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Evolution in microstructure and compression behaviour of a metallic sintered friction material after braking

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Abstract

Due to the complexity of friction materials, the characterization of the tribological properties is prioritised over the bulk material properties even though the tribology is expected to be influenced by the material behaviour. The extent of this relationship is still unknown and further knowledge is required to account for the load history and evolution of the bulk properties. With this view, the compression behaviour and microstructure of a semi-metallic friction material with reduced formulation were investigated before and after a braking program. The thermal loading was monitored with inserted thermocouples. Uniaxial compression tests coupled with Digital Image Correlation (DIC) show significant changes in the worn material, which develops a compression behaviour similar to that of a tri-layered material. The microstructural analysis indicates microcracking and carbon diffusion in the Fe-phase. The thermal loading was found to be the key parameter controlling both the friction behaviour and evolution of the material properties. The expected effects of material evolution on the contact uniformity, durability and tribology are discussed.

Keywords: Semi-metallic sintered material, Braking load history, Uniaxial compression tests, Digital image correlation, DIC, Scanning Electron Microscopy, SEM, Evolution of bulk properties

1. Introduction

Friction materials are at the heart of defining the performance of braking systems. Because these materials are 2 crucial to the transportation safety, they have to fulfil numerous requirements involving different fields: a constant 3 level of friction whatever the conditions of use to ensure the stopping distance, a low rate of wear to decrease the life-4 cycle costs, high mechanical strength for integrity, and limited noise and vibration, just to name a few. The extensive 5 requirements have led to the development of very complex materials containing many different component types 6 [1, 2]. The use of a metal-matrix composite (MMC) is needed to withstand the high temperature due the dissipation of very high energies (e.g., in the railway industry). In addition, fillers (e.g., ceramic and graphite particles) need to 8 be added to fulfil the wear and friction requirements. In the past, this complexity (material and conditions of use) has 9 led to empirical developments mostly reliant on trial-error iterations. 10 The investigation of the friction level and wear rates along with the characterization of the contact surface (usually 11

limited to a depth of $\sim 100 \,\mu\text{m}$) provides knowledge of some key mechanisms involved in the tribology. It has been reported that the material immediately beneath the contact surface (sub-surface material) has a significant influence

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on the mechanisms controlling the generation of wear sources (e.g., erosion, cracks or hot-spots) [2? -9]. Regarding 14 the bulk properties, the studies published to date mainly focus on the mechanisms involved in disc cracking. For 15 cast-iron [10, 11] and steel [12–15] materials, it was shown that the cracking is induced by phase transformations 16 occurring during the braking. In [14, 15], the transformation from austenite to martensite/bainite is observed in steel 17 discs that endured cycles of high thermal loading (800-1200°C) followed by rapid cooling. On the other hand, very 18 few studies deal with the evolution of the friction material facing the disc [7, 16]. In particular, reference [16] reported 19 a strong modification in the mechanical properties of a semi-metallic material after a high-energy braking sequence 20 was applied (e.g., the stiffness decreased from 5,800 MPa in the virgin material to 1,100 MPa in the worn one). 21

The above studies indicate that the evolution of friction materials during braking has a multilevel nature (i.e., 22 changes in the bulk and subsurface affect the contact surface). The further advancement of friction materials requires 23 in-depth understanding of how the evolution in bulk properties influences the braking performance. The present study 24 approaches the challenge by investigating the mechanisms involved in the evolution of the compression behaviour. 25 Moreover, a detailed characterization of the mechanical properties will lead to more realistic numerical simulations, 26 ultimately contributing to a reduction in the empirical dependency of the braking-process simulations. The useful-27 ness of thermomechanical modelling is shown, for example, in references [16–22]. These studies used numerical 28 simulations to investigate the influence of brake-pad design on the development of contact pressure localisations and 29 subsequent formation of hot spots. 30

Given that the multi-loading and multi-physics nature of the braking process generally obscures the elucidation of mechanisms, the present study utilizes a sintered friction material with reduced formulation (only nine components) and the mechanical characterization is limited to uniaxial compression tests. The braking program is carried out using a real-scale dynamometer, where the temperature level of the friction material is monitored with inserted thermocouples. The friction behaviour and differences in compressive properties and microstructure are examined. The mechanisms involved in the evolution of the compression behaviour are discussed along with the expected influence of these mechanisms on the braking performance.

2. Material and methods

39 2.1. Material

The *simplified* formulation (reduced to *only* nine components) is given in Table 1. This material can be considered as a (FeCu-based) metal matrix composite that contains particles of graphite, ceramics and a small percentage of

⁴² fillers and lubricant particles, which are included in the metallic matrix percentage.

Main Components	Mass (%)	Size
Metallic Matrix (MM)	70	Sintering powder
		$< 100 \mu{ m m}$
Graphites 1 & 2	20	100-600 µm
		400-1100 μm
Ceramics 1 & 2	10	100-200 µm
		100-250 µm

Table 1. Friction material composition.

The manufacturing process consists of three main steps: (i) preparation of the raw powders, including weighing and mixing, (ii) compacting of the powders (up to 1,000 tons along the Z axis) using a shaped mould to consolidate the and densify the model (also known as green compact), and (iii) sintering in a protective-atmosphere furnace for 8h at ~1,000°C. The final dimensions of the brake-pad is surface = 200 cm² and height = 22.5 mm. Note that the compacting and in-service loads are applied along the same direction .

