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Effect of vacuum heat treatment process on toughness and wear of H13 tool steel material

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ABSTRACT

H13 tool steel is most frequently used for manufacturing of the tools used for metal forming, metal machining and metal cutting. Local metal working industries complain that H13 steel do not provide better combination of wear resistance and toughness. The objective of present study is to evaluate the toughness and wear properties at different vacuum heat treatments and wear rate with the different heat treatment conditions. Heat treatments on the H13 Tool steel investigate its properties through vacuum tempering, secondary tempering and Nitriding. Microstructure and carbide particles visualized after heat treatment showed great impact on the overall properties. Microstructure of the H13 tool steel was analyzed by metallographic microscope. It was observed that different heat treatment processes significantly improves the mechanical properties like hardness, toughness and microstructure of tool steel.

Key words: Heat Treatment, H13 tool steel, Toughness, Vacuum heat treatment, Wear

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INTRODUCTION

Tool steel is any steel used to make tools for cutting, forming or otherwise shaping a material into a part or component adapted to a definite use [1-3]. The addition of relatively large amounts of tungsten, molybdenum, manganese and chromium can enable tool steels to meet the requirements and service demands therefore it can provide greater dimensional control and freedom from cracking during heat treatment [4-7]. The performance of a tool in service depends on the design of tool, accuracy with which the tool is made, choice of tool steel, and choice of heat treatment [8-9]. High quality tool steel, appropriate design and proper manufacturing methods are the essential factors determining the procedure of the heat treatment [10-13]. H13 tool steel is most frequently used for manufacturing of the tools used for metal forming, metal machining and metal cutting [14-16]. This steel possesses a combination of hot strength, wear resistance and toughness, and is predominantly based on the 0.4% C, 5% Cr compositions containing up to 1.5 % Mo, 1% V and sometimes with increased silicon [17-18].

Local metal working industries complain that H13 steel do not provide better combination of wear resistance and toughness [13]. The objective of present study is to investigate different properties of H13 Tool Steel with consideration of the relationship between current heat treatment procedures and the resultant microstructures/hardness in these materials. Moreover, the study evaluate dynamic impact toughness at several heat treatment conditions by using Charpy V-notch impact toughness tests for Hl3 tool steels. Wear test was carried out to measure different conditions of heat treatment on H13 Tool steel and wear rate. The optimum conditions for heat treatments of these materials for which the properties and life of the tools can be improved were determined.

MATERIALS AND METHODS

Chemical Analysis

Spectro Model MAXx LMM14 (SpectroMAXx) was used to perform chemical analysis. The inert atmosphere was created by using Argon gas before conducting the elemental analysis. At least three sparks were made on different positions of H13 steel sample and average composition given in Table 1 was noted.

Table -1 Chemical composition of H13 tool steel

Element	C	Si	Cr	Mo	V	Mn	P	S	Fe
Average (wt%)	0.39	1.03	5.22	1.25	0.94	0.44	0.019	0.018	Balance

Specimen for H13 Tool Steel and Heat Treatment Conditions

Before commencing heat treatment samples were assigned specific code, given in Table. 2 as per their heat treatment conditions. Thereafter all the test samples were hardened, tempered and nitrited using vacuum furnace (Model: VPT 4035/36 and VTR 5035/36) at Karachi Tools Dies and Moulds Center (KTDMC).

Table -2 Specimen ID of H13 tool steel and heat treatment conditions

S.No	**Sample ID**	**Heat Treatment Condition**
1	AR	As received
2	HT	Hard and Single tempered
3	HTT	Hard and double temper
4	HTN	Hard, Single tempered & Single Nitriding
5	HTNN	Hard, Single tempered & Double Nitriding
6	HTTN	Hard, Double tempered & Single Nitriding
7	HTTNN	Hard, Double tempered & Double Nitriding

Hardening and Tempering Recipe

Hardening and tempering parameters used are given in Table. 3. After loading the samples into the furnace chamber, of 600 x 600 x 900 mm3, the inert environment was created by purging nitrogen gas of 99.999 % purity to develop vacuum environment of 0.1 mbar. The furnace was step heated to 650oC, 850oC and 1030oC with 5oC per minute. The samples were soaked for 30 min time period at 1030oC and then quenched by purging nitrogen at pressure 3 bar using blower of high speed cooling system. Hardening was immediately followed by first and second tempering cycles in order to avoid distortion consequences. In first tempering samples were reheated to 570oC, soaked for 90 min and slowly quenched in nitrogen environment at pressure of 2 bars. In case of second tempering heating temperature was raised to 590oC while maintaining other parameters similar to first tempering.

