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Effective Heat Treatment Process to Enhance the Tool Life of H11 Steel

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Abstract

Heat treatment is the controlled heating and cooling of metals to alter their physical and mechanical properties with out changing the product shape. High carbon and high alloy steels have retained austenite at room temperature. To eliminate retained austenite, the temperature has to be lowered. The amount of martensite formed at quenching is a function of the lowest temperature encountered. Cryogenic Treatment or deep freezing is done to make sure there is no retained austenite which formed during quenching. Cryogenic tempering becomes important especially in the hypereutectoid steels. The main objective of this work is to be quantifying the improvements in wear resistance of hypo-eutectoid tool steel (H11) by sliding wear method (Pin-on-Disk) due to the degree of cooling. The overall enhancement of specific-wear resistance compared to normal treatment in each was 27%, 98%, 126% and 115% for C1 (233 K), C2 (193 K), CR1 (123 K) and CR2 (83 K) respectively. As result of this work, the difference in level of percentage-retained austenite and residual stress between CR1 & CR2 was marginal; the experimental results also shows the marginal difference in overall percentage of wear resistance improvement in CR1 & CR2 and maximum development had taken place at CR1 condition. Hence, H11 steel can be treated under Cryotreatment I (CR1) condition.

Keywords: cryogenic treatment, microstructure, wear resistance, hot working steel

1.0 Introduction

The thermal treatments of metals ought to be definitely considered as one of the most important developments of the industrial age. Researches are being continued in to making metallic components stronger and more wear resistant. One of the latest processes being used to treat metals is cryogenic tempering. Cryogenic tempering becomes important especially in the hypereutectoid steels. The temperature range for martensite formation is determined by the carbon content of the material.

Bowers R.G. (1974) [5], cryogenic process is key to continuing the transformation of retained austenite in to martensite in hypereutectoid steels. The effects of low temperatures in retained austenite, increases hardness and toughness which lead to increase product life more economically than any of the surface treatments.

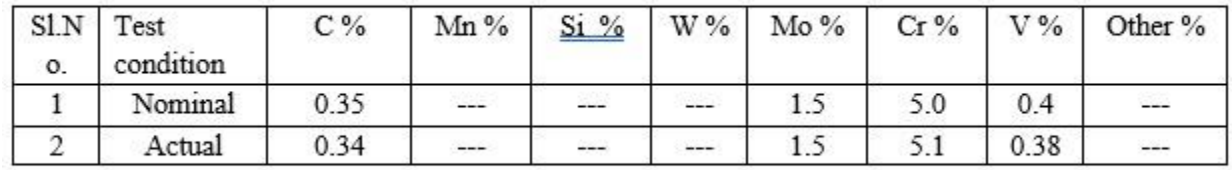
Cryogenic treatment refers to the post-heat treatment process, where the mass of products to be treated are slowly cooled to very low temperatures, usually the liquid nitrogen temperature, held at that temperature for a specific period of time and warmed back to room temperature at a specific rate indicated by M/s Controlled thermal processing, Applied Cryogenics Inc., (1986) [6], and Freeze Tempering, DSP Cryogenics, (1996) [7]. Barron R F. (1982) [4], the mechanism for the improvement was attributed to transformation of retained austenite, formation of benevolent residual compressive stress and formation of fine carbides and also Mohanlal D, *et al.* (2001) [8], depended on long soaking period. Vaccari JA (1986) [9], the transformation of retained austenite to martensite is not the only reason for prolonging tool life but the precipitation of small finely dispersed carbides in the martensite is one among the main reasons.

A typical application of Chromium hot work steel H11 of medium carbon content is required to withstand the combination of heat, pressure, and abrasion associated with operations. The main objective of this work is to quantify the improvements in wear resistance of hypo-eutectoid tool steel (H11) by sliding wear method (Pin-On-Disk) due to the degree of cooling.

2.0 Material

In ASM, tool steels (1990) [1], the major alloying element that influences almost all the cutting characteristics of the material is carbon. This is much more concerned with heat treatment and quenched hardening. Both the hardening as well as the microstructure formed during heat treatment are controlled by the carbon content of material. The nominal value of H11steel composition as per AISI (1978) and actual composition was found by wet type method is listed in Table 2.1.

Table 2.1 Composition of Tool Steels selected for study



Sl.No.	Test condition	C %	Mn %	Si %	W %	Mo %	Cr %	V %	Other %
1	Nominal	0.35	---	---	---	1.5	5.0	0.4	---
2	Actual	0.34	---	---	---	1.5	5.1	0.38	---

3.0 Experiments

In the Pin-on-Disk apparatus, ASTM, G99-95, (1996) [3], the maximum size of pin that can be accommodated is held as ϕ6 mm and 50 mm length. The specimens were machined to ϕ5 and a length of 30mm. Then the pieces were faced flat and made to contact fully on the disk (grinding wheel GC 320 K5V). Finally the specimens were finished by centreless grinding to avoid irregularity in their diameters.

After machining, the test specimens (pins) were subjected to heat treatment process ASM, heat treaters guide, (1995) [2]. Initially the stress was relieved at 300^{o}C then those were preheated in a single stage from 300^{o}C to 845^{o}C and soaked at 815^{o}C for 30 minutes then slowly heated to 1025^{o}C (i.e. austenitizing temperature) and soaked at this temperature for 15 minutes. After that process, immediately quenched in oil maintained at 300^{o}C and soaked for 2 minutes in single stage. After hardening, the hardness values of pins were 53 to 55 HRC. The quenched samples were stress relieved at 150^{o}C for 2 hours and single stage tempering at 550^{o}C for 1 hour and 590^{o}C for 15 minutes.Coldtreatment and Cryotreatment were introduced after completion of hardening process. In case of cold treatment, one part of hardened and stress relieved samples were cooled from room temperature of 30^{o}C to -40^{o}C and another set of samples were cooled from room temperature to -80^{o}C in 2 hours and 4 hours respectively then soaked at this temperature for 24 hours. Subsequently the samples were slowly heated to room temperature in 1 hour and 2 hours respectively.

