

Effect of Cooling Rate on Properties and Microstructure during Solidification and Heat Treatment of Steels

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ABSTRACT

Steel is an alloy of iron and carbon. The properties of the steel can be controlled by various means of altering the composition and/or by adding alloying elements or through heat treatment process. Steel is well known to its strength and ductility, combination of which contributes toughness. The property can also be altered by controlling the cooling rate of the liquid melt during solidification of various steel group materials.

Solidification is a phase transformation process in which liquid is transformed into solid phase upon cooling by liberating the super heat and latent heat of fusion. The properties can be controlled by controlling the microstructure obtained after solidification in turn its properties. Microstructures are at the centre of materials science and engineering. They are the strategic link between materials processing and materials behavior. Microstructure control is therefore essential for any processing activity. One of the most important processing routes for many materials, especially metals and alloys is solidification.

An attempt has been made to review various literatures from many researches related to cooling rate of the liquid metal during solidification, many heat treatment processes and its effect on micro structural changes, mechanical and tribological characteristics.

Keywords: Microstructure, End chills, Carbon steels, Retained Austenite, Morphology.

1. INTRODUCTION

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Steel with very low carbon content is generally not responsive for heat treatment and has nearly the same properties as iron. It can be easily formed as they are quite softer. As the carbon content increases becomes stronger and harder by gradually losing its ductility. The steel and cast iron can be classified based on the carbon present in it. Various techniques through which the characteristics/properties of these ferrous based materials can be altered either by controlling rate of cooling of the liquid melt, or by adding alloying elements or also through various heat treatment techniques. The properties of iron and steels are linked to the chemical composition, processing path and resulting microstructure of the material. The metal alloys solidify over a range of temperature. Solidification behavior depends on parameters such as growth rate, temperature gradient and alloy constituents. Temperature gradient and growth rate influence the solidification morphology and solidification substructure respectively.

Growth rate or solidification rate is the rate of advance of the solid /liquid interface into the liquid. The rate of movement of solidification front determines solute redistribution during solidification, scale of solidification substructure and the growth under cooling. Rapid solidification (faster movement of solid/liquid interface) minimizes the tendency of segregation of elements temperature gradients both in solid and liquid are important.

Thermal gradient in liquid is more critical as compared with solid and is strongly affected by mixing in liquid temperature gradient in solid is diffusion dependent.

2. DISCUSSION ON COOLING RATE OF FERROUS BASED MATERIALS AND EFFECT ON ITS PROPERTIES

D.Candane et al [1] studied the effect of cryogenic heat treatment on tool steel of AISI M35 grade high speed steel specimen. Comparative study on conventionally heat treated and cryogenically treated M35 grade HSS specimen has been made.

Specimens initially subjected to conventional heat treatment at austenitizing temperature of 1200°C were subsequently subjected to shallow cryogenic treatment at -84°C for 8 hours and deep cryogenic treatment at -195°C for 24 hours followed by double tempering at 200°C. Presence of retained austenite was studied at the end of each of the above treatment using XRD analyzer. An estimated 19% retained austenite present at the end of conventional heat treatment was reduced to 5% at the end of shallow cryogenic treatment, while deep cryogenic treatment practically removed all traces of austenite in the sample.

Variation in mechanical properties such as toughness and hardness has been identified. There was no change in toughness due to cryogenic treatment and it corroborates well with the results of fractography. Wear characteristics were studied using pin-on –disc wear tester. The operative modes and mechanisms of wear have been identified as severe delaminative and mild oxidative from the morphology of worn surface of pin. The results unambiguously confirm enhancement in hardness and wear resistance of cryogenically treated specimens.

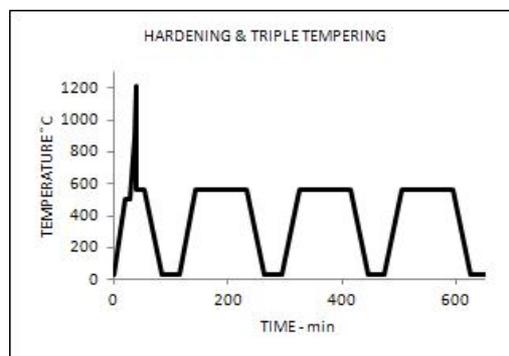


Figure. 1. Conventional heat treatment cycle followed for AISI M35 HSS.

