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A study on graphite extrusion phenomenon under the sliding wear response of cast iron using microindentation and microscratch techniques

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Abstract

This study focuses on the graphite flakes extrusion mechanism during microindenting and microscratching of cast iron. Observations on the graphite response under abrasive conditions revealed that the matrix deformation which is occurred during a sliding wear condition could have a significant influence on its lubricating performance. Simple microindentation and microscratch tests were conducted to explore the lamellar graphite contribution to tribofilm formation under abrasive wear conditions. The results obtained showed that induced plastic deformation which developed adjacent to the graphite compressed the lamellas and in turn resulting in extrusion of the graphite from its natural position. Further investigations on both indentation and scratch tests indicated that, surprisingly, the graphite began to be fractured and extruded from the centre of graphite lamellas, irrespective of the lamella size. Additionally, a mechanism was proposed to explain the self-lubricating and the extrusion behaviour of the lamellar graphite as a result of indentation.

Keywords: Lamellar graphite iron; Graphite extrusion; Sliding wear; Abrasive wear; Microindentation testing; Microscratch testing

1 Introduction

Generally speaking, cast iron can be considered to be a self-lubricating metal-base composite material [1]. Hence, traditionally lamellar graphite iron has been used in variant tribological applications, especially, in situations where encountering the sliding motion such as disc brakes and clutch systems [2, 3]. The other application where excellent tribological performance is of great importance is piston ring-cylinder liner system. Improvements in the tribological behaviour of cast iron are primarily attributed to factors such as microstructure and graphite morphology. Prasad [4]

investigated the effect of microstructures in terms of the amount of ferrite, pearlite, and graphite on wear characteristics. He also performed more studies of the influence of graphite characteristic on wear properties in terms of morphology (lamellar and spheroidal), size, content, and distribution on sliding wear surfaces. Furthermore, he indicated that lamellar cast iron (predominately that with a pearlitic matrix) offers significantly better wear resistance in both dry and oil-lubricated conditions than spheroidal graphite iron. Based on the investigations into the wear properties of cast iron performed by Sudarshan [5] and Taylor [6], a pearlitic A type lamellar graphite iron, with a limited quantity of ferrite, is a viable candidate for piston ring applications. Additionally, regarding the influence of hard phases on wear improvement, Eyre [3] and Nadel [7] performed valuable investigations, the conclusions of which were validated by other researchers [5, 8]. They stated that hard phases stand out from the matrix on a fully run-in surface, and thereby improve the wear resistance by minimizing the direct metal to metal contact during sliding.

Regarding the effective lubricating nature of the graphite in lamellar iron, Sugishita et al., in two separate works [1, 9], examined the effect of tribofilm formation under the reciprocating sliding and rolling-sliding conditions, respectively. In both studies, the friction and wear performance of lamellar cast iron were influenced by the surface graphite conditions. So that for conditions in which the graphite can easily lubricate the sliding surfaces a significant decrease, by several orders of magnitude, was observed both in coefficient of friction and wear rate. The good tribological properties of graphite is basically associated with its anisotropic structure and its weak interlayer van der Waals forces [10], as well as the fact that it provides a large lubricating surface area in the lubricant mixture during sliding. The interactions between these weak interlayers cause smearing processes and the formation of a very thin lubricating film between the sliding surfaces [10]. However, and despite this important function, the self-lubricating mechanism of lamellar iron remains ambiguous and has not been demonstrated and fully explained in scholarly literature.

During sliding, the ease with which a graphite particle can be extruded from its embedded position is directly proportional to the improvement in tribological performance [11]. In previous work [12], Ghasemi et al. found that under sliding conditions, not all graphite lamellas serve the same graphite supplying function. Some lamellas are partially or entirely covered after a given period of operational time, while others remain uncovered and lubricate the sliding surfaces. Thus, this covering tendency is of critical importance, as the consequence of a less effective smearing propensity is an increased risk of incidences of scuffing or other issues associated with poor tribological properties. In the same study, the relationship between lamellar graphite orientation and covering tendency during sliding was investigated; this demonstrated that graphite lamellas

that are parallel or close to the sliding direction are more easily covered than those oriented away from the sliding direction. However, the metal matrix deformation and consequent effect on the graphite as a result of sliding remain ambiguous.

