Quality Improvement in High Carbon Eutectoid Grade Steel Wire Rods for Tire Cord Application

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Abstract

High carbon eutectoid grade steel is widely used for tire cord application and requires low grain boundary carbides, low alumina and titanium nitride inclusions to avoid filament breaks during final wire drawing. Titanium nitride and alumina inclusions are non-deformable and have extremely high strength and could cause delamination and filament breaks during wire drawing. Cementite network at grain boundaries has been identified as a source of embrittlement in high carbon steels. Alumina and titanium nitride inclusions in high carbon eutectoid grade wire rods were therefore controlled in this study by the optimization of ladle slag chemistry, ferroalloy addition practice, argon bottom purging of ladle. Titanium nitride inclusions precipitate in steel during solidification even with low titanium and nitrogen content due to the effect of elemental segregation. This elemental segregation in billet was controlled by optimizing the continuous caster EMS parameters. Liquid steel super heat is controlled below 40 °C and continuous caster secondary cooling parameters were modified to reduce billet centerline carbon segregation and subsequent grain boundary carbide network in wire rods. Billet reheating temperature, stelmore conveyor laying head temperature and cooling pattern on stelmore conveyor at wire rod mill were also optimized to reduce the severity of grain boundary carbide network in wire rods.

Keywords: Titanium nitride inclusions, Alumina inclusions, Grain boundary carbide network, Eutectoid grade steel

1 INTRODUCTION

Tire cords are produced by drawing high carbon wire rods to fine diameters typically 0.15-0.35 mm and then bunching, twisting and stranding the wires to forms cords for tire reinforcement. High carbon eutectoid grade steel is widely used for making these tire reinforcing cords because of its high tensile strength and modulus. The presence of hard and angular non-metallic inclusions such as alumina and titanium nitride inclusions in high carbon wire rods are extremely detrimental and promotes microscopic cavities and delamination defect during wire drawing. The severity of titanium nitride inclusion of size 6 µm on fatigue properties of steel is equivalent to that of oxides with an average size of 25 µm [1]. In addition, central segregation and subsequent grain boundary carbide network in wire rods is also crucial for the improvement of product quality. Pro-eutectoid cementite network at grain boundaries has been identified as a source of embrittlement in high carbon wire rods [2]. Han et al. [2] studied the effect of carbon, silicon and vanadium on grain boundary cementite network and mechanical properties of high carbon pearlitic steels. Strecken et al [3] reported the interlamellar spacing of pearlite in high carbon wire rods in the order of 100 nm is required to achieve high drawing strains during wire drawing. Cooling rate of wire rod during stelmore cooling is therefore to be optimised to control pearlite interlamellar spacing. Yan et al. [4] investigated the effect of intensive secondary cooling, F-EMS and soft reduction on control of segregation in billets. It was reported in this study that final stirring and using an appropriate cooling rate are beneficial for effective control of network cementite and martensite in billets. Redesign of SEN, modification of refining slag, usage of aluminum free refractory during the vacuum process and improvement of the deformability of inclusions through the addition of B_2O_3 and alkali metal compounds are beneficial for the control of inclusions and helps to reduce subsequent drawing failures [4]. It has also been extensively studied the effect of chemical composition of liquid steel and cooling rate in continuous caster on the precipitation behavior of titanium nitride inclusions in high carbon steel [5-8]. However, plant operating practices and procedures are different for different steel plants and a systematic study is therefore required to improve the quality of high carbon eutectoid grade wire rods with a permissible level of alumina inclusions, titanium nitride inclusions and grain boundary carbides.

2 QUALITY IMPROVEMENT OF HIGH CARBON EUTECTOID GRADE STEEL WIRE RODS

The chemical composition of high carbon eutectoid grade SWRH 82A steel is shown in Table 1. This grade is widely used in tire reinforcement cord application due to its high tensile strength and ductility. High carbon SWRH 82A grade is produced at JSW Steel Limited, Bellary, Karnataka, India through Basic oxygen furnace (BOF) followed by argon rinsing station (ARS), ladle heating furnace (LHF) and casted into 165x165 mm square billets through continuous casting route. Billets are then reheated to rolling temperature (1000-1050 °C) in walking beam furnace and finally rolled to 5.5 mm diameter in various stages of wire rod mill followed by control cooling on stelmore conveyor to achieve required fine pearlite microstructure and specified mechanical properties. Wire rods are then drawn to 0.15-0.35 mm diameter at customer end to make tire reinforcement cord. It has been identified that high titanium nitride inclusions, alumina inclusions and continuous network of gain boundary carbides are major contributors for wire rod fractures during wire drawing. Therefore, plant operating practices have been modified in this study to control the hard, non-deformable and angular inclusions such as titanium nitride, alumina inclusions and brittle grain boundary carbide network.

