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Original Research Article

Development of a Surface Modification Procedure for Enhanced Hardness and Wear of Mild Steel

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ABSTRACT

This study developed an effective heat treatment procedure aimed at conferring enhanced hardness and wear characteristics on mild steel. The methodology entailed thermo-carburisation of mild steel using charcoal and graphite alternately at varied particle sizes (60 µm, 80 µm and 100 µm). Carburisation of the mild steel samples was carried out in a muffle furnace at a constant temperature of 1000 °C, soaked for 4 hrs and characterised for wear and hardness. Furthermore, the carburised samples' surface microstructural feature was analysed using scanning electron microscope (SEM). In comparison with charcoal-carburised samples performances, the 60 µm graphite-carburised samples had the highest hardness: (293.7 HV), wear resistance: (1.01×10^3 cm^{-2}) and lowest wear rate: (0.78×10^{-3} cm^2). These values translate to improvement in hardness, wear rate and wear resistance by 55%, 63.5% and 32.1% respectively. Contribution to these outcomes is attributed to the high carbon thermo-diffusivity of graphite resulting in inducement of fine cementite crystals which dispersed homogeneously in ferrite matrix. It is concluded that graphite is a better carburiser for improved mild steel hardness and wear characteristics. Aside other relevant stakeholders, the quest for improved surface quality of low carbon steels stands to benefit immensely from the outcomes of this work.

1. INTRODUCTION

Wear of critical machine components and tools resulting in incessant systems breakdown and failure continues to plague both manufacturers and consumers (Korotkov, 2015). As reported by Holmberg and

Erdemir, (2017), the annual global loss arising from wear phenomenon is estimated to run into several billion dollars. For example, during the production of collapsible aluminium tubes using mild steel mandrel to thread the tube head, excessive wear of the mandrel-tip often results in fracture giving rise to prolonged downtime for retooling and its attendant low productivity (Rana et al., 2016).

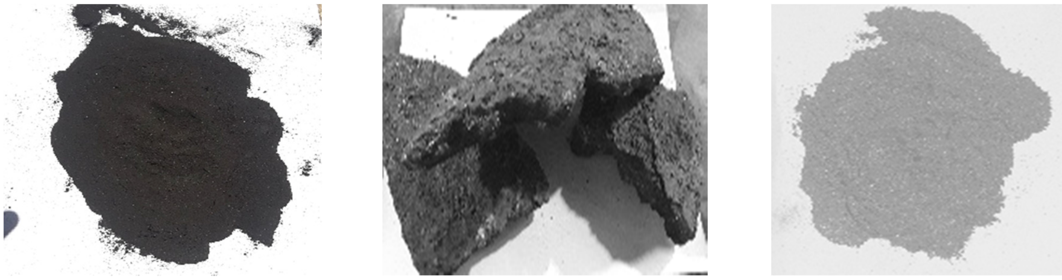
It is established that the ability of a material to exhibit adequate wear resistance depends mainly on the constituent intrinsic microstructure coupled with the surface conditions (Tusharkanta and Kaibalya, 2013). Given that mild steel is not easily amendable to heat treatment owing to its low carbon content, surface condition modification procedure is touted to be an effective solution (Shibe and Chaula, 2014; Kumar et al., 2018; Mozetic, 2019). One of the effective surface modification techniques for enhanced wear resistance of mild steel is the thermo-chemical method (Rehman et al., 2018). This entails either one of the following processes; carburising, nitriding, cyaniding, boronising and chromising. Out of these processes, carburising has been established to be the most easily adaptable in terms of technology, safety and cost competitive (Senthil et al., 2012; Mozetic, 2019). According to Thibaux et al (2007) and Tibetts (2008), carburisation is a process through which carbon from a carbonaceous material is introduced by diffusion into a solid ferrous alloy at a range of temperature above the A_3 point (ferrite to austenite phase transformation temperature on the iron-carbon phase diagram which is normally above 910 ^oC). However, for effectiveness, carburisation of mild steel requires skilful manipulation of the process parameters. These process parameters include the choice of carburiser, heating temperature regime, soaking time and case-depth control. However, the depth of carbon penetration during carburisation is dependent on the composition of the carburiser and its surface characteristics (Reza et al., 2016).

