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Load Effect on Scratch Micro-Mechanisms of Solution Strengthened Compacted Graphite Irons

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Abstract

This study investigates the scratch load effect, from 100 to 2000 mN, on micro-mechanisms involved during scratching. A pearlitic and three ferritic Compacted Graphite Irons (CGI) solution strengthened through addition of 3.66, 4.09, and 4.59 Si wt% were investigated. Good correlation was observed between hardness measurements, tensile testing, and scratch results explaining the influence of matrix characteristics on scratch behaviour for investigated alloys. A significant matrix deformation, change in frictional force and scratch coefficient of friction was observed by increase in scratch load. In all cases, microscratch depth and width increased significantly with load increasing, however pearlitic CGI showed most profound deformation, while the maximum and minimum scratch resistances were observed for high-Si ferritic and pearlitic CGI alloys, respectively.

Keywords: CGI, Si solution-strengthening, scratch testing, deformation micro-mechanisms during scratching.

1 Introduction

Abrasion caused by asperities or hard particles on component surfaces is a very important issue in various engineering applications, such as piston rings and cylinder liners. Eyre [1] estimates that roughly 50% of the total wear encountered in industrial situations is because of abrasion. Pearlitic Compacted Graphite Iron (CGI) is considered to be a suitable candidate for heavy truck and heavy-fuel marine diesel engine applications such as piston rings and cylinder liners, where a combination of excellent mechanical, thermal, and tribological properties is essential [2]. However, the low machinability of pearlitic CGI is a large drawback [3], [4], [5]. In addition, components such as cylinder liners and piston rings are exposed to severe wear conditions, in particular abrasive wear, and so abrasion resistance is a critical factor for engine manufacturers and designers [6], [7]. Abrasion is a relatively complex wear mechanism that is influenced by a variety of parameters, such as size, shape,

and attack angle of the abrasive particles, along with the hardness of the contacting materials (in terms of both the abrasive particles and workpiece) and operating conditions and environment, e.g. applied loads and relative humidity [8], [9], [10]. As hardness strongly influences abrasion behaviour of the materials, in recent decades CGI alloys with increased hardness and improved matrix structures have received significant attentions in industrial applications, particularly those in which good machinability and reasonable wear resistance while maintaining strength are crucial. Hardness can be increased through modifying the pearlitic structure, grain refining, solid solution-strengthening, and so on [11], [12]. In this study, the solid solution-strengthening mechanism, increases in hardness and improvements in tensile strength, and the yield strength of ferritic alloys have been studied through adding Si in different quantities. However, the hardness improvement was obtained at the expense of, to some extent, the thermal conductivity and ductility of the alloys [3], [13].

It is believed that the detailed micro-scale examination of the scratch behaviour could contribute to further tribological and mechanical improvements and an improved understanding of the macroscopic abrasion mechanisms of heterogeneous materials such as CGI alloy. Although substantial effort has been put into studying the abrasion mechanisms of steel and polymers by Murray et al. [14], Glaeser [15], Bates et al. [16], Kayaba et al. [17], and Calabrese et al. [18] (and a good correlation has been found between micro- and macro-scale abrasive wear mechanisms), very few studies have been performed using cast iron materials, in particular CGI alloys.

In recent years, the single-point scratch testing technique has been commonly used as a comparative means to determine a practical coating's adhesion strength and cracking failure, as well as to characterise the scratch resistance of coating and bulk materials [19]. The present technique has also been employed to evaluate the elastic and plastic deformation (permanent deformation) of materials on micro- and nano-scales [20]. The obtained results can fairly be extrapolated with reasonable accuracy to predict the global wear response of heterogeneous materials such as cast irons in order to characterise the active mechanisms of abrasion. This understanding helps to evaluate the contribution of each individual micro-constituent to the global tribological properties of the multiphase components [21]. In addition, it is believed that the knowledge obtained through scratch testing can be used to explain the role of each individual micro-constituent within the matrix in micro-machining of components [22].

This study correlates experimental results obtained from microscratch testing performed over surfaces, microstructural features and mechanical properties, in order to determine the abrasion resistance of pearlitic and Si solution strengthened ferritic CGIs. Furthermore, SEM analysis was used to study the scratch behaviour and related matrix deformation in details by investigating the interaction between the indenter, the matrix, and graphite particles as result of scratching.

2 Experimental procedure

2.1 Materials and sample preparation

A conventional pearlitic compacted iron as a ‘reference material’, and the three-developed ferritic CGIs solution strengthened with different Si contents of 3.66, 4.09, and 4.59 wt% (named low-, medium- and high-Si alloys, respectively) with chemical composition listed in **Table 1** were used in this study. More detail regarding the cast component geometry, and casting set-up can be found in our earlier work [13].

Table 1. Chemical compositions of the CGI samples used in this study (wt%).

Materials	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Cu	V	Mg	C _{eq}
Pearlitic CGI	2.73	2.04	0.420	0.015	0.016	0.033	0.007	0.038	0.019	0.791	0.011	0.020	3.24
Low-Si CGI	3.24	3.66	0.202	0.010	0.011	0.054	0.010	0.065	0.025	0.020	0.016	0.014	4.16
Medium-Si CGI	3.01	4.09	0.190	0.010	0.010	0.041	0.010	0.066	0.025	0.019	0.016	0.010	4.04
High-Si CGI	3.13	4.59	0.173	0.010	0.009	0.038	0.010	0.066	0.027	0.019	0.015	0.014	4.28

$$C_{eq} = C\% + 0.25(\%Si) + 0.33(\%P) + 0.4(\%S) - 0.027(\%Mn).$$

The as-cast samples were cut into 20×20×15 mm pieces, then metallographically ground and polished according to the standard cast iron sample preparation procedure down to 1 µm. The sample preparation process was carefully controlled as regards load and speed in order to minimise the stresses generated on the surface during grinding and polishing steps prior to scratch testing.