Table 1. Chemical composition (weight percentage) of high carbon eutectoid grade (SWRH 82A) steel

C	Mn	Si	P	S
0.8-0.85	0.45-0.6	0.15-0.3	0.02 max	0.02 max

2.1 REDUCTION OF TITANIUM NITRIDE INCLUSIONS

Titanium nitride inclusions are non-deformable and have extremely high strength and could cause filament breaks and delamination during wire drawing [8]. The number and size of titanium nitride inclusions are therefore aimed to be reduced by reducing the titanium content in steel. Titanium is not purposefully added to this grade during steel making process and considered as a tramp element. Fig.1 shows the variation of titanium and nitrogen of liquid steel during the various stages of steel making. Titanium at BOF (basic oxygen furnace) tapping is very low and is in the order of 0.0005-0.0008%. Increase in titanium (pickup) is mainly observed after ladle additions during BOF tapping and processing at ladle furnace. From the chemical analysis of ferroalloys, ferrosilicon (FeSi) and calcined petroleum coke (CPC) are identified as significant contributors for titanium pickup in the steel making ladle. Therefore, low titanium (<0.02%) containing imported ferrosilicon and graphite fines are added to liquid steel for silicon and carbon respectively to reduce titanium pickup in the liquid steel. In addition, titanium alloyed grade processed ladles are avoided for processing this inclusion critical grade to prevent infusion of titanium into liquid steel from ladle glaze of previous heats.

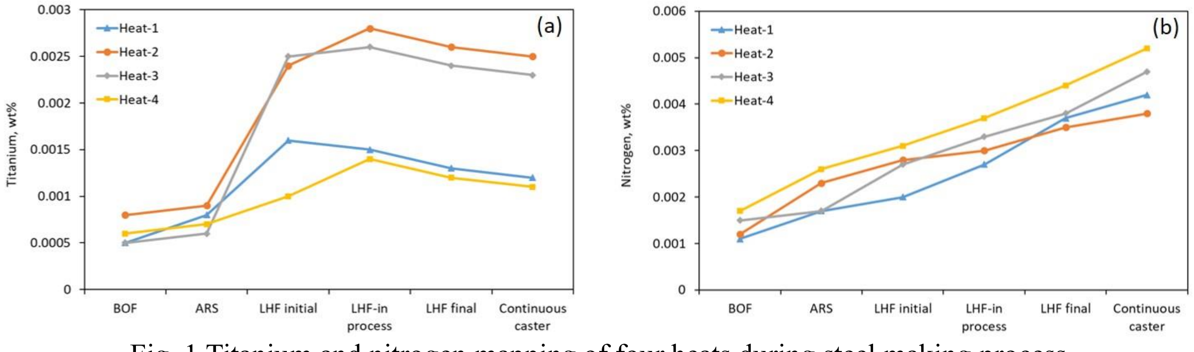


Fig. 1 Titanium and nitrogen mapping of four heats during steel making process

Titanium in liquid steel is considered as the limiting factor for the growth of titanium nitride (TiN) inclusions as it diffuses slowly in steel than nitrogen and enriches in interdendritic liquid regions [8,9]. The size of TiN inclusion in high carbon billet can be reduced by lowering the concentration of titanium in liquid steel and increasing the rate of solidification in secondary cooling zone of continuous caster. The flow rate of water in secondary cooling zone of continuous caster is therefore increased by 20% to increase the solidification rate of billet. In this study, titanium nitride inclusion size less than 4 µm was obtained by restricting the titanium in billet to 0.0015% maximum and increasing the solidification rate (cooling rate) in continuous caster greater than 50 K/min.

