Influence of plasma remelting conditions on quantitative graphite dissolution and modified surface characteristics in 500-12 ductile iron

Falalu Rabiu Jahun¹,², Zhang Benhua¹,³, Song Yuqiu¹, Wang Ruili¹ and Liu Siyao¹

¹ College of Engineering, Shenyang Agricultural University, Shenyang, 110866, People’s Republic of China
² Department of Agricultural and Environmental Engineering, Faculty of Engineering, Bayero University, Kano 999062, Nigeria
³ Author to whom any correspondence should be addressed.

E-mail: falalur@yahoo.com, zbb@syau.edu.cn, songyuqiusyau@sina.com, wangruili@syau.edu.cn and lsiyao641@163.com

Keywords: plasma remelting condition, multi-pass scan, quantitative graphite dissolution, surface modification, wear resistance, 500-12 ductile iron

Abstract

In this study, the influence of multi-pass plasma remelting conditions on quantitative graphite nodules dissolution, surface hardening and wear resistance of 500-12 ductile iron was evaluated. Surface remelting was carried out at various levels of heat inputs using high-temperature plasma beams. The graphite dissolution was uniquely quantified via image analysis approach, and then the remelted surface layer was characterized for phase transformation, microstructure, hardness and wear resistance. The remelting process parameters (arc current and scanning speed) showed significant (p < 0.01) influence, both on graphite nodules dissolution and surface hardening. Consequently, microstructure of the treated layer was modified and the surface exhibited a significant improvement in hardness and wear resistance. In addition, treatment conditions having slower scanning speed showed better surface modification. Therefore, the plasma remelting enhanced surface hardness and dry sliding wear resistance of 500-12 ferricitic ductile cast iron, which makes it suitable for application in a severe wear condition.

1. Introduction

In recent times, ductile cast iron (DCI), also called nodular cast iron (NCI) has been used extensively in a number of fields such as automobile, mining, weaponry and agricultural machinery industries [1, 2] due to its wide range of mechanical properties and low production cost compared to steel [1]. It is well known that machine parts such as crankshafts, camshafts, gears, harvester’s shaft, plowshares, and weapons have been produced from different DCI grades [1–6]. The type of metallic matrix in ductile iron determines its strength whereas the graphite nodules serve as crack arresters [7]. Thus DCI, depending on its matrix type, is characterized by good ductility and strength. However, the low hardness and poor wear resistance in its untreated (plain ductile iron) state have limited its application, especially in adverse harsh conditions like earth moving [6, 8] and sliding guides of machine parts. Therefore, conventional bulk material heat treatments such as austempering, quenching and partitioning (Q&P) [8–10] and in recent times, surface heat treatment using concentrated heat sources, like lasers and plasma beams [4, 11–17] have been employed to improve hardness and wear properties of the ductile iron parts. The surface heat treatment method has been confirmed to be more appropriate because it gives good combination of surface hardness and tough core [18], suitable for wear condition. Also, the method is flexible and cost effective [4, 17]. Several surface treatment techniques have consequently been used under different treatment conditions to modify the surface layer of DCI parts. Among these techniques, plasma transferred arc (PTA) technique has even more advantages of superior metallurgical bond formation, low operation cost, ease of
operation \[13\], high heating efficiency \[19\], minimum dimensional change and selective localized hardening tendency \[20\].

Generally, PTA heating is performed in a single scan pass or multiple scanning. The multiple scanning involves scanning the selected regions of the workpiece with plasma beam, repeatedly and continuously for the specified number of travels \[21\]. Multi-pass scanning has been reported \[6, 21\] to have advantages of reducing temperature gradient between the heated layer and hardened cast iron, and inhibiting cracks formation. Hence, it was adopted in this work.

The presence of soft graphite nodules in ductile iron has been reported by other authors \[4, 22, 23\] to be the most significant factor contributing to its low hardness and poor wear resistance. Consequently, PTA surface remelting was performed primarily to dissolve the surface graphite nodules in ductile irons \[4, 12, 22, 24\]. This significantly increased the hardness and wear resistance of the surface layer \[4, 25, 26\], depending on the degree of dissolution of the graphite nodules. The degree of graphite dissolution was found \[4, 12\] to be influenced by the remelting conditions. For instance, other authors \[4, 12, 17, 27\] achieved complete and near-complete graphite dissolution when ferritic ductile iron was remelted using high energy density and longer interaction time. Olexiy et al \[12\] found the graphite nodules dissolution to increase with increasing heat input. Also, they found that the graphite nodules dissolve quickly by heating the ductile iron above the eutectoid transformation temperature. Hua-tang et al \[4\] found that slower scanning speed enhanced the graphite diffusion. This was due to the longer interaction time between the plasma torches with substrate surface. The authors also achieved a near-complete nodules dissolution. Moreover, Karamis et al \[17\] observed that almost all the near surface graphite nodules were removed when ductile iron was remelted using high temperature tungsten inert gas (TIG) heat source. However, none of the authors has ascertained the effect of remelting conditions on the quantitative graphite nodules dissolution. To the authors’ knowledge, relationship between the remelting process variables and graphite dissolution has not yet been established. Furthermore, the dry sliding wear behavior of plasma remelted 500-12 ductile cast iron has not been analyzed in detail. Therefore, in this work, image analysis approach was used to quantitatively evaluate the graphite nodules dissolution. The interactive effect of remelting parameters on graphite nodules dissolution and remelted surface hardness was evaluated using statistical analysis (ANOVA). In addition, the dry sliding wear behavior of the remelted surface layer was investigated. The aim of the work is to establish optimum PTA process condition for achieving high graphite dissolution, enhanced surface hardness and wear performance in the 500-12 ferritic ductile iron.