Table -3 Hardening and tempering parameters

Heat treatment	**Working Parameters**	**Heating Parameters**				**Cooling Parameter**
		Rate of Heating	**Heating Temp $\pm 10^{o}C$**	**Soaking Time (min)**	**Vacuum**	**Pressure (Bar)**
Hardening	Purging	---	---	5	0.1	---
	Gas Heating	5	650	10		1.5
	Vacuum Heating	5	850	15	0.1	---
	Partial Pressure Heating	5	1030	30	0.2	---
	Blower HS Cooling	---	70	5	---	3
Single Tempering	Gas Heating	6	570	90	---	1.5
	Blower LS Cooling	---	50	5	---	2
Second Tempering	Gas Heating	6	590	90	---	1.5
	Blower LS Cooling	---	60	5	---	2

Nitriding

After hardening followed by tempering a set of samples were nitrided in vacuum environment at pressure 0.1 mbar. Nitriding was carried out by purging the ammonia gas at 490oC for 60 min, thereafter flow of ammonia was stopped and samples were left for additional 60 min in nitrogen environment created due to dissociation of ammonia gas. Here in after nitriding at above parameters is referred as first stage nitriding. In second stage nitriding only the heating temperatures were raised to 550oC while keeping ammonia purging and sample soaking time equal to 60 min. The nitriding parameters are given in Table 4.

Table -4 Nitriding parameters

Working Parameters		Heating Parameters				Cooling Parameters	Nitrogen (N)	
		Rate of Heating	Heating Temp ±10^{o}C	Soaking Time (min)	Vacuum	Quenching Pressure (Bar)	% Flow rate	Concentration Rate/ Dissociation Rate
Heating and Purging	Purging	---	---	5	0.1	---	---	---
	Gas Heating	10	360	15	---	0.05	---	---
	Ammonia Purging	10	490	60	---	---	---	---
Nitriding	Single Stage	10	510	60	---	---	100	25
	Double Stage	10	550	60	---	---	100	25
Cooling	Nitrogen Purging	10	300	40	---	0.05	---	---
	Blower Cooling	---	60	30	---	2	---	---
	Pumping	---	---	10	0.1	1.5	---	---

Wear Test

Wear test of as received and heat treated samples of 40 mm x 25 mm x 5 mm dimensions was conducted using Pin on disc machine (Model: 3905, Maid stone England). The sample was fixed in a sample holder vice and electro coated water proof silicon carbide abrasive paper of 150 grades was fixed on rotating disc of 150 mm dia. The sample at 700 kg load was placed on abrasive paper mounted on disc and disc was rotated at 200 rpm. Water was continuously poured on disc to create wet wear environment. After every 10 min the abrasive paper was replaced with fresh one, and weight of sample was noted. In present experimental work total test time was set 70 min, while speed, and sample load were kept constant. The wear rate of each sample for each time interval was calculated.

Charpy V-Notch Dynamic Impact and Hardness Test

The toughness tests were carried out using Charpy impact toughness testing machine Tinsu Olsen model impact 104.The samples was placed on working table and pendulum was raised to 145^{0} lifting angle. Thereafter pendulum was released to hit the sample and the toughness of sample(s) was noted from dial. Charpy impact test samples from the H13 tool steels obtained from the different conditions of heat treatment as mentioned in Table 2. The specimen was machined according to ASTM size as given specification to diameter of 10 mm by a finish length of 55 mm (Tolerance ±0.5 mm) with a notch of radius 45^{o}. Charpy impact testing was conducted at room temperature by using The Tinsu Olsen Impact Machine model impact 104, which has 48 ft-lb maximum capacity and is accurate to t .l ft-lb. Rockwell hardness testing machine (Model Type 4150) was used to measure hardness of the samples at 150 kgf load.

Metallography

Metallographic preparation of Hl3 tool steels is relatively difficult due to the large amount of carbides. The test pieces were cut from the different heat treated samples using an abrasive cutting wheel. Due to the large amount of massive carbide particles, it was very difficult to cut the samples, even when in annealed condition, so extreme care had been taken during the cutting process.

