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RESEARCH

Effect of Microstructure on the Wear Behavior of Heat Treated SS-304 Stainless Steel

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A B S T R A C T

Sliding wear characteristics of some heat treated SS-304 stainless steel against EN-8 steel in dry condition have been studied in the present experimental work. Samples of SS-304 stainless steel have been heated in a muffle furnace in desired temperature and allowed to dwell for two hours. The heated specimen are then cooled in different media namely inside the furnace, open air, cutting grade oil (grade 44) and water at room temperature to obtain different grades of heat treatment. Microstructures and corresponding micro hardness of the samples have been measured along with Feritscopic studies. Wear characteristics have been studied in a multi tribo-tester (Ducom) in dry sliding condition against EN-8 steel roller. Speed, load on job and duration of test run have been considered as the experimental parameters. The wear of the samples have been obtained directly from 'Winducom 2006' software. Mass loss of the samples before and after operation has also been considered as the measure of wear in the present study. All the samples have been slid against EN-8 steel roller with fixed experimental parameters. The data have been plotted, compared and analyzed. Effect of microstructures as well as micro hardness on the wear behavior has been studied and concluded accordingly.

1. INTRODUCTION

Study of friction and wear Study of friction, wear and associated lubrication in case of interacting surfaces under relative motion is the purview of tribology. Wear determines the useful life of a part/product. Product quality as well as reliability also depends on wear. Hence the study of friction, wear and their control plays an important role in different engineering applications [1,2,28].

For the functional reliability of any component it is very important to control the friction and wear in case of sliding contact. A fundamental knowledge base is helpful to achieve the control. Friction and wear are system dependent properties [3]. There are several research based models and formulations in the field of friction and wear. However, majority of them are not suitable to predict the tribological behavior in a particular work situation. This practical limitation necessitates the iteration of friction and wear data through practical experimentation in a particular situation and for a particular tool-work materials combination. Present work has been carried out to compare the sliding wear behavior of different heat treated SS-304 stainless steel against EN-8 stainless steel in dry working condition. The microstructures and micro hardness of the heat treated samples have also been studied and attempt has been made to find out the relation, if any, on the wear at large.

Stainless steel is a major component for industrial, commercial and consumer products. SS-304 stainless steel is an austenitic iron-nickel-chromium alloy and can't be heat treated for hardening purpose. However, annealing is possible and annealed SS-304 stainless steel has several use in industries like chemical, refrigeration, paper and food processing, beverage, screws, machinery parts, car headers, architecture and many more [27]. This may also be used for bellows, flexible metal hose, spinning, tubing and numerous other stainless applications. Study of friction and wear behavior is thus important in the characterization of annealed SS-304 stainless steel along with the evaluation of other mechanical properties.

Block-on-roller configuration of a multi tribotester "TR-25" (DUCOM, India) has been utilized for the experimental evaluation of wear [17]. Normal load on the sample, sliding speed (rpm) of the wheel and duration of the test run have been considered as the design factors for the evaluation of tribo characteristics. The experiments have been conducted in dry condition, that is, without any lubricants.

2. EXPERIMENTAL

AISI SS-304 stainless steel has been selected as test material for the present experimental work. The material has been procured directly from the market. Samples of size 20 × 20 × 8 have been prepared as per the dimension requirement for the experiments in tribo tester. The chemical composition of the 'as received' material has been indicated in Table 1.

2.1 Heat treatment

Heat treatment of the samples has been done in a microprocessor controlled electric muffle furnace [make: N.R. Scientific Kolkata]. The dimension of the furnace is 250 × 127 × 127 with a maximum temperature limit of 1100°C. The set up is shown in Fig. 1. It is not out of place to mention here that stainless steel SS-304 can't be hardened by heat treatment as no phase changes occur on heating and subsequent cooling. However, softening is possible. Several ways of softening processes have been discussed by different researchers in their research studies [4-14, 30]. Based on the feasibility of studies in the laboratory some basic heat treatment processes have been selected. These operations have been discussed in the following sub-sections.