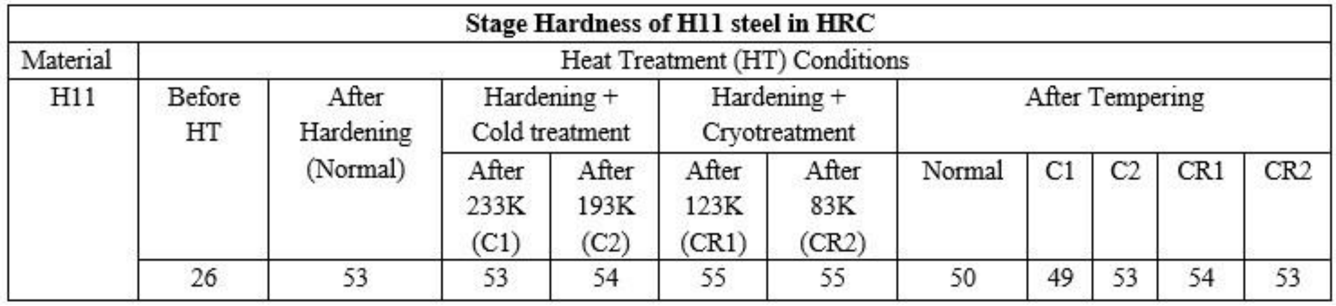
In case of Cryotreatment, one set of hardened and stress relieved samples were cooled from room temperature of 30^{o}C to -150^{o}C and another set of samples were cooled from room temperature to -190^{o}C in 6 hours and 8 hours respectively then soaked at this temperature for 24 hours. Subsequently the samples were slowly heated to room temperature in 4 hour and 6 hours respectively.

On completion of all the Heat treatment process, Treatment conditions considered for wear resistance are recorded as:

1. Untreated **[NORMAL]** (Hardened & Tempered)
2. Coldtreated - I **[C1]** (Hardened + Coldtreated at -40^{o}C (233K) + Tempered)
3. Coldtreated - II **[C2]** (Hardened + Coldtreated at -80^{o}C (193K) + Tempered)
4. Cryotreated - I **[CR1]** (Hardened + Cryotreated at -150^{o}C (123K) + Tempered
5. Cryotreated - II **[CR2]** (Hardened + Cryotreated at -190^{o}C (83K) + Tempered)

The hardness was measured at the end of each heat treatment process. Finally the hardness of five classified samples of H11 was tabulated in Table 3.1

Table 3.1 Stage Hardness of H11 steel in HRC



Stage Hardness of H11 steel in HRC											
Material	Heat Treatment (HT) Conditions										
H11	Before HT	After Hardening (Normal)	Hardening + Cold treatment		Hardening + Cryotreatment		After Tempering				
			After 233K (C1)	After 193K (C2)	After 123K (CR1)	After 83K (CR2)	Normal	C1	C2	CR1	CR2
	26	53	53	54	55	55	50	49	53	54	53

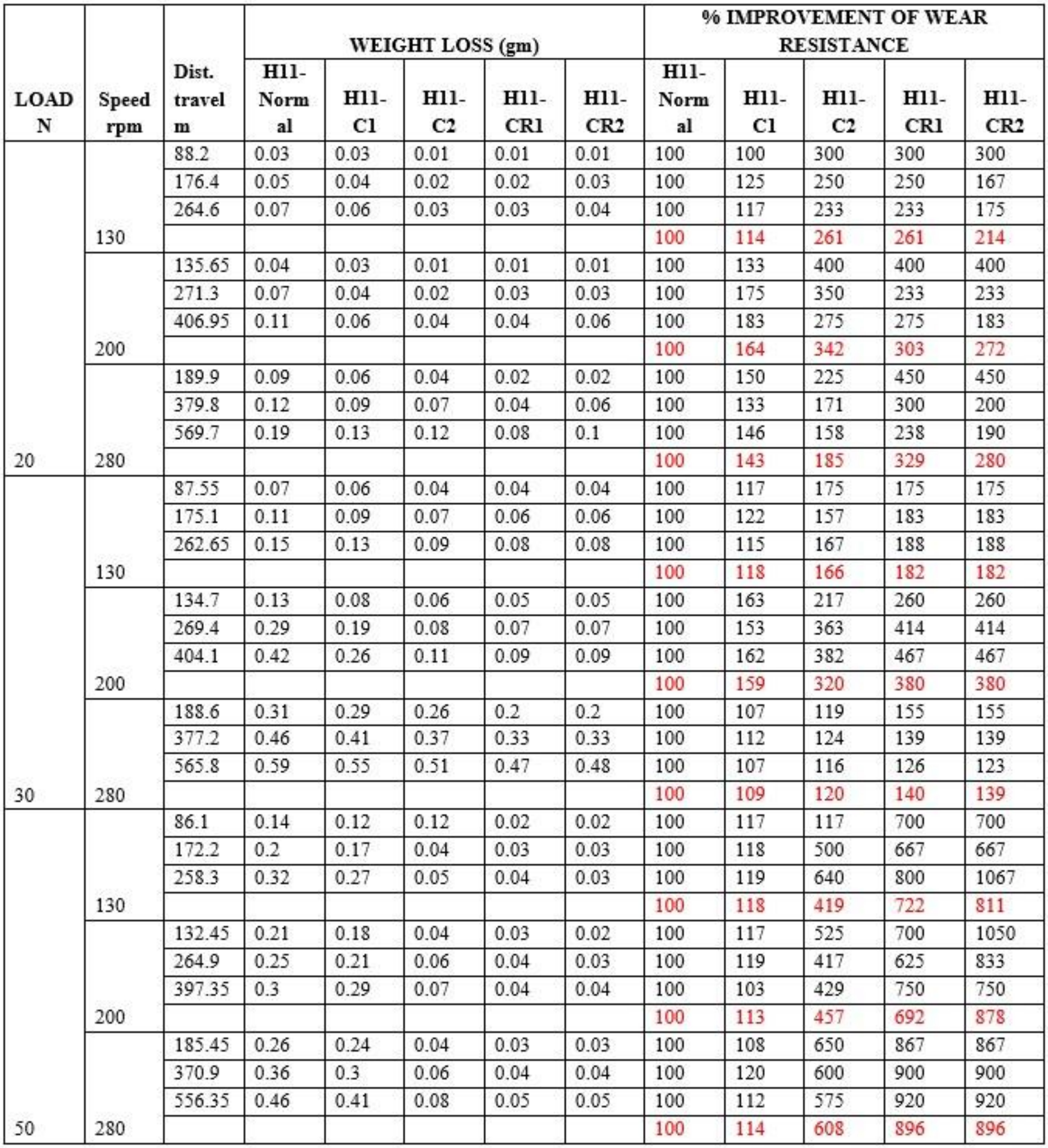
3.1 Pin-on-Disk Wear Test

The method describes a laboratory procedure for determining the wear of materials during sliding using a Pin-on-Disk apparatus. Materials are tested in pairs under abrasive conditions. The wear test of the tool steels were carried out by changing the following parameters.

1. Load acting over the pin (20 N, 30 N, and 50 N)
2. Linear velocity of the pin by adjusting the rpm and radial distance
3. Continuous sliding time (5, 10 and 15 minutes)

The wear test was conducted for three separate sliding timings for the speed of 130 rpm, 20 N and weight loss readings were noted down for each pin with respect to travel timing. Then the rpm was set to 200 and 280 and the same procedure was repeated for each case. Subsequently the wear test was conducted for 30 N and 50 N loads.