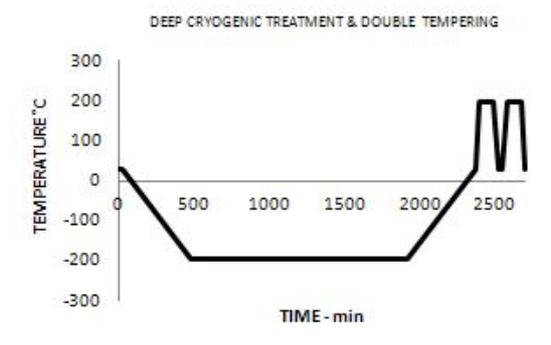


Figure 2 Cryogenic treatment applied to conventionally heat treated AISI M35 HSS specimens

D.Szeliga et al [13] designed the cast part with gating system and selected the SiC chill material. Cast specimen prepared by ceramic shell mould. On the basis of the temperature distribution measurements, the kinetics of the solidification process was determined in the thickened part of the plate cast. The authors present the mechanism of solidification and formation of shrinkage defects in casts with and without chills. It was found that the applied chills influence significantly the hot spots and the remaining part of the cast. Their presence allows creating conditions for solidification of IN-713C nickel super alloy cast without shrinkage defects.

Researcher reported that by using chills, it is possible to control the solidification process of the thickened part of the cast and create conditions for proper feed of the remaining cast volume. No shrinkage defects in the areas with larger walls thickness were detected despite the significant difference in thickness between them and the remaining part of the cast. Introduction of chill from one side of the thickened part of the cast allows obtaining the required cooling rate and creating conditions for cast solidification without formation of shrinkage defects.

P.R.Rao [2] attempted to discuss some of the available techniques of rapid solidification and the impact of rapid solidification on structure. Rapid solidification has yielded grain sizes as small as a few hundred angstroms. In most alloys grain diameters of the order of $0.01\mu\text{m}$ are attainable. Researcher also reported that many investigations have demonstrated the importance of fine grain size in reducing segregation, improving strength, ductility, fracture toughness and inducing super plasticity. there are two routes by which grain refinement can be achieved and they depend on either rapid heat extraction or on inducing large under cooling.

Sathees Ranganathan [3] experimented on rapid solidification of alloy systems and observed the characteristic behavior of these alloy systems. The rapid solidification behavior for alloys in the Fe-Cr-Mn-Si-Mo-C system was investigated for different system and cooling rates. Initially a complete featureless homogeneous phase was obtained for 2.85mm diameter rod for 72.8Fe-8Cr-6Mn-5Si-5Mo-3.2C alloy composition. In an attempt to extend the featureless phase by changing Mo, Cr and Mn yielded around 7.5mm diameter rod with complete featureless phase for 61.8Fe-15Cr-7Mo-8Mn-5Si-3.2C alloy composition. It is understood that featureless phase could be ϵ phase or austenitic solid solution through the experimental results and the literature review. However still an extensive crystallographic analysis should be performed to find out the exact phase. Heat treatment performed on the featureless phase revealed that bainite and secondary fine carbides starts to precipitate at low temperatures and almost complete around 700°C . Precipitation of bainite ferrites and fine carbides reveals that the best use of this material could be achieved with suitable heat treatment. High hardness observed in the featureless phase could be exploited to use in high strength materials. Production of featureless powders by water atomizing shows that extension of the featureless phase could be achieved for large dimension by sintering them together. However effect of temperature during sintering has to be investigated.

Yisheng Zhao and Xinming Zhang [4] investigated the effect of cooling rate on metallographic and hardness of bearing B-steel. The results showed that it consists of ferrite and pearlite formed in the initial stage of the transformation in the studied bearing B-steel fig.3 (a) cooled at $0.5^{\circ}\text{C}/\text{Sec}$. For the higher cooling rate than $10^{\circ}\text{C}/\text{sec}$, it consisted of bainite and martensite formed in the initial stage of the transformation fig (3b). All the results showed that the Hardenability of this alloy was sufficient to avoid any elevated temperature transformation which steel sheet was expected to be cooled at rates from 0.5 to $20^{\circ}\text{C}/\text{sec}$. The hardness of specimens was rapidly increased for the higher cooling rate than $10^{\circ}\text{C}/\text{sec}$. The peak value of the hardness was captured corresponding to the cooling rate of $20^{\circ}\text{C}/\text{sec}$ though its invariant then as depicted in fig(3e).