2. Materials and methods

2.1. Material

The material used in this study was a plain ductile cast iron. The microstructure of the substrate has graphite nodules surrounded mainly by ferrite phase and few pearlite phases (figure 1). The chemical composition and mechanical properties of the DCI base material are presented in table 1. Samples were cut from the cast blocks into cubic shapes with a thickness of 10 mm (10 mm² cross-section). The surfaces of the samples were ground progressively using #320 to #600 abrasive papers to remove surface oxides and then polished with Al₂O₃ suspension to obtain mirror-like surface.
2.2. Determination of graphite nodule morphological features

The graphite nodule morphological features (spherical nodule count, circularity, and nodularity) were characterized using image analysis. This approach was used in order to establish the total nodule count in the samples before and after the plasma treatment. The image processing was performed using algorithms programmed with HALCON12.0 software [28]. First, the metallographic images of the polished samples were obtained along the track length, tiled [7] into 1 mm² (figure 2), using a high-resolution optical microscope, Keyence-VHX 5000 model at a magnification of x200. The image processing steps employed in this work consist mainly of separating the nodules from the background through image noise filtering, image binarization [29], and nodule extraction. Reconstruction transformation filtering method [29] based on Fourier transformation (since the noise manifest itself as a high frequency fluctuation of the gray values in the image) was used to suppress pulse noise in the image. The segmentation algorithm based on circularity (figure 3) was employed to separate the nodules in the binarized image from the background. Therefore, from the binary images shown in figures 4 (aii) and 5 (bii), the required nodule morphological parameters could be estimated easily. The segmented nodule contour is then fit into a circle (by minimizing the sum of square error) using a Huber algorithm [28], which enhances the robustness of square error loss function to discrete points. Relevant shape parameters values for each nodule are computed and obtained from the program output. The segmentation process output with sampled nodules shape parameters values is shown in figures 4 and 5.

The obtained nodule shape parameters from the segmentation process output for the entire image of the sampled tile (9th tile at condition C1 S3), referred to in figures 4(aii) and 5(bii), are: before plasma remelting: mean circularity = 0.755 53, mean diameter = 35. 4551 μm, nodularity = 100%, and spherical nodule count = 59; and then after plasma remelting: mean circularity = 0. 66 713, mean diameter = 33. 6637 μm, nodularity = 71. 4285% and spherical nodule count = 5.

Circularity, $C = \frac{A}{\pi D_{\text{max}}^2}$

Where $A$, is the area of the elemental domain and $D_{\text{max}}$ is maximum distance from the domain boundary point to center point.

The fitting loss function is expressed as [28]:

$$L_\delta(y, \hat{f}(x)) = \frac{1}{2}(y - \hat{f}(x))^2$$

Where $\delta$, is the residual error, $y$ measured value ($x$), the predicted value and $L_\delta(y, \hat{f}(x))$ is the fitting loss. And accordingly, the enhanced square error function (Huber function) is given as [28]:

$$L_\delta(y, \hat{f}(x)) = \frac{1}{2}(y - \hat{f}(x))^2, \quad |y - \hat{f}(x)| \leq \delta$$

$$y - \hat{f}(x) \delta + \frac{1}{2}\delta^2, \quad |y - \hat{f}(x)| > \delta$$

Table 1. The chemical composition (w%) and mechanical properties of the substrate ductile iron.

| Chemical composition | Mechanical properties |
|----------------------|-----------------------|
| C | Si | Mn | S | P | Mg | Fe | UTS (N mm$^{-2}$) | E (%) | Microhardness (HV$_{0.5}$) | Impact strength (J) |
| 3.85 | 2.75 | 0.15 | 0.048 | 0.05 | 0.003 | Bal. | 500 | 12 | 219 | 13 |

UTS = ultimate tensile strength, E = percentage elongation.
2.3. Experimental design

Full factorial design of experiment was used to generate the planned order for performing the plasma surface hardening. The design has three levels each of scanning speed \((S)\) and transferred arc current \((C)\). The factor levels of the parameters used are \(S_1\) (100 mm min\(^{-1}\)), \(S_2\) (200 mm min\(^{-1}\)), \(S_3\) (150 mm min\(^{-1}\)), \(C_1\) (45 A), \(C_2\) (40 A), and \(C_3\) (35 A). The parameters levels were set after initial trial runs and based on literature [30] and the device potential. This was to ensure that workable factors levels that are not too high to damage the surface and adequate for the required surface hardening, were used. Accordingly, the design has 9 treatments, and thus a
total of 9 experiments were performed and three replicated data were analyzed using analysis of variance (ANOVA).