The same procedure was followed for the wear test on untreated, Coldtreated [C1, C2] and Cryotreated [CR1, CR2] specimens and the results were tabulated in Table No. 4.1.



LOAD N	Speed rpm	Dist. travel m	WEIGHT LOSS (gm)					% IMPROVEMENT OF WEAR RESISTANCE				
			H11-Normal	H11-C1	H11-C2	H11-CR1	H11-CR2	H11-Normal	H11-C1	H11-C2	H11-CR1	H11-CR2
20	130	88.2	0.03	0.03	0.01	0.01	0.01	100	100	300	300	300
		176.4	0.05	0.04	0.02	0.02	0.03	100	125	250	250	167
		264.6	0.07	0.06	0.03	0.03	0.04	100	117	233	233	175
								100	114	261	261	214
	200	135.65	0.04	0.03	0.01	0.01	0.01	100	133	400	400	400
		271.3	0.07	0.04	0.02	0.03	0.03	100	175	350	233	233
		406.95	0.11	0.06	0.04	0.04	0.06	100	183	275	275	183
								100	164	342	303	272
	280	189.9	0.09	0.06	0.04	0.02	0.02	100	150	225	450	450
		379.8	0.12	0.09	0.07	0.04	0.06	100	133	171	300	200
		569.7	0.19	0.13	0.12	0.08	0.1	100	146	158	238	190
								100	143	185	329	280
30	130	87.55	0.07	0.06	0.04	0.04	0.04	100	117	175	175	175
		175.1	0.11	0.09	0.07	0.06	0.06	100	122	157	183	183
		262.65	0.15	0.13	0.09	0.08	0.08	100	115	167	188	188
								100	118	166	182	182
	200	134.7	0.13	0.08	0.06	0.05	0.05	100	163	217	260	260
		269.4	0.29	0.19	0.08	0.07	0.07	100	153	363	414	414
		404.1	0.42	0.26	0.11	0.09	0.09	100	162	382	467	467
								100	159	320	380	380
	280	188.6	0.31	0.29	0.26	0.2	0.2	100	107	119	155	155
		377.2	0.46	0.41	0.37	0.33	0.33	100	112	124	139	139
		565.8	0.59	0.55	0.51	0.47	0.48	100	107	116	126	123
								100	109	120	140	139
50	130	86.1	0.14	0.12	0.12	0.02	0.02	100	117	117	700	700
		172.2	0.2	0.17	0.04	0.03	0.03	100	118	500	667	667
		258.3	0.32	0.27	0.05	0.04	0.03	100	119	640	800	1067
								100	118	419	722	811
	200	132.45	0.21	0.18	0.04	0.03	0.02	100	117	525	700	1050
		264.9	0.25	0.21	0.06	0.04	0.03	100	119	417	625	833
		397.35	0.3	0.29	0.07	0.04	0.04	100	103	429	750	750
								100	113	457	692	878
	280	185.45	0.26	0.24	0.04	0.03	0.03	100	108	650	867	867
		370.9	0.36	0.3	0.06	0.04	0.04	100	120	600	900	900
		556.35	0.46	0.41	0.08	0.05	0.05	100	112	575	920	920
								100	114	608	896	896

3.2 Microstructure Analysis

The polished specimen (Mount) shown in figure 3.1 was used for studying the texture and viewing the grain boundaries. Figures 3.2 to 3.6 show microstructure of H11 steel used in this investigation consisted of a Fe- rich matrix with 0.34% of C, and alloy of Chromium and vanadium carbides. This was apparent from the microstructure taken at 1000X, the dark block spots were indicative of Fe distribution, white colour needle shapes were indicating carbide distribution and rest was carbon distribution (black colour dots). The transformation of retained austenite into martensite can be seen in the Cold treatment C1.The size and shape of carbides were reduced to maximum up to the cold treatment. The number of alloy carbides formed were more uniform and rounded in shape; martensite matrix appeared homogeneous and saturated at CR1 treatment condition. The microstructure of CR2 was not showing any further development of chromium carbides, but carbide precipitation had taken place, which offered more wear resistance for higher loads with high velocities. As compared to the structure of normal treatment and cold treatment, the cryogenic treatment CR1 showed larger number of secondary carbides, which resulted into uniform hardness and toughness on the surface. Also, it reflected in the improvement of specific-wear resistance at CR1 condition.