2.2 REDUCTION OF ALUMINA (AL_2O_3) INCLUSIONS

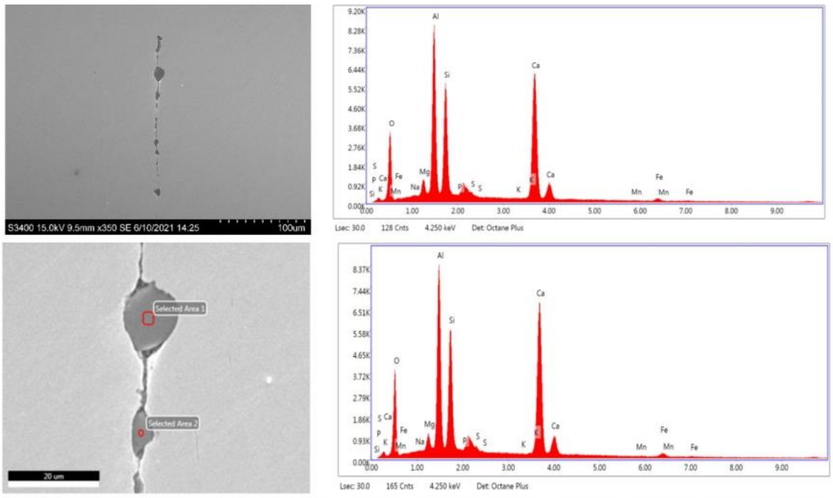
The breakage of wire rod during drawing is attributed to the presence of non-deformable inclusions with an incoherent boundary, centreline segregation, centre porosity and precipitation of grain boundary carbides in eutectoid and hyper eutectoid grade steels. Non-deformable inclusions in tire cord steel is therefore to be controlled to maximum 5 µm size with spherical morphology and should be uniformly distributed [10]. In this study, the size of non-deformable alumina inclusions was measured in 12 wire rod coils (diameter 5.5 mm) from three different heats of grade SWRH 82A and presented in Table 2. It can be observed from this table that many coils in each heat have alumina inclusions size greater than 5 µm which is detrimental for tire cord making through wire drawing.

The process of inclusion removal from liquid steel involves the growth and clustering of individual inclusions, movement of inclusions to top slag and absorption of inclusions by the liquid slag. Bath stirring and dragging action of rising bubbles are considered as two prominent factors that influence the removal of inclusions from liquid steel [11]. Grade SWRH 82A is silicon killed and aluminium is not purposefully added during the steel making process. However, alumina inclusions are significant problem in this grade which comes from ladle additions and refractory material. Argon purging/rinsing of liquid steel after ladle treatment with optimum stirring energy is an effective method of removal of non-metallic inclusions from liquid steel. Therefore, ladle bottom purging with argon for minimum 3 minutes with flow rate 10 Nm^3 with top slag refining is found to be effective method for the removal of non-metallic inclusions form liquid steel. Refining liquid steel through top slag with optimised slag composition is widely used method of achieving good plasticity of inclusions. With higher basicity and higher alumina content of slag, soluble aluminium increases. This is mainly because the increase in basicity, increases the activity of alumina in slag which is not conducive to reduce acid soluble aluminium in steel [12]. Therefore, the final inclusion composition should be controlled by controlling the slag alumina and basicity.

Table 2 Size of alumina (Al_2O_3) inclusions in high carbon SWRH 82A grade steel

Coil No	Size of Al_2O_3 inclusions, µm		
	Heat-1	Heat-2	Heat-3
1	5.2	3.65	7.84
2	10.3	8.02	-
3	-	3.04	15.6
4	2.1	-	2.58
5	-	9.54	9.74
6	6.5	4.42	-
7	14.1	6.26	4.23
8	3.7	3.54	6.75
9	-	12.03	-
10	4.3	5.25	3.45
11	-	2.05	8.20
12	2.5	-	10.45

Inclusion composition in wire rods is obtained in this study from SEM-EDS analysis and presented in Fig. 2. This figure shows that inclusions mainly consists complex oxides of Ca, Al and Si. This inclusion composition is plotted in SiO_2-CaO-Al_2O_3 ternary diagram as shown in Fig. 3 (a). It can be seen from this ternary diagram that inclusion composition is matching with di-calcium silicate stable region with melting temperature of 1500-1600 °C. This inclusion composition is aimed to be shifted towards pyroxene-anorthite stable region as indicated in Fig. 3 (a) to decrease the inclusion melting temperature to 1300 °C and increase the plasticity of inclusion. To achieve this, ladle slag composition is therefore modified from predominately di-calcium silicate to merwinite as indicated in Fig. 3 (b).