2.4. Experimental procedure
The plasma transferred arc treatment was carried out using PTA machine (DML-V02BD model, Duomo Co. Ltd, Shanghai), with rated power of 6 KVA and 120 A maximum current. Argon was used at 7 bar, both as shielding and plasma gas. Details of the experimental parameters are shown in table 2. The plasma remelting was performed in multiple (three) scans via a six degree of freedom, automatic control plasma torch motion. Experiments were run according to the factor levels setting and combinations indicated above. The run for each specimen was carried out along the center-line throughout the specimen’s full length in the longitudinal direction. The treated samples were allowed to quench in the air at room temperature of 10°C–12°C (winter period). All experiments were conducted during the daytime. After the experiments, specimens were ground and polished again to remove oxides and obtain good surface finish for subsequent characterization. Changes in the specimen dimensions due to thermal distortions were measured after the plasma treatment.

2.5. Determination of quantity of dissolved graphite nodules
The quantitative graphite nodules dissolution was determined in this work based on the percentage of dissolved graphite (%DG) in the melt, which was calculated using equations (4) and (6). Prior to this, the graphite nodule quantitative morphological parameters of the treated samples were also determined based on the previously described image analysis procedure. Consequently, mean parameters values, such as spherical nodule count, nodularity and nodule size were calculated for all the samples. It is important to note that dimensional changes

### Table 2. PTA treatment experimental parameters.

| Parameters                               | Values                  |
|------------------------------------------|-------------------------|
| Transferred arc current/A                | 45, 40, 35              |
| Scanning speed/mm. min⁻¹                 | 100, 150, 200           |
| Plasma gas flow rate (Ar)/L. min⁻¹       | 0.7                     |
| Diameter of torch/mm                     | 4                       |
| Gun height/mm                            | 3.8                     |
| Tungsten electrode diameter/mm           | 2.36                    |
| Travel distance/mm                       | 20                      |
| Number of passes/no.                     | 3                       |

Figure 5. Characteristic segmented images of sampled 1 mm² tile (9th tile treated with condition of C₁ S₃) after plasma remelting with circularity threshold of 0.6: showing (b₁) original image and (b₁i) segmented nodules. Remark: A = individual nodule pixels, D = individual nodule diameter, C = individual nodule circularity and only nodules with circularity equal or greater than the threshold of 0.6 are segmented.
in the samples due to thermal distortions after the heating were adequately accounted for while acquiring the images for the second time.

\[
DG_i(\%) = \frac{NC_{bi} - NC_{ai}}{NC_{bi}} \times 100
\]

(4)

Where \( DG_i(\%) \) is the percent dissolved graphite of \( i \)th tile and \( NC_{bi} \) (no. mm\(^{-2}\)) is the total nodule count of \( i \)th tile before remelting, \( NC_{ai} \) (no. mm\(^{-2}\)) is total nodule count of \( i \)th tile after the remelting. The total nodule count was calculated from the ratio of nodularity to spherical nodule count using equation (5) \(^{[29]}\).

\[
Total \ nodule \ (N) = \frac{Spherical \ nodule \ count \ (NC)}{Nodularity(\%)}
\]

(5)

\[
DG(\%) = \frac{\sum_{i=1}^{n} DG_i(\%)}{n}
\]

(6)

Where \( DG \) is the average of dissolved graphite per specimen per treatment, \( n \) is the \( n \)th tile and \( N \), the number of tiles per sample.

### 2.6. Microstructure characterization

The microstructural investigations were carried out after plasma treatment on the transverse cross-sections of the samples to study the phase transformations and determine remelted zone thickness. Specimens were prepared for the microscopic examinations according to standard procedure in ASTM 2001\(^{[31]}\). The polished surfaces were etched with 5% Nital to reveal the grain structures. After the etching, optical microscope (Keyence - VHX5000) was used to examine the microstructures and determine the remelted zone thickness. X-ray diffraction (XRD) analysis was also performed according to ASTM E975-84 standard \(^{[32]}\) using Rigaku D/Max 2500PC, Tokyo, Japan diffractometer. Cu–Kα radiation was used in the 2θ scan from 20 to 90 degrees and diffraction patterns of the remelted layers were used to identify the types of phases present.

### 2.7. Hardness characterization

The microhardness tests were first performed on the unsectioned sample using Vickers microhardness tester (H XD-1000TMC/LCD model) at a constant load of 500 g and 15 s indentation time according to ASTM E384 standard \(^{[33]}\). The hardness was measured along the remelt surface center-line at 1 mm intervals throughout the track length. Variations in the hardness distribution along the track were estimated from the coefficient of variation (CV) index using equation (7). Consequently, the surface hardness distribution uniformity was established from the coefficient of variation values, a low CV value signifies uniform hardness distribution.

\[
CV = \frac{\sigma}{\mu}
\]

(7)

Where \( \sigma \) is the standard deviation of the hardness distribution and \( \mu \) is the mean hardness distribution along the melt track.

The specimens were then cut through the middle in a direction perpendicular to the track. The cross-sectioned surfaces were ground and polished for the hardness characterization. Hardness measurement along the case depth direction was done at an average distance of 0.11 mm from the surface using a Vickers microhardness test procedure as previously described.

### 2.8. Analysis of data

To assess the effect of plasma arc current and scanning speed on graphite dissolution, case layer hardness, and hardness uniformity, the obtained data was statistically analyzed using ANOVA. The mean differences were also evaluated for significance using Tukey’s -b test. All the analysis was accomplished by IBM-SPSS 19.0 version software and using MS Excel.

