Effect of austempering treatment on lubricated sliding contact of compacted graphite iron

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Received: 17 July 2020 / Accepted: 27 October 2020 / Published online: 5 November 2020
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Abstract

Significant research has been done to improve the wear properties of the components used in internal combustion engines. Excessive wear is observed in components such as cylinder liners and rings, which can lead to lower volumetric efficiency of the engine, increase oil consumption, polluting emissions, and scuffing related issues. Since tribological systems in internal combustion engines are complex, the different wear mechanisms involved need to be investigated to improve the life of components. Cast irons are commonly used for engine components, especially compacted graphite irons (CGI) for piston rings and gray cast irons (GCI) for cylinder liners. This work aims to evaluate the tribological behavior of two different microstructures of CGI (pearlitic and ausferritic), sliding on pearlitic GCI. The samples of CGI with different microstructures and hardness were evaluated in a short-stroke reciprocating sliding tester, using Petronas Urania SAE 30 API CF lubricant oil at 100 °C for four hours. The characterization of worn surfaces was made using a scanning electron microscope (SEM) and 3D roughness measurements. The coefficient of friction (COF) comparison between the two CGI microstructures showed very similar results with COF = 0.11. The pearlitic CGI showed more severe wear than the austempered one, confirmed by SEM images and the difference in topography parameters before and after the tests. Phosphorus, sulfur, and zinc were detected by EDS analyses in the samples' worn-out regions, indicating the formation of tribo-films, which was further confirmed by the friction tests.

Keywords Compacted graphite iron · Austempering · Piston rings · Cylinder liners

1 Introduction

Cast iron is a relevant material due to its low cost and good tribological behavior; however, it lacks suitable mechanical properties. Hence, it is commonly applied for conditions where a combination of wear and heat is important considerations, such as piston ring-cylinder liner systems [1, 2]. Much research has been carried out on cast iron to improve the microstructures to fulfill both its superior mechanical and tribological performance in such severe wear applications, mainly where the weight reduction and saving energy are extra essential considerations [3–5]. By showing superior fatigue limit together with excellent thermal conductivity,

compacted graphite iron (CGI) is an alternative replacing gray cast iron (GCI) [6] in wear application in both piston rings and cylinder liners. These excellent combined mechanical, physical, and tribological properties of CGI are associated with the graphite particles' inter-connected coral-like morphology between nodular and lamellar shapes. Some strategies have been adopted to improve wear resistance of the CGI alloys, using the laser hardening technique [7], the addition of pearlitic stabilizer elements [8], surface alloying [9], and induction hardening [10]. Another possible route for increasing mechanical properties is austempering heat treatment. Previous studies demonstrated a significant influence of microstructure on improving CGI wear resistance

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after austempering, especially compared to the pearlitic metallic matrix under dry sliding conditions [11]. Ghasemi et al. [12] studied austempering parameters of a ferritic CGI and noticed the significant influence of temperature on microstructure: For 275 °C, there was lower ausferrite transformation; on the other hand, when austempering was conducted at 375 °C, the resulting ausferrite was coarse, due to higher diffusion rate of carbon. The same authors showed a small influence of processing times, concluding that only for times as long as 120 min, the hardness got a slight increase because of the carbides' formation.

By the fact that CGI employment is recent and austempering could be used to improve its combined mechanical and tribological properties, it is necessary and reasonable to conduct more studies about the influence of the resulting austempering microstructure. In this scenario, this paper aims to compare the wear behavior of pearlitic and austempered microstructures of CGI through ring-on-cylinder lubricated tests.

2 Materials and methods

2.1 Reference-CGI

An alloyed CGI was produced in a marine diesel engine piston ring with a diameter of 800 mm. This piston ring was cut into cylindrical pieces of 30 mm in diameter and 30 mm in height for the austempering process.

A spark optical emission spectrometer analyzed the CGI composition, and it is listed in Table 1. The as-cast material had a hardness of $328 \pm 17 \text{ HV}_{0.1}$ and will be called "Reference-CGI." Its microstructure is shown in Fig. 1, where the pearlitic metallic matrix and compacted graphite can be seen.