The wide range in the size of particles leads to a complex multiscale material. As shown in Fig. 1a, only Graphite 2 particles can be observed at a macroscopic scale length (*i.e.*, with the naked eye). In addition to Graphite 2 particles, Graphite 1 (also shown in black but with smaller sizes) and the ceramic particles are distinguishable at the mesoscale (Fig. 1b). Finally, the two principal phases of the metallic matrix (ferrite iron and copper) can be observed at the microscopic scale length (Fig. 1c-d). Note that some pearlite colonies are observed in Fig. 1c-d. These colonies

⁵⁴ during the sintering process.



Figure 1. Microstructure of the studied friction material: (a) Light-optical image at the macroscopic scale length. (b) Backscattered Electron (BSE) image at the mesoscopic scale length (c) Secondary Electron (SE) image of the metallic matrix at the microscopic scale length and (d) same as previous image but in BSE mode. Here, the pearlite colonies are almost indistinguishable. The surface was etched with Nital reagent for 5 s.

The density of the virgin material was measured using the Archimedes method, which compares the sample 55 weight when measured in dry air and saturated in ethanol, giving information about the open porosity. For the studied 56 material, the porosity content is 14% (mass percentage). Synchrotron Computed Tomography (SCT, carried out at 57 ID19 beamline of the European Synchrotron Radiation Facility (ESRF), Grenoble, France) was performed to gain 58 qualitative knowledge about the porosity. The investigated sample was a cylinder 5 mm in diameter and 18 mm in 59 height (where the height is parallel to the compression direction). The scanning was performed at 110 KeV with 60 a voxel size of 3.425 µm³. The images were reconstructed from projections by applying Paganin's phase retrieval 61 algorithm [23] and subsequent filtered back-projection reconstruction for parallel beam. The SCT images correspond 62 to the mesoscopic scale length. 63

In the reconstructed images (Fig. 2a), the two types of graphite particles and Ceramic 1 particles exhibit similar 64 dark-grey levels. Ceramic 2 particles are shown in light-grey and the metallic matrix is close to white (grey-scale 65 values of 220-255). Close observation of the graphite particles indicates that these particles contain some crack-like 66 intrinsic porosity (Fig. 2b). It is likely that the intra-porosity only occurs within Graphite 2 particles, as this type of 67 porosity is not observed within the smallest Graphite 1 particles. The SCT images also show some interface-porosity (considered as a discontinuity, Fig. 2c) between the metallic matrix and the graphite/ceramic particles, as well as 69 between individual graphite/ceramic particles. It is, therefore, probable that the porosity distribution throughout this 70 friction material is complex and heterogeneous. Moreover, it is likely that this mesoscale porosity only accounts for a 71 portion of the 14% measured by the Archimedes method. Nevertheless, a detailed study of the nanoporosity content 72 within the metallic matrix is outside of the scope of this study. 73

74 2.2. Methods

75 2.2.1. Braking set-up and protocol

A real-scale railway dynamometer, replicating those used in TGV high-speed trains, was used to apply realistic 76 braking loads to the friction pair. It was equipped with a solid disc (non-ventilated) made of a 28CrMoV5 steel 77 (Fig. 3a) and brake pads made of the friction material described in Section 2.1. The brake pads and disc were brought 78 into contact by a pneumatic caliper actuated with a pressure-control system. In addition to the standard output data 70 usually acquired during typical laboratory experiments (e.g., actuator pressure, torque and rotation speed), thermal 80 monitoring was carried out to record the temperatures induced in the friction pair. The pad thermocouples were 81 inserted from the back side of each brake pad at two different depths (3 and 6 mm, measured from the virgin contact 82 surface). The thermocouple locations are shown in Fig. 3b. Regarding the disc, three rubbing thermocouples were 83



Figure 2. (a) 2D slice (transverse plane) of the reconstructed synchrotron CT data corresponding to the material mesoscopic scale length. (b) Detail of a Graphite 2 particle showing some intrinsic porosity. (c) Detail of the metallic matrix, where the porosity/discontinuity between the matrix and the graphite/ceramics particles in addition to some porosity at the interface between individual graphite/ceramic particles can be observed.

⁸⁴ used (see Fig. 3a) as well as an infrared camera located in front of the disc surface to record the occurrence of thermal
 ⁸⁵ localisations.

The test program was specifically designed to investigate the level of thermal loading induced by an increasing energy dissipation during braking and follows the guidelines of the Union Internationale des Chemins de fer (UIC, 87 International Union of Railways is the translation in English). An initial S0 braking sequence, not noted in Fig. 3c, 88 was performed prior to the program. This initial braking was performed with the same normal force used in S1V0. 89 Once 80% of the contact surface was worn away by friction (the verification was done visually), the program shown in 90 Fig. 3c was started. This braking program consisted of three blocks (series) of low, medium and high braking energy 91 (S1, S2 and S3). The increment in braking energy was achieved by increasing the inertial mass to be stopped (see 92 fifth column in Fig. 3c). The braking force applied on the pad (defined as normal force in Fig. 3c) was also increased 93 in each series. Before the starting of each series, five low-energy braking sequences were carried out as a breaking-94 in procedure, meant to resettle the contact surfaces for the following series. These series were subdivided into five 95 braking sequences, where the speed (equivalent to the speed of an in-service train) was progressively increased. Each 96 braking sequence is noted as S(i)V(i). 97