### 2.9. Dry sliding wear tests

The dry sliding wear behavior of the modified DCI surface was determined in the remelted region as per ASTM G77-05 standard \(^{[34]}\). Sample with the highest achieved value of surface hardness was selected for the sliding tests, the specimen dimension was 19 × 12 × 12 mm\(^3\). A block on ring tribometer (MRH-3 model, Jinan Yihua Tribology Testing Technology Co. Ltd, China) was used for the tests. The block was held against a hardened alloy steel ring (counter surface) of dimension 50 × 13 × 5 mm\(^3\), having hardness 47 HRC (Rockwell hardness on C scale). Chemical composition (weight percent) of the ring material is: (C) 0.42%–0.50%; (Si) 0.17%–0.37%; (Mn) 0.5%–0.8%; Cr < 0.25%; Ni < 0.30% and Cu < 0.25. Prior to the tests, both block sample and the counter surface were polished with abrasive papers of 600 and 1000 grit sizes. The specimens were then weighed using digital weighing balance having accuracy of 0.001 g, and the experiment was conducted at ambient environmental conditions. Applied normal forces of 20 N and 90 N were selected as the minimum and maximum loads respectively, which are also within the range of those used by other researchers \(^{[18, 26]}\), for the
3.1. Effect of treatment parameters on quantitative graphite dissolution

The effect of various treatment parameters on the percentage graphite dissolution is shown in Table 3. It was found that the treatment parameters significantly influenced the graphite dissolution. Among the parameters tested, the scanning speed and arc current were found to have the most significant effect on graphite dissolution. At higher scanning speeds, the amount of graphite dissolution increased, which was associated with lower heat accumulation due to the increased sliding speed.

3.2. Results and discussion

3.2.1. Effect of treatment parameters on quantitative graphite dissolution

The influence of PTA remelting on percentage graphite dissolution is depicted in Table 3 and shows the mean % DG to vary with the treatment conditions. The trend shows more increase of graphite dissolution at slower scanning speeds, which facilitates the diffusion and dissolution of graphite into the melt. Accordingly, the results showed that all samples treated using the same lowest speed (100 mm min⁻¹) relatively displayed high percentages of graphite dissolution. In contrast, samples treated using the same highest scan speed (300 mm min⁻¹) were found to have the lowest % DG. Regarding the arc current influence on % DG, the results showed no obvious influence, since even at the highest current of 45 A, a very low % DG was obtained, when used together with the highest value of scanning speed. This is because the high arc current value was not enough to compensate for the high value of scanning speed used. In addition, a linear relationship between the % DG and scanning speed exists (figure 6), where it can be seen that the % DG decreased continuously as the scanning speed increased. This indicates lower graphite dissolution at higher scanning speed and is associated with low heat accumulation because of the shorter plasma beam interaction with the substrate, which in turn caused less graphite melting. Further, the relationship between % DG and remelt layer hardness is shown in figure 7. A polynomial relationship was established, and it gives a good function relation with coefficient of determination, \( R^2 = 0.92 \). The hardness was
seen to increase steadily with an increase in % DG at the beginning, and then becomes much steeper as more graphite dissolved. This explains that with more graphite dissolving into austenite, the negative effect of graphite nodules was eliminated and the surface became expectedly harder, since the re-formation of more graphite was suppressed in favor of the eutectic hard carbide. The results of statistical analysis (ANOVA) in table 3 indicate significant \( p < 0.01 \) influence of the combined process parameters on % DG for all the treatments combinations. This implies that the % DG obtained for each of the parameter’s combinations were not the same. Also, the LSD results showed that there is a significant difference \( (P = 0.05 \text{ level}) \) in the mean % DG of all treatments and the mean values were found to increase both with decrease in scanning speed and increase in arc current.

### 3.2. Effect of treatment parameters on microstructure

The metallographic examination of treated samples shows obvious microstructural change due to the effect of treatment conditions. Obvious changes were observed in samples treated using scanning speed in the range of 100 to 150 mm min \(^{-1}\). On the other hand, there was no significant microstructural change in the samples treated at the highest scanning speed of 200 mm min \(^{-1}\), regardless of the arc current value. The significant effect of lower scanning speeds on the microstructural change observed could be due to higher thermal accumulation associated with the prolong interaction time. Therefore, for samples that showed appreciable microstructural changes, three distinct zones were revealed: remelt zone (RZ), heat affected zone (HAZ) and the substrate zone (SUB). This is consistent with the observations of other researchers [4, 11, 22, 36, 37]. For the samples treated with lowest scanning speed value of 100 mm min \(^{-1}\), the remelt zone thickness was observed to vary between 0.395 mm to 0.996 mm (table 3), and the maximum thickness of 0.996 mm was obtained with treatment condition: 40A and 100 mm min \(^{-1}\). A closely similar result was obtained by Hua-tang et al [4], who studied the effect of scanning speed on the microstructure of nodular cast iron during PTA remelting. They obtained a depth of melt zone of 0.93 mm for a treatment condition: 90 A and 300 mm min \(^{-1}\). Moreso, it is seen in figure 8 that the remelted zone is nearly nodule-free, suggesting that substantial graphite dissolution has taken place, and also emphasizes the effect of higher energy density associated with the slower scanning speed.