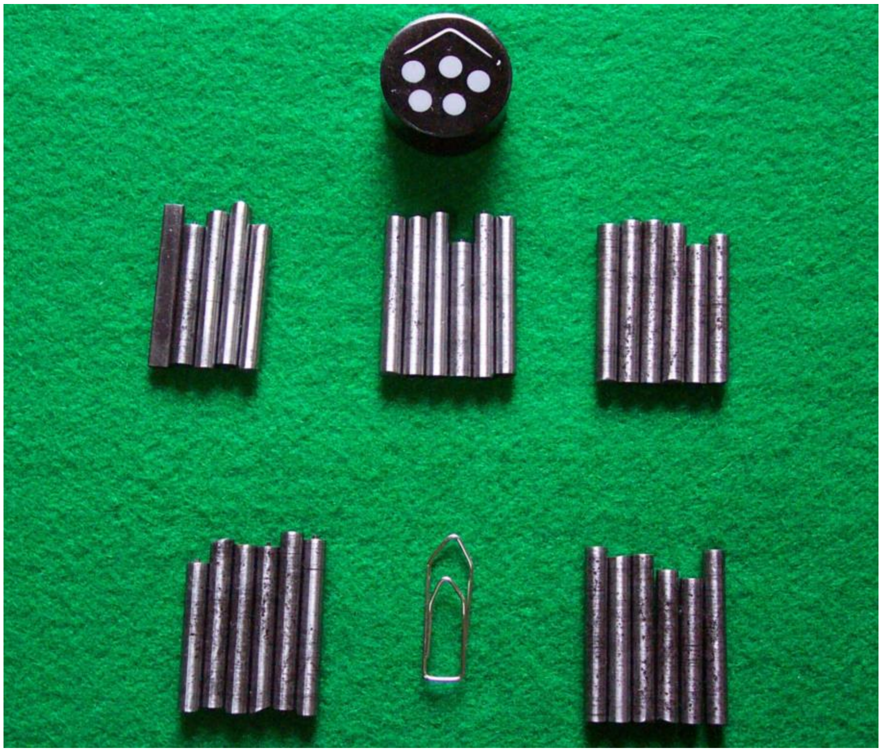


Fig. 3.1 Tested specimen of H11 with Mount

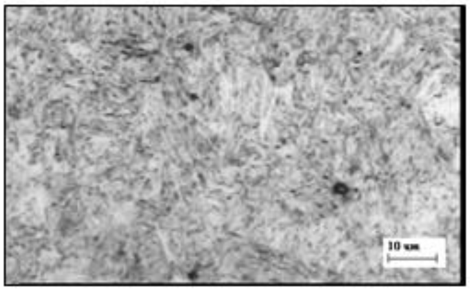


Fig. 3.2 Normal treatment

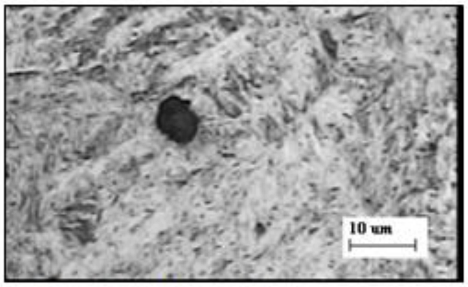


Fig. 3.3 Cold treated (-40 °C)

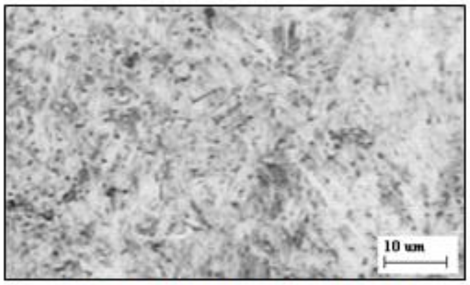


Fig. 3.4 Cold treated (-80 °C)

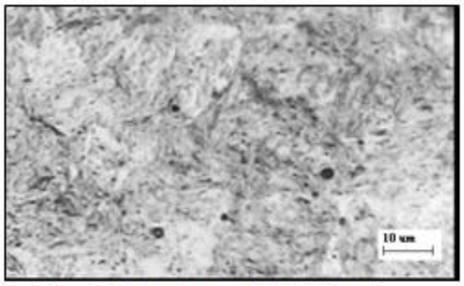


Fig. 3.5 Cryo treated (-150 °C)

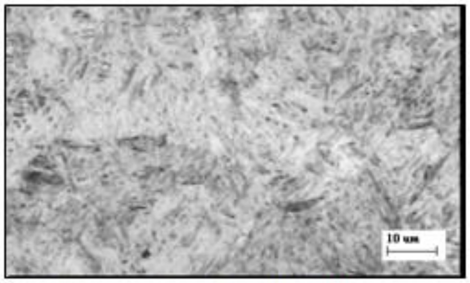


Fig. 3.6 Cryo treated (-190 °C)

4.0 Experimental Results and Discussions

H11 is Chromium hot work steel with a composition of medium carbon content of 0.34% and alloy of molybdenum 1.5%, chromium 5.1% and vanadium 0.38%. Chromium has property of good resistance to heat softening and also combined with its alloy creates great toughness. Similarly, chromium and vanadium combined with carbon creates more wear resistance even at high temperature. Since the carbide forming elements, chromium, vanadium did appear in the form of their carbides at low temperature, the specimens were treated stage-by-stage to cryotreatment namely Normal, C1, C2, CR1 and CR2 and maintained for 24 hours soaking period for all the categories.

From the microstructure of H11 steel shows in figures from 3.2 to 3.6, after being subjected to cold treatment as well as cryogenic treatment, the atomic spacing with in the freshly formed martensite decreases by the degree of cooling. The particle size distributed in Normal, C1, C2, CR1 and CR2 were not similar, which influenced the migration of carbon to fill the developed voids during the transformation of retained austenite to martensite. The size and shape of the elements were reduced and newly formed carbides were precipitated in the entire material.

A good amount of transformation of retained austenite to martensite took place at C2 treatment. Cryogenic treatment further reduced the presence of retained austenite to nearly negligible value. The migration of carbon got just started in C1 and ended in the cryotreatment CR1. The level of retained austenite present in the steel was evaluated and shown in figure 4.1. Cryogenic treatment was more responsible for the formation of secondary carbide like chromium and vanadium to create more toughness to the material, so that uniform hard martensite structure was formed. The precipitation of fine carbides and formation of secondary carbide as a result of cryotreatment CR2 was accountable for the improved wear resistance.

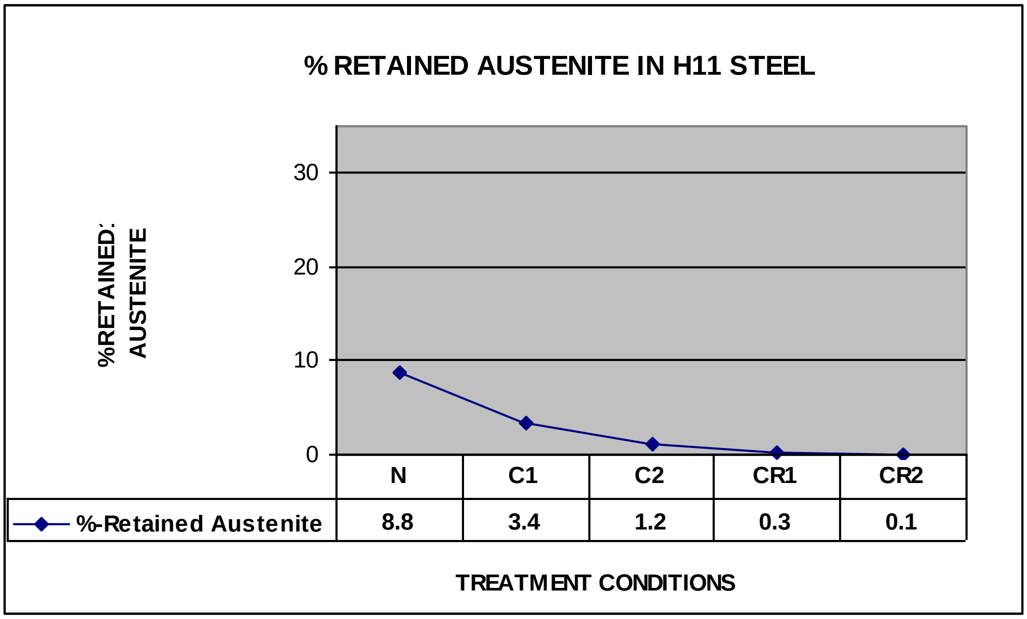


Fig. 4.1 Variation of %Retained Austenite with Treatment conditions in H11 Steel

The long soaking period helped the surface to get uniform temperature, which produced different thermal contraction as well as dislocation in the structure. So the free carbon molecules were developed and transported to fill in the voids. During high temperature treatment, such above mentioned transportation of molecules had not taken place. Since the formation of martensite generally not started while cooling, hence, internal stress got generated and created the voids, same voids again got filled up by the balance amount of retained austenite.