Fig. 1. Photographic view of muffle furnace.

For sensitization, samples have been heated at 660 °C and allowed to dwell for 30 minutes at that temperature inside the furnace. Samples have been cooled in an open environment, that is, air cooling.

For annealing some samples have been heated from room temperature to 950 °C and allowed to dwell for 2 hours at that temperature inside the furnace. At the end of the operation the furnace has been switched off and the samples have been allowed to cool inside the furnace, that is, furnace cooling.

Table 1. Chemical composition of AISI SS-304 stainless steel.

Constituents	C	Si	Mn	P	S	Ni	Cr	Fe
Percentage	0.08	1.00	2.00	0.04	0.03	8.00-10.50	18.00-20.00	Balance

Some test specimens have been heated from room temperature of approximately 30 °C up to a temperature of 950 °C and allowed to dwell at that temperature for 2 hours (soaking time). Rate of increment of temperature has been selected as 10° per minute for all the cases. At the end of the operation specimens have been taken out of the furnace and allowed to cool in open air to attain room temperature. This is the process of normalizing.

Sudden cooling from elevated temperature is known as quenching. This has been accomplished by dipping the heated samples in an oil bath maintained at room temperature. Samples have been heated up to a temperature of 950 °C with a dwelling time of 2 hours as before and then dipped immediately inside an oil bath maintained at room temperature. Cutting grade oil (grade 44) has been used as the quenching medium in this case.

For solution annealing some sensitized samples are reheated at temperature of 1040 °C with a soaking time of 30 minutes and then allowed to cool rapidly in water maintained at room temperature. This is, however, completely opposite to martensitic steels where this type of heat treatment would harden the steel instead of softening. Solution annealing of SS-304 dissolves any precipitated carbide phase at high temperature resulting a homogenous microstructure with a minor content of δ-ferrite [13].

2.2 Observation of microstructure

Physical properties and mechanical behaviour, in particular, of a material depends on the microstructure. An optical or electron microscope can be used to reveal the microstructure. In the present study a trinocular microscope (Radical) has been used for the purpose. The surface of each sample has been polished using five grades of emery papers, courser to finer. Finally a felt polishing machine has been used to polish the surface with aqueous aluminium oxide (Al_2O_3) solution to give a shinny, smooth and mirror like appearance of the surface of the sample. The surface roughness has not been measured in the present work. Standard nital solution, a mixture of 3 % nitric acid and 97 % ethyl alcohol, has been used to etch the polished surface of the samples for microscopic observation as well as for the measurement of micro hardness.

2.3 Micro hardness measurement

Micro hardness of the samples has been measured in a micro hardness tester 'LM 248 AT' (LECO, serial no. XM8 116, Michigan). Included angle of the pyramid shaped diamond indenter is 136°. All the tests have been conducted at a magnification of 500X, test load of 100 gf and dwell time of 10 sec. Two indentations have been made at two different points on each sample and the average value has been considered as the representative micro hardness. Figure 2 shows the set up for micro hardness measurement.

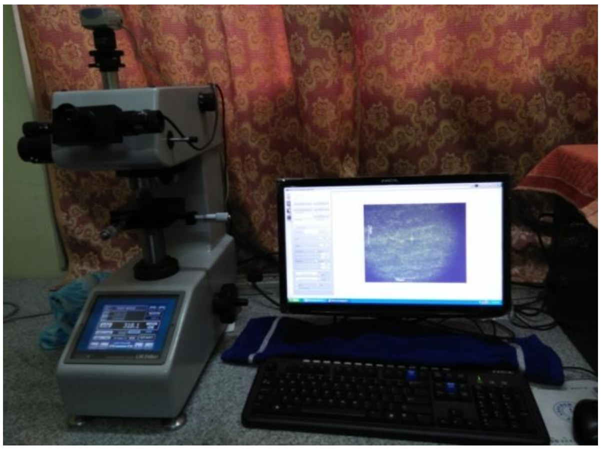


Fig. 2. Set up of the micro hardness measurement.