Figure3(a) it consists of ferrite and pearlite forms due to lower cooling rate $5^{\circ}\text{C}/\text{sec}$.



Figure 3(b) Bainite phase formed ($5^{\circ}\text{C}/\text{sec}$) consists of feathery needle like structure of upper or lower bainite formed.



Figure 3(c) consists of bainite (the light crystals) ($8^{\circ}\text{C}/\text{sec}$) and martensite (the dark crystals) formed in the stage.



Figure 3(d) consists of martensite structure (20°C/Sec) corresponds to a smaller number of crystals with granular morphology in the structure.

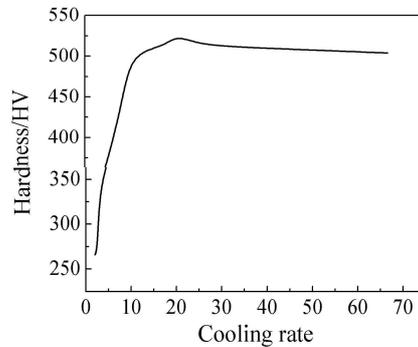


Figure 3(e) Effects of cooling rate on hardness of bearing-B Steel

Z.Zhuang et al[5] effort has been made to increase the cooling rate of solidifying melt by using water cooling mould. Researcher reported that both dendrite structure and equiaxed grain structure are obtained in cast Co-Cr-Mo alloy by control of the cooling rate of castings during the solidification process, to determine whether a significant improvement in the mechanical properties of the alloy can be obtained. The different structural characteristics of the two kinds of casting are examined by optical microscopy, SEM and TEM. These two kinds of castings produced through sand mould with air cooling and a metallic mould with water cooling of cast Co-Cr-Mo alloy, commercially known as Vitallium or H.S 21, were used. Tensile and fatigue tests as well as hardness measurement are carried out using individually cast test-pieces. Fracture surface appearance characteristics of tensile and fatigue specimens are also studied. Researcher concluded that the mechanical properties, including both transient and permanent properties of the equiaxed grain structure castings obtained by fast cooling are superior to those found in the coarse dendritic structure castings.

B.N. Leela and K.V.Srinivasa Rao [6] made an attempt to vary the cooling rate of Al-B4C composites cast using stainless steel cast iron and copper end chills. the microstructure and micro hardness of the chill cast specimens are analyzed and reported. It is observed that the chill material has a significant influence on the microstructure and properties of the cast specimens. Finer structure and better mechanical properties were observed with the specimens cast using copper chills, whereas, cast iron and stainless steel chills gave rise to coarse structure with reduced mechanical properties.

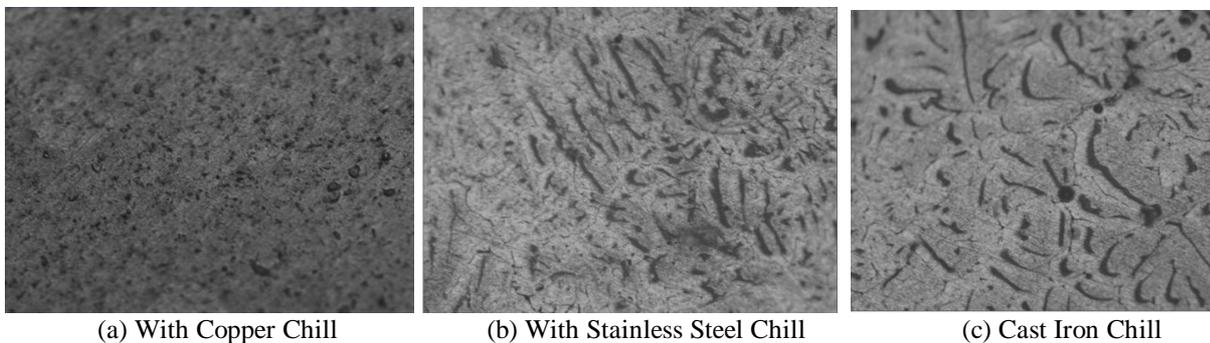


Figure 4. Microstructure of Cast Composites

The effect of cryogenic cooling on the mechanical properties of metal matrix composites has been studied by **Panchakshari H.V [7]**, the specimens which were prepared by liquid metallurgy technique were subjected to deep cryogenic treatment and properties studied and compared with as cast specimens. After deep cryogenic treatment of samples at liquid nitrogen temperature, the microstructure of specimens shows the change in distribution of

precipitates. The precipitate particles almost dissolved in the matrix and obtained very fine grain boundaries. The modification of microstructure of Al/Al₂O₃ MMC's due to cryogenic treatment shows significant improvement in mechanical properties. The preferred orientation of grains was sufficiently corroborated by XRD results of Al/Al₂O₃ composite before and after cryogenic treatment.