The abrasive wear behaviour of lamellar irons has been studied, using scratch testing techniques, by Mendas et al. [13]. They investigated the effect of microstructure, applied various loads and attack angles of conical indenter on coefficient of friction and wear characteristics. According to the results obtained, graphite plays an important role in affecting the wear performance of the specimens. Additionally, Nakamura et al. [14], in examining lamellar iron abrasion patterns, demonstrated that sliding surfaces which are in contact only interact within a micro localized area. They further demonstrated that the graphite film that is formed on the micro scale is identical to that on the macro-scale. Consequently, it derives the attentions for those who attempt to conduct further research into the nature of graphite film formation and its corresponding mechanisms to do so on the micro, rather than macro scale. A single micro contact, when subjected to a typical abrasive particle, can cause the macroscopic contact, and so the interaction of a single hard particle can be used to study and explain the macroscopic wear characteristics of a used lamellar iron component.

Any circumstance which results in or exacerbates a scratch on the matrix, particularly in the vicinity of the lamellar graphite, can cause major elastic and plastic deformation in the matrix during sliding. This can be seen in Fig. 1(a) which corresponds to a real worn surface achieved after 16,000 h operates as marine diesel engine piston ring. As known, the hard abrasive particles present in the piston ring-cylinder liner sliding system are originated either from the fuel catalyst (CAT) fines [15] or produced as the consequence of adhesive or corrosive wear (i.e. oxides/debris) [16] which were analysed by EDS technique and shown in Fig. 1(b) and (c), respectively. CAT fines are considered to be impurities which are present in heavy fuel oil, and result from catalytic cracking during the oil refining process. Hard aluminium silicate particles in various forms, sizes, and hardness ranges are used during the catalytic cracking process [15]. This interaction between the hard particles, matrix, and graphite should be thoroughly taken into consideration in a situation where a continuous supplying of graphite is required. According to Jones et al. [17], a moderate to high concentration of these hard particles may significantly impact on the wear behaviour of the sliding surfaces, in turn resulting in excessive wear to fuel pumps, cylinder liners, and piston rings. From this viewpoint, a single asperity or piece of debris may either indent a mating elastic surface, or become flattened during sliding [18].

Fig. 1. SEM image indicating the presence of a CAT fine and wear debris entrapped in the lamellar graphite, along with corresponding EDS spectra.

The present study investigates the interactions between the hard abrasive particles and graphite lamellas. Microindentation and microscratch testing were carried out in order to understand and explore the matrix and lamellar graphite response, as well as the ways in which graphite may act to lubricate sliding surfaces when it is subjected to abrasive particles.

2 Experimental procedure

2.1 Materials

Two pearlitic lamellar graphite iron piston ring segments, which were sectioned from a marine diesel engine running with heavy fuel oil, were used in the study. Table 1 presents the chemical composition of the tested materials.

Table 1. Chemical composition of the lamellar iron tested samples (wt.%).

C	Si	Mn	Ni	Mo	Cu	Ti	Cr	V	S	P	Fe
3.30	1.55	0.85	0.35	0.60	0.85	0.07	0.15	0.15	0.08	0.10	Bal.

2.2 Microstructural analysis

To examine the microstructure, the specimens were ground and then polished metallographically using diamond paste, following a standard sample preparation procedure down to a 3 μm finish. A 2% Nital solution was used for etching. The etched microstructures were studied using optical microscopy. Fig. 2 shows the microstructure of the etched cast iron specimen. The scanning electron microscopy (SEM) in secondary electron (SE) mode was used to study the deformation pattern of the microstructure after microindentation, and microscratch

tests. In addition, energy dispersive X-ray spectroscopy (EDS) was employed to study the chemical composition of the constituents which caused the abrasion.

Fig. 2. Optical photomicrograph illustrating hard phases in a lamellar cast iron; the remainder of the matrix is pearlite (2% Nital solution).

2.3 Microindentation testing

The microindentation test was performed on 10 mm thick specimens in order to make plastic deformation in the cast iron matrix. The deformation was induced close to the graphite lamellas. The indenter used was based on a Vickers microhardness test, with a typical diamond pyramid with apex semi-angles of 136° . In addition, the microhardness value corresponding to the pearlitic matrix and the hard phases was measured by applying a load of 200 g and dwell time of 15 s. In addition to the hardness measurement, several indentations were made at different locations on the cast iron matrix, and deformation characteristics were compared with indentations made close to the graphite lamellas. The applied test load for indentations was 500 g on a diamond polished surface for a dwell time of 15 s.