A soft grade of cutting wheel, a copious supply of coolant, and a slow cutting speed were used to avoid overheating and breaking the carbide particles. Each of these effects can lead to misinterpretation of the observed microstructure. An Allied mounting machine was used to mount the samples, which were attached with mounting powder; a pressure of up to 3000-psi and a temperature of up to 80oC were used during mounting. After mounting, grinding and polishing of the specimen was carried out in several steps. Motor-driven disk grinders were used with 240, 320,400, 600, and1200 mesh size grit grinding papers. After fine grinding, polishing produced a surface that was scratch-free, and mirror-like in appearance. For polishing, the specimen was introduced to a cloth, and 1pm alumina particle spray was introduced for a short time at a low initial pressure. The pressure was increased for the main polishing time and then reduced toward the end. To polish the scratch-free surface, and smaller quantities were applied as required during the polishing stage. The polishing time was kept short and the pressure was kept low. A long polishing time or high pressure can result in the formation of relief. Finally samples were etched in 2% Nital prepared by adding 2 % concentrated nitric acid in methanol solution. All the samples were observed under Metallurgical Microscope Model GX51 Olympus & Stereo Micro scope LEICA Model EZ403 optical microscope.

Scanning Electron Microscopy

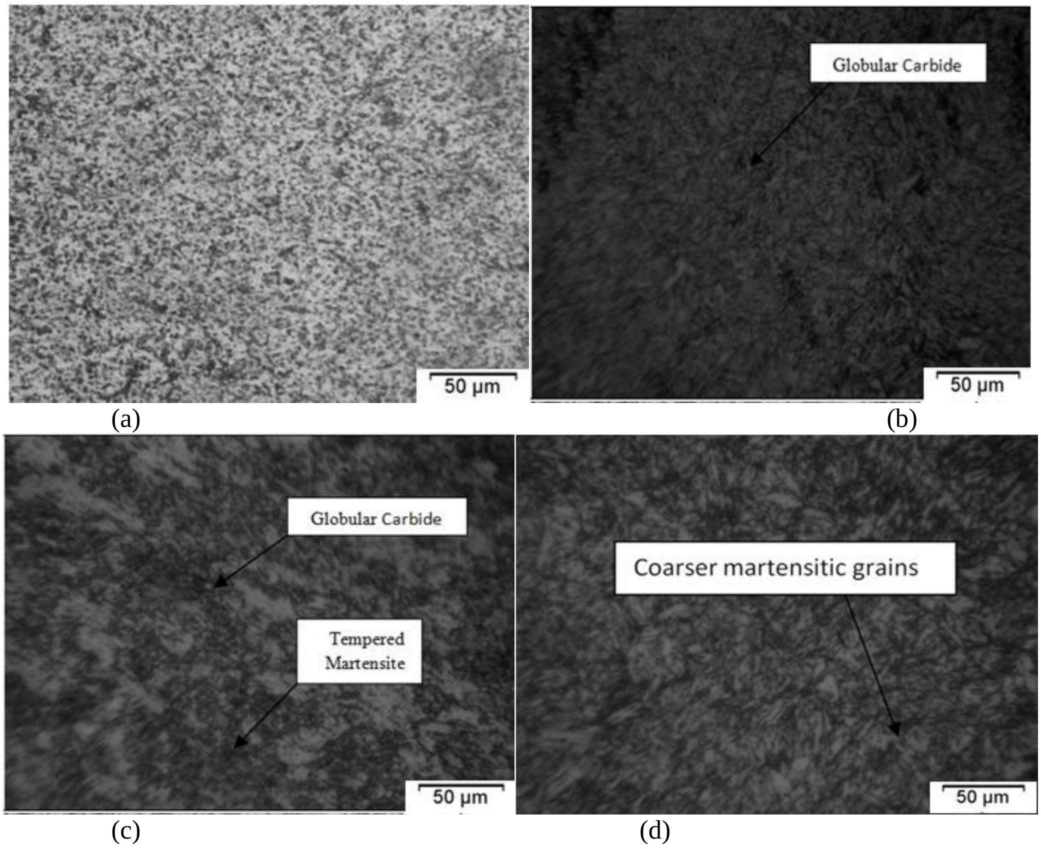
In order to thoroughly understand the effects of different heat treatment conditions including tempering & nitriding behavior on the impact properties of Hl3 tool steels, the fracture surfaces of selected specimens were examined using a Quanta 21Z FEI Scanning Electron Microscope (SEM) operating at an accelerating voltage of up to 20 kV. Representative images of the observed features on these fracture surfaces were recorded.