Tensile testing, according to ASTM-E8-98, was carried out at room temperature using a Zwick Z100 machine to obtain Young's modulus (E) of the CGIs. Tensile specimens prepared with 6 mm in diameter, a gauge length of 24 mm, and a radius of fillet of 6 mm. The strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ was used in this study. An in-house MATLAB™ script was used to evaluate the tensile curves and determine ultimate tensile strength (UTS), yield strength (YS), and Young's modulus (E), as used in earlier works [12, 13]. The tensile curve for the Reference-CGI can be seen in Fig. 2.

2.2 Austempering treatment

The cylindrical pieces (Ø30 mm and 30 mm height) were austenitized at 900 °C for 90 min, which was enough time to complete the matrix's transformation into austenite. After the austenitization, samples were transferred quickly to a salt bath (Duferrit AS 140–50%KNO₃ and 50%NaNO₃) at 325 °C. They were maintained at this temperature for 120 min. After that, samples were air cooled to room temperature. This heat-treated condition will be called "Austempered-CGI."

Cutting, grinding, and polishing were carried out following the standard samples preparation procedure for cast irons. The microstructure was analyzed with an optical and scanning electron microscope (SEM). Vickers hardness test was conducted using a load of 100 gf (1 N) both for as-cast Reference-CGI and Austempered samples.

2.3 Tribo-tests—samples preparation

The Reference-CGI and Austempered-CGI cylinders were cut into a coin-like samples 3 mm thick, and then, the perimeter was polished. After that, the samples were cut using a wire EDM and then a diamond blade (to avoid microstructural changes), with the shape and dimensions shown in Fig. 3.

2.4 Tribo-tests—counter-bodies

Commercial cylinder liners of FC35 gray cast iron class were used as counter-bodies. These liners were supplied by MAHLE GmbH and had an initial diameter of 131 mm, being similar to those tested elsewhere [14].

Fig. 1 The microstructure of the Reference-CGI sample

Table 1 Composition of Reference-CGI

C	Si	Mn	Cu	Mo	Ni	Cr	P	S	Ti	Mg
3.59	1.75	1.03	1.28	0.51	0.45	0.38	0.014	0.001	0.001	0.017

Fig. 2 The tensile curve for Reference-CGI **Fig. 3** Shape and dimensions of CGI samples for the friction tests **Fig. 4** Counterbodies of CGI for the tribo-tests

Samples were cut from the liners into small coupons of $(8.3 \times 10.1 \times 24.0) \text{ mm}^3$ (Fig. 4) and had the honed surface characterized by surface roughness measurements. The matrix hardness of this gray cast iron is $400 \pm 75 \text{ HV}_{0.1}$.

The holder of the counter-body has a cavity, which is filled with lubricant oil. In this configuration, the lubricant covers the counter-body surface completely. There is a resistance inside the holder, responsible for heating and maintaining the temperature during the tests. A thermocouple is placed inside the lubricant cavity to control the temperature. The schematics of assembly for the tribo-tests can be seen in Fig. 5. It shows the sliding direction and the holders' position for the samples and the counterbodies.

2.5 Tribo-tests—equipment

Friction tests were carried out using a CETR UMT-2 reciprocating module. The normal load and frequency were 125 N and 5 Hz, respectively. The stroke length was 10 mm, and the tests were performed for 4 h at 100°C , using PETRONAS Urania SAE 30 CF as a lubricant. Tests were repeated three times for both tested materials. Friction coefficients were obtained with the average values of up and down semi-cycles with a data acquisition rate of 100 Hz. After

Fig. 5 Counterbodies holder

tests, the worn surfaces were analyzed using a Zeiss EVO-MA15 scanning electron microscope.

2.6 Roughness measurements

Surface roughness measurements were taken before and after the tribo-tests using an optical interferometer Taly-surf CCI Lite—Taylor Hobson, with 1024×1024 pixels. In the CGI samples, the polished perimeter had the roughness measured, and in the case of the counter-bodies, the honed surface was analyzed before the tests. After the tests, worn regions were measured associated with the mid-stroke. The dimensions of each measurement were

$0.8 \times 0.8 \text{ mm}^2$. The 3D roughness parameters analyzed belong to the structure height parameters (Sk family), besides the root-mean-square height (Sq), the skewness (Ssk), the kurtosis (Sku), and the mean summit curvature (Ssc).