2.4 Microscratch testing

To simulate elementary abrasive wear of the lamellar cast iron and examine the contribution of the abrasive particles to the self-lubricating behaviour of the graphite lamellas a microscratch test was conducted. The test was performed under a constant load. The present investigation only simulates the circumstances in which a single hard particle moves over the cast iron matrix during sliding. The same indenter as was used for the microhardness measurement was employed for the microscratch test. Various range of loads could be selected, however here only the results achieved under applying a load of 200 g is presented so that this is representative of actual particle loading on real system. In both microindentation and microscratch tests, the

distance between the indentations/scratches was carefully controlled, being maintained at three times the length of the indentation diagonal, so that the stress field effect in the vicinity of the indentations/scratches was eliminated. All indentations and scratches were made under ambient laboratory conditions.

3 Results and discussion

3.1 Microstructure of the polished lamellar cast iron surface

As shown in Fig. 2, the lamellar cast iron microstructure has three distinct constituents. The matrix is mostly pearlite (a fine mixture of ferrite and cementite), with a very limited quantity of free ferrite. Randomly oriented and distributed graphite lamellas, varying in thickness and length, are also observed in the microstructure. Moreover, there exist some hard phases, which are revealed as white areas and dispersed throughout the matrix. As stated above, the improvement in wear resistance of the piston rings and cylinder liners is somehow related to the amount of these hard particles, which minimize the direct metal to metal contact [3, 5, 7, 8].

Table 2 presents the average microhardness of the pearlite and white hard phase. In order to ascertain the repeatability of the measurements, the average of six indentations was calculated. The average microhardness of the hard phase present in the structure was 753 HV which, in comparison with the microhardness of pearlite (292 HV), increases the overall hardness of the cast iron. The effect of the hard phase on wear resistance will be further explored in section 3.3. However, one should consider that the data presented for pearlite microhardness does not take into account the pearlite lamellar orientation and coarseness of the pearlite, which may have caused a differing hardness value for a given coarseness and lamellar pearlite orientation.

Table 2. Vickers microindentation hardness results for the constituents of the lamellar cast iron samples investigated.

Constituent	Length of diagonal [mm]±SD	Vickers microhardness HV _{0.2} [kg/mm ²]±SD
Pearlite	0.0357±0.0017	292.25±26.72
Hard phase	0.0225±0.0025	752.58±166.56

3.2 Microindentation testing

Given the above observations, it was considered to be of value to study in detail the mechanism behind the self-lubricating process of the graphite, and its contribution to the formation of the tribofilm. Consequently, the microindentation test, together with a microscopic analysis, was performed. Indentation leaves a permanent impression on the material surface, and induces both compression stresses and shear stresses on the four-sided slant impression surfaces. This

is believed to facilitate a better understanding and interpretation of the related deformation mechanisms, particularly those which occur close to the graphite lamella. Moreover, it could serve to simulate the deformation caused by an abrasive particle travelling along the actual sliding parts, albeit to an extremely exaggerated degree.

3.2.1 Lamellar graphite's response when subjected to microindentation testing

Fig. 3(a) presents a cross-section of a typical graphite in a lamellar cast iron polished surface, prior to indentation. This being done near to the graphite caused stress to be transferred towards the lamellar graphite through deformation of the metallic matrix. The close-up view of the indentation at 500 g is shown in Fig. 3(b) at a higher magnification.

Fig. 3. (a) Optical micrograph of the lamellar graphite before indentation; (b) SEM image showing the same area at a higher magnification, in order to further illustrate extruded graphite as a result of indentation.

When comparing the graphite feature prior to indentation, the SEM micrograph allows clear imaging of the surface response of the cast iron indicating that the lamellar graphite was compressed and extruded from its original pocket during loading due to the induced compressive stress which is generated as a result of indentation.