Critical factors that affect wear include microstructure, temperature, surface condition and hardness. These factors are usually factored into carburizing process parameters such that better wear characteristics are conferred on the mild steel. Thus, a large body of works have been conducted and reported in literature aimed at achieving improved wear characteristics of mild steel (Oyetunji and Adeosun, 2012; Elzanaty, 2014; Verma, 2015; Fayomi, et al., 2016; Supriyono, 2018). However, it was observed that the outcomes of most of these works show a relatively marginal improvement in mild steel wear properties. Furthermore, it was discovered that non variation of the carburising agent particle sizes is responsible for the short comings. This appears to be the major drawback of the works of previous researchers in the area of wear characteristics enhancement of mild steel. Based on Hall-Petch relationship, variation of carburiser's particle size reduction-wise has the potential to achieve better outcomes (Kato, 2014). This is premised on the established principle that fine particle size aids diffusion of higher concentration of materials due to increase in surface area (Helmut, 2007; Zang et al., 2020). Therefore, the current work intends to carburise mild steel using varied particle sizes of the carburisers with a view of enhancing its hardness and wear resistance characteristics.

2. MATERIALS AND METHODS

2.1. Materials Preparation

Materials used for the study include five (5) meters length of mild steel (AISI 1020) supplied by African Steel Mill, Ikorodu, Lagos, Nigeria. The steel samples elemental composition shown in Table 1 was obtained using an optical emission spectrometer; (model ARL 3460, Thermo Fisher, USA). Wood charcoal and graphite were sourced from local vendors while barium carbonate ($BaCO_3$) was purchased from a commercial chemical shop at Ojota, Lagos State, Nigeria. The as-received carburising media i.e., charcoal and graphite (Figure 1: a-c) were prepared by pulverisation to obtain fine powders using a laboratory crusher and then sieved to varied particle sizes (60 µm, 80 µm and 100 µm). This was followed by thorough blending of each carburiser with $BaCO_3$ as energiser in the ratio 80:20 of carburiser and energiser respectively.



(a) (b) (c)

Figure 1: Pictures of carburisers used for the study (a) charcoal (b) as-received graphite lump (c) graphite powder

2.2. Methods

2.2.1. Carburisation and tempering of mild steel samples

Prior to carburisation of the mild steel samples, the carburisers i.e., charcoal and graphite were subjected to composition analysis using X-ray fluorescence (XRF) analytical tool. Subsequent to this exercise, the prepared samples were thoroughly cleaned with methylated spirit to remove contaminants like oil and grease. Then, the samples were completely buried in the carburiser blend containing charcoal and energiser ($BaCO_3$) and placed in a steel box. To prevent air infiltration, the steel box was covered with clay and then charged into an electric muffle furnace; (model AF2, Vecstar, UK). The muffle furnace was heated to 1000 ^oC while the samples were soaked in the furnace for 4 hrs. This process was also repeated for the graphite blend with $BaCO_3$. At the end of soaking, the samples were brought out of the furnace and quenched in water to retain carbon on the surfaces to a certain depth. This was followed by tempering at 550 ^oC for 180 min, held in the furnace at that temperature for 60 min and then cooled in air. Tempering was done to relief the internal stress that might have built up in the samples after quenching in water.