3 Results

3.1 Initial characterization

The CGI microstructure resulting from the austempering treatment at 325°C can be seen in Fig. 6. Ferrite has grown

Fig. 6 Matrix microstructure of Austempered-CGI

with a feathery morphology immersed in austenite not fully transformed. An important amount of more stable unreacted or blocky austenite is present, which is usually related to micro-segregation issues. This microstructure delivered a hardness of $520 \pm 19 \text{ HV}_{0.1}$, representing an increase of approximately 60% compared to the untreated microstructure.

3.2 Friction behavior and chemical analysis

The coefficient of friction was measured throughout the four hours of the test (Fig. 7), and the average value was 0.11 ± 0.02 for both cases, showing that different microstructures did not influence these results. A previous investigation found no differences in COF between pearlitic and austempered microstructures in different cast irons under dry sliding tests [11].

Considering the differences in the test setup, Vale et al. [15] have found a similar value for CGI liners tested against rings manufactured with nitrided steel. These researchers used the same lubricant oil but under milder conditions

(75 N of applied load and oil temperature of 40°C). Surprisingly, this average COF was the best value described by Vale et al., which is a good indicator of the austempered condition's tribological performance.

All worn surfaces were analyzed using EDS to detect phosphorus, sulfur, and zinc after reciprocating tests. These elements can be associated with the formation-removal-formation mechanisms of zinc dialkyldithiophosphate (ZDDP) tribo-films [16]. The elements' content was not similar for both tribological pairs, as can be seen in Fig. 8. Therefore, considering the qualitative analysis given by the EDS technique, they can be considered in a similar range of values, justified by the friction coefficients, which were the same for both conditions of CGI.

However, the amount of elements present in ZDDP films is higher on the surface of the rings. This result can be associated with the higher deformation on the softer material. Looking at the findings reported by Vale et al. [15], they found a more considerable amount of these elements on the liner surface. However, in that case, the rings were much harder, manufactured with a hard nitrided layer. The more intense formation of the ZDDP tribo-film is associated with regions with more deformation [10].

Additionally, magnesium's significant presence in the EDS analysis of all worn surfaces (10% at rings and 1.2% at cylinder surfaces) was detected. This element is related to detergent additives in the oil formulation. A discussion on this topic is out of this investigation's scope but certainly could be explored in the future.

3.3 Wear behavior

The wear behavior was evaluated by analyzing the variation in the surface roughness measured before and after the sliding tests, as listed in Table 2. For the honed surfaces (liners), there was a reduction in S_q and S_k parameters, representing a flattening of the surface after the tribo-tests. However, the behavior of the parameter S_{ku} for the liners was different from each other. The liners tested

Fig. 7 Friction coefficients curves obtained for Reference-CGI and Austempered-CGI

Fig. 8 Friction coefficients versus Zn + P + S contents after the tests **Table 2** Roughness parameters before and after tribo-tests

Sample	Period	Ssc	Sq	Ssk	Sku	Spk	Sk	Svk	Variation						
									Sq	Ssk	Sku	ΔSk	Δ (Spk + Sk)	ΔS3k	ΔS3k
		mm ⁻¹	μm						%			μm			%
Liners against Reference-CGI	Before	771	0.80	-2.94	15.55	0.34	0.96	1.94	-53	-12	1	0.32	0.34	1.48	-46
	After	195	0.38	-2.60	15.64	0.32	0.64	0.80							
Liners against Austempered-CGI	Before	781	0.69	-2.86	20.26	0.36	0.88	1.63	-42	-32	-30	0.10	0.15	1.00	-35
	After	207	0.40	-1.94	14.13	0.31	0.78	0.78							
Reference-CGI	Before	434	0.37	-2.52	19.42	0.28	0.54	0.73	16	-58	-66	0.33	0.32	0.38	24
	After	1120	0.43	-1.06	6.53	0.27	0.87	0.79							
Austempered-CGI	Before	226	0.33	-1.32	7.77	0.25	0.65	0.59	6	-33	-27	0.06	0.08	0.05	3
	After	1114	0.35	-0.88	5.67	0.27	0.71	0.56							

against the Reference-CGI did not present variation in the Sku parameter. On the other hand, the liners tested against the Austempered-CGI showed a significant reduction in Sku value, which means a flattening in the distribution, reducing the high peaks and deep valleys on the surface of the liners. This behavior can be seen in the example of height distribution curves shown in Fig. 9 for liners before and after the Austempered-CGI tests. It can be attributed to the higher hardness of the austempered condition in comparison.