98 2.2.2. Uniaxial compression tests

Compression tests required the extraction of cubic samples ($\sim 20 \times 20 \times 20 \text{ mm}^3$, a volume considered large enough 99 to be representative of the macroscopic behaviour) from the central region of the brake pad (see Fig. 4a). A testing 100 machine equipped with a 50 kN load cell was used with a loading rate of 0.01 mm/s. The loading/unloading direction 101 corresponds to the in-service braking direction. No important barrelling effects were observed during the experiments 102 and, therefore, the cross section dimension was kept constant at 20×20 mm². The top and bottom surfaces were 103 ground to ensure parallel alignment, with a misalignment error lower than 20 μ m. In the case of the virgin material, 104 two conventional strain gauges were glued to the lateral surfaces to obtain macro-strain curves. The front surface was 105 ground down using P500, P1200, P2500 and P4000 grit paper (without water) and subsequently polished using 3 μ m 106 diamond paste and 0.04 μ m colloidal silica suspension before etching the surface with Nital reagent for no longer than 107 5 s. This surface was sprayed with black paint to create the background before applying an airbrushed white speckle 108 pattern. More details about the uniaxial experiment can be found in [16]. 109

¹¹⁰ During the compression tests, two optical fibres illuminatee the sprayed surface while a digital camera (16-bit ¹¹¹ digitisation, 2048×2048 pixel array) recorded macroscopic images of the frontal surface ($\sim 20 \times 20 \text{ mm}^2$) every 500 ms ¹¹² (see Fig. 4b). The pixel size was $\sim 100 \,\mu$ m. YaDICs free software [24] was used to calculate the displacement fields.



Figure 3. (a) Image of the real-scale dynamometer set-up. (b) Localisation of the pad-inserted thermocouples located at 3 and 6 mm depth from the contact surface. (c) Table summarising the program carried out during the braking experiment.

¹¹³ This software uses the entire image as a sampling grid (*i.e.*, total sampling), and uses a bicubic interpolation for the

¹¹⁴ grey level evaluation at non-integer coordinates. The Optical Flow elastic transform based on the Finite Element

¹¹⁵ Model (OF-FEM) kinematics method is used with a discretized pixel subset of 14×14 pixels. The OF-FEM method ¹¹⁶ reduces measurement uncertainties by ensuring the continuity of the found displacement field over the region of

reduces measurement uncertainties by ensuring the continuity of the found displacement field over the region of interest (ROI, see Fig 4b) [25]. The strain was computed from the displacement field using an in-house Python script.

¹¹⁷ interest (ROI, see Fig 4b) [25]. The strain was computed from the displacement field using an in-house Python script. ¹¹⁸ For the sake of simplicity, the study of the mechanical behaviour was limited to the normal strain (ε_{zz}). A good

For the sake of simplicity, the study of the mechanical behaviour was limited to the normal strain (ε_{zz}). A good agreement was found between the DIC stress-strain curves and those obtained using gauges (although the DIC results

tended to exhibit slightly lower strain values, >10%) and no significant out-of-plane effects were observed [22].



(a)

6



Figure 4. (a) Image of a $20 \times 20 \times 20 \text{ mm}^3$ cube, where a speckle pattern has been applied to the front surface and two strain gauges have been glued to the lateral surfaces. (b) Image of the experimental configuration of the compression test showing the ROI used for the computation of the vertical (Z) displacement field (expressed in units of pixel).

121 3. Results

122 3.1. Braking experiment

123 3.1.1. Thermal measurements

Fig. 5a shows the mean temperature obtained from the rubbing thermocouples (located on the disc and denoted in 124 the figure by filled bars) and the 3 mm inserted thermocouples (located on the pad and denoted in the figure by empty 125 bars). Some precautions must be taken when analysing these measurements. First, the disc-rubbing thermocouple 126 results correspond to the mean of the measurements at the inner, medium and outer radius (see Fig. 3a). Apart 127 from the fact that this provides an average value over the radius and the circumference, the main downside of these 128 measurement is the fact is the contact continuity between the thermocouples and the disc is interrupted when the disc 129 surface is not flat. This situation is observed during high-energy braking sequences, where thermo-mechanical effects 130 usually lead to the formation of hotspots and undulating distortions [8]. Also, these perturbations are more significant 131 with increasing speeds. Therefore, the values from the rubbing thermocouples have to be considered with caution, 132 particularly in the case of high speed braking, where the thermocouples tend to measure values that are lower than the 133 actual surface temperatures. 134

Wear of the brake pads induces a reduction in the distance between the contact surface and the inserted thermocouples. This effect becomes more intense with each subsequent braking series. Nevertheless, it is considered that, for similar heating levels, the reduction of thickness does not have a great impact on the temperature values because the thermal diffusivity of the sintered metallic matrix is known to be high. This point is confirmed by the small temperature difference (~100°C) observed between the recordings of 3 and 6 mm inserted thermocouples (Fig. 5b-c). Note that the wear reduction led to the destruction of one of the 3 mm inserted thermocouples (which occurred during S3V5, see bottom graph of Fig. 5b).