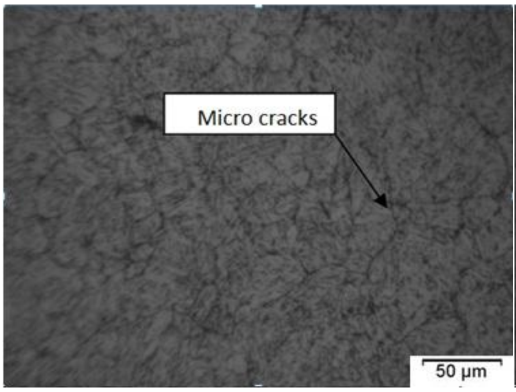
RESULTS AND DISCUSSION

Effect of Heat Treatment on Microstructure

The results revealed that the heat treatment in the vacuum furnace and the control ted atmosphere furnace, at appropriate austenitizing temperatures, gave uniforrn microstructure, and no decarborization and scaling was observed. The microstructure reveals tempered martensite in which coarse carbides (The carbides those do not dissolve during austenitizing) dispersion coexists with fine carbides (The carbides those precipitate during tempering) dispersion. The coarse carbide particles are expected to be M7C3 (chromium carbides). Coarse carbides act as barriers to austenite grain growth and are responsible to a large degree for the high wear resistance. The shape and distribution of these carbide particles are believed to be responsible for the anisotropic mechanical properties. The micrograph also shows the fine carbide particles that are precipitated after tempering.

In H13 tool steel should have an optimum combination of high hardness, good Wear resistance and sufficient fracture resistance or toughness for a given application. H13 tool steels should be designed to retain a significant volume fraction of spheroidized carbides to produce austenite in balance composition. The retained carbides also contribute significantly to wear resistance during service and uniform distributions of carbides are necessary to prevent grain coarsening and abnormal grain growth during austenitizing. [3] The optical micrograph of Hl3 tool steel specimen heat treated in the vacuum furnace followed by tempering shows martensite with small preexisting austenite grain boundaries and spheroid carbide particles that are distributed throughout the matrix. These martensitic structures were very uniform and exhibited minimal signs of alloy segregation. H13 steel is hot working steel, and therefore a high austenitiang temperature without causing grain growth is important to improve the red hardness and the high dynamic impact value.





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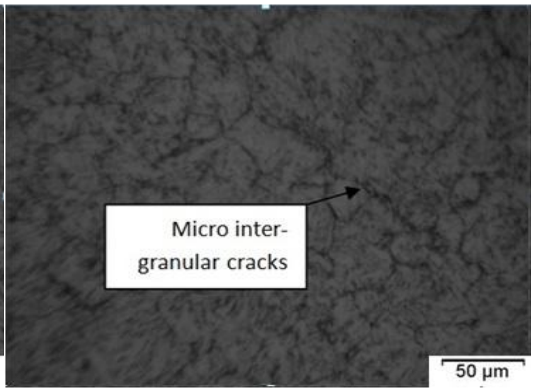

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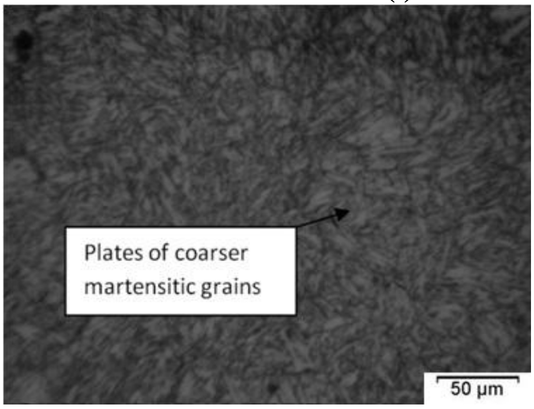


(g)

Fig. 1 Microstructure of (a) AR sample -500X (b) HT Sample -100X (c) HTT sample -100X (d) HTN sample -100X (e) HTNN sample -100X (f) HTTN sample -100X (g) HTTNN sample -100X

Effect of Heat Treatment on Hardness *and* Toughness

Rockwell C hardness testing was performed on hardened, tempered & nitrited specimens of H13 tool steel measured after Charpy V-notch impact testing in order to establish a relationship between hardness of the specimen and energy absorbed by the material. The average results are shown in Fig. 2. The results indicate that an increase in temperature for a material and followed austenitization, tempering and nitriding resulted in increased hardness. Tempering temperatures of 500°C and 530°C used for Hl3 tool steel samples show similar behavior when tested.

