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Research Article

High Temperature Tensile Properties of Cryogenically treated AISI 4340 Steel

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Abstract

Deep cryogenic treatment is now extensively adopted as a supplementary process for many engineering applications such as for improving wear resistance, for improving stability of precision measurement equipment, in press tools, etc. In this study, the tensile properties of quenched, tempered and cryotreated AISI 4340 Ni-Cr-Mo low alloy steel are investigated under different process parameters including time and temperature. This steel finds application in press dies, landing gears of aircraft, crankshafts, connecting rod, heavy duty axles, etc. The results have demonstrated that the hardness of test samples is been increased by 6.366% for HCT (Hardened-cryotreated-tempered) samples and by 19.36% for HTC (Hardened-tempered-cryotreated) samples with reference to 377 Hv of HT (hardened-tempered) samples. Improved tensile properties are observed at elevated temperatures (200 and 400^{0}C) for cryotreated samples. This is because the internal stresses developed due to martensite transformation of hardened samples are relieved to a large extent. The improved tensile strength and reduced %elongation can be attributed to coarsening and homogenization of carbide in microstructure.

Keywords: *AISI 4340 steel, Cryogenic heat treatment, High temperature tensile properties, Mechanical Properties.*

1. Introduction

AISI 4340 steel is considered as the standard with which other ultrahigh-strength steels are compared. It combines deep hardenability with high ductility, toughness, and strength. It has high fatigue and creep resistance. It is often used where severe service conditions exist and where high strength for heavy sections is required. In thin sections, this steel is air hardened; in practice, it is usually oil quenched. It is especially immune to temper embrittlement. It does not soften readily at elevated temperatures (ASM Metal handbook; 2000).

AISI 4340 alloy steel is a heat treatable and low alloy steel containing chromium, nickel and molybdenum as prime elements. The addition of Molybdenum prevents the steel from being susceptible to temper embrittlement (Y.M. Shivaprakash; 2005). Gurumurthy *et.al* studied the characteristics evaluation of normalized and conventionally hardened AISI 4340 steel where it was found that the normalized steel has got lower hardness than conventional steel. Rajesh *et.al* carried out a abrasive wear test on hardened and tempered specimen. The abrasive wear volume loss decreased with increase in tempering temperature, which was attributed to coarsening of martensite. Najeeb *et.al* studied austempering on AISI 4340 steel. The hardness and tensile strength at different austempering temperatures and cooling times with the best combination of these improved mechanical properties for better and optimum utilization of this grade of steel was established. Microstructural observations revealed that the carbide precipitates had plate-like structure at low temperatures, but shown spheroidal structures at high temperatures (ASM Metal handbook; 1993, W.S. Lee; 1999). Zhirafar *et.al* Investigatedthe effects of cryogenic treatment on the mechanical properties and microstructures of AISI 4340 steel. It was found that the hardness and fatigue strength of the cryogenically treated specimens were higher whereas, the toughness of the cryogenically treated specimens was lower when compared to that of the conventionally treated steel. Employing 1200^{o}C and 870^{o}C austenitizing temperature on AISI 4340 steel resulted in 80% increase in the toughness. The yield strength was unaffected by austenitizing temperature (G. Y. Lai; 1994, G.E. Dieter). Dhokey *et.al* investigated the Wearrate on multiple tempering after cryogenic treatment of D-3 tool steel. Conventional quenching (950^{o}C) and tempering (150^{o}C) treatments were given

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along with intermediate cryogenic treatment (−185°C). Improved results were obtained by cryogenic treatment. Karthikeyan *et.al* studied the effect of shallow cryogenic treatment (SCT) on hardness, toughness and the amount of retained austenite present in the structure of EN24 steel. In this study it was found that the hardness and ductility increased by 15%, and 55% respectively. In the present study, the AISI 4340 steel is subjected to conventional and cryogenic heat treatment processes and its mechanical properties were analyzed.

For this study, austenitizing temperature of 900°C and tempering temperature of 500°C is set. The effect of cryogenic treatment (−185°C) on high temperature tensile properties along with the microstructural changes is described also the effect of these heat treatments on hardness and wear rate was investigated

2. Experimental work

The material used for the study was a round bar of AISI 4340 steel. The chemical composition (wt %) of the steel as determined by Optical Emission Spectrometer is given in Table 1.

Table 1 Chemical composition (in weight %) of AISI 4340 steel

% Element	Spectrometer Observation
% C	0.38
% Mn	0.51
% Cr	1.02
% Mo	0.24
% Ni	1.48
% Si	0.29
% S	0.02
% P	0.017
% Fe	Bal.