2.2 Mechanical properties’ measurements

The tensile properties of four grades of CGIs were investigated through tensile testing using a Zwick Z100 machine according to the ISO 6892-1:2009 standard. In addition, Vickers microhardness measurements were performed locally by applying a load of 600 mN just over the pearlite colonies and ferritic matrix. The main idea of performing the microhardness measurement is to identify the effect of adding Si on solution strengthening of the iron metallic matrix (reasonably small volume) and furthermore its influence on scratch resistance of the fully ferritic matrix CGI alloys, excluding any graphite particles effects. In addition, Brinell measurements (using a load of 750 kg and 5 mm taken using a tungsten carbide ball) were also conducted to correlate the tensile mechanical properties and general hardness values as well as the effect of hardness on the transition of the abrasive wear mechanism of ferritic Si solution strengthened CGIs. Since the indenter used in Brinell hardness measurement is big, the obtained hardness values are significantly influenced by both the metallic matrix and graphite particles present in the microstructure. Moreover, a larger volume of the matrix is influenced by the Brinell hardness measurements.

The scratch (abrasion) resistance is strongly influenced by the strain hardening capability of the materials of the substrate and the sub-surface layer generated during scratching [23], [24]. Due to the complexity of the work hardening phenomenon during scratching tensile strain hardening behaviour of the microstructures was analysed using the Ludwigson model, as given by **Eq. (1)** [25]:

$$\sigma = K_H \epsilon_{pl}^{n_H} + \Delta \quad \text{where} \quad \Delta = e^{(k_L + n_L \epsilon_{pl})} \quad (1)$$

where K_H is the strength coefficient, n_H represents the strain hardening exponent, K_L and n_L are the coefficients obtained through plotting strain versus the natural logarithm of the Δ -term which resulted in a straight line. The n_L - and K_L -values are the slope of the line for small strains, and the the interception point at zero plastic strain, respectively.

2.3 Microscratch testing

A Micro Materials NanoTest system equipped with a sphero-conical diamond indenter with a tip radius of 50 μm and induced cone angle of $2\beta = 90^\circ$ (as shown in **Fig. 1**) was used to conduct the microscratch testing. The details of the equipment can be found in our other study [20]. The scratch tests were

Fig. 1. SEM image showing the used sphero-conical diamond indenter with a conical tip (50 μm radius) and induced cone angle of $2\beta = 90^\circ$.

performed under various constant loads ranging from 100 to 2000 mN. All scratches were performed over a length of 1000 μm , at scratch speed of 10 $\mu\text{m/s}$, and at room temperature $\sim 25^\circ\text{C}$. Each scratch test involved a pre-scan and post-scan procedure under a very low profiling load of 0.2 mN to provide a reference surface position, profile the surface topography, and obtain the true depth of penetration. A third scratch pass was used to profile the scratch depth after elastic recovery. The applied normal load, tangential force, and resulting microscratch depth were continuously recorded during scratching. **Fig. 2 (a)**

and **(b)** show schematically a typical scratch test diagram and a groove profile produced by scratching a flat specimen, respectively. More details on the equipment used, set-up of the scratch experiment, and scratch-depth measurements performed during and after scratching can be found in [20]. Three scratches were performed for each loading condition, and the width and depth of each scratch were measured at several locations along the scratch length using Alicona G4 InfiniteFocus Light Optical 3D measurement Microscope (LOM). Averages of at least 15 measurements are reported in this study. Once the tensile and scratch tests were completed, the fractured and scratched surfaces and the featuring deformation induced by scratch testing, were investigated using a JSM 7001F Scanning Electron Microscope (SEM).

Fig. 2. (a) Schematic diagram of the scratch test; (b) side view of a typical scratch profile resulting from scratching.

2.4 Degree of penetration

As presented by Hokkirigawa et al. [9] and Kayaba et al. [17], the degree of penetration D_p , as defined by Eq. (2), was calculated to obtain a fundamental understanding of the effect of scratch normal load on abrasive wear behaviour and study the involved mechanisms during scratching of pearlitic and ferritic metal matrix. The D_p , as the ratio of the groove depth, h , to half of the contact width, W_1 , during scratching, defines the severity index of contact, so that an increase in D_p causes the abrasive wear mechanism to change from a ploughing mode through wedge formation to a cutting mode.

$$D_p = \frac{2h}{W_1} \quad (2)$$

2.5 Scratch hardness

With the indenter's tip geometry (sphero-conical shape), Eq. (3) can be used to calculate the recovered microscratch depth [26]:

$$W_1 = 2\sqrt{2Rh - h^2} \quad (3)$$

where W_1 , h , and R are the recovered scratch width (radius of contact), depth of penetration, and radius of the indenter's tip, respectively.

In current study, the scratch width, W_2 , was measured using the LOM to compute scratch hardness values. Scratch hardness is defined as the ratio of the normal scratch load to the projected contact area. For general viscoelastic plastic materials such as CGI alloys, scratch hardness is approximately calculated using Eq. (4) proposed by Sinha et al. [26], Williams [27] and Briscoe et al. [28] for viscoelastic plastic materials:

$$HS_p \approx \frac{F_N}{A_{LB}} \approx \frac{8F_N}{\pi W_2^2} \quad (4)$$

where F_N represents the normal load applied to the indenter's tip, and A_{LB} is the projected load-bearing area that is obtained by measuring W_2 , which is the width of the groove on the scratched surface after elastic recovery.