	O	Na	Mg	Al	Si	P	S	K	Ca	Mn	Fe
Area-1	45.92	0.00	1.41	16.18	11.80	0.48	0.86	0.00	22.40	0.05	0.91
Area-2	47.65	0.00	1.09	15.35	11.06	0.44	0.75	0.00	22.49	0.03	1.14

Fig. 2 SEM-EDS analysis of inclusions in wire rods

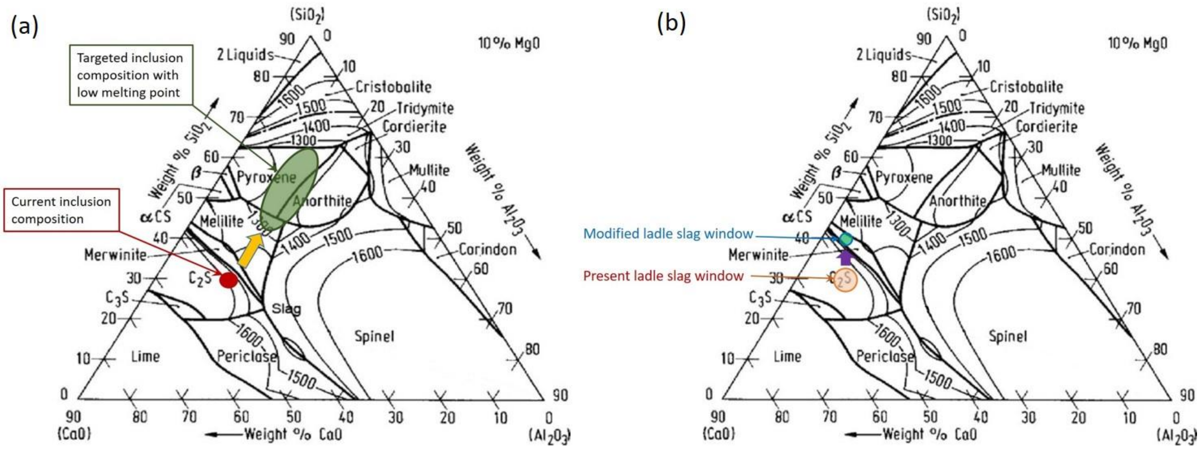


Fig. 3 CaO-SiO_2-Al_2O_3 Ternary phase diagram showing (a) current and targeted inclusion composition (b) present and modified slag composition

Ladle slag basicity (CaO/SiO_2) is maintained between 1.1-1.4 and alumina in slag is aimed at 2-5% to achieve the required slag window for making high carbon eutectoid grade steel. Quartzite is added to liquid slag during the initial stage of ladle processing to maintain required slag basicity and silica in slag in the range 35-40% as per the CaO-SiO_2-Al_2O_3 ternary diagram. In addition, ladle with alumina spinal refractory lining is replaced by MgO-C to avoid alumina inclusions from ladle refractory. Non-deformable alumina inclusions in wire rod are therefore reduced to acceptable level by ladle slag chemistry optimisation and maintaining the bottom purging of ladle with argon gas for minimum 3 minutes after ladle treatment.

2.3 PREVENTION OF GRAIN BOUNDARY CEMENTITE NETWORK

High carbon eutectoid grade steels are widely used in tire card application because of its high strength and modulus. However, continuous network of grain boundary cementite in the microstructure of this steel is a significant problem as it contributes to filament breaks during wire drawing and hence to be avoided. In this study, metallographic samples were prepared from wire rods of eutectoid grade steel as per the standard procedure given in ASTM E3. Samples then etched with a mixture of sodium hydroxide and picric acid at temperature 55-60 °C for 20 minutes for grain boundary carbide etching. Optical micrograph of grain boundary cementite network thus obtained in wire rod of eutectoid grade steel is shown in Fig. 5. Grain boundary precipitation of cementite as a continuous network is effected by chemical composition of steel, carbon segregation and rate of cooling from austenite temperature. Carbon content when increased to higher than 0.8% (eutectoid composition), cementite network forms at grain boundaries and causes embrittlement. Silicon when added to this grade inhibit the nucleation and growth of pro-eutectoid cementite during transformation. The formation of pro-eutectoid cementite at grain boundaries is a nucleation and growth process and certain minimum cooling rate is required to supress the formation of pro-eutectoid cementite network during continuous casting of billets and wire rod cooling on stelmore conveyor [12].

