low magnification optical microscopy image of the core taken from as-braked surface. (b) SEM image of friction surface of the core. (c) SEM image of a cross-section of the friction surface produced with FIB. Region A and B were further examined with TEM. (d) TEM image of the cross section, showing the white grey region in SEM image is composed of transferred materials (TM).

Figure 4. Schematic illustration of the structure of friction surface (a) before and (b) after braking testing. TM represents transferred materials and FL the friction layer. Before braking testing, the surface is composed of SiC, Si, and pyrolytic carbon and carbon fibre (PyC/C_f). After completing bedding braking tests, the surface is composed of SiC and TM regions, plus a thin layer of FL on top of all SiC and TM regions.

Figure(s)

(a)

(b)

(c)

(d)

(e)

(f)

Figure 5 Microstructure and chemical details of the transferred materials on the friction surface of C_f/C-SiC composite brake disc after completing bedding braking tests. (a) In the transferred materials region, a friction layer (FL) was developed with a structure different from the underneath layer (UL). (b) & (c) Selected area diffraction pattern acquired from the FL and UL region, respectively. (d) Identified FeO crystallites inside deeper position of a transferred materials region. (e) & (f) EDS spectrum acquired from the FL and UL, respectively.

Figure(s)

(d)

(b)

Figure 6 Microstructure and chemical details of SiC regions on the friction surface of C_f/C-SiC composite brake disc after completing bedding braking tests. (a) Bright field TEM image of SiC region that is composed of SiC crystal and a thin friction layer (FL) on its top. Dislocations/twins were activated in SiC crystal, and the primary chemical elements are shown in a EDS spectrum in (b).

(a)

(b)

Figure 7 A representative interface structure between transferred materials (TM) and a C_f/C region. The structure around the interface is shown in (a), and A, B and C indicate the positions where EDS spectrum was acquired and displayed in (b) respectively.

(a)

(b)

Figure 8 A representative interface structure between transferred materials (TM) and a SiC/Si region. The structure around the interface is shown in (a), and A, B,C and D indicate the positions where EDS spectra were acquired and displayed in (b), respectively.

Table I Details of testing conditions for each sub-section designed based on AK Master guideline

Subsection	Pressure applied on master cylinder (bar)	Braking Speed (km/h)	Temperature (°C)
Green	30	80-30	Ambient to 100
Bedding	15-51	80-30	At 100
Characteristics	30	80-30	At 100
Pressure series	10	40-5	At 100
	20	80-40	
	30	120-80	
	40	160-130	
	50	200-170	
	60		
	70		
	80		

Table 2 Quantitatively estimated area fraction of main constituents on the as-finished friction surface of the carbon ceramic composite disc.

	Area fraction of different regions in C _f /C-SiC composite (%)		
Brake	SiC	Si	C _f /C
Brake-H	41.78±5.8	11.34±4.8	47.28±6.4

Table 3 Chemical composition on as-finished surface of a pad, detected with EDS

Chemical element	Fe	Cu	C	O	Si	Al	Mg
Weight %	33.0± 10.5	22.4± 8.8	11.9±2.9	15.4±4.2	1.6±1.6	2.1±0.8	2.0±1.4