The net force applied to the graphite during indentation was a direct result of both the elastic and plastic deformation of the matrix. However, following the relief of the applied force, only the plastically deformed area, visible as an impression on the surface, remained on the affected region. The present investigation revealed that the graphite lamellas were sufficiently compressed (due to elastic/plastic strain) in the planes during indentation to precipitate the initial displacement of the graphite. Furthermore, according to the studies performed by Liu et al. [11] on aluminium alloy-graphite particle composite, and Sarmadi et al. [19] on copper-graphite

composites, subsurface deformation during sliding plays an important role in graphite lubricating film formation on tribosurfaces.

In another example shown in Fig. 4, which is similar to the earlier illustration, observations indicated an extrusion relationship between the lamellar graphite and indentation. The cracks appeared in the centre of the lamellar graphite, which can be confirmed by an examination of the marked area by the black circle near the edge of the graphite shown in Fig. 4. The bifurcation into two halves, indicated by two white arrows, with differing orientations is also clearly seen in Fig. 4. One explanation for this phenomenon could be related to the fact that the lamella is not perfectly straight, and instead takes a shape rather like a twisted plate, meaning that indentation, and in turn stress distribution, around the lamellar graphite caused to extrude it in two different planes. This latter observation immediately raises the question as to whether there exists any tendency towards a specific relationship between the lamellar graphite structure and the extrusion pattern which is represented here.

Fig. 4. The extrusion mechanism of the lamellar graphite caused by microindentation.

In addition, two distinct regions may be identified after indentation; the unaffected area near the matrix-graphite interface, and a sharp, clean, and shiny edge between the displaced graphite and the near-untouched region of the graphite as shown in Fig. 4. This individual characteristic is suggestive of the fact that, during indentation, the extrusion is largely governed by the deformation of the matrix in the area around the lamellar graphite (predominately near to the end line), as seen in Fig. 3(b) and Fig. 4, rather than at the metal matrix-lamellar graphite boundary. Therefore, this simple experiment enables discussion regarding the natural behaviour of lamellar graphite when subjected to shear stress.

One possible explanation for such an observed interaction may be related to the nature of the bonding which exists within the graphite layers, particularly at, or close to, the centre, when compared to the unaffected graphite-matrix interface. The appearance of the sheared graphite in this region can presumably, however, be related to the weaker bonds between graphite lamellas and other phases such as defects and imperfections in general. As has been discussed in literature, and with regard to the point that the lamellar graphite nucleation begins heterogeneously on complex silicates, oxides [20-22], intermetallic compounds [23, 24] and sulphides (MnS) [25, 26], this allows the formation of coherent/semi-coherent low energy interfaces between the substrate and the graphite [20]; i.e. the presence of some weak incoherent bonds becomes more likely [27]. Such conditions provoke the shear stress, and may ultimately lead to lamination of the graphite lamellas.

Fig. 5. The response of two graphite lamellas to the similar microindentation, made nearby; whilst they vary in size, both display the same extrusion mechanism.

On the other hand, no evidence for graphite delamination was detected in the metal matrix-graphite interface, which could be the proof of the existence of far stronger bonds between the cast iron matrix and graphite constituent so that is much easier for the graphite to be fractured and extruded from the centre, rather than that of graphite-matrix interface. In order to generalize the present observation and ascertain whether this tendency was dominant, the microindentation test was conducted for plenty of lamellas of approximately the same size; however, and rather surprisingly, this was apparently the most commonly observed patterns in all cases.

The fracture correspondence of two lamellas, which varied in length but were subjected to the similar indentation conditions is shown in Fig. 5. As Fig. 5(a) demonstrated in lamellas with

larger size, the 500 g applied load affected only some parts of a lamella while, for a smaller lamella, the same load was sufficient to significantly impact the whole graphite (see Fig. 5(b)).

Fig. 6. SEM image of the worn surface of a marine diesel engine, operated for 16,000 h.

In both cases, the similar behaviour was observed, with the graphite displaying an extrusion tendency from the centre. Compared to reality, this extreme situation might not be exactly identical to situation in real piston ring during sliding in terms of applied load, particle size and the tested environment, however it could give valuable information on the way of the graphite interaction with abrasive particles [28]. Fig. 6 shows a real worn piston ring surface, wherein the surface damage, in the form of scratches from hard particles, is clearly visible. The interaction between the scratches [12] and graphite flakes including the extraction of the graphite from their pockets, and covering the lamellas due to matrix deformation, are pointed out by white arrows in Fig. 6.