Fig. 5a shows that the pad temperature is close to that of the disc at low speeds, with increasing difference at higher

¹⁴³ speeds. These measured differences could be due to the contact interruptions between the rubbing thermocouples and



Figure 5. (a) Maximum temperatures in the disc (mean value of the rubbing thermocouple measurements) and pad (mean value of the measurements from the inserted thermocouples at 3 mm) for the three braking series. (b) 3 mm inserted thermocouples measurements for the three braking series at maximum speed (V5). (c) 6 mm inserted thermocouple measurements for the three braking series at maximum speed (V5). The legend for (b) and (c) is shown within the schematic brake-pad at the top of the graphs.

the disc, but also due to the fact that the disc temperature measurements are the mean of measurements at different 144 radius lengths. The difference of measured temperature between the inner and outer radii is, thus, not accounted for 145 and could be comparatively high. Nevertheless, a thermal gap between the disc and pad surfaces is often reported in 146 braking applications. This is because the contact is permanent for the pad but not for the disc, which can be considered 147 as an open thermodynamic system with alternating heating and cooling cycles. Therefore, higher temperatures are 148 usually expected at the pad contact surface. Fig. 5a also shows increasing temperature levels, which correspond to 149 increasing dissipated energies. Moreover, similar heating levels are observed between series of different speed and 150 inertial mass when the dissipated energy and friction power have a similar magnitude (e.g., S1V5, S2V4 and S3V3). 151

Fig. 5b shows the temperature recordings at maximum speed (V5) for the thermocouples located at 3 mm depth 152 from the contact surface. The highest maximum temperature was reached during the S3 series ($\sim 1,000^{\circ}$ C), which 153 corresponds to the highest dissipated energy and friction power. The S3V5 results exhibit a plateau stage that can 154 be related to saturation. Also, significant temperature variations are observed among the thermocouple locations 155 for all series. These variations probably correspond to macroscopic discontinuities at the contact surface. It has 156 been observed that the dynamic process of heating-dilatation-wear induced by friction leads to the loss of contact 157 uniformity for brake-pad surfaces as extensive as the ones studied in this work [17, 26]. Similar contact alterations 158 are observed in the case of 6 mm inserted thermocouples (Fig. 5c), although to a lower extend. Note that these 159 levels of temperature, and corresponding thermomechanical stress, are able to induce material changes such as phase 160 transformation or damage [18]. 161

162 3.1.2. Friction

The instantaneous coefficient of friction (μa) and the mean friction coefficient (μm), noted as COF hereafter, was calculated following the UIC standard [27]. μa is calculated for any instant of braking by the ratio of the tangential

force (F_t) to the normal contact $(F_b, \mu a = F_t/F_b)$. The tangential force is calculated by the ratio between the torque 165 and the mean of the contact radius. The normal force is obtained from the pneumatic actuator pressure. The evolution 166 of the COF during all braking sequences is shown in Fig. 6a. The fluctuations in friction during braking are higher in 167 S1 as well as in the low speed sequences of S2 and S3 series. Such steep growths in the COF at low speed (e.g., S1V1) 168 are often observed when the evolution in friction mechanisms promotes adhesion effects [6]. The average COF in each 169 braking sequence is shown in Fig. 6b, where two main tendencies can be observed. First, a stabilizing trend is observed 170 over the series, with S3 exhibiting the lowest differences in COF. This stabilizing trend over the series indicates that 171 the 3^{rd} body is evolving. Given the high level of thermal loading, it is likely that during S3 the tribological circuit 172 is strongly activated, with various wear mechanisms occurring simultaneously (i.e, generation of wear sources from 173 material damage and/or adhesion, debris accumulation as well as ruptures in the 3rd body). Secondly, the friction level 174 tends to be similar in for sequences with equivalent levels of energy dissipation (e.g., S1V5, S2V4 and S3V3). 175



Figure 6. (a) Evolution of COF during braking for the three series. (b) COF average values (with error bars) for all braking series.

176 3.1.3. Surface analysis

Fig. 7a shows the contact surface for the right half-pad at the end of the braking program. This half-pad exhibits 177 a macrocrack on the top-left region which seems to be caused by the presence of a 3 mm depth thermocouple. The 178 main part of the contact surface seems to be covered by a thick layer of 3^{rd} body. In general, the examination of 179 the contact surface indicates that the tribological circuit was fully developed when the program ended. The analysis 180 at high magnification shows a compacted tribo-layer on the right of Fig. 7b, and some of the components (mostly 181 graphites) lying on the left, where there is also a whole indicating the development of detachments. The red dashed 182 square in Fig. 7a indicates the location from where the compression cube was carved out. Note that the disc exhibits a 183 non-uniform deposit of 3^{rd} body material which spreads over the entire contact surface. Moreover, the infrared images 184 confirm that the deposit heterogeneity is induced by the occurrence of hotspots. 185