The cryogenic treatment changes the morphology of precipitate in both aluminium alloy and Al/Al₂O₃ MMC's. the coarse eutectic β phase present in the matrix material in MMC's resulted in the improvement of hardness of cryogenic treated samples compared with as cast samples. The fracture surface of cryogenic treated Al and Al MMC's is qualitative to shear band pattern which determines the homogeneity of deformation compared to as cast condition.

Titov Banerjee et al [22] addressed about the stone crushing and associated problems like environmental pollution with dust cloud, health hazards, noise and requirement of large quantity of electrical power. Researcher reported the alternative approach for stone crushing and size reduction process which would drastically reduce the above said problems. The authors assumed that in cryogenic temperature amorphous materials like building stones require very less impact energy for crushing with minimizing dust cloud, noise pollution, other pollutants and manual labour. the purpose here to establish an analytical model for estimation of energy requirement for crushing the stone under cryogenic conditions and also want to predict the critical time of exposure to cryogenic environment for accomplishing the crushing operation. The experimental results were tabulated by assuming the critical time of exposure 40 to 50 minutes to achieve thermal equilibrium to accomplish crushing operation.

Martin Herrea Treijo et al [8] experimented on solidification path and contraction behavior of three carbon steels and concluded that, the two of the carbon steels were of hypo-peritectic chemical composition, with 0.11%C and 0.15%C, while one of the carbon steels were hyper-peritectic with 0.16%C. the steels with 0.11%C and 0.16%C solidified as expected due to their chemical composition. In contrast, the chemically hypo-peritectic steel with 0.15%C solidified as hyper-peritectic steel, which was associated with the micro segregation of Mn. For the steel exhibiting a hypo-peritectic solidification path, peritectic transformation occurred at solid fraction values higher than 0.9, where it was assumed that the contraction generated in the mushy shell cannot be fed by the liquid. However, for steels exhibiting a hyper-peritectic solidification path, peritectic transformation began at solid fraction values lower than 0.9, where the contraction generated by the peritectic transformation was partly fed by the liquid. Hence, the highest cracking susceptibility was associated with the hypo-peritectic solidification mode.

W.puttgen et al [9] studied on rapidly cooled semi-solid state steels. This research reported that Iron based alloys quenched from the freezing range (semi solid state) exhibit microstructures which are quite different from their conventionally heat treated counterparts and display very interesting physical and mechanical properties. For the purpose of semi solid processing, it is important to know the microstructural parameters present in the semisolid state, in particular the fraction of liquid phase. Detailed structural characterization supported by the thermodynamic and diffusion calculations, demonstrate that in the quenched state their exact analytical determination is not viable. Even at very high cooling rates, the mobility of the phase boundary is very high and a differentiation of primary and secondary phases is impossible by means of conventional Metallography and electrode probe microanalysis (EPMA) measurements.

Semi solid metal forming requires precise knowledge concerning the microstructural parameters in the mushy state. For many light metals, the liquid and solid fraction and the size, shape and contiguity of the solid phase can be evaluated easily in the quenched from the freezing range condition. For iron based alloys, however, determining them is more difficult or even impossible because steels may undergo different phase transformations during cooling. Due to the high processing temperatures, diffusion during quenching is also of more importance. Here the researcher describes the phase formation during rapid cooling from the semi solid state of two different steel grades; tool steel X210CrW12 and bearing steel 100cr6. For both of these steels, the microstructures in the as-quenched state does not directly reflect the condition in the semi solid state and thus no metallographic evaluation of the microstructural parameters is possible. It is also shown here that the microstructure of the semi solid processed steels is completely different from that of the conventionally treated species.