2.2.2. Property characterisation of carburised mild steel samples

Carburised steel specimens were specially prepared and characterised for relevant properties including hardness and wear. In addition, the samples were subjected to microstructure analysis using scanning electron microscope (SEM). Hardness characterisation entailed the use of 8 cm length specimens (Figure 2) whose surfaces were ground using 150, 220, 400 and 600 grit sizes of emery papers. The surfaces were then indented using a quadrangular diamond-shaped pyramid indenter; (model Matsuzawa MMT-X, Japan). The hardness measurement was carried out on Vickers scale using a load of 100 gf with a dwell-time of 10 s. Using the prepared wear test samples (Figure 3), wear characteristics test was conducted on a manual Pin-on-Disc machine which involved holding the specimen stationary against a rotating wheel with a 60 grit emery paper fixed. The specimens wear characteristics data were obtained under a constant applied load of 11.38 N at 250 rpm rotating speed.

Figure 2: Hardness test samples

Figure 3: Wear test samples

The mass of the specimens was measured using a four digital micro weigh balance before commencement of test after 30s, 60s and 90s. The wear volume, wear rate and wear resistance were computed respectively using Equations 1-3. The carburised samples' worn surfaces were analysed using a scanning electron microscope (SEM) model JEOL JSM-6480LV under 20 kV potential. The SEM images were obtained at 30X and 150X magnifications.

$$\text{Wear volume} = \text{weight loss/density} \tag{1}$$

Where density is 7.86 gcm^{-3}

$$\text{Wear rate} = \frac{wear\ volume}{sliding\ distance\ (s)} \tag{2}$$

Where sliding distance = $\frac{2\pi RN}{60}$ x time; R (Radius of abrasive wheel) = 7.25cm and N (rpm) = 300

$$\text{Wear resistance} = \frac{1}{wear\ rate} \tag{3}$$

2.2.3. Process flow chart

Figure 4 shows the developed procedure through which the mild steel sample surfaces were modified to achieve improved hardness and wear characteristics.

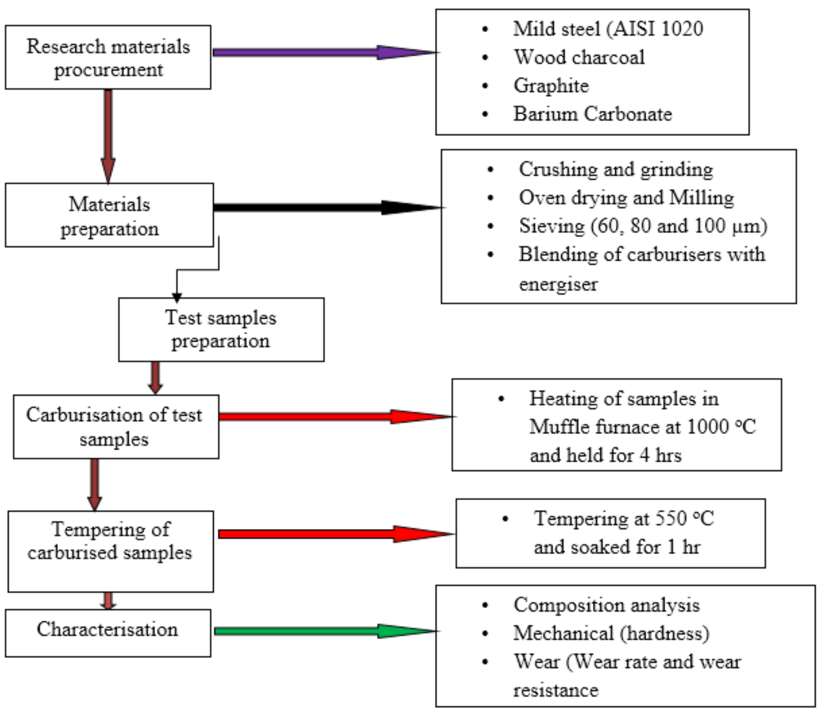


Figure 4: Mild steel surface modification process procedure

3. RESULTS AND DISCUSSION

3.1. Chemical Composition

The as-received mild steel elemental composition analysis result presented in Table 1 affirmed the grade as mild steel given the carbon, silicon and manganese contents. It is established that owing to the relatively low carbon content (≤ 0.3%) of mild steel, hardening heat treatment is rarely performed (Jha, et al., 2017). Thus, innovative surface modification procedure to enhance the hardness and wear properties is considered to be most appropriate. This underscored the approach adopted in the current study.