Figure 4.2 shows overall percentage improvement of wear resistance with respect to the level of retained austenite in the material due to different low temperature. In Normal treatment, there was no cluster of carbides present in the structure. In case of C1, due to the cooling, the retained austenite just started to get transformed to martensite, which can also be seen as migration of carbon surface in the microstructure. Moreover, the formation of carbide as well as migration of carbon had not yet started. But in case of C2, almost maximum amount of retained austenite get transformed into martensite. The level of retained austenite was slightly higher as observed. At the same time, formation of spheroidal shape carbides had just started. This indicated that improvements of wear resistance in C2 were caused by the transformation of retained austenite and little amount of carbide formation. Hence medium amount of improvement of wear resistance generated by C2 treatment.

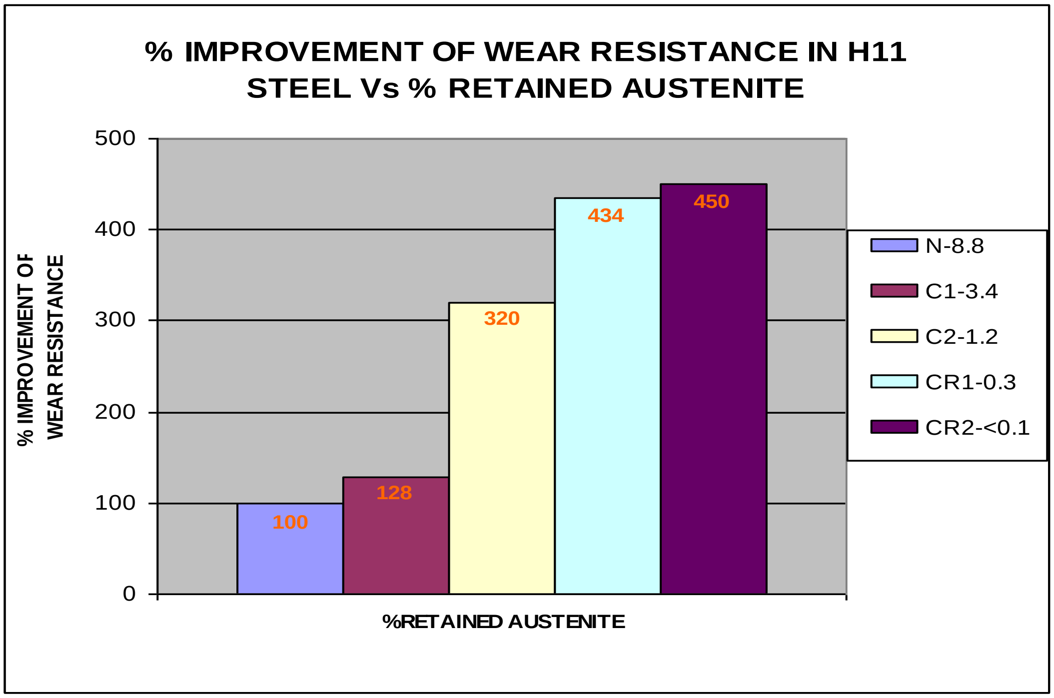


Fig. 4.2 % improvements of wear resistance against %-retained austenite in H11 steel.

In CR1, the improvement of wear resistance was quite high with less retained austenite compared to C2. This result showed that the improvement of wear resistance was not only due to the transformation of retained austenite; but also, it indicated other phenomenon like development of more carbides, reduction in size of carbide and other molecules that took place in the material under cryogenic treatment. The migration of carbon was completed at CR1. In case of CR2, carbide precipitation took place and surface of the material was hard martensite structure and homogeneous. Cryogenic treatment produced **very high** improvement in wear resistance even though percentage of carbon content was less.

Since the material was of a low carbon and high chromium alloy elements, the development of new carbides, migration of carbon molecules and saturation of transformation of retained austenite into martensite were completed in the CR1 treatment itself. The precipitation of alloy carbides along with carbon created more toughness to the material and produced hard martensite structure. Though the maximum wear resistance of the material was produced by CR2 treatment, the difference in percentage improvement of CR1 and CR2 was marginal. The extra percentage of wear resistance improved by the CR2 treatment on tools could easily offset the advantages gained when compared to time consumption and productivity of the same under CR1 treatment.

During the heat treatment, high residual stresses were generated at the time of critical temperature, which caused the material yield stress. Moreover, the tools having residual stress will cause damage in the tools and will reduce the life of the tool. Hence, tool and die steel are required to undergo low temperature treatment to get relieved of its residual stresses.

The level of residual stresses in the material under various low temperature treatments were evaluated and shown in figure 4.3. From the residual stress graph, it can be observed that cryotreatment drastically reduced the residual stress formed in the steel. Very high tensile residual stress was found in NORMAL and C1 treatments. However, tensile residual stress was reduced to maximum in cryotreatment and only a marginal difference was observed between CR1 and CR2. Since the %retained austenite and residual stress were of a smaller amount in cryotreatment, wear resistance was improved to highest value compared to Normal treatment.