Muzzamil Ahmed S. et al [10] studied on alloyed cast iron wear behavior. Researcher cast the specimen by using sand mould. Experiment conducted on wear behavior for all the 4 types of specimens viz, grey cast iron (GCI), chilled cast iron (CCI), alloyed cast iron (ACI) alloyed chilled cast iron (ACCI) surfaces. Their microstructures also studied by using different sizes of mild steel external chills to give chilling effect during solidification of cast iron and the conclusion were made that the wear is less within the combined effect of alloyed chilled casting. Only MSC (mild steel chill) effect leads to increase in the wear by two times than alloyed chilled cast iron and printed out that addition of only chromium is preferred with mild steel chill to increase the wear resistance.

It is also observed that machining near the chill end needs to have skill in selection of machine tool, tool material etc .It is observed by the micrographs and validations to the work done by other researchers that chilling effect drastically decreases after 8-10mm from the chill end.

Yogesh K.B and Joel Hemanth [11] reported that chilled austempered ductile iron has better tensile strength and hardness compared to casting obtained without using chill. Researcher conducted the strength test of the specimens obtained by sand mould technique using copper, graphite chill and without chill by adding Cu alloying element and concluded that tensile strength and hardness of the austempered chilled ductile iron is highly dependent on the chilling rate as well as addition of Cu content. An increase in the rate of copper chilling compared to graphite chill and without the chill material and increase in the Cu content of the material both result in an increase in tensile strength and hardness due to increase in the nodule count especially at the chill end. These properties are also significantly affected by bainite content of the material.

Also pointed out that the optimum tensile strength can be obtained in cast iron by increasing the amount of graphite-free area (in most cases, primary austenitic dendrites), refining eutectic cell size and establishing the pearlitic matrix structure.

Treating the base metal with commercial inoculants and neutralizing certain elements which inhibit graphite nucleation can result in the formation of more eutectic cells. By increasing the eutectic cell count, the effective span and stress concentrating effect of the graphite can be reduced, thus improving the tensile strength.

K.B Yogesh and Joel Hemanth [12] reported that the structure and properties of ductile iron are highly dependent on the solidification mechanism and chills are used to promote directional solidification to get sound castings. Researchers examined the series of micro structures and conducted the strength test and hardness for their material austempered chilled ductile iron containing Cu content varying from 0.5% to 4.2% which was sand cast using a variety of end chills (such as metallic and non metallic chills).The effect of cooling rate on the nodular size, nodularity, ultimate tensile strength (UTS) and hardness were evaluated. It was found that tensile strength and the hardness are highly dependent on the rate of chilling. Also predicted that strength properties and hardness of ADI cast with metallic chills has high values compare to non-metallic chills and cast without any chills.

Adnan Calik [14] investigated that the differences in the cooling rate appear to provide dramatic effects on the micro hardness of steels, depending on the carbon content of steels. the microhardness increases with the increasing cooling rate and carbon content due to solid solution hardening and formation of the martensite phase. Thus, heat treatment is one of the method to obtain desired properties of steels such as improving the toughness, ductility or removing the residual stresses.

Researchers experimented on the three type of steels, that are AISI 1020, AISI 1040 and AISI 1060 steels heat treated to 1250°K with a soaking period of 4hours and subsequently were cooled by three different methods. Different cooling rates, namely at room condition water quenching at room condition air cooling and furnace condition temperature cooling were applied to steels to observe the effect on the microstructures and micro hardness of steel. The researchers claiming that the increase in the micro hardness is due to the delay in the formation of ferrite which promotes the formation of pearlite and martensite at high cooling rate. Thus, the increase of micro hardness with the water quenched steels is because of increasing relative volume of pearlite and martensite after quenching.

Researcher S.O Seidu et al [16] investigated the effects of mould additives on the hardness property of grey cast iron. Five moulds with 25% iron dust,25% coal dust,25%saw dust and 25%iron dust, coal dust and saw dust additives were prepared respectively with silica sand using bentonite as a binder in wooden mould boxes. The melt was prepared with selected scraps which after superheated to 1555°C were tapped while 0.2%innoculant (Fe-Si) was added to the stream to allow uniform dissolution of the inoculants in the melt. Thereafter, the melt was quickly poured into the prepared moulds to avoid gasification of the inoculants where cooling and solidification occurred.

The highest hardness volume of 61.10HRC was revealed by mould with saw dust additive and lowest with coal dust additive with 42.10HRC.