In the present study various combination of heat treatment cycles are carried out. In first trial Conventional heat treatment was used as a reference. In which samples were subjected to conventional hardening including, austenitizing at 900°C for 30 min in a muffle furnace, followed by oil quenching. Then tempering was carried out at 500°C for 4 Hrs. In second heat treatment cycle cryogenic heat treatment was incorporated after hardening and tempering (HTC). In case of third trial cryotreatment was done in between hardening and tempering (HCT). The cryogenic heat treatment comprises of slowly cooling the samples to approximately -185°C and holding at this temperature for 16hr and gradually bringing back to room temperature. Cryogenic treatment was proposed to carry out in a Cryo-bath (−185 °C, Make: Sanmar, Navi Mumbai). In this study, five different heat treating sequences were used to evaluate the response of the steel to cryogenic treatment (Table 2). High temperature tension test and other mechanical test including hardness and wear tests for the samples in all sequences are carried out.

Table 2 Heat treatment cycles

Sequence	Heat Treatment
AR	As Received
H	Hardening at 900°C
HT	Hardening at 900°C and Tempering 500°C
HTC	Hardening at 900°C, Tempering 500°C and Cryo -185°C
HCT	Hardening at 900°C, Cryo -185°C and Tempering 500°C

Tensile test at elevated (200 and 400°C) and room temperature was conducted on MUTC-20 (20 tonnes) Series Universal Motion INC. Model Universal Testing Machine. The dimensions and geometry of the samples and the testing procedure is in accordance with ASTM standard E8 for tension test. Samples were prepared with the gauge length of 44mmand diameter of 9 mm. The mechanical properties, such as yield strength, % elongation are obtained from the stress-strain diagram obtained from the tensile test. Vickers hardness tester is used to evaluate the hardness. Flat surface was prepared by using SiC-emery paper.

A minor load of 10 kg was first applied to set the indenter, then load of 30 kg was applied for 15 seconds and indentation was recorded. An average of three readings is noted as a measure of hardness. Microstructural examination is done by scanning electron microscopy (SEM) (Sigma Field Emission). Pin-on-Disc test machine (make: Magnum Engineer, Bangalore) was used for analyzing dry sliding wear in which counter face of rotating disc was made of SAE 52100 with hardness of 59 HRC. The wear tests of all samples were carried out for a period of 600 sec, giving a total sliding distance of 1131 m. The rpm of the disc was kept constant by keeping 3kg load which gives a velocity of 1.885 m/s.

3. Results & Discussion

3.1 Hardness Measurements

Vickers hardness (30 Kg load-15 sec. dwell time) at three points is taken and an average value of it is plotted as function of different heat treatment cycles conducted. Fig.3.1 shows variation of Vickers hardness as the function of various heat treatment cycles used. It is noted from fig. 1 that the quenched samples demonstrated the highest value of 541Hv hardness. This can be attributed to phase transformation of steel during quenching process, where the lattice structure of steel changes from a face centered cubic to a body centered tetragonal (martensite).

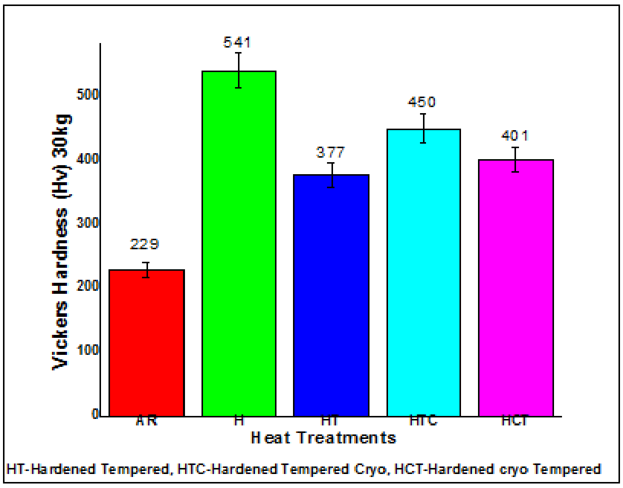

Fig. 3.1 Effect of various heat treatments used on hardness values of AISI 4340 steel