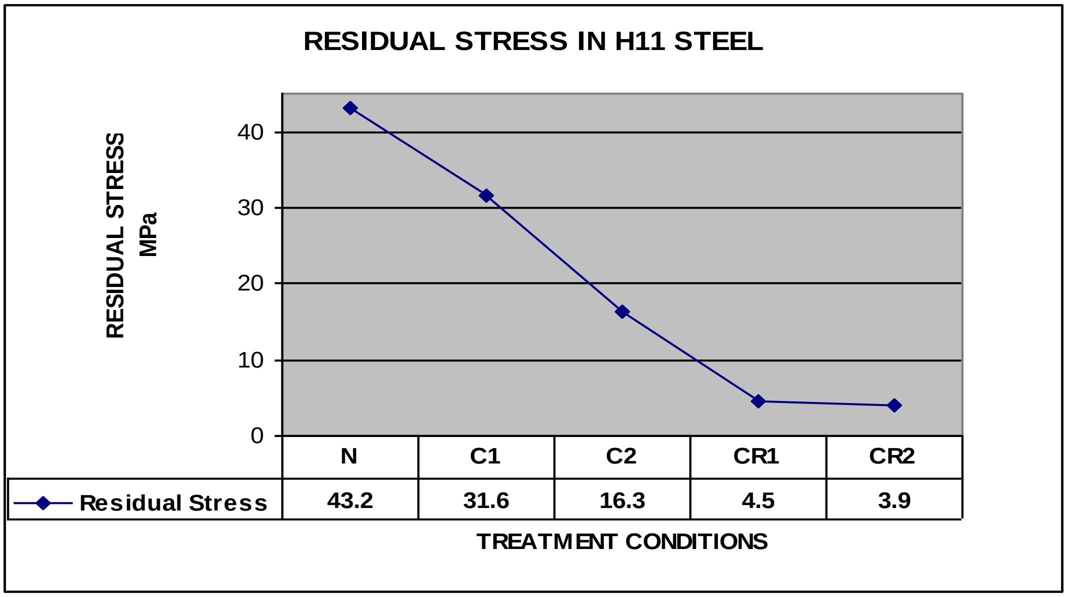


Fig. 4.3 Residual stress vs Treatment conditions in H11 Steel

Wear Rate of H11 steel with Distance Traveled

The experimental data at table no. 4.1 list the trends in abrasive wear behaviour of Normal treatment, cold treatment and cryogenic treatment of H11 specimen with different load, linear velocity and duration. With load of 20N at 17.64 m/min velocity, the wear rate was very less in C2 and CR1 than the cryotreatment CR2. The wear rate of C2 and CR1 were same for all the distances. In case of load of 20 N at 27.13 m/min which gave almost same behavior for C2 and CR1, 37.98 m/min gave less wear rate on CR1 than other treatment. Hence the cold treatment C2 was more suitable for 20 N load for all linear velocities than other sub-zero treatments.

With load of 30 N at 17.51 m/min, 26.94 m/min and 37.72 m/min, the wear rate of CR1 and CR2 were same for all speeds. For the load of 50 N at 17.22 m/min, 26.49 m/min and 37.09 m/min, the wear rate pattern was as equivalent to wear rate of 30 N load. The wear rate was same for CR1 & CR2 specimens and C2 was very close to CR1.

Fig 4.4 to 4.6 shows the bar graph of overall percentage improvement of wear resistance in H11 steel at 20 N, 30 N and 50 N loads respectively. The bar graph very clearly reveals that high wear resistance was observed for higher speeds for the load of 20N. In case of 30N load, up to 26.94 m/min offered maximum wear resistance after that it had failed to produce good wear resistance for all treatment conditions. Hence, it can be concluded that load of 30N, 26.94 will produce maximum wear resistance. But in case of 50N load, cryotreatment offered maximum wear resistance for the speed of 37.09 m/min. So, it is under stood that the performance of material will react at higher loads and high speeds. The maximum percentage improvement obtained at cryotreatement CR1 and CR2. The overall enhancement of wear resistance compared to normal treatment in each case was **28%, 120%, 334% and 350%** for C1 (233 K), C2 (193 K), CR1 (123 K) and CR2 (83 K) respectively. The maximum improved wear resistance was experimentally evaluated in cryogenically treated specimens because of the development of toughness and formation of uniform hardness surface, which could result the tool long life. Hence, this evaluation study of cryogenic treatment resulted that most of the development happened at CR1 treatment condition.