186 3.2. Uniaxial loading-unloading tests

Fig. 8a shows the uniaxial compression protocol followed during the testing of the worn material. The virgin protocol is identical but with the pre-load is established at $\sigma_{min} = 0.1$ MPa instead of $\sigma_{min} = 1$ MPa. The tests start with a loading-unloading sequence at $\sigma_{max} = 3$ MPa, (five cycles are applied, $\Delta \sigma = 2$ MPa) before complete release of the compression load. 2 to 3 minutes are necessary in-between loading/unloading cycles to change both the loading



Figure 7. (a) Image of the contact surface of the right half-pad after the braking program. The red dashed square indicates the extraction location of the worn compression cube. (b) SEM (SE) image of the contact surface showing a compacted 3^{rd} body and some graphite particles (in black).

and the image recording parameters. The maximum is subsequently increased to $\sigma_{max} = 5$ MPa, 10 MPa, 15 MPa, and 20 MPa. For the shake of simplicity, only the stress-strain curves at 20 MPa are shown in Fig. 8b (in this figure the strains are shown in negative values while stresses are kept positive).

Remarkably, the stress-strain curves shown in Fig. 8b have strong similarities with those reported in the literature for ceramics and rocks [28, 29]. Also, the yield point at which non-linear behaviour similar to plastic deformation first occurs was observed to first occur at 10 MPa in the virgin material. However, considerably micro-plasticity (due to the presence of graphite and ceramics inducing stress concentration in the metallic matrix) is expected to occur in the metallic matrix even at the lowest stress levels.

199 3.2.1. Virgin material

- ²⁰⁰ The virgin stress-strain curve (shown in blue in Fig. 8b) exhibits four main stages:
- At B1, the curve has an initial nearly perfect linear elasticity followed by yielding (B2 stage, with a $\Delta \varepsilon_{zz} = -0.27\%$) induced by the first loading at the 15-20 MPa stress level.
- B3 stage corresponds to the switching from loading to unloading and exhibits an apparent increase in stiffness.
- At the end of unloading (B4), the stiffness is 8% lower than in the forward B5 loading.
- The forward loading within the hysteresis loop (B5) is also nearly linear, with a slightly lower stiffness than that observed at B1.

Note that the loading/unloading cycles exhibit a strain *stays in hysteresis loop* behaviour ($\Delta \varepsilon_{zz} = -0.33\%$) and the residual strain at the minimum stress during B is $\varepsilon_{zz} \approx -0.2\%$.

The DIC ε_{zz} strain map (computed for a B5 loop-loading branch between 1 and 20 MPa, Fig. 8c) indicates a het-

erogeneous macroscopic deformation. The lowest strains ($\varepsilon_{zz} \approx -1.2\%$) are concentrated within and around Graphite 2



Figure 8. (a) Graph showing the different stages of the uniaxial loading/unloading protocol (compression loads are shown in positive values). (b) Stress-strain curves obtained from the DIC results for both virgin (in blue) and worn (in red) materials at 20 MPa stress level. (c) DIC map of the normal strain (ε_{zz} , expressed in %) for the virgin material. The map corresponds to a loading loop between 1 and 20 MPa (indicated by two red dots in (a)). (d) DIC map of the normal strain (ε_{zz} , expressed in %) for the vormal strain (ε_{zz} , expressed in %) for the worn material. The top of the map corresponds to the contact surface.

particles [30], leading to the formation of deformation bands. In this case the bands are more pronounced at the bottom of the sample, but the examination of other samples of the same material indicates a random location of the bands depending on the positioning of Graphite 2 particles. The formation of deformation bands has been reported in the literature for a cellular Al alloy [31] as well as for a low-density porous copper material [32]. In addition, the effect of strain localisation induced by the presence of graphite particles has been observed in cast iron materials [33, 34], although in this case the strain localisation occurs at the microscopic scale length.

217 3.3. Worn material

The worn stress-strain curve (shown in red in Fig. 8b) exhibits five main stages with three main differences compared to the virgin material:

- At A1, the curve has an initial **settling down** phase followed by a nearly linear region at higher load levels.
- Yielding is also observed to occur during the *first* loading at the 15-20 MPa stress level (A2 stage, with a $\Delta \varepsilon_{zz} = -$ 0.23%). Note that, during an actual braking situation, the mean normal load is not expected to reach these high values (15-20 MPa), although it is likely that these values could occur locally due to the material heterogeneity.
- A3 stage corresponds to the switching from loading to unloading and also exhibits a relatively increased stiffness.
- At the end of the unloading (A4), the **stiffness is similar** to the *settling down* phase of the forward loading A1.
- The forward loading within the hysteresis loop (A5) is **non-linear**.

In this case, the general stiffness is lower and therefore higher values of strain are induced ($\Delta \varepsilon_{zz} \approx -0.53\%$ is observed within the loading/unloading cycles). Moreover, the area within the hysteresis loop is smaller than in the virgin material and the loading/unloading branches do not follow the exact same paths between cycles; in other words, the strain *stays in hysteresis loop* behaviour is less noticeable in the worn material. The amount of yielding and residual strains ($\Delta \varepsilon_{zz} \approx -0.25\%$ and $\Delta \varepsilon_{zz} \approx -0.2\%$, respectively) seems to be similar for both materials. Note that the occurrence of the non-linear settling down phase (observed in A1 and A5) is usually related to the presence of damage. In the case of cracking media such as brittle rock, this phenomenon is linked to the closure of microcracks [35].