Table 1: Mild steel elemental composition

Element	C	Si	P	S	Mn	Cu	Cr	Ni	Sn	V	W	Fe
wt. %	0.283	0.192	0.046	0.044	0.603	0.246	0.138	0.114	0.093	0.011	0.172	98.23

3.2. XRF Analysis

Using an XRF analytical tool, the results of composition analysis on charcoal and graphite are as presented in Tables 2a and 2b respectively. The XRF analysis confirmed the presence of Fe_2O_3, SiO_2, MgO and Al_2O_3 as major constituents of the carburisers. These compounds have the potential for effective steel carburization (Mozetic, 2019). Other oxides present in trace amounts include K_2O, CaO, K_2O and TiO_2 are known to provide support for improved case hardening (Kumar et al., 2018).

Table 2a: Composition of charcoal

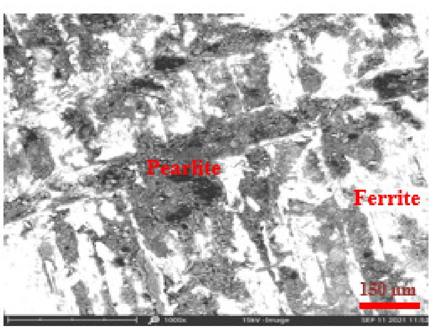
Compound	Fe_2O_3	SiO_2	P_2O_5	Al_2O_3	MgO	CO
Wt. %	1.65	2.81	0.14	0.23	0.48	94.69

Table 2b: Composition of graphite

Compound	Fe_2O_3	SiO_2	P_2O_5	Al_2O_3	MgO	CO
Wt. (%)	5.91	33.48	1.63	7.02	1.05	50.91

3.3. Microstructure of Carburised Mild Steel

The carburised steel samples were subjected to microstructure analysis using scanning electron microscope. Figure 5a shows the microstructure of the as-received mild steel sample while Figures 5b and 5c displayed the structures induced in the samples sequel to their carburisation using 80 µm charcoal and graphite respectively. The SEM micrograph of the as-received sample shows inhomogeneous dispersion of pearlite in bands within ferrite matrix which may give rise to a relatively poor hardness and a rather unmitigated high wear. Similarly, the micrographs of both charcoal and graphite carburised samples show two main phases including pearlite/cementite and ferrite. However, the preponderance and dispersion of these phases vary with the carburiser's particle size. While the crystals of 80 µm charcoal carburised show fringe presence of pearlite (Figure 5b), the 80 µm graphite carburised has its crystals coalesced into lumps (Figure 5c). Furthermore, the 100 µm charcoal carburised crystals appear segregated towards the periphery with directional inclination (Figure 5d). Clustered and segregated crystal dispersion are known to be precursors of structure stratification in metals and alloys which tend to weaken inter-crystal cohesion (Mott, 2018; Liu et al., 2018). This type of microstructure has the potential to impair the mild steel hardness and wear properties. In contrast to the foregoing observations, the micrographs of 60 µm graphite carburised micrograph exhibited higher fractions of pearlite/cementite homogeneously dispersed within ferrite matrix (Figure 5e). Thus, almost the entire samples' surfaces were covered with the cementite/pearlite crystals. This structure presents promising features that promote better hardness and wear characteristics in mild steel.