At the same time, a large amount of distortion occurs during the formation of martensite having a very high dislocation density (causing dislocation tangles during subsequent processing), leads to high hardness and high work hardenability (W.S. Lee; 1999). In case of HT (Hardened-Tempered) samples the hardness value decreases because of tempering. This is because high temperature tempering at 500°C reduces residual stresses and promote to carbides to precipitate and coarsen, thereby increasing their average size accompanied by decrease in their total population. This causes reduction in dislocation interaction occurring during deformation at the time of hardness measurement and decrease of hardness values. The values are still higher than those obtained in case of as received samples. The cryogenically treated samples showed the best results of hardness ($Hardness_{HTC}$>$Hardness_{HCT}$) values. Increase in hardness due to cryogenic treatment following hardening can be attributed to increased residual stresses as a result of phase transformation of retained austenite to martensite (W.S. Lee; 1999). Tempering after cryogenic treatment (HCT) relieve the residual stresses and reduce retained austenite and provides the driving force necessary for precipitation of secondary carbides. By comparison, the hardness of the quench cryotreated and tempered sample (HCT) is lower due to precipitation of carbide and reduction in tetragonality of martensite due to recrystallization during tempering. Cryo-treatment after tempering (HTC) produces a higher hardness than quench-cryo-tempered (HCT) samples as a result of a double effect of conversion of retained austenite to martensite and higher precipitation of ε ($Fe_{2.3}C$) or η-carbides (Fe_2C) during low temperature tempering. Further tempering may produce very small spheroidal carbide precipitation, which is important with respect to wear resistance but may not result in improved hardness.

3.2 Microstructure Analysis by SEM

The SEM microphotographs of the various samples in etched condition studied are shown in fig 3.2 (a-e). The SEM microphotograph of as received samples (fig 3.2a) showed ferrite, pearlite and carbides in its matrix. The ferrite getting lightly etched appears in front whereas the unresolved pearlite getting deeply etched appears to be present on back. Diagram shows the coarser carbides seen to be evenly distributed along the grain boundaries. For the hardened samples, a lath martensitic structure distributed in different packets is observed. The size and inter-lamellar spacing of the initial martensite flakes even on tempering appears to be much finer in case of the cryotreated samples. The tempered martensite appears to be distributed finer in case of HCT samples than HTC samples. However the hardness value noted is higher for HTC samples may be because of much finer distribution of carbide in case of these samples.

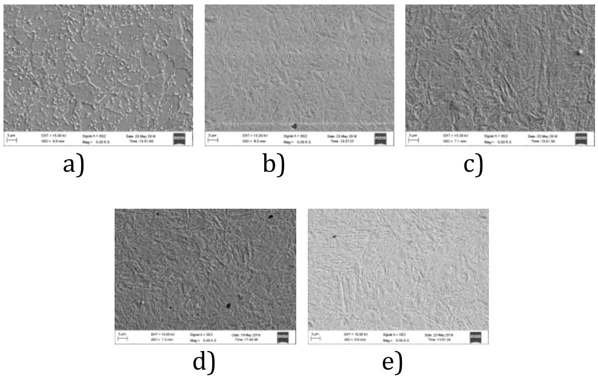

Fig. 3.2 Scanning Electron Micrograph of AISI 4340 High strength alloy steel material used (a) As Received, (b) H, (c) HT, (d) HTC, (e) HCT (Magnification 5000X).

3.3.1 Yield strength

A material is said to be failed once its yield strength is reached. The yield strength of material under tension and compression is assumed to be same. Fig.3.3 shows yield strength values for different heat treated samples.

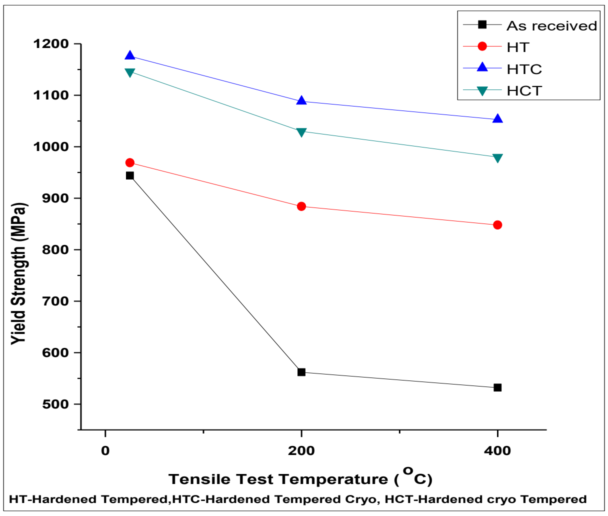

Fig. 3.3 Yield Stress for different heat treatments at test temperatures 200 and 400°C

For all samples tested, there is marginal reduction in yield strength at elevated temperatures. These values range from 1146Mpa for HCT samples to 1176Mpa for HTC. Imparting a cryogenic treatment to these samples giveS much higher tensile strength values. This can be attributed to transformation of retained austenite to martensite. The highest value for the HTC sample can be attributed to the occurrence of greater dislocation density in these samples than HCT samples. In particular for the HT samples beyond a test temperature of 200°C, the yield stress values are even lower than that of original YS value as per which design of a die is made.