3 Results and discussion

3.1 Mechanical properties

In order to clarify and investigate the influence of matrix characteristics and mechanical properties on scratch behaviour, Brinell and Vickers hardness measurements and tensile testing were performed on both the pearlitic and the three fully ferritic solution strengthened CGI alloys, as the results of which are shown in **Table 2**. It should be noted that the presented Vickers microhardness values represent only the measurements that were carried out on the pearlitic colonies and ferritic metallic matrix, i.e. other micro-constituents such as graphite particles and hard phases are excluded. This was done in order to characterise the impact of Si addition on the strengthening of the ferritic iron matrix.

As shown in **Table 2**, increase of Si content from low- to high-level resulted in a clear increase in Vickers micro-hardness values – from approximately 230 to 287 HV – which corresponds to an increase of about 25% as well as increase of roughly 23% for Brinell hardness values. It is interesting to note that, approximately 7% improvement was also noticed in Young's modulus, E , of the solution strengthened ferritic alloys compared to the pearlitic CGI, while the difference in E between the low-, medium-, and high-Si CGIs was negligible.

Table 2. Vickers and Brinell hardness measurements, and tensile test results of the investigated CGI alloys.

Materials	Hardness measurements		Tensile test properties				Ludwigson equation parameters			
	HV [0.06]	HBW [5/750]	R_m [MPa]	$R_{p0.2}$ [MPa]	E [GPa]	ε_F [%]	n_H [-]	K_H [MPa]	n_L [-]	K_L [-]
Pearlitic CGI	219±2.8	205±2.8	410±2.1	308±4.2	145±0.7	1.28±0.1	0.125±0.23	820±9.5	-2586±198	3.5±0.05
Low-Si CGI	230±2.5	182±3.8	408±0.7	364±1.3	157±0.1	2.32±0.3	0.053±0.011	518±6.4	-1404±130	4.4 ±0.25
Medium-Si CGI	258±2.5	198±4.7	451±1.5	415±1.6	155±0.4	1.71±0.8	0.045±0.001	557±21.9	-1528±114	4.7±0.08
High-Si CGI	287±3.7	223±4.1	516±0.4	480±0.8	154±0.8	1.09±0.1	0.058±0.002	691±46.1	-1789±129	4.9±0.15

Not only the hardness values, but also both the $R_{p0.2}$, and the R_m of the Si solution strengthened CGIs were improved significantly, by approximately 29% and 25%, respectively, through the addition of Si. However, a more profound influence of Si addition was observed on improved $R_{p0.2}$ than R_m as compared to E . This considerable increase in micro- and macro-hardness values, improvements in yield strength, and tensile strength are principally due to the solid solution-strengthening effect of Si through hindering dislocation mechanisms of plastic deformation. The obtained values for the three Si solutions-strengthened CGIs are in good agreement with earlier studies performed by Stets et al. [29] and Glavas et al. [30] on fully ferritic solid solution strengthened ductile cast iron, except the R_m trend, in that they reported the solution embrittlement's start point and a drop in R_m as the Si content exceeded 4.22 wt%, however such phenomenon was not observed in this study for high-Si CGI. In spite of increase in Vickers and Brinell hardness measurements, as well as improvements in $R_{p0.2}$, and R_m values; a brittle failure (see **Fig. 3**) was observed, particularly for the high-Si and pearlitic CGIs, together with a

significant drop in elongation to failure as a result of increasing Si content, so that high-Si CGI showed the lowest elongation to failure among the CGIs.

The strain hardening exponent as a measure of the resistance to plastic deformation is affected by multiplication factors of plastic deformation and dislocations in the metal [31]. In cast irons alloys, in addition to the chemical composition effect, the graphite roundness, and graphite fraction play important roles in strain hardening exponent (plastic deformation rate), so that, strain hardening exponent increases by decrease of roundness, and increases of the graphite fraction by considering this fact that the strain hardening exponent can be explained as a multiplication factor of plastic deformation and dislocations [31]. **Table 2**, shows a significantly lower n_H values for the solution strengthened ferritic CGIs than that of pearlitic CGI. This represents materials with very small (or almost zero) strain-hardening behaviour at room temperature (note that an ideal plastic material has an n_H value of zero). However, the pearlitic CGI's n_H value – approximately 0.125 – showing some strain hardening capability during tensile testing. Among the ferritic CGI alloys, increase of Si content from low- to medium- resulted first in a decrease of n_H value, which is in good agreement with the other studies [32]. However, a further increase of Si content to high-level caused an increase in the n_H value which needs a detailed study – considering alloys' composition, ferrite grain size, graphite roundness and graphite fraction – to be able to explain this happening [31]. Interesting to notice that a fair relationship was observed between the C_{eq} and n_H values, which could partly explain the observed trend in CGIs tested materials. The other possible reason could be related to the maximum about 4 wt% solubility limit of the Si in ferritic matrix [33], and about 2 wt% in pearlitic matrix which results in matrix hardening via solution-strengthening [12]. Strength coefficient K_H – representing the material strength – showed an increase through increasing Si content from low- to high-level due to matrix strengthening, although the values were lower compared to pearlitic grade in good agreement with yield stress data (see **Table 2**).

The Δ -term containing the two new n_L and K_L parameters, added by Ludwigson to the Hollomon equation, compensates the non-uniform linear behaviour of the double logarithmic plot. As the plastic strain increases the Δ -term becomes smaller, such that the Ludwigson equation approaches the Hollomon equation for larger strain values. The K_L is used to adjust the initial plastic stress value calculated by the Hollomon equation, which can be either positive or negative depending on the sign of the Δ -term. With this in mind, a larger K_L value is poorly approximated by the Hollomon equation at just above zero plastic strain [34].