2.4 Determination of delta ferrite

Amount of delta ferrite on the base material has been de by magneto inductive testing equipment 'Fischer Feritscope FMP 30 (Finland)' as shown in Fig. 3. A feritscope is used to measure the ferrite content in austenitic and duplex steel.

Fig. 3. Feritscope FMP 30 (Finland) with probe.

Magnetic induction method is the basis of such measurement [15,16,26]. Proportions of delta ferrite, strain induced martensite and any other ferrite phases can be measured by this method. Testing has been done on heat treated samples of size 20 × 20 × 8. Ten readings have been taken in ten different places on the surface of each sample and the average value represents the corresponding reading. This test is based on the fact that the austenite is nonmagnetic and the delta ferrite is magnetic.

2.5 Friction and wear test

Friction and wear tests of the samples have been carried out in a multi tribo tester "TR-25" (DUCOM, India) supported with 'Winducom 2006' software. Block-on-Roller configuration of the tester has been utilized for this purpose. The samples have been slid against EN-8 stainless steel roller of 50 mm diameter and hardness of 55HRC. Wear is measured in terms of displacements in microns with the help of a linear voltage resistance transducer in this set up [17]. All the tests have been carried out at an average room temperature of 30 °C and humidity of approximately 85-90%. All the samples have been cleaned thoroughly with acetone to remove any trace of oil, grease, dirt or dust. Based on different research studies [3, 18-22 and references there in] rotational speed of the wheel (rpm), load on job (N) and duration of test run (sec) have been identified as the most important parameters for the wear test in multi tribo tester and corresponding values of 375 rpm, 25 N and 1800 sec of the said parameters have been selected for the wear test. Weights of all the specimens have been measured before and after the wear test using an electronic analytical and precision balance (Sartorius BSA 223S, Germany; maximum range is 220 gm with a readability of 0.001gm). Difference of the two values indicates the weight loss of the specimen due to wear [23].

3. RESULTS AND DISCUSSION

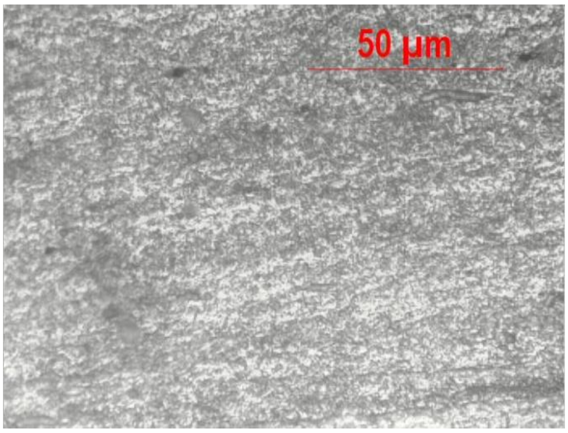
Micro hardness values of the heat treated samples at different matrix or region have been furnished in Table 2. The values of the micro hardness indicate the probable microstructures of the particular region. This is further supported by corresponding micrographs. The micrographs of all the heat treated samples at two magnifications namely 500× and 400× have been furnished in Fig. 4 (a) – (j).

It is well known that AISI 304 stailess steel undergoes a martensitic transformation under cyclic loading conditions [16,24,25]. There are three prominent matrix or zone in the microstructures namely white, black and mixture of white and black. Micro hardness values have measured in those zones or regions. Comparing the micrographs and corresponding micro hardness values, as tabulated in Table 2, following analysis can be made:

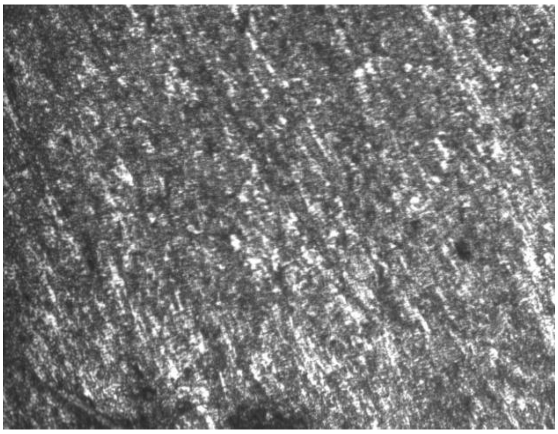
Table 2. Micro hardness at different matrix of heat treated samples.