The DIC ε_{zz} strain map of the worn material (Fig. 8d) also indicates a heterogeneous macroscopic deformation with a marked strain band occurring at ~4 mm from the contact surface. In this case, the presence of such large deformation bands cannot be exclusively correlated to the presence of Graphite 2 particles. On the contrary, the bottom region of this sample exhibits strain localisation occurring at/around Graphite 2 particles, similar to the one observed in the virgin material.

Highly strained deformation band (HSDB). The effects on the mechanical response induced by the presence of this 240 band has been studied in detail by comparing the DIC ε_{zz} strain maps computed for loop-unloading branches at 241 different stress levels (Fig. 9). An additional DIC computation is performed between the stages noted as 10 min and 242 2 s after the sample is fully unloaded (Fig. 9a). At 3-1 MPa (Fig. 9b), the mean strain unloading values are close to 243 0.1%, although a small band showing 0.35% values is observed on the right middle of the image. At 5-1 MPa (Fig. 9c), 244 the strain localisation only takes place within the region identified as HSDB. The highest strain values observed in this 245 region are close to 1.0 %. At 10-1 MPa, 15-1 MPa and 20-1 MPa (Fig. 9d-f), the strain localisation is progressively 246 extended to the bottom region of the sample. Interestingly, a time-dependent unloading effect is observed in Fig. 9g, 247 mainly occurring within the HSDB region. The strain values occurring in this region are close to 0.1%, and the mean 248 ε_{zz} calculated from the image yields a negligible unloading of 0.016%. Finally, note that the regions exhibiting the 249 highest strain levels (for instance, red in Fig. 9f) during the loop-unloading correspond to the regions with the lowest 250 strain levels during the loop-loading (blue in Fig. 8d). These results indicate that the compression behaviour of the 251 worn material is mainly controlled by the HSDB region where the strain localisation is maximal. 252



Figure 9. (a) Graph showing the different stages of the uniaxial loading/unloading protocol. The red dots indicate the images used for the computation of ε_{zz} strain maps: (b) at 3-1 MPa, (c) 5-1 MPa, (d) 10-1 MPa, (e) 15-1 MPa, (f) 20-1 MPa, and (g) between an image taken 10 min after sample unloading and one taken immediately after sample unloading.

Layered bulk behaviour. The use of layered ROIs (Fig. 10a) allows the study of the local mechanical behaviour

at three different depths (noted as top, middle and bottom). The stress-strain curves obtained from the local DIC computations are shown in Fig. 10b. The top layer behaviour shows an initial and relatively small *settling down*

²⁵⁶ phase followed by a nearly linear region at higher load levels. No plastic deformation and very small residual strain

 $\Delta \varepsilon_{zz} \approx -0.05\%$) are observed. The hysteresis can be considered as non-existent. The behaviour of the middle layer is

very similar to the worn curve shown in Fig. 8b but with higher strains observed within the loading/unloading cycles

 $\Delta \varepsilon_{zz} \approx -0.8\%$ against $\Delta \varepsilon_{zz} \approx -0.42\%$). The hysteresis seems to be slightly increased. This result additionally confirm the above conclusion about the HSDB region controlling the macroscopic mechanical behaviour of the worn material. By contrast, the behaviour of the bottom layer is quasi-linear and exhibits lower stiffness when compared to the virgin material. This reduction in the stiffness could be explained by the development of microcracks. Further, the bottom *stays in hysteresis loop* behaviour is less noticeable when compared to that of the virgin material.



Figure 10. (a) Light-optical image of the worn sample showing the ROI locations used in the DIC computations of the top, middle and bottom layers. (b) Stress-strain curves of the top, middle and bottom layers at 20 MPa stress level. The virgin curve is added in blue for comparison purposes. (c) SE image and (c') BSE image of the microstructure within the top layer. (d) SE image of the microstructure within the bottom layer. See Fig. 1c-d for comparison with the virgin material.

Microstructural changes. The analysis of the microstructure indicates a carburization-like effect occurring in the Fephase. In the top layer, the Fe-phase is completely covered by pearlite (Fig. 10c-c'). In the middle layer, the colonies of pearlite are homogeneously distributed throughout the Fe-phase (Fig. 10d). The microstructure in the bottom (Fig. 10e) layer is similar that observed in the virgin material (Fig. 1c-d).

Microcracking. The visual evaluation of the SEM images does not allow a thorough investigation of the microcrack density. Nevertheless, it is sufficient to determine an increased content of microcracks within the middle layer

(Fig. 11). No macrocrack is observed, although the possibility of a bulk 3D linkage between microcraks must be considered. The content of microcracks within the top and bottom layers seems similar, although this statement is conjectural. Note that the virgin material also exhibits some microcracking after the uniaxial compression test. The orientation of cracks seems random, as horizontal, oblique and vertical cracks can be indistinctly observed. These microcracks tend to occur at the thin walls of metallic matrix laying between large Graphite 2 particles. Finally, some out-of-plane features can be observed in (Fig. 11). These out-of-plane features are also observed within the top and between the second second

²⁷⁶ bottom layers although they are less noticeable.



Figure 11. (a) SE image of the mesostructure within the middle layer. Charging effects are observed on some ceramic particles. (b) Detail of the microcracks. See Fig. 1b for comparison with the virgin material.