V-notch impact tests were carried out because H13 tool steel heavy-duty machine knives and blades, dies and tool materials are used under dynamic impact loading conditions. The objective of this to determine which heat treatment condition produced a high impact value and what is the effect of tempering temperature and number of heat treatment under V-notch impact loading conditions. H13 tool steels samples were prepared from the said samples and heat treated in different conditions tempering temperature.

The results of the room temperature Charpy V-notch impact testing are plotted as a function of austenitizing temperature, tempering temperature, and number of tempering. Heat treatment decrease the impact toughness values as expected. The impact toughness values after tempering were found to be lower than the impact toughness values observed before tempering at 500°C and 533°C. H13 tool steel heat treated at the above-mentioned austenitizing temperatures followed by tempering at has a trend that impact toughness of the steel increases with increasing the number of temperings. The results show that tempering three times versus one or two gives high impact toughness values. The increase in toughness value is also evident from the observation made by SEM of greater ductility on the fractured surfaces after the third tempering.

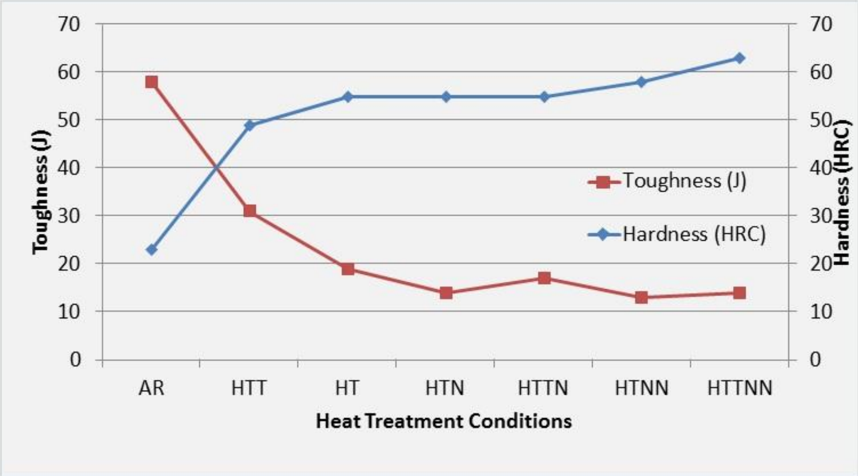


Fig. 2 Effect of different heat treatments on toughness and hardness of H13 tool steel

Effect of Heat Treatment on Wear Rate

Following results shows that the development friction through the wear test. Several phases of increasing & decreasing friction were examined during calculation of wear rate.