3.2.2 Characterization of microindentation shapes

Various indentation shapes were examined and classified by Young and Millman [29]. The indentation response of the pearlitic lamellar iron under discussion revealed a clear difference in indentation shape for different graphite lamellas, and Fig. 7 shows two graphite lamellas with markedly different indentation responses. The applied load was 500 g in both instances. Fig. 7(a) shows two perfectly square and clear indentations so that the graphite was not influenced by the two microindentations which were made, despite them being made very close to the graphite. These indentation shapes are very similar to those made in matrix, but are further away from the lamellas. However, Fig. 7(b) illustrates a different situation, in which the graphite was extruded as a result of the damage (fracture and/or deformation) caused by indenting. The Nomarski microscopy technique was used to further increase contrast and show the area

deformed as a result of indentation. It may be observed that the indentation edges near to the graphite became concave. They were affected to a much greater degree than the two edges where no interaction was detected with the graphite, while the latter are almost straight. The present response could closely correspond to a small plastic deformation occurring in this region of the matrix; therefore, it is reasonable to conclude that this alteration in indentation shape is most likely associated with the lamellar graphite extrusion behaviour. However, as discussed by Mendas [13], the change in indentation shape could also be related to the presence of hard phases, such as carbides, close to the indentation area.

Fig. 7. (a) SEM photomicrograph and optical image, showing the microindentation shape close to the graphite lamellae as (a) unaffected; (b) extruded, respectively.

3.2.3 The proposed mechanism for graphite extrusion tendency

The most uniquely shaped indentations observed during the present study are schematically depicted in Fig. 8. In most cases, a near-perfectly square indentation was obtained as shown in Fig. 8(a). They typically were associated with indentations made either in the matrix away from the graphite lamellae, or close to the graphite but without extrusion response (see Fig. 7(a)).

Fig. 8. Microindentation shape characteristics; (a) straight edges, (b) concave edges.

However, as indicated in Fig. 5(a) and (b), where the graphite were come out, the concave shape of the edges close to the graphite was obtained after unloading. Dissimilar behaviour was observed when compared to the other three edges, and is schematically depicted in Fig. 8(b). As has been mentioned above, the emergence of the graphite in the vicinity of the indentation mark causes a change in indentation shape. Among those mechanisms which may influence the wear process, one which may explain the extrusion mechanism of the lamellar graphite is exhibited in Fig. 9. As discussed earlier, the SEM image also displays three distinctive, clear-cut regions; a metallic matrix, graphite which is unaffected, and graphite which has been extruded.

At the first stage of the extrusion process, the graphite is unable to withstand the high strain applied by the indenter due to its low strength [30]; meanwhile, the metal matrix is compressed towards the graphite. This compression stems from the combination of both the elastic and plastic strains. The presence of such compressive forces on graphite lamellas results in the graphite becoming thinner and, simultaneously, the shear force becomes sufficient to cause the graphite lamellas to slip over each other. Thus, the graphite is displaced from its pocket, as shown in Fig. 3(b) and Fig. 4.

During the real operational conditions, this issue may be exacerbated due to sufficiently large particles, in the form of either CAT fines or relatively coarse metallic wear debris, which cause indenting and scratching on the sliding surfaces [7, 16]. Due to this interaction, the subsurface is squeezed and causes to extrude the graphite onto the sliding surfaces. Then, the formed debris of graphite is smeared between the sliding contacts, resulting in tribofilm formation. Meanwhile, some part of the removed metallic matrix forms metal debris, which consists primarily of α -Fe (ferrite), as illustrated in Fig. 1(c) [7, 31].

Fig. 9. SEM image showing three distinct areas as a result of an indentation made close to the graphite lamella.

3.3 Microscratch testing

As shown in Fig. 6, abrasive particles can cause severe damage (failure/deformation) to tribological surfaces, primarily affecting the topmost matrix surface layers and graphite lubricating capability. Fig. 10(a) and (b) demonstrate the SEM morphologies of two scratch grooves and their interactions with hard phases and graphite lamellas, respectively; in both cases, a ploughing mode was observed. No metal chips, and cracks were observed at the bottom of the grooves. Moreover, the contribution of the hard phases in improving the wear resistance during scratching is well-discussed in literature [13]. This mechanism is illustrated by the SEM micrograph in Fig. 10(a). As seen, gliding occurs on hard phases where the indenter crosses the hard phase, and a narrower scratch is formed with no displacement of the metal matrix.