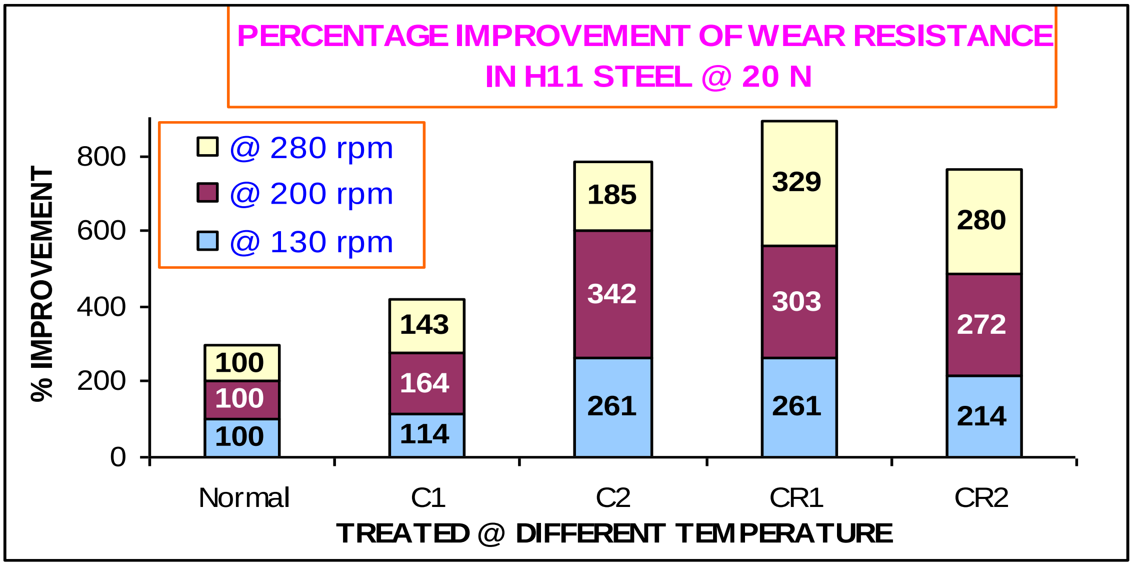


Fig. 4.4 Percentage Improvement of Wear – Resistance in H11 – 20 N

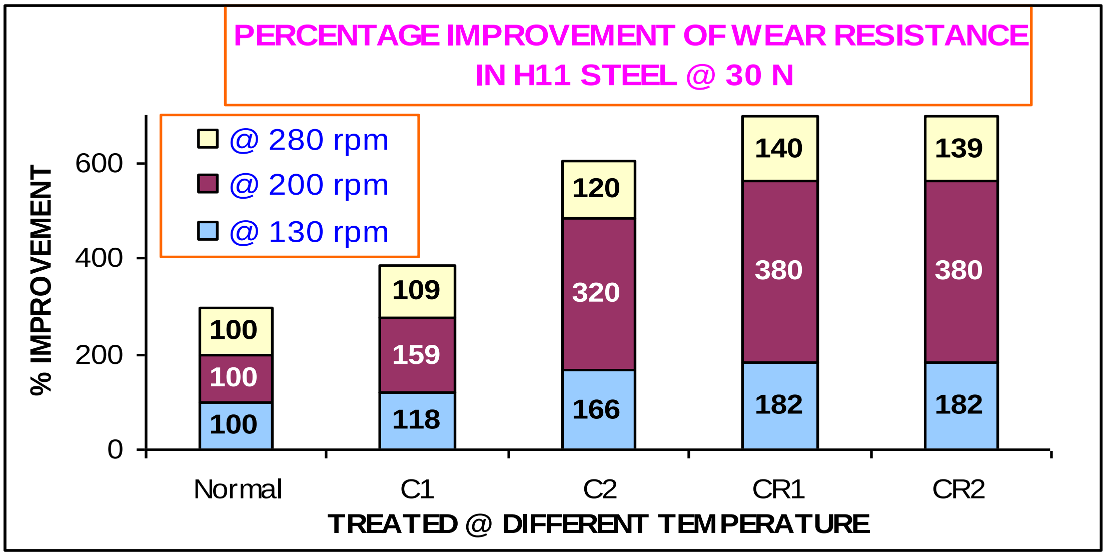


Fig. 4.5 Percentage Improvement of Wear – Resistance in H11 – 30 N

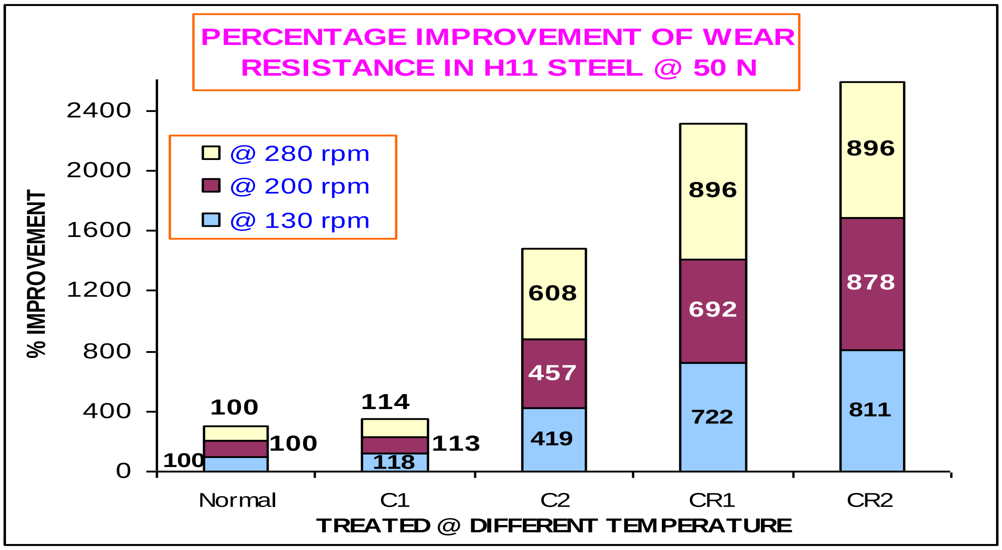
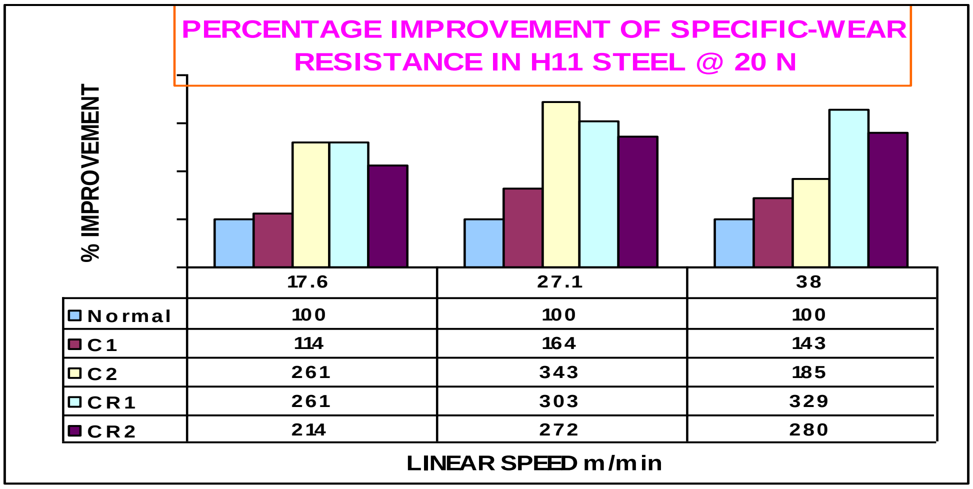


Fig. 4. 6 Percentage Improvement of Wear – Resistance in H11 – 50 N

Specific Wear rate of H11 Specimen with Linear Velocities

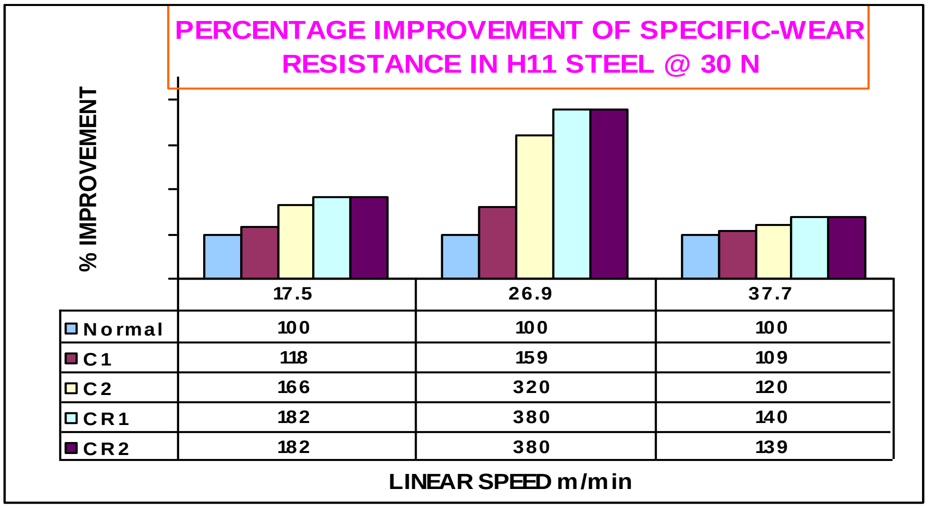
From the observation table no. 4.1, it was observed that the specific wear of the specimen for 20N, 30N and 50N decreases up to 27 m/min and then increases further for 20N and 30N loads. But in case of 50 N load, the specific wear had decreased further for higher velocities for all the five considered categories of the specimen due to the effect of chromium, vanadium and molybdenum at higher temperature. The gap of specific wear between normal treatment and cryotreatment was quite large for all cases.

Figures 4.7 to 4.9 show the bar graph of overall percentage of specific wear in H11 for the load of 20 N, 30 N and 50 N respectively. CR1 and CR2 shows almost equivalent performance in all cases and cold treatment II performance was just closer to cryotreatment CR1 specimens. **The maximum specific-wear resistance was obtained at cryotreatment I.** The overall enhancement of specific-wear resistance compared to normal treatment in each case was **27%, 98%, 126% and 115%** for C1 (233 K), C2 (193 K), CR1 (123 K) and CR2 (83 K) respectively.