The Sq values for the rings increased, which is explained by the initial polished condition that degrades after the sliding test. However, the Austempered-CGI sample showed a lower increase in Sq, ascribed to the higher hardness resulting from austempering with the perlite one, causing more damage to the liner's surface sliding against it.

For helping the wear evaluation, the structure height (S3k = Spk + Sk + Svk), already used in previous investigations, especially for characterizing the so-called almost zero wear [14, 17–22], will be considered. In this study, the structure height decreased for the liners, in agreement with the flattening reported for honed surfaces. However, for the rings, there was an increase in S3k value for the Reference-CGI, indicating more severe wear in this condition. When considering the Austempered-CGI, it was not detected significant changes in topography parameters, confirming CGI's superior wear behavior after the austempering process.

According to the variation of roughness parameters (Table 2), the Austempered-CGI sample aspect in Fig. 10b shows less wear than the Reference-CGI sample (Fig. 10a). In the cylinder liners' images, it was possible to identify grooves and plateaus related to the honed finishing

Fig. 9 Comparison of height distribution of the liners before and after the reciprocating tests against the Austempered-CGI

Fig. 10 SEM images of worn surfaces, **a** Reference-CGI, **b** Austempered-CGI. GCI Liner against, **c** Reference-CGI, and **d** Austempered-CGI. Marked areas show EDS analysis locations

(Fig. 10c, d). This aspect will be better discussed in the next section.

4 Discussion

Figure 10 presents the aspects of worn surfaces, revealed in SEM.

The first aspect to consider is the scar left on the ring surfaces. The diameter determined in Fig. 10a is higher than that observed in Fig. 10b. This finding agrees with all roughness variations described before, meaning that the variation in S3k is a good indication of wear.

Another exciting aspect can be observed, looking at the worn surfaces of liners. The region deformed plastically is much more intense on the surface where the Reference-CGI passed on. In this way, the dark region corresponding to the zone, where a tribo-film was formed, is much smaller than observed in Fig. 10d. Consequently, the content of elements present in the tribo-film cannot explain alone the equality for the COF.

To discuss these findings better, we estimate first the level of plastic deformation in each tribological pair. The mean pressure of contact (P_m) was 131.6 MPa, considering the contact area obtained from the round deformed region restrained by the circle seen in the SEM image for the Reference-CGI after the tests (Fig. 10a).

Besides the contact pressure, the model provided by Cheng-Etsion-Bogy [23], suitable for micro-contact cases, was considered. The asperities are considered spherical in their summits, and their radius was described by the Ssc parameter, which represents the arithmetic mean summit curvature [24]. An equivalent value of Ssc was used as a radius combining Ssc from the liner and the Reference-CGI before the tests. The interference (ω) and the inception of plastic deformation (ω_0) were calculated, taking into

account the Poisson's ratio of 0.27 [25] and Young's modulus of 142 GPa for the CGIs. The Poisson's ratio and Young's modulus for the liners were 0.3 and 120 GPa (information supplied by the manufacturer).

The interference (ω) for the "Liner-Reference-CGI" pair is about three orders of magnitude higher than the critical interference at the inception of plastic deformation (ω_0). This result supports the plastic deformation verified in the SEM images (Fig. 10a, c). This plastic deformation was responsible for reducing the summit curvatures of the liners, according to the flattening of the honed surface.

Remember that no doubt about the more intense plastic deformation (and wear) occurred in the test with the Reference-CGI. As expected, the reasons for low friction can be discussed. The possibility of adhered material acts as a lubricant should be taken into account in this case. The direct contact with graphite can lead to a distinguishable reduction in friction, as observed by Vale et al. [4] during scratch tests of compacted graphite iron. Moreover, Vadiraj et al. [26] showed fascinating results for different cast irons in dry sliding tests where the COF was lower than that for a lubricated test. This result could be attributed to graphite ejection as debris particles, which acted as a lubricant in the contact between the surfaces [26, 27].