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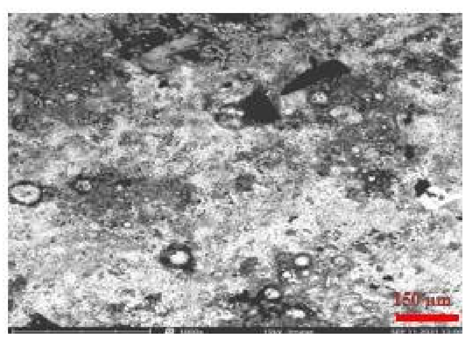

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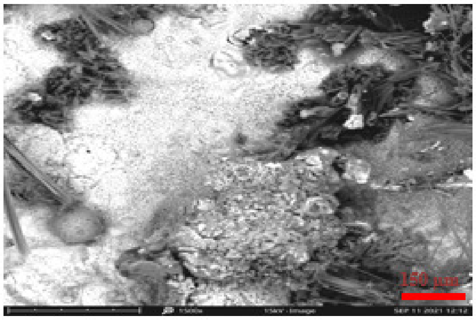

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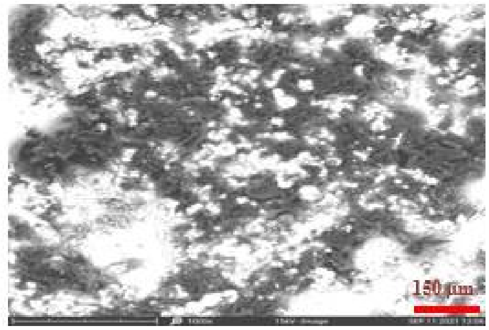

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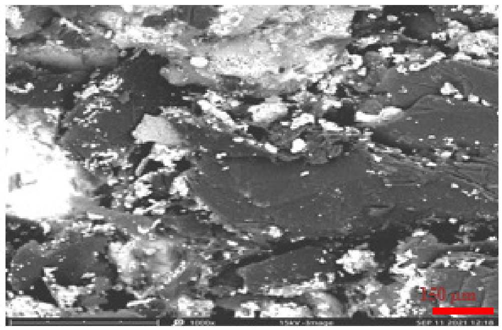

(e)

Figure 5: SEM micrographs of mild steel samples (a) as-received (b) 80 µm charcoal-carburised (c) 80 µm graphite-carburised (d) 100 µm charcoal-carburised and (d) 60 µm graphite-carburised

3.4. Wear Characteristics of Carburised Mild Steel

Results of wear characteristics test conducted on the charcoal and graphite carburised mild steel samples are presented in Tables 3 and 4. The data in Tables 3 and 4 were used to illustrate the wear properties of the samples at varied dry sliding speed spanning 30-90 seconds. The as-received steel sample is identified as ST while the graphite carburised samples using varied particle size of 100 µm, 80 µm and 60µm are labeled as ST_G_{100}, ST_G_{80} and ST_G_{60} respectively. Similarly, the charcoal carburised samples are identified as ST_C_{100}, ST_C_{80} and ST_C_{60}.

Table 3: Wear properties data of graphite carburized mild steel

Sample ID	Sliding time (s)								
	30			60			90		
	Wear volume (cm^3)	Wear rate ($cm^2 \times 10^{-3}$)	Wear resistance ($cm^{-2} \times 10^3$)	Wear volume (cm^3)	Wear rate ($cm^2 \times 10^{-3}$)	Wear resistance ($cm^{-2} \times 10^3$)	Wear volume (cm^3)	Wear rate ($cm^2 \times 10^{-3}$)	Wear resistance ($cm^{-2} \times 10^3$)
ST	19.1	2.81	0.15	36.2	2.65	0.38	46.5	2.27	0.44
ST_G_{100}	13.8	2.02	0.37	20.8	1.52	0.66	35.3	1.72	0.58
ST_G_{80}	7.21	1.13	0.41	14.9	1.09	0.92	21.2	1.03	0.97
ST_G_{60}	4.72	0.71	0.53	12.1	0.89	1.12	15.1	0.74	1.35