The mould additives were found to convert essentially cooling and solidification rates of casting in the respective moulds investigated and since the rates were found to vary from mould to mould, microstructures with different morphologies were obtained. Hence effects of these additives on hardness characteristic of the cast iron were found to be equally different with the highest hardness value revealed by mould with saw dust additive and lowest with coal dust additive. However, the synergic effect of saw dust, iron dust and coal dust was found to be disadvantageous as excessive eutectic cells which are detrimental to improve hardness are produced.

Also the chemical composition of the scrap used for their study is shown in below table.

Table 1. Chemical composition of scrap from auto parts (wt. %)

C	Si	Mn	P	S	Cr	Ni	Al	Cu	Mo
3.9	1.9	0.8	0.08	0.13	0.16	0.05	0.05	0.13	0.01
7	4	7	8	1	3	8	8	7	5

Table 2. Chemical composition of the Fe-Si inoculants used

Element	Si	Ca	Al	Zn
composition	74.22	2.44	1.21	1.21

Table 3. Chemical composition of 0.2% inoculated sample

C	Si	Mn	P	S
2.783	3.252	0.313	0.158	0.063
Cr	Ni	Mo	Al	Cu
0.110	0.050	0.011	0.002	0.162

Simran preet singh gill et al [18] studied on cryogenically treated tool steel and reported that during cryogenic treatment of tool steel, the process modifies the carbon present in the tool steel. However, cryogenic treatment has not been widely adopted by the cutting tools industry due to lack of understanding of the fundamental metallurgical mechanisms and due to the wide variations in reported research findings. The researcher expressed that, the reasons for improving the mechanical properties of tool steels are transformations of retained austenite to martensite and precipitation of fine carbides.

Chang-jiang sang[17] investigated that the structure evolution and the solute distribution of 2mm thick strips of Fe-(2.6,4.2,4.7,7.9wt%)Ni peritectic alloy under a near-rapid solidification condition, which were in the regions of δ -ferrite single-phase, hypo-peritectic, hyper-peritectic and γ -austenite single-phase, respectively. The highest area ratio of equiaxed grain zone in the hyper-peritectic of Fe-4.7wt% Ni alloy strip was observed, while other strips were mainly columnar grains. The lowest micro-segregation was observed in the Fe-7.9wt% Ni alloy strip, while micro-segregation in the Fe-4.7wt% Ni alloy was the highest. As opposite to the micro-segregation, the macro-segregation of all the Fe-Ni strips was suppressed due to the rapid solidification rate.

3. CONCLUSION

Researchers[1] studied on effect of cryogenic treatment on properties of HSS grade steel material and compared to conventionally treated one and reported the enhancement of hardness and wear resistance observed in cryogenically treated specimens. Some researchers[13] worked on nickel super alloy (IN-713C) studied and reported the importance of chills to avoid shrinkage defects particularly at larger thicknesses relative to remaining/adjacent part of the cast. Few researchers [4] experimented on cooling rate of B steel and revealed the different microstructures obtained by varying the cooling rate in turn its effect on properties. Here researchers focused on solid-state transformation achieved during heat treatment with different cooling rates. Some other researchers[5] made an effort to vary the cooling rate of the solidifying liquid melt by using both sand mould and metallic mould and concluded that equiaxed grain structure obtained by metallic mould has superior properties than coarse grain structure castings obtained by sand mould with air cooling. Some of the researchers [6] experimented on aluminium composite to study the properties obtained by controlling cooling rates during solidification by using different chills made of copper, cast iron and stainless steel. Observed that copper chill has a significant influence on better properties than the other chills. An attempt has been made by few researchers [12] on cast iron material by using different chills (metallic and non-metallic chills) and reported that metallic chills given more significant effect on improving mechanical properties. Some of the researchers [10] also studied on cast iron material by using chills and by adding alloying elements, reported that, better properties obtained with alloyed chilled cast iron. Some researchers [16] worked with different mould additives employed in

preparing moulds and reported that mould prepared with saw dust additive is more effective to achieve high hardness than other additives. Also few researchers [14] made an attempt to vary cooling rate by employing different heat treatment processes on three types of carbon steels and suggested that quenched steels are having better properties.

There is an insufficient investigation found in the field of techniques to control the solidification of plain carbon steel group material by various means particularly by employing chills to achieve the desired microstructure in turn reflects on tailoring the properties. Hence in order to fill the void, further investigation is required to study the chilling effect on strength properties and wear characteristics during phase transformation from liquid to solid upon cooling the metal.

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