In this case, the subsurface graphite should be analyzed after wear tests. Figure 11 shows the cross sections of Reference-CGI and Austempered-CGI samples.

One can observe carbides' presence (a significant amount of carbon was detected), which is revealed with white color in the cross sections. However, this constituent is not present at the subsurface in enough distance to contribute to the tribological events. Besides, at least two morphologies of graphite are detected in Fig. 11. One of them resembles spheroidal graphite, while the other one is typical of compacted graphite iron. On the contrary of carbides, the graphite is very close to the

Fig. 11 Cross-sectional SEM images of worn surfaces, **a** Reference-CGI, **b** Austempered-CGI. EDS analysis location is relative to carbide

Table 3 Summary of events affecting friction behavior. Arrows indicate the effect of increasing (↑) or reduction (↓)

Event	Reference-CGI/liner tribo-pair	Austempered-CGI/liner tribo-pair
Plastic deformation	Applicable ↑	Non-applicable ↓↓
Ejection of graphite	Applicable ↓	Non-applicable (neutral)
Tribo-film formation	Non-applicable ↓	Applicable ↑

surface in significant volume fraction, especially those with vermicular morphology. Many more areas are close to the Reference-CGI surface, looking at the subsurface graphite frequency, turning more probable its ejection in this case. Additionally, the ejection would be less intense if the spheroidal graphite was predominant [27], which is not the case here.

The reduction of tribo-film can be associated with more intense wear, which can reduce the dark region, observed in Fig. 10d. In this way, the most probable is that the material adhered to the liners may be a mixture of ejected graphite, deformed debris, and tribo-film.

Another issue is related to the presence of elements that form ZDDP, Zn + P + S. Recently, Fernandes et al. [14] described that the detection of these elements on tested liners' worn surfaces was associated with an increase in the friction. This hypothesis can be applied to the observed in Fig. 10d.

In summary, a balance of all tribological events described here is enough to justify the equality in friction values. Each parcel of contribution is presented in Table 3, considering plastic deformation, ejection of graphite, and tribo-film formation as the tribological phenomena involved during the reciprocating tests.

The indications presented in Table 3 show that reduction in friction is predominant, and a balance of terms is proposed, justifying the similar values in friction coefficient.

Despite the microstructural differences between the pearlitic CGI and the Austempered-CGI, no difference in the COF occurred. Nevertheless, the analysis of SEM images and roughness parameters determined a superior wear behavior of the Austempered-CGI compared to the pearlitic one. This scenario raises opportunities for studying different austempering parameters and testing conditions for CGI used for cylinder liner and piston ring pairs.

5 Conclusions

In the present work, the effect of austempering treatment—(325 °C/120 min) applied to an as-cast pearlitic compacted graphite iron was studied on the tribological behavior. From the above-mentioned results, the following conclusions can put forward:

- The austempering promoted an increase of 30% in the hardness of pearlitic compacted graphite iron, but this effect was not enough to change the average friction coefficient after four hours of the test;
- The friction behavior was dependent on the presence of typical elements of tribo-films for Austempered-CGI, and for the Reference-CGI, the most probable reason for low friction was the presence of ejected graphite on the mixed adhered material;
- Surface roughness measurements of Reference-CGI worn specimens showed considerable variation when compared to original surfaces, indicating a significant level of wear; and
- Higher hardness of the Austempered-CGI contributed to improving the wear property.

Acknowledgements The material provided in this collaborative work was partly supported by the AusCGI project [funded by Stiftelsen för kunskaps- och kompetensutveckling (KK-Foundation)], Sweden, under the Prospekt scheme [GNR. 20170021]. Federal-Mogul AB and Bodycote are greatly acknowledged for their materials and helping with the heat treatments, respectively. G. Pintaude thanks CNPq for a scholarship through Process 308416/2017-1. The authors also thank the Multi-User Center for Materials Characterization (CMCM) of the UTFPR SEM-EDS analysis and MAHLE METAL LEVE for supplying samples.

Compliance with ethical standards

Conflict of interest The author(s) declare that they have no competing interests.

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