Heat Treatment	Matrix Type	Microstructure	D_1 (μm)	D_2 (μm)	HV (H)	Avg. HV
Sensitized	White	Martensite	24.72 μm	21.64 μm	345.1	331.6
	White	Martensite	25.37 μm	22.92 μm	318.1	
	Black	Martensite	21.24 μm	19.96 μm	437.0	432.3
	Black	Martensite	20.37 μm	21.28 μm	427.6	
Solution Annealed	White	Austenite	30.48 μm	32.16 μm	189.0	202.1
	White	Austenite	29.87 μm	29.87 μm	215.2	
	Black	Ferrite	23.50 μm	29.29 μm	266.2	270.8
	Black	Ferrite	24.88 μm	27.02 μm	275.4	
Oil Quenched	White	Austenite	28.51 μm	28.35 μm	229.4	223.1
	White	Austenite	30.84 μm	27.65 μm	216.8	
	Black	Ferrite Band	26.66 μm	25.45 μm	273.2	273.2
	Black	Carbide(CrC)	16.28 μm	14.04 μm	806.9	806.9
Annealed	White	Carbide(CrC)	15.88 μm	16.63 μm	701.8	734.6
	White	Carbide(CrC)	15.16 μm	15.93 μm	767.4	
	Black	Ferrite	25.36 μm	25.44 μm	287.4	291.3
	Black	Ferrite	24.99 μm	25.14 μm	295.2	
Normalized	White	Austenite	29.54 μm	29.30 μm	214.8	214.8
	White	Ferrite	26.14 μm	27.23 μm	257.8	257.8
	Black	Carbide(CrC)	16.55 μm	16.23 μm	690.3	690.3
	Black	Mixture of Martensite and Carbide	20.54 μm	19.86 μm	454.4	454.4

Figure 4 (a) and (b) indicates the microstructures of sensitized sample where white and black matrix in both the figures indicates the formation of martensite. Mixture of martensite and carbide is present in the black matrix or region and some part contains ferrite, where as white matrix or region is austenite in case of air cooled samples as indicated in Fig. 4 (c) and (d) respectively. Microstructures of annealed samples have been depicted in Fig. 4 (e) and (f). Figure 4 (e) indicates that austenite has been transformed into ferrite and carbide (chromium carbide). White matrix in Fig. 4 (f) reveals the presence of austenite within grain boundary. Carbide deposition is also prominent on the grain boundary. Oil quenched sample shows the presence of ferrite as evidenced as black band [cf. Fig. 4 (g)]. The entire black matrix here represents carbide. Figure 4 (h) shows the existence of austenite (white), ferrite and carbide. In case of solution annealed samples the existence of austenite and formation of ferrite has been revealed from Fig. 4 (i). Figure 4 (j), on the other hand, indicates the formation of martensite inside the grain boundaries. It is needless to be mentioned that austenite already contains carbon.