Fig. 10(b) highlights the commonly observed graphite morphologies and their influence on tribological properties when subjected to a scratch. A similar extrusion behaviour was detected when compared to earlier observations made as a result of the microindentation testing. As regards a lamellar graphite which lies in the direction of scratching, the passage of the indenter causes compression of the lamellar graphite and extrudes it from its pocket; the graphite then follows the indenter by sticking to its tip, causing less scratching. Other investigation [14] led to the realization that the decrease in the friction coefficient occurs due to the presence of a smeared graphite layer over the scratch groove. However, when the indenter penetrates deeper, through application of higher loads, it acts on a consistent volume of the material and thus the corresponding damage is more significant. The study performed by Mendas et al. [13] indicated

that the portion of the graphite which is in front of the sliding is fractured, extruded and smeared out onto the sliding surface. Further matrix deformation causes the graphite to be covered.

Fig. 10. SEM illustration representing the microscratch interaction developing on a cast iron matrix with: (a) hard phases (load 50 g), (b) graphite lamellas with different orientations (load 200 g).

Besides that, the graphite parallel to the scratch also began to be compressed and extruded from its natural position. However, it should be emphasized here that the extrusion tendency related to the lamellar graphite depends upon its size (as previously discussed in 3.2.1) and orientation with respect to the scratching direction (as demonstrated in Fig. 10(b)).

4 Conclusions

The microindentation and microscratch tests that were performed on lamellar graphite iron demonstrated a similar mechanism is involved in the contribution of the graphite to lubricate the sliding surfaces. Furthermore, it has been demonstrated by the indentations made close to the graphite lamellas that the deformed matrix caused the cracks to be formed in the centre of the graphite within the lamellas, meaning that the graphite is being compressed and then extruded from its natural position. This particular behaviour was demonstrated in other ways as well, notably by combining the scratch testing. In all cases, it was observed that the graphite was extruded from the centre of the graphite pocket. These observations could confirm that there is a weak bond in place between the graphite layers when compared to the graphite-matrix interface, due to the fact that there was no evidence for extrusion response from the graphite-matrix interface area.

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Figure caption

Fig. 1. SEM image indicating the presence of a CAT fine and wear debris entrapped in the lamellar graphite, along with corresponding EDS spectra.

Fig. 2. Optical photomicrograph illustrating hard phases in a lamellar cast iron; the remainder of the matrix is pearlite (2% Nital solution).

Fig. 3. (a) Optical micrograph of the lamellar graphite before indentation; (b) SEM image showing the same area at a higher magnification, in order to further illustrate extruded graphite as a result of indentation.

Fig. 4. The extrusion mechanism of the lamellar graphite caused by microindentation.

Fig. 5. The response of two graphite lamellas to the similar microindentation, made nearby; whilst they vary in size, both display the same extrusion mechanism.

Fig. 6. SEM image of the worn surface of a marine diesel engine, operated for 16,000 h.

Fig. 7. (a) SEM photomicrograph and optical image, showing the microindentation shape close to the graphite lamellas as (a) unaffected; (b) extruded, respectively.

Fig. 8. Microindentation shape characteristics; (a) straight edges, (b) concave edges.

Fig. 9. SEM image showing three distinct areas as a result of an indentation made close to the graphite lamella.

Fig. 10. SEM illustration representing the microscratch interaction developing on a cast iron matrix with: (a) hard phases (load 50 g), (b) graphite lamellas with different orientations (load 200 g).

Table caption

Table 1. Chemical composition of the lamellar iron tested samples (wt.%).

Table 2. Vickers microindentation hardness results for the constituents of the lamellar cast iron samples investigated.

Highlights

- The interaction between graphite lamellas and abrasive particles is discussed.
- Matrix deformation showed a major effect on lubricating performance of graphite.
- A mechanism on self-lubricating behavior of graphite lamellas is proposed.
- Under matrix deformation all lamellas are fractured and pushed out from the center.