The values of K_L were, as shown in **Table 2**, relatively matrix-dependent. Thus, the pearlitic CGI showed the minimum value, while no dramatic variation was observed for the Si solution strengthened ferritic alloys. It is interesting that, among the developed ferritic CGI alloys, K_L was not affected

dramatically as result of metal matrix hardening in the same way as, for example, the coefficients in the Hollomon equation, although a somewhat lower value was observed for the pearlitic CGI. As shown in **Table 2**, the coefficient n_L (absolute value) increased with an increase in Si content, showing that a larger total plastic strain value generally resulted in a smaller n_L value (low-Si CGI), whereas smaller total plastic strains caused larger n_L values (high-Si CGI), which is in agreement with the literature [34].

In contrast with the improved mechanical properties, Si addition caused a decrease of roughly 53% in elongation to failure at room temperature for the ferritic Si solution strengthened CGI materials. **Fig. 3 (a)-(d)** are the SEM images of the tensile fracture surfaces of all four grades of investigated CGIs. As is obvious from the fracture surfaces, and in contrast to lamellar and spheroidal graphite iron alloys [35], the fracture mechanism of CGI alloys is comprised of a mixed mode of ductile and brittle fractures which is explained by the irregular shape of the graphite particles, which cause different triaxial stress fields around the compacted graphite particles [36].

Fig. 3. SEM images showing the effect of silicon on fracture pattern of; (a) pearlitic CGI, (b) low-Si, (c) medium-Si, and (d) high-Si ferritic solution strengthened CGIs.

The fracture surface of the pearlitic CGI (**Fig. 3 (a)**) was comprised of a facet pattern with transgranular fractures, together with multiple micro-void coalescence, which is a strong indication of a dimple rupture. However, as can be observed in **Fig. 3 (b)-(d)**, an increase in Si content resulted in the fracture

mechanism changing considerably, from a dimple pattern to the predominance of cleavage fractures with the appearance of an embrittlement issue, as can be seen most clearly in the high-Si CGI, (**Fig. 3 (d)**), where both transgranular and intergranular cleavage mechanisms dominated the fracture mechanism. It can thus be deduced that, in the investigated ferritic CGIs, the brittle cleavage mechanism was the most preponderant fracture mechanism. Such fracture behaviour can be explained by the small total plastic deformation that occurs in high-Si solution strengthened CGI. The cleavage facets blend into areas of dimple ruptures, and the cleavage steps became tear ridges.

Fig. 4. SEM image showing the tensile fracture surface of the medium-Si ferritic CGI. It is dominated by decohesive ruptures and intergranular cracks.

The increase in Si content in ferrite matrix caused a decrease in the ferritic grain boundary's strength. Furthermore, this nature resulted in weakening of the grain boundaries' cohesion and an increase in the transition temperature [37], such that the fracture mechanism changed from a dimpled and moderate transgranular cleavage fracture to intergranular and transgranular severe brittle cleavage. This can be confirmed by the tensile fracture surfaces shown in **Fig. 3** and **Fig. 4**, as well as previous studies of steels and other types of cast iron alloys which claim that,

during the solidification process, Si atoms are segregated in the grain boundaries. As shown in **Fig. 4**, an increase in Si content to the medium-level caused the creation of many decohesive ruptures and intergranular cracks, with the crack propagation occurring at the grain boundaries. The grain boundary will always fail in preference to cleavage due to the slip planes' orientation and low-index surface energy, which favours cleavage fracture occurrence along crystallographic planes over intergranular failure in metals with body-centred cubic (bcc) crystals [37], [38].

3.2 Microscratch analysis

As a viscoelastic plastic-metallic material, CGI alloy undergoes both elastic and plastic deformation during scratching, and so the scratch depth measured during loading is a combination of both deformations. After unloading, however, the measured scratch width and depth values represent the matrix after elastic recovery. Although progressive load scratch testing is a quick method of studying the scratch behaviour of coating and bulk materials, constant load scratch testing is generally used to investigate critical scratch load, which causes cohesive failures, such as cracking, or plastic

deformation, which affects the overall tribological performance of the material [39]. Thus, in this study several scratch tests were performed under loads ranging from 100 to 2000 mN.

The actual penetration depth at the time of scratching (scratch depth), post-scan topography (recovered depth), and frictional force profile versus the scratch length were recorded and are presented for a constant load of 600 mN on the low-Si alloy by solid black, blue dash, and green dash-dot-dot lines, respectively, in **Fig. 5 (a)**.

Fig. 5. (a) Scratch depth and frictional force profiles measured during scratching, post-scan topography vs. scratch length, (b) SEM micrograph shows a scratch performed under a constant load of 600 mN on low-Si solution strengthened CGI. The scratch direction is from left to right.

The SEM image corresponding to the scratch described by the graph is shown in **Fig. 5 (b)**. As can be concluded from **Fig. 5 (a)** and **(b)**, the induced stress caused by scratching was sufficiently high to cause plastic yielding in the localised regions of the matrix. A uniform groove was observed as a result of the scratching of the material up to a length of roughly 200 μm. Such a uniform scratched groove, which was created due to the uniform hardness resulting from solution-strengthening, suggests a profound positive impact on the machinability of these alloys, as was found by another study [40]. By following the scratch depth, a noticeable variation was identified in the actual depth of penetration profile, which is associated with the effect of individual microstructural features such as graphite particles and carbides, on wear resistance under low-severity conditions (i.e. a small abrasive size and a low load). These effects are also seen in the frictional force profile. It should be noted that the grain

boundaries could act as effective barriers, changing the response of the material to the crossing of the indenter. This is visible in **Fig. 5 (a)** in the region of the grain boundaries in terms of scratch depth and width, and with regard to frictional force curves, which is in agreement with the findings of Moore [41]. In addition, some degree of "stick-slip" phenomenon; when the indenter passes over different micro-constituents; which resulted in a non-uniform depth scratch groove during slip showed which increased with the increase of scratch load magnetified as shown and discussed in **Section 3.2.2**.