(a)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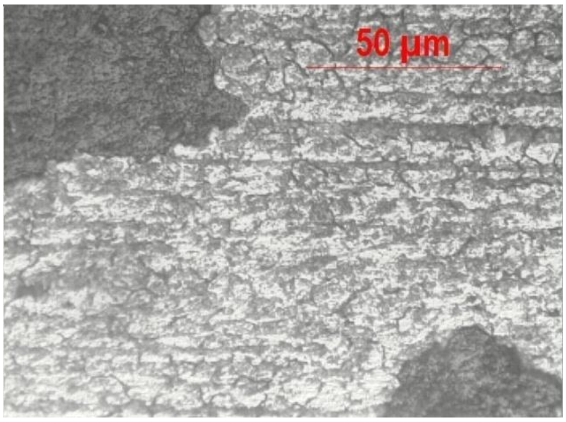
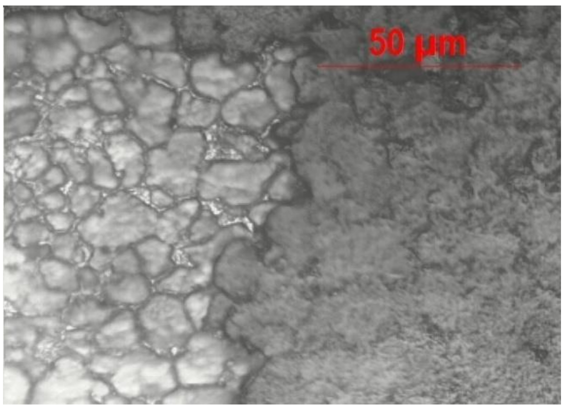
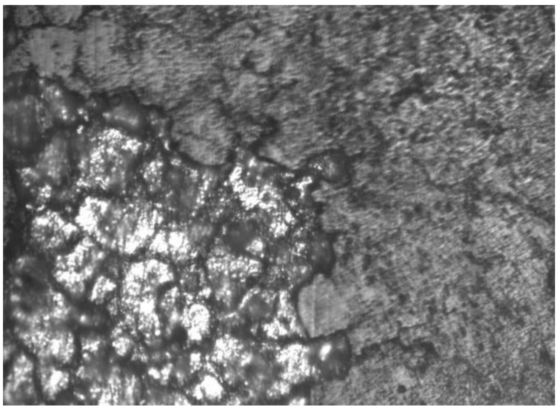


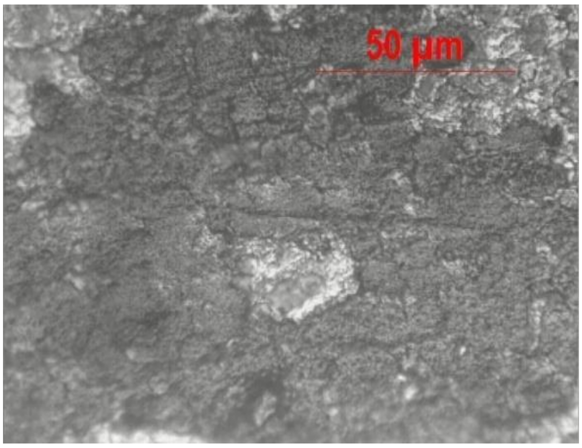
(c)


(d)

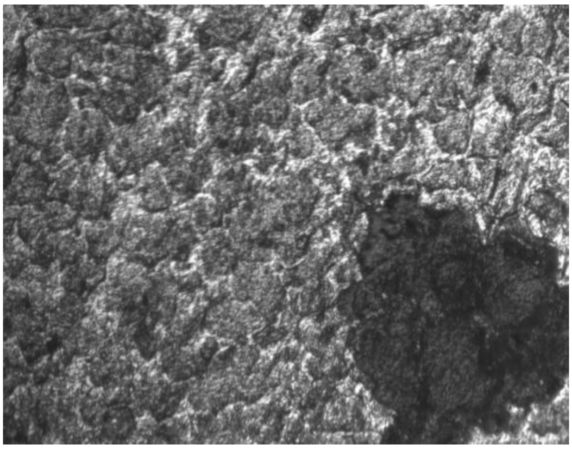


(e)