277 Hardness. Vickers hardness tests were performed using an instrumented indentation machine (with about 10 points

per layer and up to 120 N of multi-cycling). The reported values correspond to the Martens hardness, computed from

the maximum displacement of the load-displacement curves (see Table 2). The highest values (+30%) are observed

in the top layer. The hardness of middle and bottom layers is similar to that of the virgin material. These results are in

281 good agreement with the increased content of carbon observed in the Fe-phase of the top layer. The high dispersion

of these measurements is related to the material heterogeneity, which gives a different response depending on which

phase/particles are being indented, as well as on the arrangement of these phases/particles within the volume.

Virgin	Worn Material			
Material	Тор	Middle	Bottom	
0.30(0.21)	0.46(0.31)	0.31(0.18)	0.32(0.20)	

Table 2. Martens hardness in GPa (standard deviation) of the virgin and worn materials. The worn material is subdivided into the three regions identified in Fig. 10a.

284 **4. Discussion**

285 4.1. Evolution of the compression behaviour related to the microstructure

Fig. 12 summarizes the main findings concerning the evolution of the compression behaviour and microstructure. 286 The virgin material exhibits a complex mechanical behaviour where Graphite 2 particles, which are softer than the 287 matrix, induce strain localisation in the form of macroscopic deformation bands (Fig. 12a, shown in green and blue). 288 After the compression, some microcracking can be distinguish on the SEM images (Fig. 12c). Additional compres-289 sion tests, not shown here for the sake of simplicity, indicate that the Graphites 2 particles control the formation of 290 hysteresis (the graphite material is known for having intrinsic hysteretic behaviour [36]). Nonetheless, there are other 291 factors (e.g., energy dissipation induced by friction between Graphite 2 particles and the metallic matrix walls) that 292 might also contribute, in some extent, to the hysteresis loop. 293



Figure 12. (a) Schematic illustration of the strain localisation occurring in the virgin material (b) and the worn material when tested in a looploading branch between 1 and 20 MPa. (c) Schematic illustration of the microcracking observed in the virgin material at the end of the compression test. (d) Schematic illustration of the microcracking and level of carbon diffusion observed in the worn material at the end of the compression test. The arrow indicates that the carbon content in the Fe-phase decreases along the depth. The temperature gradient is an estimation based on the thermocouple results.

The mechanical response of the worn material is even more complex than that of the virgin one. The changes 294 induced by the braking program led to the formation of a tri-layered-like material. Strain localisation is also observed, 295 but there are variations depending on the layer (Fig. 12b). The carbon diffusion is more important in the top layer 296 (Fig. 12d, temperatures as high as 800-1000°C). This is consistent with the fact that the austenitization occurring at 297 $800-1000^{\circ}$ C enhances carbon diffusion (the solubility of C is greater in austenite compared to ferrite [37]). Thus, we 298 consider that the mechanical changes observed in the top layer (increased Young's modulus and hardness) are mainly 299 induced by carbon diffusion. Furthermore, the lack of yielding observed in the top layer (Fig. 10b) would be due to a 300 carbon-induced increase in the elastic limit (σ_v) of the Fe-phase. 301

As is the case in geological materials, the pronounced non-linearity (Fig. 10b) exhibited by the middle layer is 302 probably related to the high content of microcracks. Moreover, it has been reported [29] that an increased microcrack 303 content leads to the increase of the area enclosed within the hysteresis loop. Also, the reduced reversibility in the 304 hysteresis loop of the middle layer (*i.e.*, the stays in hysteresis loop behaviour is less noticeable) could be linked to 305 enhanced crack closure effects such as higher friction between the crack lips. On the other hand, it is probable that the 306 time-dependent recovery observed in Fig. 9g is controlled by the intrinsic hysteretic behaviour of Graphite 2 particles 307 308 rather than by crack closure effects. Even if we were not able to confirm this point, the possibility of 3D macrocracks resulting from the coalescence of microcracks should be contemplated (Fig. 12d). Note that the compression curve of 309 the bottom layer (Fig. 10b) also suggests an increased microcrack density when compared to the virgin material. 310

The combination of mechanical loading (shear and compression) with constrained thermal expansions and microstructural transformations could explain the higher microcrack density observed in the middle layer. Neverthe-

less, the Fe-phase microstructural transformation (*i.e.*, higher content of C within the lattice structure combined with austenitization cycles) seems to be the key parameter controlling the increase in microcrack density. This is because the depth of the middle layer mainly corresponds to the range of temperature (800-700°C) where the Fe-phase transitions from ferrite to austenite (Fig. 12d). This transition would induce a boundary effect, which promotes the localisation of stresses. In any case, the elucidation of the mechanisms leading to the increase of microcracks observed in the middle layer needs further research mostly concerning material science (*e.g.*, the use of diffraction techniques for phase analysis).

Finally, given that the virgin material is considerably porous (see Section 2.1) and its tendency to microcrack, the residual stresses induced by Fe-phase transformation as well as the different dilatation coefficient of components are

residual stresses induced by Fe-phase transformation as well as the different dilatation coefficient of considered to play a minor role in the evolution of the compressive behaviour.