The values for the wear rate of graphite-carburized mild steel are presented in Table 3 which shows that the uncarburised sample (ST) demonstrated the highest wear rate, 2.81×10^{-3} cm^2 while the ST_G_{100} exhibited a slightly lower wear rate of $2.02 \times 10^{-3} cm^2$. This was followed by 1.13×10^{-3} cm^2 computed for ST_G_{80} samples. However, the lowest wear; 0.71×10^{-3} cm^2 was exhibited by the ST_G_{60} samples. The downward reduction in wear rate shown by the samples may be attributed to the carburisers particle sizes. It was observed that the finer the carburiser's particle size, the higher the volume of carbon diffused resulting in improved surface modification and corresponding reduction in wear rate. This behaviour underscored the trends shown by the samples in terms of their wear resistance performances.

As shown in Table 4, the charcoal carburised mild steel samples' wear characteristics demonstrated similar trend as the case with graphite carburised samples. However, the carburisers' particle sizes significantly influenced the samples wear behaviour. For example, the ST_C_{60} carburised samples show a relatively low wear rate (1.6×10^{-3} cm^2) and wear resistance (0.68×10^3 cm^{-2}). Comparatively, the 60 µm graphite carburised samples demonstrated lower average wear rate of $0.78 \times 10^3 cm^{-2}$ against $1.6 \times 10^{-3} cm^2$ for the charcoal-carburised samples. Similarly, graphite carburised samples show higher resistance to wear to the tune of $1.0 \times 10^{-3} cm^2$ against $0.68 \times 10^3 cm^{-2}$ for charcoal-carburised samples. These outcomes appear to compare well with improvement achieved by most previous researchers (Supriyono, 2018; Falat et al., 2019) in terms of hardness and wear characteristics of mild steel (Eksi, 2020).

Table 4: Wear properties data of charcoal carburized mild steel

Sample ID	Sliding time (s)								
	30			60			90		
	Wear volume (cm^3)	Wear rate ($cm^2 \times 10^{-2}$)	Wear resistance ($cm^{-2} \times 10^2$)	Wear volume (cm^3)	Wear rate ($cm^2 \times 10^{-3}$)	Wear resistance ($cm^{-2} \times 10^3$)	Wear volume (cm^3)	Wear rate ($cm^2 \times 10^{-3}$)	Wear resistance ($cm^{-2} \times 10^3$)
ST	17.3	7.61	0.13	21.1	3,14	0.32	46.5	3.41	0.29
ST_C_{100}	7.58	3.32	0.30	20.8	2.98	0.34	25.7	1.88	0.53
ST_C_{80}	5.51	2.42	0.41	19.5	2.86	0.35	17.3	1.27	0.79
ST_C_{60}	4.69	2.06	0.49	9.6	1.41	0.71	16.1	1.18	0.85

3.5. Hardness

The influence of the carburisers particle size on hardness is illustrated in Figure 6. Apparently, the uncarburised sample has the lowest average hardness value, 209.2 HV. It was observed that the samples hardness increased progressively as the carburisers particle size decreased. Specifically, for the graphite carburised samples, average hardness increased from 257.3 HV to 264.1 HV and 293.7 HV using 100 µm, 80 µm and 60 µm respectively. Similar hardness value trend was observed with the charcoal carburised samples. Overall, the 60 µm carburiser particle size exhibited the highest hardness both for graphite (293.7 HV) and charcoal (281.4 HV). The hardness performance by the ST_G_{60} and ST_C_{60} samples can be attributed to the fine particles which enhanced carbon diffusion rate resulting in higher carbon volume retained after quenching in water (Liu et al., 1991; Akanji et al., 2015; Reza et al, 2016; Eksi, 2020).