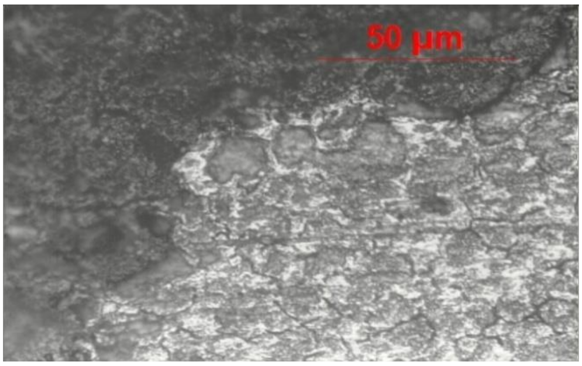

(f)



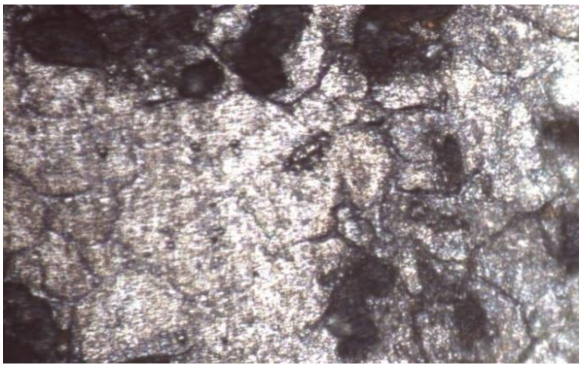
(g)



(h)



(i)



(j)

Fig. 4. Micrographs of different heat treated SS-304 samples showing the microstructures of (a) sensitized sample at 500× (b) sensitized sample at 400× (c) normalized sample at 500× (d) normalized sample at 400× (e) annealed sample at 500× (f) annealed sample at 400× (g) oil quenched sample at 500× (h) oil quenched at 400× (i) solution annealed sample at 500× and (j) solution annealed sample at 400×.

Table 3. Percentage of bcc phase as obtained from Fischer Feritscope.

Heat Treatment Type	**% Reading of bcc Phase**										**Average (bcc Phase)**
	1	2	3	4	5	6	7	8	9	10	
Sensitized	0.25	0.15	0.14	0.26	0.25	0.12	0.17	0.16	0.24	0.18	0.192
Annealed	0.67	0.75	0.68	0.58	0.72	0.88	0.70	0.51	0.63	0.65	0.676
Oil Quenched	0.61	0.51	0.55	0.60	0.49	0.61	0.43	0.59	0.45	0.58	0.542
Annealed	1.0	0.87	0.93	0.97	1.1	0.81	0.90	1.0	0.91	0.99	0.948
Normalized	0.51	0.53	0.51	0.71	0.78	0.59	0.80	0.53	0.71	0.64	0.631

Different types of heat treated SS-304 stainless steel contain mainly austenite (approximately 95 %), carbide, martensite (less than 1%) and δ-ferrite (less than 1%). Fischer Feritscope measures the bcc percentage in the heat treated samples [26]. Presence of bcc structure is mainly due to martensite and δ-ferrite. Table 3 indicates the percentage of bcc phase in different heat treated samples. Ten measurements have been taken in ten different places on the surface of each sample and the average values have been considered as the representative data in this regard. It has been revealed from the above table that quantity of δ-ferrite is less than 1% in all the cases. Thus, δ-ferrite has very little effect on the hardness of heat treated samples. The hardness of the heat treated samples may be assumed to be affected by austenite, carbide and martensite (very little). Hardness, again, is related to micro hardness of ferrite austenite, carbide and austenitic phases. Micro hardness values have already been presented in Table 2.

Weight loss data due to wear have been furnished in Table 4, whereas Table 5 indicates wear (micron) data as obtained directly from 'Winducom 2006' software at different time intervals. Corresponding curves have also been drawn in Fig. 5.

Table 4. Weight loss of the specimens due to wear.

Sample No.	Weight Loss (w_1-w_2) in gm				
	Sensitized	Furnace Cooled	Air Cooled	Oil Cooled	Solution Annealed
I	0.076	0.069	0.057	0.098	0.092
II	0.074	0.063	0.061	0.093	0.088
Avg.	0.075	0.066	0.059	0.096	0.090

Table 5. Wear (micron) of different heat treated samples in regular time intervals.