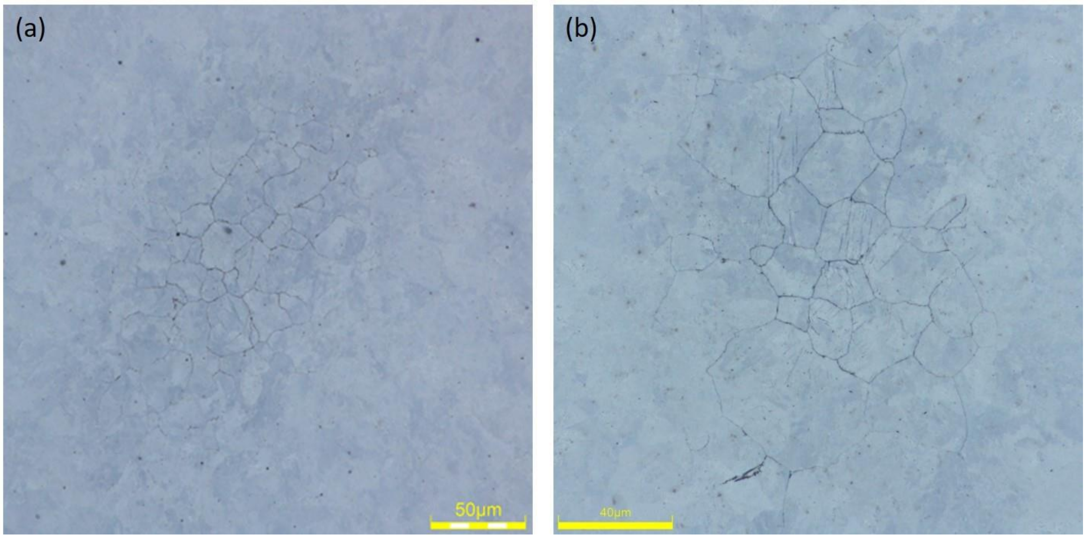


Fig. 4 Micrograph showing grain boundary network of cementite in wire rods of eutectoid grade steel

Precipitation of cementite at grain boundary is effected by carbon segregation in billets which in turn depends on liquid steel super heat, stirring intensity of electromagnetic stirrers and the cooling intensity in secondary cooling zone of continuous caster. Therefore, electromagnetic stirrer current was increased in this study from 250A to 350A and frequency was reduced from 3 Hz to 2 Hz. Zone wise secondary cooling water of continuous caster was increased by 20% to increase the cooling rate of billet and to reduce carbon segregation. Liquid steel super heat was maintained at 40 °C maximum to reduce carbon segregation in billets. In addition, laying head temperature of stelmore conveyor at wire rod mill was increased from 850 °C to 870 °C and cooling rate on stelmore conveyor was increased by fully opening blowers and increasing the stelmore conveyor speed from 45 m/min to 65 m/min. Increase in stelmore conveyor speed increases the ring spacing of coils on stelmore conveyor with less overlaps facilitating higher cooling rate for the suppression of grain boundary cementite network [12]. The ring spacing of coils on conveyor was also modified by optimising the individual speeds of different zones of stelmore conveyor. Continuous grain boundary network formation of cementite in high carbon eutectoid grade steel was thus eliminated by the modification of operating parameters of continuous caster and wire rod mill.

3 CONCLUSIONS

The presence of alumina and titanium nitride inclusions and continuous network of grain boundary cementite in high carbon eutectoid grade steel wire rods impose significant problem of filament breaks during wire drawing. Therefore, the following critical actions were implemented during the steel

making, continuous casting and wire rod rolling to improve the quality of wire rods.
(1) Low titanium nitride inclusions with size lower than 4 μm was obtained by controlling the titanium in liquid steel to 0.0015% maximum and increasing the cooling rate in continuous caster greater than 50 K/min.
(2) Alumina inclusions in liquid steel were reduced by optimising the slag chemistry and maintaining soft argon purging for minimum 3 minutes after ladle treatment with MgO-C lined ladles.
(3) The severity of grain boundary cementite network was controlled by optimising the liquid steel chemistry and controlling the operating parameters of continuous caster and stelmore conveyor.

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