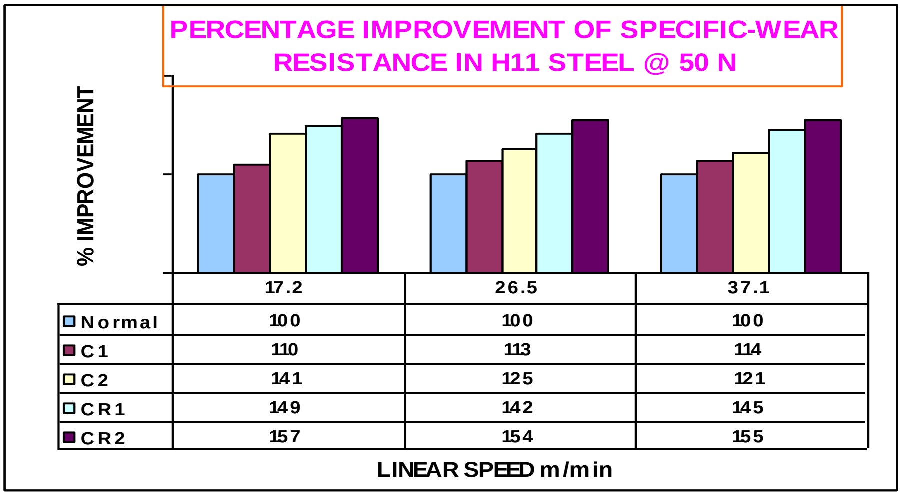
	17.6	27.1	38
Normal	100	100	100
C1	114	164	143
C2	261	343	185
CR1	261	303	329
CR2	214	272	280

Fig. 4.7 Percentage Improvement of Specific-wear Resistance in H11 at 20 N



	17.5	26.9	37.7
Normal	100	100	100
C1	118	159	109
C2	166	320	120
CR1	182	380	140
CR2	182	380	139

Fig. 4.8 Percentage Improvement of Specific-wear Resistance in H11at 30 N



	17.2	26.5	37.1
Normal	100	100	100
C1	110	113	114
C2	141	125	121
CR1	149	142	145
CR2	157	154	155

Fig. 4.9 Percentage Improvement of Specific-wear Resistance in H11at 50 N

Figure 4.10 shows the overall specific wear rate of H11 steel specimen in different treatment condition with respect to axial load, which was evaluated from the observation table. It was understood that the treatment condition plays a major role in the material to resist wear rate with respect to load applied on. Wear rate of NORMAL was 2.46 times at 20 N, 1.89 times at 30 N and 1.55 times at 50 N approximately higher than of CR2. Also, the wear rate of NORMAL was 2.38 times at 20 N, 1.64 times at 30 N and 1.27 times approximately higher than C2. These improvements had happened due to reduction in retained austenite as well as residual stress, formation of new carbides and secondary carbides in the material. Experimental results showed that Specific wear rate pattern was uniform and CR1 & CR2 were almost merged together for all loads.

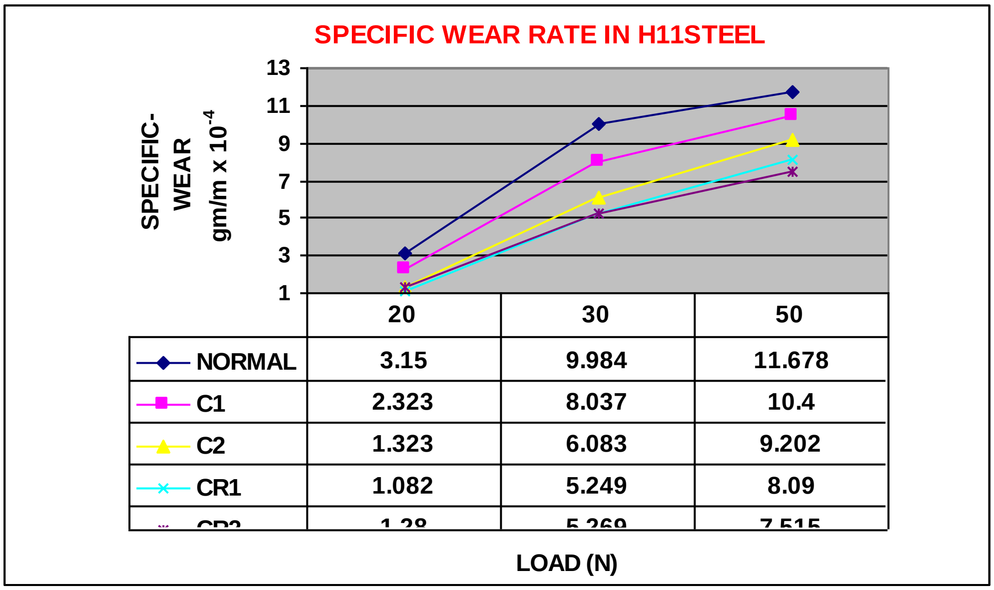


Fig. 4.10 Specific wear rates with different load in H11 steel

As a result of this work, the difference in level of percentage-retained austenite and residual stress between CR1 & CR2 was marginal; the experimental results also shows the marginal difference in overall percentage of wear resistance improvement in CR1 & CR2 and maximum development had taken place at CR1 condition. Hence H11 steel can be treated under Cryotreatment I (CR1) condition.

Conclusion

In this paper, the investigations on the effectiveness of Cold treatment and Cryotreatment at various low temperature conditions for 24 hours soaking period on wear resistance performance and hardness of H11has been reported.

1. The required degree of cooling was experimentally evaluated for obtaining maximum wear resistance in the material.
2. The results of study indicate that the metals, such as tool steels, which can exhibit retained austenite at room temperature, can have the wear-resistance significantly increased by subjecting the metal at C2 treatment condition.
3. It can be seen that there is a negligible amount of retained austenite and residual stress present in the cryogenically treated specimen that have undergone high temperature tempering.
4. There is a large-scale formation or dissolution of carbides present in the specimens at CR1 treatment condition.
5. Even though the cost of the Cryotreated tools is more than the untreated tools, the life enhancement due to this offsets the increased cost.
6. It is found that H11 tool when treated at 123K (24h soak), the texture got micro-refined and carbide were uniformly distributed.
7. The maximum percentage of life improvement obtained in H11 steel at cold treatment I was 183% at 20 N, 27.13 m/min, cold treatment II was 650% at 50 N, 37.09 m/min, cryotreated was 900% at 50 N, 37.09 m/ min and cryotreated II was 1067% at 50 N, linear velocity of 37.09 m/ min.

8. The overall percentage improvement of wear resistance of H11 steel at cold treatment I was 128%, cold treatment II was 320%, cryotreatment I was 434% and cryotreatment II was 450%, **so it is suggested that H11 steel may undergo for cryotreatment I instead of cryotreated II.**

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