Moreover, frictional force dropped when the indenter passed over the graphite particles (see **Fig. 5 (a)**) [20]. This phenomenon could provide a valuable micro-level explanation of how graphite particles contribute to the lubricating of tribosurfaces under sliding conditions when small abrasive particles in the tribo-system face the graphite phase, causing fracture behaviour and the extrusion of graphite particles from their pockets [42], [43].

3.2.1 Microscratch depth and width, and SCOF measured during scratching

Fig. 6 (a) and **(b)** show the indenter's actual depth of penetration (contact depth) and scratch width (contact width, W_I) with 95% confidence band during scratching. It should be noted that the scratch contact width curves were calculated using **Eq. (3)**. As can be clearly seen in **Fig. 6 (a)** and **(b)** for all of the investigated materials, an increase in the scratch load resulted in the scratch contact depth and contact width tending to increase. By coupling microscratch depth and width with hardness measurements (see **Table 2**), it can be concluded that the greatest scratch depth was found in the pearlitic CGI alloy, followed by the low-, medium- and high-Si alloys, respectively. Medium- and high-Si alloys both showed a significantly shallower scratch depth. This behaviour is in good agreement with the micro-hardness Vickers measurements, in that the minimum matrix hardness was observed for pearlitic CGI, supporting this observation. While a good correlation exists between the Brinell hardness data and the scratch behaviour, no clear explanation exists regarding the pearlitic CGI's high hardness value and poor scratch resistance. As is obvious from **Fig. 6 (a)**, the microscratch depth of the high-Si alloy is only slightly lower than that of the medium-Si CGI. The lower the scratch depth, the less material loss and the greater scratch resistance, as in good agreement with the tensile and scratch data given in **Table 2** and **Table 3**.

Fig. 6. (a) Microscratch contact depth and (b) microscratch contact width, W_t , corresponding to the scratch testing performed under constant loading conditions ranging from 100 to 2000 mN (95% confidence band). The error bars indicate one standard deviation.

On the effect of hardness on scratch resistance, it was observed that the pearlitic CGI and high-Si CGI with the lowest and highest microhardness and Brinell hardness values showed the lowest and highest scratch resistances, respectively. However, no conclusive relationship was found between the hardness profile and friction response for the investigated CGI alloys. In scratch test, the ratio of tangential force to normal force is usually called as SCOF (scratch coefficient of friction) instead of COF (coefficient of friction) [44]. As shown in **Table 3**, the scratch coefficient of friction generally increased with an increase in the normal load for all investigated alloys [9]. It is obvious that no significant variation in SCOF exists for low normal load scratches, but applying high loads resulted in more significant variation in the SCOF values which did not correlate with micro-hardness profiles. It is worth pointing out that a more uniform SCOF (see **Table 3**) was observed for pearlitic CGI than the three developed ferritic CGI alloys.

Table 3. SCOF measured during the scratching of pearlitic, low-, medium-, and high-Si CGI alloys under applied constant normal loads ranging from 100 to 2000 mN.

Normal load (mN)	Pearlitic CGI	Low-Si CGI	Medium-Si CGI	High-Si CGI
100	0.19±0.011	0.12±0.012	0.12±0.001	0.12±0.003
200	0.20±0.006	0.16±0.004	0.14±0.004	0.14±0.003
400	0.21±0.003	0.18±0.003	0.17±0.002	0.16±0.002
600	0.22±0.017	0.19±0.003	0.19±0.006	0.18±0.002
800	0.23±0.005	0.21±0.022	0.20±0.003	0.19±0.001
1000	0.23±0.002	0.22±0.015	0.21±0.001	0.20±0.007
1200	0.24±0.024	0.24±0.005	0.21±0.001	0.22±0.008
1400	0.25±0.014	0.28±0.009	0.22±0.002	0.22±0.010
1600	0.27±0.003	0.31±0.022	0.26±0.016	0.24±0.011
1800	0.27±0.015	0.34±0.009	0.30±0.027	0.27±0.040
2000	0.28±0.018	0.39±0.033	0.36±0.033	0.33±0.022

Under low-load scratching conditions, i.e. below about 1000 mN, the scratching of pearlitic CGI showed higher SCOF values than the three developed ferritic CGI alloys, while no sizeable difference was observed between the three solution strengthened alloys with fully ferritic matrixes. For the three ferritic CGIs, a further increase in the applied load resulted in a notable and rapid increase in SCOF, while for the pearlitic CGI a smaller increase in SCOF was observed such that, over a load of 1600 mN, it remained constant with small fluctuations. In addition, under high scratch loads the low- and high-Si CGIs showed the minimum and maximum tangential forces and SCOF values, respectively (see **Table 3**). The lowest SCOF value was achieved by the high-Si CGI alloy, for which the scratch depth was lower than that of the low- and medium-Si alloys. In order to describe this behaviour in relation to the SCOF, a detailed microstructural analysis was conducted using observations of the scratched surfaces. The results of this are presented in **Section 3.2.2**.

3.2.2 Scratch feature characterisation

Fig. 7 (a)-(d) show SEM micrographs of the scratches produced under four different constant loads over the pearlitic, low-, medium-, and high-Si CGI alloys, respectively. It is possible to reasonably compare the scratch resistance of the fully ferritic Si solution strengthened alloys under various applied loads to that of the conventional pearlitic CGI looking into the **Fig. 7 (a)-(d)**. The amount of induced plastic deformation caused by the scratching is indicative of the scratch resistance of each individual matrix feature. As is apparent in **Fig. 7 (a)**, applying a load of 200 mN to the pearlitic CGI resulted in scratches with a shallow groove and very small deformation over the matrix, with no considerable pile-up formation aside from the groove. The produced microscratch had a depth, h , and width, W_2 , of approximately 0.37 μm and 18 μm , respectively. The pearlitic CGI showed a smooth change in micro-wear mechanisms, from mild to moderate micro-ploughing (characterised by the absence of wear particles) as a result of an increase in load from 100 to 2000 mN (see **Fig. 7 (a)**), which aligns well with the obtained SCOF results (see **Table 3**). Here, the increase in applied low load caused the SCOF to increase; a further increase to 2000 mN caused the SCOF to remain approximately constant during the subsequent sliding processes. This is obvious in **Fig. 7 (a)**, where only ploughing continued to be steadily high and a groove with ridges on both sides was formed.