323 4.2. Evolution of the friction behaviour related to the contact surface and sub-surface material

At low temperature (S1V1-S1V3, S2V1-S2V2, S3V1-S3V2, see Fig. 6b) the COF values are higher and more 324 variable when compared to those occurring at high temperature (S1V4-S1V5, S2V3-S2V5, S3V3-S3V5). These re-325 sults correlate well with the fact that the friction tends to be more abrasive at low temperature and more adhesive 326 at high temperature [38]. They also provide a demonstration of the role that temperature level plays in COF stabi-327 lization. For instance, a significant stabilization can be observed during the S1V4 braking sequence. During this 328 sequence, a temperature higher than 700°C is reached for the first time at the contact surface (more than 600°C is 329 registered by thermocouples at 3 mm, Fig. 6b). As discussed in Section 4.1, ferrite to austenite transformation starts 330 at temperatures above $\sim 700^{\circ}$ C. At this temperature, the copper is able to endure higher levels of plastic yielding. It is 331 generally accepted that the increase of copper ductility contributes to the development of a tribolayer that facilitates 332 the stabilization of the COF [39]. Thus, opposing mechanisms develop during the braking program: the hardness of 333 the sub-surface material increases due to carbon diffusion while the copper contributes to an increase in the ductil-334 ity of the tribolayer. The effect that an increasing sub-surface hardness has on the ductility of the tribolayer has yet 335 to be determined. However, an increase in the brake-pad hardness usually leads to an increase in the wear rates of 336 the counter-face (i.e., the disc). This aspect could not be investigated in the present study because it would require 337 long-term tests, but the results of endurance tests conducted by the industrial partner confirms this trend. 338

339 4.3. Limitations and open questions

The combination of thermal monitoring and bulk properties characterization has led to an in-depth understanding of the mechanisms involved in the evolution of the compression behaviour. Nevertheless, so far we can only conjecture about the influence of the material evolution on the braking performance. Some of the most relevant issues concern:

Contact uniformity. The results shown in Fig. 5 indicate the development of different thermal gradients depending on the pad location. A spatially heterogeneous mechanical behaviour can be therefore expected over the contact surface.

The spatial variation of the mechanical properties usually leads to a reduced contact uniformity and subsequent thermal

localisations. In this work, however, the mechanical heterogeneity could be neutralized by the reduced stiffness of the

³⁴⁷ middle layer.

³⁴⁸ *Durability.* The damage localisation observed in the middle layer can be considered as highly detrimental to the ³⁴⁹ brake-pad durability. It would be interesting to investigate whether this damage could eventually lead to the final ³⁵⁰ failure of the brake-pad.

Vibrations. A reduction in vibrations could be expected given the decrease in stiffness (Fig 8b) and the dominant role of Graphite 2 particles in the middle layer (Fig 9). However, a decreasing effect resulting from the increase in stiffness

³⁵³ and hardness of the top layer should be considered.

Tribology. The spatial heterogeneity of the thermal loading most likely affects the 3^{rd} body composition. As is the case for contact uniformity, one could argue that a spatially heterogeneous 3^{rd} body can hamper the stabilization of COF and even increase wear rates by lowering the cohesiveness of the layer.

Ideally, the evolution of material properties should contribute to the further stabilization of the COF and 3^{rd} body. Further investigation on the subject requires the design of highly instrumented dynamometers accounting for the monitoring of other parameters (*e.g.*, vibrations), the implementation of multi-loading tests (*e.g.*, to investigate the effect of shear load), as well as a detailed characterization of the 3^{rd} body composition (*e.g.*, the use of Focused Ion Beam (FIB-SEM) to map the chemical composition and microstructure over the contact surface).

363 5. Conclusions

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The changes in bulk properties induced by a braking program were investigated using a *simplified* semi-metallic friction material. The use of inserted thermocouples enabled the monitoring of the thermal loading while the characterization of the compressive and microstructural properties was carried out using compression tests coupled with DIC and SEM analysis. The following conclusions can be drawn:

1. The thermal loading plays a dominant role in the evolution of the friction behaviour and material properties.

 The stiffness of the worn material is lower and the evolution in the material properties leads to a compression behaviour similar to that of a tri-layered material.

37. 3. The changes in the worn material compression behaviour are related to the microstructure:

- (a) The increased microcrack density of the central portion of the brake-pad thickness leads to the formation
 of a deformation band containing, in turn, the maximum (during unloading) or minimum (during loading)
 strain values.
- (b) An increase in the carbon content (in the form of perlite colonies) is observed in the Fe-phase. The increase in carbon content is particularly important in the top layer, which exhibits the highest stiffness and hardness.
- 4. The results indicate that the material evolution has deleterious effects on the contact quality (spatial variation of the mechanical behaviour over the brake-pad cross section), durability (localisation of damage favouring material fragmentation) and tribology (spatial variation of the 3^{rd} body composition).

Even if the tribological aspects are to be prioritised, the bulk behaviour should also be considered when developing new formulations. Understanding the interactions between components at the microstructural level gives valuable insight into possible ways to improve the material formulation. For example, the results presented here indicate that the carbon diffusion has negative effects. Thus, new formulations ensuring reduction in carbon diffusion can be envisaged. Hence, the further investigation of the evolution of friction material properties with special emphasis on the quantification of the subsequent effects on the braking performance is recommended.

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