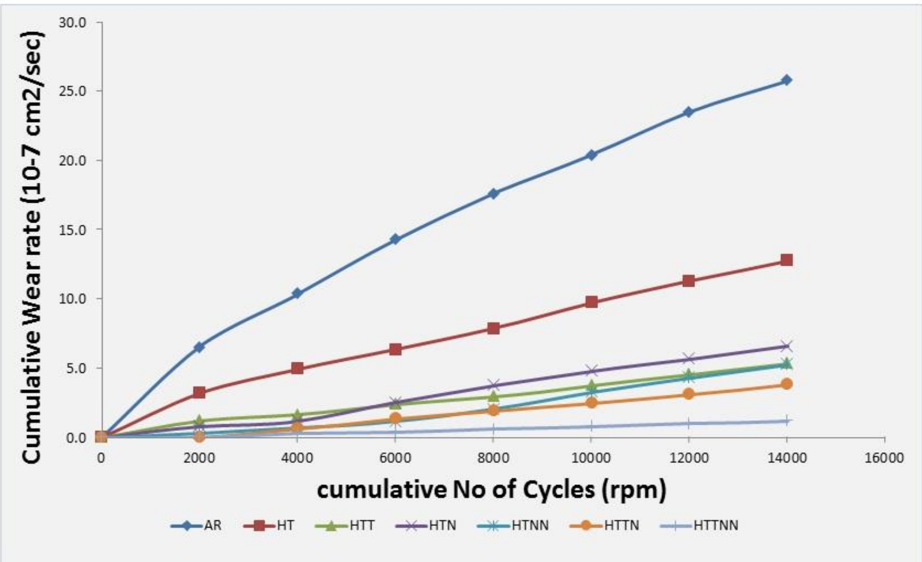


Fig. 3 Effect of heat treatment conditions on wear rate of H13 steel

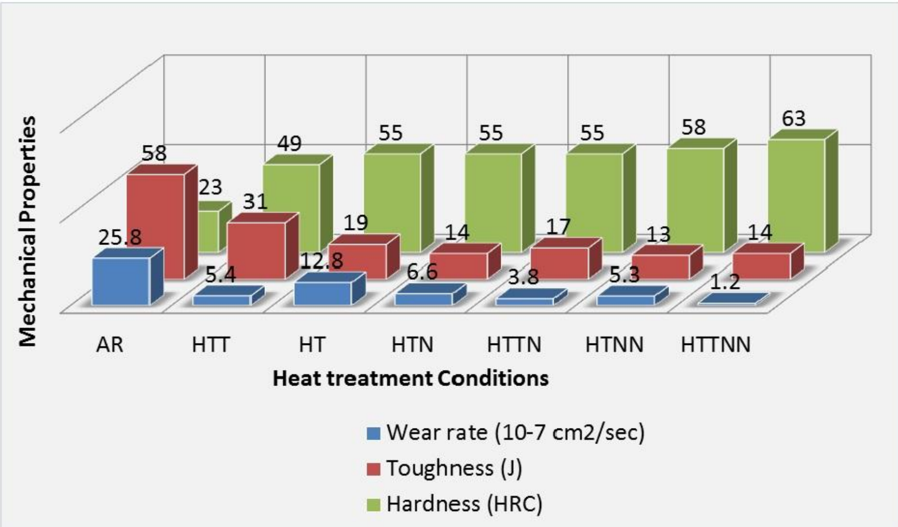
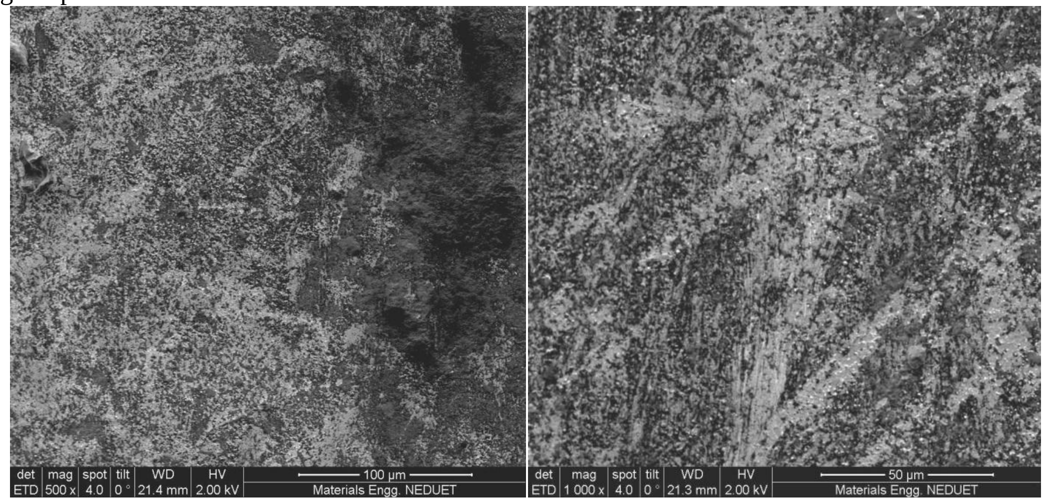