The phase transformation in all the observed PTA remelted zones was characterized mainly by the formation of martensite, retained austenite, eutectic carbides, and few undissolved graphites, as similarly reported by Ishida [11], who investigated the microstructural changes in the PTA-melt boundary area of nodular cast iron. The martensite phase is formed in the solid state zone due to rapid cooling rate and the dissolution of carbon from the graphite nodule into the austenite matrix. The carbon-enriched austenite, which is due to the dissolution and diffusion of graphite nodules leads to both decrease in martensite start transformation.
temperature and finish transformation temperature, as a consequence, untransformed austenite is retain as residual at room temperature. For the eutectic carbide phase, this is formed at the melt zone due to fast cooling rate and the absence of effective graphite nuclei during the eutectic transformation. Moreover, treatment conditions having the lowest scanning speed gave coarse structure in the remelted zones with large primary dendrite and thick cementite plates, like the one seen in figure 8(ii). The coarse structure could be due to the lower cooling rate, which was associated with the slower scanning speed, and in this sense, more time for the transformed austenite to coarsen. On the other hand, the HAZ, which experienced quenching, and therefore a substantial amount of austenite transforming into high carbon martensite, is evident by the dark martensitic phase seen to have spread across the layer (figure 8(i)). Also undissolved graphite, ledeburite and ferrite were observed, with evidence carbide surrounding the graphite.

The x-ray diffraction patterns shown in figure 9 confirmed the microstructural changes. Comparison of the peak position in ferrite for the untreated (base material) and PTA treated samples showed that the ferrite iron dominates in the base material. This is reasonable since no surface heating has taken place for the untreated sample, and the material was already characterized as ferritic. In contrast, the treated surfaces, with exception to samples treated at the highest speed, exhibit strong peaks of cementite and martensite. The surface treated at 200 mm min\(^{-1}\) which was also the highest scanning speed shows peaks similar to those of the base material, signifying that no phase transformation has taken place even after the treatment. This confirms our earlier finding that samples treated at the scanning speed of 200 mm min\(^{-1}\) do not show obvious microstructural change. Furthermore, comparing the diffraction patterns for samples treated using scanning speeds of 100 and 150 mm min\(^{-1}\), it was observed that more peaks of martensite were formed in the former, whereas cementite peaks dominate the sample treated at 150 mm min\(^{-1}\). This observation could be explained by the difference in solidification rates in the two samples; the slower scanning speed of 100 mm min\(^{-1}\) resulted in lower cooling rate, which implies lower solidification rate and more time available for substantiating the amount of austenite transformation into martensite \cite{4,38}. On the other hand, the relative higher scanning speed of 150 mm min\(^{-1}\)
would lead to higher solidification rate, which suggest higher cooling rate and reduced diffusion rate. This, therefore, would facilitate more precipitation of cementite crystal both during non-equilibrium and eutectic solidifications.

3.3. Effect of treatment parameters on hardness
The hardness distribution across the hardened zone for the different parameter settings are shown in table 3. The microhardness measurement was made in the surface zone above the thickness of 1.1 mm. The hardness was seen to improve significantly due to the plasma remelting, this could be associated to the microstructural modifications in the remelted layer. The previously observed cementite and martensite phases in the modified microstructure are known to significantly enhance the hardness of the remelted layers. The hardness vary with the treatment conditions, and the scanning speed was observed to have more influence on the hardness values. An appreciable increase in the hardness was observed in samples treated at scanning speed range of 100 to 150 mm min$^{-1}$, which were also those that showed the most graphite dissolution. However, hardness increase at a scanning speed of 200 mm min$^{-1}$, which exhibited the least graphite dissolution was very negligible, even at the highest value of arc current. The maximum hardness of 719.1 HV$_{0.5}$ was observed for a sample at processing condition; 45 A/150 mm min$^{-1}$, which was approximately 3.3 times greater than the substrate hardness (219 HV$_{0.5}$). Hua-tang et al [4] reported to have obtained their maximum hardness as 3.1 times greater than the substrate hardness when ferritic nodular iron was remelted at condition; 90 A/300 mm min$^{-1}$, thus showing a good agreement with the finding in the present study. Also, Xiu et al [22] found the PTA melted surface microhardness of ductile iron to increase from 315 HV$_{0.1}$ to 933 HV$_{0.1}$, when treated with an arc current of 55 A, which is about 2.97 times more than the substrate hardness. This is also very consistent with the result in this work. The significant improvement in hardness for samples treated at low scanning speed is related to the formation of cementite and martensite phases in the samples, as observed in the XRD patterns. Regarding the combined parameters effect, the statistical analysis results in table 3 shows that all the treatment combinations had a significant effect ($p < 0.01$) on the surface layer hardness, implying that the sample hardness values obtained for each of the parameters combinations were not the same. Further, the LSD test results show that there is a significant difference (at $p = 0.05$ level) in the mean hardness of all treatments and were seen to increase with a decrease in scan speed, and also increase with arc current. The microhardness as a function of case depth for different parameter settings is presented in figure 10. However, only samples that showed significant hardness variation with the case depth are presented. Therefore, microhardness distribution profiles in depth direction show an approximately uniform trend across the remelt and heat affected zones. The profiles showed microhardness decreasing with the penetration depth and then level off at the initial hardness of the substrate, a similar trend was reported by [4, 11, 13] who stated that phase transformation and grain refinement due to localized remelting are responsible to the increase in hardness. The significant increase in hardness at the remelted surface could be attributed to the grain refinement and the absence of graphite, together with the formation of hard cementite phase in the iron-carbon eutectic. Hardness improvement observed at the heat affected zone could be attributed to the presence of austenite transformed, martensite phase. Furthermore, the hardness penetration depth across the samples was seen to range between 0.65 and 0.90 mm in the remelted zone, and the sample with a treatment at 40 A/100 mm min$^{-1}$ displayed the highest depth of 0.9 mm.
The hardness uniformity determines how consistent the hardness is distributed along the remelted layer track and in this work, it was characterized by the hardness distribution coefficient of variation (CV). A low value of CV index signifies a uniform hardness distribution, and in contrast, high CV value indicates non-uniform distribution. The effect of process conditions on the hardness uniformity is presented in table 3, where the mean CV was seen to vary among the treatment conditions. Considering the single parameter effect, the CV was seen to decrease with increasing scanning speed, which means that, samples treated at lower scanning speeds give better hardness uniformity. On the other hand, the trend on the effect of arc current on the hardness uniformity is not clearly defined, since the variation coefficient was seen to fluctuate with arc current. The excellent hardness uniformity exhibited in samples treated at lowest scanning speed (100 mm min⁻¹) could be due to the high %DG value observed earlier in these samples, since a homogenous and fine structure is usually obtain when more graphite carbon dissolved into the matrix phase of ductile cast iron [39].