Heat Treatment	Wear (micron) in different time intervals (sec)					
	300	600	900	1200	1500	1800
Sensitized	86.152	94.262	107.793	125.043	151.501	164.972
Annealed	111.040	117.157	116.397	117.201	120.968	123.966
Normalized	31.712	40.092	48.325	60.933	73.133	91.218
Oil quenched	139.965	177.901	204.43	218.06	224.054	231.011
Solution annealed	134.974	168.633	189.968	199.537	205.777	210.34

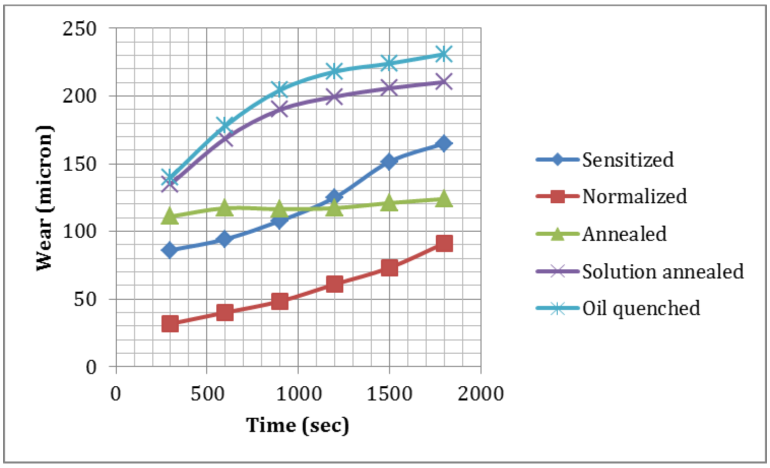


Fig. 5. Comparative charts of wear (micron) against time.

From Table 2 it has been revealed that normalized sample has good hardness owing to the presence of austenite, carbide (CrC) and mixture of martensite and carbide. So it has lesser amount of wear than all other heat treated samples. The wear follows almost a straight line path (cf. Fig. 5). Sensitize samples contain higher amount of martensite. Thus, more hardness is obtained in sensitized samples resulting less wear rate as is evidenced in Table 5. The wear is, however, gradually increasing with time (cf. Fig. 5). On the contrary, annealed samples have more amount of carbide. These samples also have good amount of hardness. Initial wear is somehow more than that of sensitized samples. But the wear is almost stable with time and follows a straight line path. Sensitized, annealed and normalized samples have less amount of wear than oil quenched and solution annealed samples as the hardness of oil quenched and solution annealed samples are far less than sensitized, annealed and normalized samples. Ferrite and austenite phases are prominent in majority of the portions of oil quenched and solution annealed samples. Though oil quenched sample contains carbide (CrC) but quite likely the percentage is very low. Hence, this sample has little resistance against wear. Weight loss data (gm) of the worn samples have been given in Table 4. It has been revealed that the weight loss data and the wear (micron), as obtained directly from the 'Winducom 2006' software, are in good agreement. Thus, it is justified to consider the weight loss data as a measure of wear.

4. CONCLUSION

Some important findings of the present experimental work have been furnished below:

- Some important heat treatments of SS 304 stainless steel have been done utilizing in house facilities. This is a great value addition in conceptual learning process.
- Microstructures and corresponding micro hardness of different matrix has been appraised and evaluated accordingly.

- Presence of δ- ferrite and martensite, that is, bcc structure is less than 1% in all the heat treated samples.
- Normalized samples have minimum wear whereas oil quenched samples have maximum wear. Wear of normalized samples is minimum, however there is a gradually increasing trend.
- Annealed samples show medium wear with a stable trend.
- Weight loss data are in good agreement with wear data, as obtained directly from 'Winducom 2006' software, in the present study.

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