However, a significant change from mild micro-ploughing to severe micro-wedge formation was observed for the other three ferritic alloys under a load of 2000 mN (see **Fig. 7 (b)-(d)**), particularly low-Si CGI. As can be seen in **Fig. 7 (b)**, under a load of 2000 mN the scratching of the low-Si CGI resulted in severe deformation and pile-up formation next to the grooves, as well as the pushing of a large volume of deformed material in front of the indenter. This was then ploughed to the sides or appeared as burr formation, clearly demonstrating the occurrence of severe micro-wedge formation

[46]. Under high load conditions, i.e. 2000 mN, high SCOF (see **Table 3**) and strong adhesion exacerbated the wear modes (see **Fig. 7 (b)**).

Studies performed by Abele et al. [4] and Heck et al. [45] illustrated the importance of the graphite morphology on wear mechanisms during the machining of cast iron materials. Investigations by Dawson et al. [3] and Berglund et al. [11] showed that CGI has significantly better machinability than spheroidal graphite iron (SGI) with a similar metallic matrix. On the micro-level, the improved machinability could be linked to some extent to the better lubricating performance of the compacted graphite particles during machining (i.e. micro-cutting during scratching) than that of the spheroidal ones, which could partially explain the higher friction in SGI than CGI alloys during machining. As can be seen in **Fig. 7 (d)**, for instance, the scratching of the high-Si alloy under a constant load of 1000 mN resulted in the fracturing, extrusion, and smearing of the compacted graphite particles in the path of the scratch, while the nodular graphite particles remained largely intact even though the scratches under 2000 mN were made very close by.

As shown in **Fig. 7 (a)**, an increase in the scratch load from 100 to 2000 mN produced more severe and deep scratches in the pearlitic CGI than in the three Si solution strengthened ones' (see **Fig. 7 (b)-(d)**), while the matrix deformation mechanism was principally controlled by an increase in the penetration depth of the indenter under low-load conditions (100 to 400 mN). However, a higher load (600 to 1000 mN) resulted in scratches that caused more considerable plastic deformation, without substantial pile-up formation. This behaviour indicates a common transition from a very smooth to a moderate micro-ploughing wear mode [46]. A severe micro-ploughing formation was observed as a result of a further increase in the normal load to 2000 mN, which resulted in significant matrix deformation and large pile-up formation on the edges of the microscratch grooves. It is worth mentioning that the occurrence of the stick-slip phenomenon was more severe, visible and significant for scratch loads greater than 1200 mN in all studied CGIs. This phenomenon was even more visible in recorded frictional force measurements. On the influence of strengthened matrix phase on stick-slip phenomenon of CGI it was observed that for low-Si CGI the matrix deformation is more dominant by the delamination of the matrix, visible on the bottom of the scratch grooves, while increase of Si content to high-level resulted in more stick-slip phenomenon in particular for high scratch loads as shown in **Fig. 7 (d)** [47].

Fig. 7. SEM microscratch micrographs taken under constant normal loads of 200, 600, 1000, and 2000 mN: (a) pearlitic, (b) low-Si, (c) medium-Si, and (d) high-Si solution strengthened CGI alloys. The scratch direction was from left to right.

The ferritic matrix that was solution strengthened with low-Si content showed another type of wear under high load scratching (compare the two scratches produced under a load of 2000 mN; **Fig. 7 (a)** and **(b)**). As can be observed in **Fig. 7 (b)**, many thin pile-ups (burr formation) at the middle and end of the groove were almost detached (loose wear debris).

It was observed that a 200 mN load was insufficient to produce even a noticeable scratch in the high-Si CGI (see **Fig. 7 (d)**). This is confirmed by the fact that almost no visible grooves were produced on the surface. However, increasing the normal load caused to form scratches, with considerable deformation. For scratches under 1000 mN, the measured scratch depth for low-, medium-, and high-Si CGI alloys showed a similar value and trend, in that scratch depth increased with an increase in the applied load. However (see **Fig. 9**), slightly different micro-mechanisms were involved in controlling the scratching (see **Fig. 7 (b)-(d)**).

The differences between the pearlitic and solution strengthened CGI alloys became more evident for higher applied normal loads. A closer examination of the grooves produced under various loading conditions showed a clear change in the scratch mode, from micro-ploughing to micro-wedge formation, as a result of hardness increase, which was caused by the solution-hardening mechanism (which is in agreement with the literature [48]). The effect of matrix hardness was more visible at high

scratch loads in that, for scratches performed under a scratch load of 2000 mN, low-Si CGI showed a very severe matrix deformation together with significant burr and pile-up formation, as can be seen in **Fig. 7 (a)**. However, for high-Si CGIs the impact of the load increasing was considerably lower (although severe deformation of the matrix did occur) due to the greater resistance to abrasion. Moreover, profound delamination and shear fracturing of the matrix was observed at the bottom of the grooves created under a scratch load of 2000 mN on the low-Si alloy, while no significant delamination was detected for pearlitic CGI. This behaviour could be associated with the brittle behaviour of solution strengthened ferritic CGI alloys, as investigated and discussed in **Section 3.2.2**.

Fig. 8. (a) Degree of penetration as a function of scratch load; (b) SCOF vs. degree of penetration (scratch contact width and depth values calculated based on the data recorded during scratching). The error bars indicate one standard deviation.