The statistical analysis in table 3 depicts the combined parameters effect on hardness uniformity along the treated surface layer. The results show that all the parameter settings had a significant effect (p < 0.01) on the surface layer hardness uniformity. That is, the sample CV values obtained for each of the parameters combinations are not the same. Further, the LSD test results show that there is a significant difference (at p = 0.05 level) in the mean CV of all treatments and were seen to increase with scanning speed, but the change with arc current was fluctuating.

3.4. Wear resistance and worn surface morphology

The resulting wear characteristics based on weight loss and wear rate are presented in figures 11(a) and (b). The results showed both the weight loss and corresponding wear rate to decrease significantly due to the PTA remelting. Weight loss in the treated sample was reduced by 86.5% compared to the untreated substrate. The wear rate also was 7.38 times less. This result is in agreement with the findings of Hua-tang [4], who reported the weight loss and wear rate of PTA remelted ferritic DCI during sliding wear test to be respectively 90.7% and 9.76 times less than that of substrate. The improvement in wear resistance could be explained by the enhanced surface hardness which was attributed to the grain refinement and formation of hard eutectic carbide and martensite phases in the remelted surface layer. Consequently, this makes the remelted surface difficult to be deformed and worn out. For the friction coefficient, the untreated sample exhibited higher friction coefficient (0.390 55) compared to the plasma treated, having friction value of 0.234 11. The lower friction coefficient for the treated sample also suggest a better wear resistance due to the enhanced surface layer formed by the plasma remelting. Considering the effect of applied load, the wear resistance was seen to decrease with increasing applied normal load (figure 11). This could be attributed to the low load - bearing capacity of the material which could result to severe plastic deformation as the applied load increases. Also, at higher loads more heat due to friction is generated under the dry sliding condition, this reduce the material strength and accelerate the wear damage.
These findings are similar to that reported by Ayday and Durma [25] who studied the sliding wear performance of ductile iron after electrolytic plasma hardening. Gadag et al [40] also reported the increase in wear with applied load, while investigated the dry sliding wear behavior of laser-treated ductile iron. Rashid et al [41], who studied dry sliding wear behavior of untreated and treated sugar palm fiber filled phenolic composites, reported the increased wear rate with increased applied normal load. On comparison, the wear resistance of the treated specimen under the highest normal load of 90 N is 2.4 times less than that of the one tested at the lowest loading condition. While for the untreated substrate, the wear resistance even at the minimum load of 20 N is 3.07 times lower than that of the PTA treated specimen tested under the maximum load of 90 N. This emphasizes the significant effect of surface remelting in ductile iron on improving the wear resistance. Wear tracks of the worn surfaces were examined using scanning electron microscopy (SEM) analysis. The micrographs of SEM, together with EDS of the worn surfaces for the treated and untreated samples are shown in figures 12 and 13. The worn surface of the plasma treated specimen shows presence of shallow grooves, adhesion and some oxides debris, indicating mild adhesive and oxidative wear mechanisms. The presence of oxides, which was due to heat generated under the applied normal load, resulted in oxides film forming between the tribo-pair surfaces. The oxide film acted as lubricant [25] and protect the treated surface from severe wear, consequently reduced the wear rate significantly. The EDS result testifies to the formation of oxides layer and also confirmed the adhesion of tribo-pair contact surfaces, since presence of chromium (counter surface element) is manifested. On the other hand, the worn surface of the untreated ferritic DCI shows presence of wide and relatively deep abrasion grooves which could be due to high penetration by the much harder counter surface asperity. Also, large amount of material was seen to have been dislodged from the surface together with delaminated craters as a result of the severe plastic deformation. Oxides debris and some adhesion particles were also evident on the worn surface layer as confirmed from the presence of oxide and counter surface element (copper) in the EDS results. The wear mechanism is thus characterized by abrasive, delamination, oxidative and severe adhesive wear.