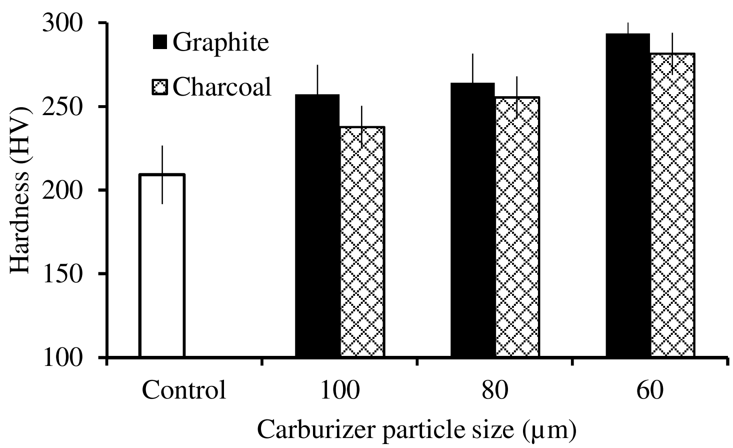

Figure 6: Influence of carburiser particle size on hardness of mild steel

4. CONCLUSION

Surface modification of mild steel using thermo-carburisation technique has been carried out. The carburised mild steel samples' hardness and wear properties were affected by the carburising agents' particle sizes. Specifically, the 60 µm particle size graphite and charcoal carburised samples demonstrated better performances. However, the graphite carburised samples performed better than the charcoal carburised samples in terms of wear rate (0.78×10^{-3} cm^2), wear resistance (1×10^{-3} cm^{-2}) and hardness (293.7 HV). These values represent 63.5%, 32.1% and 55% improvement over conventional mild steel wear rate, wear resistance and hardness respectively. Improved outcomes by graphite carburised sample are predicated on the cementite phase developed on the sample surface given that cementite is harder than the pearlite formed by charcoal. Furthermore, carbon thermo-diffusivity of graphite is established to be higher than that of charcoal. It is concluded that the quest for improved surface quality of low carbon steels stands to derive immense benefits from the outcomes of this study.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

REFERENCES

Akanji, O., Fatoba, O., and Aasa, A. (2015). The Influence of Particle Size and Soaking Time on Surface Hardness of Carburized AISI 1018 Steel. *Current Journal of Applied Science & Technology*, *7*(1), pp. 37-44.

Eksi, S. (2020). Effect of Different Heat Treatments on Mechanical Properties of AISI Steels. *Sakarya University Journal of Science,* 24(3), pp. 479-486.

Elzanaty, H. (2014). The Effect of Carburization on Hardness and Wear Properties of Mild Steel. *International Journal of Innovation and Applied Studies,* 6(4), pp. 995-1001.

Falat, L. Dupon, M. Tavodova, M. Hnilica, R. and Durisinova, K. (2019). Microstructure and Abrasive Wear Resistance of Various Alloy Hard-facings for Application on Heavy-Duty Chipper Tools in Forestry Shredding and Mulching Operations. *Materials,* 12, pp. 2212-2228.

Fayomi, O. S. I. Popoola, A. P. I. and Monyai, T. (2016). Improving the Properties of Mild Steel by Ternary Multilayer Composite Coating via Electrodeposition Route. *Chemical Society of Ethiopia,* 30(3), pp. 449-456.

Helmut, M. (2007). *Fundamentals, Methods, Materials, Diffusion-Controlled Processes.* Springer Series in Solid-State Sciences, vol. 155, Springer, Berlin.

Holmberg, K. and Erdemir A. (2017). Influence of Tribology on Global Energy Consumption, Costs and Emissions. *Friction,* 5(3), pp. 263-284.

Jha, R. Ranjan, S. Kumar, R. and Sharma (2017). Investigation of Heat Treatment on Mechanical Properties of Mild Steel. *International Journal of Engineering Sciences,* 25, pp. 24-28.

Kato M. (2014). Hall-Petch Relationship and Dislocation Model for Deformation of Ultrafine-Grained and Nanocrystalline Metals. *Materials Transactions,* 55(1), pp. 19-24.