3.3.2 % Elongation

Fig. 3.4 shows values of % elongation (% E) for the as received and the samples subjected to different heat treatment cycles. The room temperature values of the %E are 14.77 for the as received samples and 8.59 to 9.18 for the heat treated samples agree with the values obtained in other researches. The difference in stretchability of as received and the heat treated samples is considerably high, which increases further with the increase in test temperature to 200 and 400 °C respectively, thereby indicating that the as received material is not suitable for high temperature applications. The HTC samples are best suitable as they show a minimum change in %E with respect to rise in temperature.

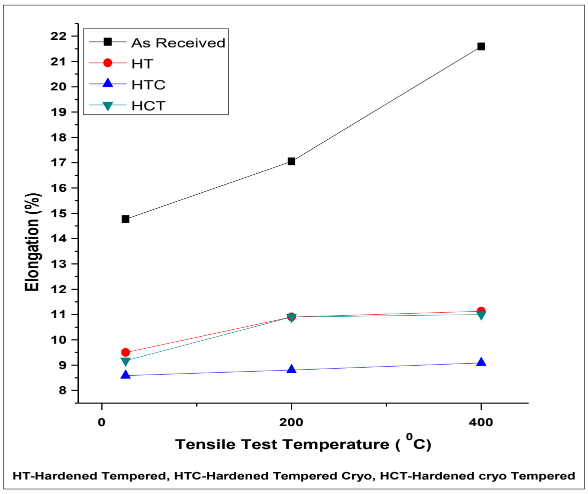

Fig. 3.4 % Elongation for different heat treatments at test temperatures of 200 and 400°C

3.4 Wear Rate Measurement

Wear rate for different heat treated batches is as shown in fig 3.5. From Fig. 3.5 it is noted that the wear rate of as received sample is highest (1.83 x 10^{-3} mm^3/mm). These samples show relatively soft phase in its microstructure like ferrite and pearlite, which increases their wear rate. The low hardness value of these samples justifies the higher wear rate noted. The hardened samples demonstrate the lowest wear rate of 0.48 x 10^{-3} mm^3/mm associated with their highest hardness values. An intermediate wear rate of 1.27 x 10^{-3} mm^3/mm is observed in case of HT samples, followed by lowering of wear rate to 0.93 x 10^{-3} mm^3/mm for HTC and to 0.63 x 10^{-3} mm^3/mm for the HCT samples. The additional amount of fine carbide nucleated during cryogenic treatment, homogeneous distribution along with elimination of residual stresses are the factors which are likely to contribute therein.

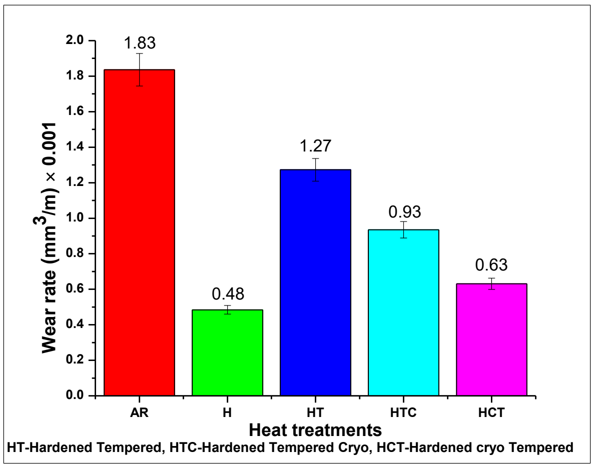

Fig.3.5 Effect of various heat treatments used on wear rate values of AISI 4340 steel

Conclusions

In present work different samples were prepared for tension test, wear test, hardness and microstructural analysis. Cryo-treatment was done on samples which were hardened at 900°C for 30 min. and tempered at 500°C for 4 h. Hardness test gave better results for HTC samples in comparison to HCT and HT samples. Microstuctural investigation shows annealed (as received) structure containing coarse carbides along the grain boundaries. After hardening, it gets converted into martensitic structure showing needles of martensitic laths under microscope. Tempered martensite produced after tempering appears much finer in cryotreated samples. Yield strength goes on decreasing as test temperature increases. There is drastic fall in tensile and yield strength for as received and HT heat treated sample at 400°C. The cryotreatment of the samples caused improvement almost 30% in room temperature tensile strength as well as high temperature tensile strength. The HTC samples showed the best results in high temperature tensile test. The % Elongation goes on increasing as test temperature increases. The % elongation of 15% value was highest for as received sample which was reduce to 8-9% after the HTC heat treatment. The wear rate value was noted 1.83 x 10^{-3} mm^3/mm for as received and 1.27 x 10^{-3} mm^3/mm for HT samples. The wear rate value was noted 0.63 x 10^{-3} mm^3/mm for

HCT samples which was lower than HTC (0.93 x 10^{-3} mm^3/mm), as received and HT samples.

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