4. Conclusions

The influence of remelting condition on graphite nodules dissolution and surface hardening was investigated for 500–12 ductile cast iron. The combined effect of the process parameters proved promising in achieving adequate graphite dissolution and improved surface hardness, particularly with the combination of lower scanning speed and high arc current. The graphite dissolution was particularly seen to increase linearly with the decrease in scanning speed. The presence of remelted zone was only evident in samples treated at a scanning speed range of 100 to 150 mm min \(^{-1}\), and maximum remelted zone thickness of 996 \(\mu\)m was obtained. The surface hardening was observed to vary with the graphite dissolution, and polynomial relationship was established. The hardness increases to maximum, after which it start decreasing with any further increase in graphite dissolution. The surface hardening effect was observed to be more at the remelted zone and then decreases towards the solid state transformed zone. The microhardness of samples that underwent surface remelting ranges between 627.8 and
720.9 HV$_{0.5}$ and the maximum hardness value was approximately 3.3 times greater than substrate hardness. The hardness distribution was more uniform in samples treated at slower scanning speed which also have the highest percentage of dissolved graphite.

Further, the statistical analysis determined significant ($p < 0.01$) effect of the combined process parameters on the observed responses. Remelting condition; 40 A/100 mm min$^{-1}$ gave best surface modification. Moreso, wear resistance of the modified surface was found to increase significantly (approximately, 7.4 times). This was due to the enhanced surface hardness, as a consequence to the formation of hard cementite and martensite phases.

Figure 12. SEM micrographs (a), (b) together with EDS analysis (c) of worn surfaces at 90 N normal load, 300 rpm sliding speed and 2828 m sliding distance for the untreated ductile iron.
This work has uniquely established relationships between graphite nodules dissolution and the remelting conditions. The findings can be significant for ductile iron surface engineering design, particularly the ferritic ductile iron.

Acknowledgments

This project is based on: ‘National Key R&D Program of China’, Project number: 2018YFD0300300. Also, the first author acknowledges the support of Bayero University, Kano, Nigeria. In addition, special thanks go to Mr Mustafa Haider and Mr Ibrahim Muhammad of the Institute of Metal Research (IMR), Shenyang for their technical supports in conducting the X-ray diffraction experiment.

ORCID iDs

Falalu Rabiu Jahun @ https://orcid.org/0000-0001-9193-6629

References

[1] Cai Qizhou and Wei B 2008 Recent development of ductile cast iron production technology in China China Foundry. 5 82–91
[2] Paczkowska M 2013 The possibility of selected surface layer modification of nodular iron engine parts by laser boronizing Vehicle Engineering (VE) 1 64–9
[3] Zheng Y 2007 Application and development forecast of Austempered ductile iron in China Chinese Journal of Casting Equipment and Technology 3 60–6
[4] Cao H-T, Dong X-P, Huang Q-W, Pan Z, Li J-J and Fan Z-T 2014 Effect of scanning speed during PTA remelting treatment on the microstructure and wear resistance of nodular cast iron Int. J. Miner. Metall. Mater. 21 363–70
[5] Jahun F R and Jatau J S 2013 Design and production of single-throw nodular cast iron crankshaft Bayero Journal of Engineering and Technology (BJET) 8 1–12
[6] John R K, Dorn T, Kathy L H, Vasko P, Steven S and Arron R 2009 Agricultural applications of austempered iron component Met. Cast. Design. Purchase. 3 28–31

[7] De Santos A, Di Bartolomeo O, Iacoviello D and Iacoviello F 2008 Discrete image model and segmentation for microstructure features identification in ductile irons International Journal for Computational Vision and Biomechanics 1 203–13

[8] Zimba J D, Simbi and Navara E 2003 Austempered ductile iron: an alternative material for earth moving components Cem. Concr. Comp. 25 643–9

[9] Melado A C, Authur S N, Helio G, Michael A G and Philippa A S R 2017 Effect of microstructure on fatigue behaviour of advanced high strength ductile cast iron produced by quenching and partitioning (Q&P) process Int. J. Fatigue 104 397–407

[10] Wanga B, Barbera G C, Tao C, Sunb X and Ran X 2018 Characteristics of tempering response of austempered ductile iron Journal of Material Research and Technology IMRTEC 7 198–202

[11] Ishida T 1983 Local melting of nodular cast iron by plasma arc J. Mater. Sci. 18 1773–84

[12] Oleksiy A T 2016 Kinetic of graphite shape transformation International Journal of Engineering and Research 5 109–15 (www.ijert.org)

[13] Roman S and Janek G 2011 The influence of retained austenite on residual stresses in laser remelted cast iron Journal of Material Engineering and Performance 20 1671–7

[14] Angelini V, Borrelli I, Martini C, Scheuer C J, Cardoso R P, Brunatto S F and Ceschini I 2016 Dry sliding behavior (block-on-ring tests) of AISI420 martensitic stainless steel, surface hardened by low temperature plasma-assisted carburizing Tribol. Int. 103 555–65

[15] Scheuer C J, Cardoso R P, Zanetti F L, Amaral T and Brunatto S F 2012 Low-temperature plasma carburizing of AISI 420 martensitic stainless steel: influence of gas mixture and gas flow rate Surface & Coatings Technology 206 5085–90

[16] Tadeusz S and Krzyztof C 2006 Surface hardening of plain cast ductile iron Journal of Polish CICMAC Gdańsk University of Technology Poland 5 319–26