Kumar, K. R. Shivasabrani, C. M. Sudherson, D. S. and Jinu, G. R. (2018). Surface modification of mild steel: investigation of basic mechanical properties. *International Journal of Computer Aided Engineering and Technology,* 10(1), pp. 54-63.

Korotkov, V. A. (2015). Wear Resistance of Carbon Steel with Different types of Hardening..*Journal of Friction and Wear*, 36(2), pp. 149-152.

Liu, J. Tennessen, E. Miao, J. Huang, Y. James , L. Rondine, M. and Heinz, H. (2018). Understanding Chemical Bonding in Alloys and the Representation in Atomistic Simulations. *Journal of Physical Chemistry,* 122, pp. 14996-15009.

Liu, W.J. Brimacombe, J.K. Hawbolt, E.B. (1991). Influence of composition on the diffusivity of carbon in steels. *Acta Metallugica et Materialia,* 39(10), pp. 2373-2380.

Mott, N. F. (2018). The Cohesive Forces in Metals and Alloys. *Progress in Physics,* 25(1), pp. 218-230.

Mozetic, M. (2019). Surface Modification to Improve Properties of Materials. *Materials,* 12, pp. 441-449.

Oyetunji, A. and Adeosun, S. (2012). Effects of Carburizing Process Variables on Mechanical and Chemical Properties of Carburized Mild Steel. *Journal of Basic and Applied Sciences*, 8, pp. 319-324.

Rana, R. Batra, M. Sharma, V. K. and Sahni, A. (2016). Wear Analysis of Brass, Aluminium and Mild Steel by using Pin-on-disc Method. *Proceedings of the 3rd International Conference on Manufacturing Excellence,* New Delhi, India, pp. 68-75.

Reza, S. Hosseini, E. and Li, Z. (2016). Pack Carburizing: Characteristics, Microstructure and Modelling. *Encyclopedia of Iron, Steel and Their Alloys.* Taylor and Francis, pp. 1-24, New York.

Rehman, M.A. Munawar, M.A. Nawaz, Q. and Anwar, M.Y. (2018). *Design of Experiment Approach in the Industrial Gas Carburizing Process.* In Statistical Approaches with Emphasis on Design of Experiments Applied to Chemical Processes.

Senthil, B. Kumar, T. and Ajiboye, K. (2012). Effect of Heat Treatment Processeson the Mechanical Properties of Medium Carbon Steel. *Journal of Minerals Characterisation and Engineering,* 11(2), pp. 143-152.

Shibe, V. and Chaula, V. (2014). A Review of Surface Modification Techniques in Enhancing the Erosion Resistance of Engineering Components. *International Journal of Research in Mechanical Engineering & Technology,* 4(2), pp. 89-95.

Supriyono, S. (2018). The Effect of Pack Carburizing using Charcoal on Properties of Mild Steel. *Journal of Technique,* 19 (1), pp. 38-42.

Tibetts, G. C. (2008). Diffusivity of carbon in iron and steels at high temperatures. *Journal of Applied Physics,* 51(9), pp. 4813-4825.

Thibaux, P. Metenier, A. and Xhoffer, C. (2007). Carbon Diffusion Measurement in Austenite in the Temperature Range 500 °C to 900 °C. *Metallurgical and Materials Transactions A,* 38(6), pp. 1169-1176.

Tusharkanta, D. and Kaibalya, M. (2013). Effect of Heat Treatment on Wear Properites of Plain Carbon Steel. Thesis submitted to the department of Metallurgical and Materials Engineering, National Institute of Technology, Rourkela.

Verma, M., Dhillon, K. S., and Verma, M. (2015). Improvement in the Wear Resistance and Mechanical Properties of Carburized Mild Steel by varying Carburization Temperature and constant Tempering Temperature. *International Journal of Innovation and Scientific Research*, 15(2), pp. 379-388.

Zang, J., Wang K. and Yu Y. (2020). Effects of Particle Size on Diffusion Kinetics. *Processes,* 8, pp. 514-523.