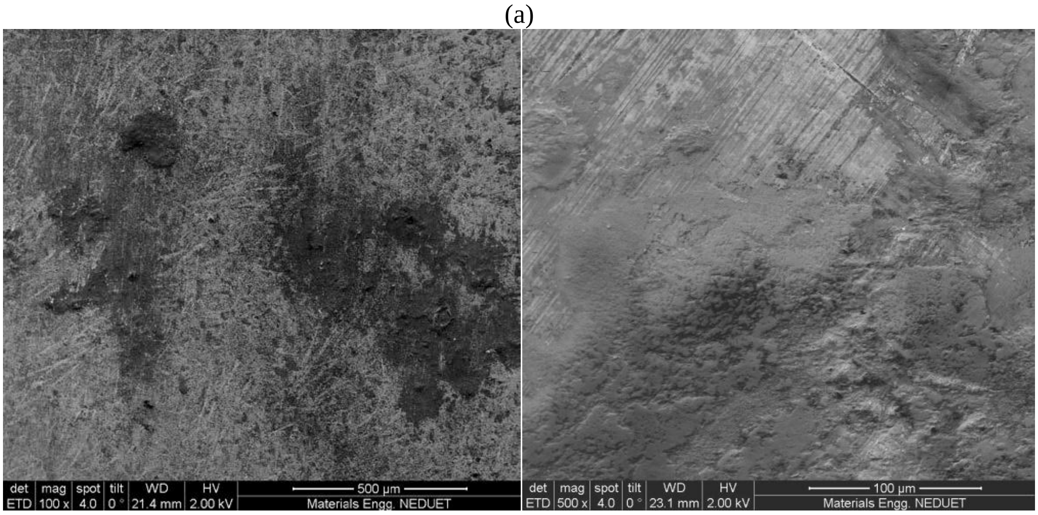
Fig. 4 Hardness, toughness and wear rate at different heat treatment conditions

Fractured Surface Analysis

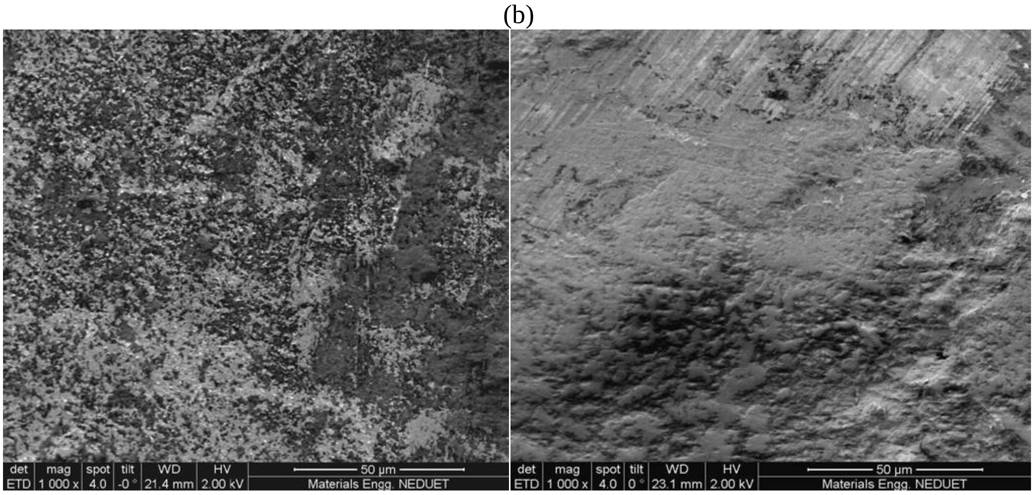
The Charpy impact fractured surfaces show different morphology for the samples tempered once and nitrited at the tempering temperature of 500 °C & 530 °C. It is clear in the fractro graph taken at different magnification shown in Figure 5.The results of H13 impact toughness test show a clear trend that toughness of the material increases as the number of temperings increases, and therefore tempering three times after austenitizing gives higher toughness than tempering once or twice. Analysis of the fracture surfaces from fully martensitic impact specimens tempered at 500°C & 530 °C and tested at roorn temperature revealed that a distinct change in fracture morphology occurs as the austenitizing temperature is increased.



(a)



(b)



(c)

Fig. 5 H13 Tool steel (a) tempered showing the matching part of fractured carbide (b) nitrited showing the matching part of fractured carbide at 530^{o}C (c) tempered twice at 500°C and tempered thrice at 530°C

CONCLUSION

This is based on the results carried out through the extensive test conducted and investigation performed on the tool steel H13.Results indicated that with increasing the tempering stage the volume fraction of chromium carbide precipitates increased, which increased the wear resistance of H13 steel. Nitriding stage further augmented the wear resistance. Encouraging results in terms of minimum wear rate (1.2x10-7cm2/sec) with 14 J toughness has been achieved in HTTNN sample, which showed that the hardening and double tempering followed by double nitriding heat treatment is the best heat treatment for H13 tool steel. Results, in the present investigation, suggest that cooling rates after nitriding treatment play significant role in the embrittlement of steel substrate, so it must be studied carefully. The resultant microstructure of H13 steels after the Heat treatment including tempering, double tempering, nitriding and double nitriding process gives better plasticity. It is found that H13 exhibits excellent mechanical properties after vacuum tempering and Nitriding.

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