[17] Karamis M B and Yıldızli K 2010 Surface modification of nodular cast iron, a comparative study of graphite elimination Journal of Material Science and Engineering A 527 5225–30

[18] Ceschini L, Campagna G, Panico N and Angelina V 2016 Effect of laser surface treatment on the dry sliding behavior of the ENGS400-12 ductile cast iron Tribol. Int. 104 542–51

[19] Pan W X, Meng X, Li G, Fei Q X, Fei C K and Wu C K 2005 Feasibility of laminar plasma jet hardening of cast iron surface Surf. Coat. Techn. 197 345–50

[20] Giordano L, Tiziani A and Zambon A 1991 Characterization of surface chromium and molybdenum alloying on gray cast iron obtained by the plasma-transferred arc technique Mater. Sci. Eng. A 140 727–32

[21] Ismail M S and Taha Z 2014 Surface hardening of tool steel by plasma arc with multiple passes International Journal of Technology 1 79–87

[22] Cheng X, Hu S, Song W and Xiong S X 2014 A comparative study on gray and nodular cast iron surfaces remelted by plasma beam Vacuum 101 177–83

[23] Andriollo T, Thorborg J and Hattel J H 2015 The influence of the graphite mechanical properties on the constitutive response of a ferritic ductile cast iron—a micromechanical F analysis Proc. of the XIII Int. Conf. on Computational Plasticity: Fundamentals and Applications: Int. Center for Numerical Methods in Engineering (CIMNE) ed E Orate et al 632–41

[24] Benyounis K Y, Fakron O M A, Abboud J H, Olabi A G and Hashmi M J S 2005 Surface melting of nodular cast iron by Nd·YAG laser and TIG J. Mater. Process Techno 170 127–32

[25] Aydai A and Durma M 2019 Wear performance of ductile iron after electrolytic plasma hardening Kovace Mater. 57 19–26

[26] Mohammadzadeh H, Saghatly H and Kheirandish Sh 2009 Sliding wear behavior of a gray cast iron surface remelted by TIG J. Mater. Sci. Technol. 25 622–8

[27] Guizar A, Akhter JI, Ahmad M, Ali G, Mahmoon M and Ajmal M 2009 Microstructural study and wear behavior of ductile iron surface alloyed by Inconel 617 Appl. Surf. Sci. 255 8527–32

[28] Liu S, Xing Z, Wang Z, Tian S and Jahun F R 2017 Development of machine vision system for gap inspection of muskmelon grafted seedling based on HALCON PLoS One 12 1–12

[29] Morales-Hernandez L A, Terol-Villalobos I R, Dominguez-Gonzalez A, Manriquez-Guerrero F and Herrera- Ruiz G 2010 Spatial distribution and spheroidization characteristic of graphite nodules based on morphological tools Journal of Material Processing Technology 210 335–43

[30] Konstantinova M V, Balanovska A E, Gobenco V E, Karga Poltsev S K, Karlina A I, Shtayerg M G, Guseva E A and Kuznetsov B O 2019 Application of plasma surface quenching to reduce rail wear wear IOP Conf. Series: Materials Science and Engineering 560 012146

[31] ASTM E3-01 2001 Standard Procedure of Surface Preparation for Microscopic Examination, ASTM International,100 Barr Harbor Drive, West Conshohocken, PA 19428-2595. USA: ASTM

[32] ASTM E975-13 2013 Standard Practice for X-ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation (West Conshohocken, PA: ASTM International)

[33] ASTM E384-17 2017 Vickers microhardness standard test procedureStandard test method for microindentation hardness of materials, ASTM International, West Conshohocken, PA, USA (www.astm.org)

[34] ASTM G77-05 2005 Standard test method for ranking resistance of materials to sliding wear using block on ring wear test, ASTM International, West Conshohocken, PA USA, (www.astm.org) (https://doi.org/10.1520/G0077-05)

[35] Jayaprakash N, Yang C-H, Duraiselvam M and Prabu G 2019 Microstructure and tribological evolution during laser alloying WC-12% Co and Cr3C2–25%NiCr powders on nodular iron surface Results in Physics 12 1610–20

[36] Hemant Deore M and Rathod V D 2017 Hiwarkar, Influence of laser surface hardening on microstructure and mechanical properties of austempered ductile iron Int. Conf. on Ideas, Impact and Innovation in Mechanical Engineering (ICIIIME) 5, 1126–32

[37] Packowska M 2016 The evaluation of the influence of laser treatment parameters on the type of thermal effects in the surface layer microstructure of gray irons Optics & Laser Technology 76 143–5

[38] Yan M and Zhu W Z 1997 Surface treatment of 45 steel by plasma-arc melting Surf. Coat. Technol. 91 183

[39] Rathod M J and Deore H A 2014 Laser surface hardening of ductile irons International Conference on Automotive Materials and Manufacturing 28 1–5

[40] Gadag S P and Srinivasan M N 1994 Dry sliding wear and friction; laser-treated ductile iron Wear 173 21–9

[41] Rashid B, Leeman J, Jawaad M, Ghazali M J, Ishak M R and Abdelgani M A 2017 Dry sliding wear behavior of untreated and treated sugar palm fiber filled phenolic composites using factorial technique Wear 380–381 26–35