The transition between different abrasive wear modes can be interpreted using the degree of penetration measure. **Fig. 8 (a)** and **(b)** show D_p as a function of scratch load, and the correlation between the SCOF and degree of penetration, respectively, for the studied CGI alloys. As can be seen in **Fig. 8 (a)**, D_p value generally increased alongside the increase in scratch load, although it should be noted that under low-load conditions (below approximately 1000 mN), all four grades showed almost the same trend for an increase in D_p value. However, a more rapid increase was observed for the pearlitic CGI than the three developed ferritic ones when the scratch load increased above approximately 1000 mN. In agreement with the SEM images shown in **Fig. 7**, it is clear from D_p data presented in **Fig. 8 (a)** that wear became severe with increasing h/a . In addition, where very shallow ploughing occurred, D_p was so small as to be negligible, while for mild- and severe-ploughing and further wedge formation D_p was noticeable. In the case of the wedge-type wear of solution strengthened CGI alloys, under scratch loads of above approximately 1600 mN the adhesion between the indenter and the flat surface played an important role in the wedge-formation mechanism, such that it can be considered to be a severe wear phenomenon from the viewpoint of adhesive wear [17].

More interestingly, it is clear that an increase in D_p resulting from the scratch load caused the SCOF to increase for all of the CGI alloys (see **Fig. 8 (b)**). For scratch loads of under 1000 mN, pearlitic CGI showed higher SCOF values with regard to the degree of penetration; for scratch loads of over 1000 mN, a more profound increase in SCOF occurred for solution strengthened CGI than pearlitic CGI, in that a very small increase in the SCOF values for pearlitic CGI was observed as a result of an increase in the scratch load from 1000 to 2000 mN (see **Fig. 8 (b)**).

3.2.3 Recovered microscratch depth and width

Fig. 9 (a) and **(b)** show the microscratch width values W_1 and W_2 after plastic recovery, measured using the Alicona microscope, for the investigated CGI alloys. If we consider the CGI alloys to be viscoelastic plastic materials and know the indenter's geometry and scratch width, W_1 , the microscratch depth can be calculated using **Eq. (3)**. The obtained results are shown in **Fig. 9 (c)**. Note should be taken of the fact that the scratch depth after unloading can also be measured using post-scan topography measurement by passing the indenter along the scratch path by means of the used instrumented scratch test machine. Good agreement between the Alicona microscope measurements and the post-scan measurements (see **Fig. 9 (d)**) was observed for applied loads of lower than 1000 mN. However, the post-scan measurement results deviated notably from the Alicona measurements for high scratch load levels. A very rough groove with lots of delamination of the matrix, primarily for solution strengthened CGI alloys (as shown in **Fig. 7 (b)-(d)**), limited the usage of post-scan topography measurement for high-load scratch conditions. The W_1 data presented in **Fig. 9 (a)** shows that the residual groove width on the scratched surface increased with an increase in load for all of the investigated CGI alloys. However, no significant difference was observed between the three solution strengthened CGIs under low-load scratching conditions (see **Fig. 9 (a)**). The difference was more significant and evident under high-load conditions, with the maximum and minimum scratch widths obtained for low-Si and pearlitic CGIs, respectively.

Fig. 9. (a) W_1 and (b) W_2 , measured using scratch width with the Alicona microscope; (c) computed scratch depth (using W_1 data) after elastic recovery, and (d) measured scratch depth using post-scan topography for pearlitic, low-, medium-, and high-Si CGI alloys. The error bars indicate one standard deviation.

Both the matrix deformation behaviour and measured scratch depth strongly influence the microscratch (abrasion) behaviour of metallic materials [49]. In addition to the qualitative SEM scratch analysis (see **Fig. 7 (a)-(d)**), the quantitative scratch-depth measurements (**Fig. 9 (c)**) show the depth variation corresponding to the scratches created under various load levels. **Table 4** presents the fitted parameters for recovered scratch depth measured after scratching using Alicona microscope. As can be seen in **Fig. 9 (c)** and **Table 4**, for the pearlitic CGI material, the scratch depth followed a linear relationship (representing a very small a -coefficient value of 0.6×10^{-7}) with scratch applied load and increased significantly with the increasing scratch load, whereas for the solution strengthened CGIs the scratch depth increase was of the second order. Moreover, it is obvious from the fitted parameter presented in **Table 4** that the a -value decreased as Si content increased, from low through medium to high. The largest scratch depth was found for low-Si CGI, followed in descending order by medium-Si CGI, high-Si CGI, and pearlitic CGI.

To more specifically examine this behaviour, we need to compare the strain hardening capability of the studied CGI alloys. So that, this behaviour could be related to the highest strain hardening exponent and largest elastic recovery of the pearlitic matrix. As seen in **Fig. 6 (a)**, the pearlitic CGI showed the

maximum scratch depth during scratching for the applied loads ranges from 100 to 2000 mN while after elastic recovery two different behaviour are obvious for scratch loads of lower or above approximately 1000 mN. This is obvious not only from the SEM images, but also from the scratch depth measurements (see **Fig. 9 (c)**) that clearly a transition is observed at approximately 1000 mN scratch load with change in wear micro-mechanism from micro-ploughing to micro-wedge formation.

Table 4. Polynomial fitted parameters for depth of penetration corresponding to scratches produced under constant normal load on pearlitic, low-, medium-, and high-Si CGI alloys.

Materials	$y = ax^2 + bx + c$	R^2
Pearlitic CGI	$y = 0.6 \times 10^{-7}x^2 + 1.1 \times 10^{-3}x + 0.16$	0.99
Low-Si CGI	$y = 9 \times 10^{-7}x^2 + 0.2 \times 10^{-3}x + 0.17$	0.97
Medium-Si CGI	$y = 6 \times 10^{-7}x^2 + 0.5 \times 10^{-3}x + 0.13$	0.98
High-Si CGI	$y = 4 \times 10^{-7}x^2 + 0.6 \times 10^{-3}x + 0.07$	0.96

In more details, the scratch depth trend for scratches performed under loads of approximately 1000 mN, could be explained by the n_H , and K_L with regard to this fact that, the deepest scratches were found in the pearlitic CGI followed by the solution strengthened CGIs. The lower the K_L , the more elastic recovery and the higher n_H the higher strain hardening capability (see **Table 2**). Note that under low load conditions, the difference between the low-, medium-, and high-Si CGI alloys is very small, so that it is very difficult to draw a distinctive conclusion to correlated to the Ludwigson fitted parameters.

However, a different scenario was observed for higher scratch loads, over 1250 mN in particular. By comparing the tensile testing results (**Table 2**) and scratch depth measurements (**Fig. 9 (c)**), it is possible to conclude that for the scratch loads over 1250 mN, the scratch depth follows the same trend as the K_H and n_L translating that by decrease in K_H and increase in n_L values, the scratch depth increased. This conclusion, at least, is valid for the scratch loads between 1000 to 2000 mN where a large difference was observed between the pearlitic and solution strengthened CGIs. Interesting to note that, by decrease in K_H and increase in n_L the elastic recovery after scratching was decreased (see **Table 2**, **Fig. 6 (a)** and **Fig. 9 (c)**).

3.2.4 Scratch hardness

It is well known that the deformation behaviour of a material during scratching is dependent on the material's local mechanical properties. However, scratch resistance is not an intrinsic material property, and changes in response to the tribo-system changes in multi-parameters such as applied load [50]. Hence, it is very important to define which parameter is of interest in order to investigate the impact of mechanical properties on scratch hardness. In this study, scratch width, W_2 , values measured at five positions along the length of each individual scratch were selected and used for scratch hardness calculations using **Eq. (4)** to quantify the influence of applied load on scratch resistance. The mean values of all positions are plotted as a function of scratch applied under normal loads for the

investigated materials in **Fig. 10 (a)** and **(b)**. As can be seen in **Fig. 10 (a)**, scratch hardness values increased for pearlitic CGI as the applied load increased. For Si solution strengthened CGIs (see **Fig. 10 (b)**), however, scratch hardness initially increased with an increasing applied load, from 100 to 600 mN, but it then decreased with further increase in applied load. From the large one standard deviation values, it can be concluded that the *SHp* value strongly varies under different loading conditions.

Fig. 10. Scratch hardness of (a) pearlitic and (b) low-, medium-, and high-Si CGI alloys, computed based on the data obtained from the scratch widths after elastic recovery.

It is interesting to note that, under an applied load of approximately 1000 mN, pearlitic CGI showed less scratch resistance than ferritic solution strengthened CGI. However, a clear change was observed for loads greater than 1000 mN, where the scratch resistance of pearlitic CGI was higher than that of even the high-Si CGI. This behaviour could be related to the considerable deformation hardening phenomenon that occurs in the matrix during scratching, whereby the strength of a ductile metal increases when it is plastically deformed. As observed for 2000 mN loads, the results revealed that the work hardening of metallic materials plays a significant role in the development of pile-up in particular, and thus greatly affects the active micro-mechanisms.

Fig. 11. The average scratch hardness values of pearlitic, low-, medium-, and high-Si CGI alloys.

Fig. 11 shows the average calculated scratch hardness values for pearlitic, low-, medium-, and high-Si CGI alloys using W_2 . The observed differences in the scratch hardness values for the pearlitic and

solution strengthened CGI alloys mean that the ratio F_N/W_2 (see **Eq. (4)**) was affected by the change in the matrix and strengthening through the addition of Si. As can be seen, the scratch hardness data for the solution strengthened CGI materials exhibited an increasing trend as a function of Si level in the matrix, in that an increase in Si content resulted in improved scratch hardness and scratch resistance. The resulting scratch hardness was lowest for the low-Si CGI alloy. A greater scratch resistance means that, under similar loading conditions, less material is removed by each scratch. This is perhaps the reason why pearlitic CGI is more difficult to machine than Si solution strengthened ferritic CGIs.

4 Conclusions

Based on the tensile testing, microhardness measurements, microscratch testing using a sphero-conical diamond indenter performed under various constant loads, and SEM observations – all using a typical pearlitic CGI and three developed ferritic Si solution strengthened CGIs – the following conclusions can be drawn:

- The scratch resistances of the ferritic solution strengthened CGIs increased with increasing Si content, and high-Si CGI showed the greatest resistance to scratching.
- An increase in scratch load resulted in an increase in the depth and degree of penetration for all of the investigated CGIs.
- The influence of an increase in load on the depth of penetration was most profound for the low-Si CGI and least noticeable for the high-Si CGI. This could be related to high-Si CGI having the highest microhardness and Brinell hardness among the investigated CGI alloys.
- As the scratch load increased, the pearlitic CGI showed a transition from mild to moderate ploughing, while the three ferritic CGI alloys, particularly low-Si CGI, showed a transition from a moderate micro-ploughing through wedge formation to a severe wedge formation.
- The scratch behaviour, scratch hardness and SEM observations are in good agreements with the tensile fracture surface analyses, which showed that the solution-strengthening of CGI using Si resulted in brittle failure mode during scratching.

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6 References

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Highlights

- Effect of scratch normal load on matrix deformation and scratch hardness were discussed.
- Microscratch behaviour, and scratch resistance of pearlitic and ferritic solution-strengthened ferritic CGs were examined.
- Scratch micro-mechanisms were correlated with microstructural and mechanical properties.
- The microscratch depth, width, frictional force, and friction coefficient were measured and compared.