Root cause analysis of formation of Black/Brown patch defect on cold-rolled steel sheet surface

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Abstract: Current thoughts as for thing quality rely upon the standard of best satisfaction of customer essentials. Leading the market by meeting the quality standards, is prime goal for any steel industry. Cold Rolling is assumed to have a significant role because of its exceptionally unpredictable procedures and its product end use in automobile industry. During the manufacturing of cold rolled steel sheet, different defects and imperfection are generally observed. Among them formation of "Black/Brown patch" defects on cold-rolled steel sheet surface, degrades the production quality yield which results in huge loss in term of customer satisfaction. In this paper root cause analysis of Black /Brown patch defect is evaluated by characterization using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Metallography, Destructive Hardness testing and wet chemical analysis are done for process parameters analysis. On basis of characterization techniques and process parameters data evaluation, root cause is gripped along with suggestion including controlling of different process parameters.

Index Terms- Cold Rolling Defects, Black Patch, Brown Patch, Rolling defects. Surface defects, Steel surface cleanliness, Defect Free Cold Rolling

INTRODUCTION

Current concepts regarding product quality are based on the principle of best satisfaction of customer requirements [1]. Rolling is not just thinning of material it actually enhancing the mechanical properties by altering the properties through strain hardening and by high pressure. In rolling material is deformed by the help of work roll along with backup roll. A certain pressure is set to control rolling process. Indeed, Cold rolling is counted among imperative process in metallurgical industry. Its operation directly marks the quality of the end product. The quality of steel sheets is as important in aesthetic as it is in its properties. This process consisting of multi-disciplines including, computing techniques, automatic controls, mechanics, materials engineering and many [2]. Control on the surface finish is an important concern in the manufacturing of cold rolled steel strip. Prior to cold rolling of hot rolled coil, coil is pickled in HCl concentration by passing at a specific speed through three tanks with different concentration of HCl to remove oxide layers from surface of steel. Reason for using three tanks of different concentration is that after the hot rolling of steel, as it happens at higher temperatures that is above re-crystallization temperature, oxide layers are formed on surface and these layers consist of three well defined layers of iron oxides. Adjacent to the base steel is the thick layer comprises of whustite having an estimated composition of FeO. The in between layer contain magnetite (Fe3O4) while the last layer is hematite (Fe2O3). The thickness of these layers is dependent majorly on the temperature of the strip at the exit of the final mill, temperature of the coiling and the accessibility of oxygen at the strip surface. The layer at the surface of the strip is richest in term of oxygen and constitutes 0.5 % to 2 % of scale thickness. The layer at the metal surface is richest in term of iron and constitutes about 85 % of the scale thickness. The intermediate layer of scale constitutes around 12 % to 14.5 % of scale depth [3].

The different iron oxides and metallic iron get reacted with Hydro chloric acid results in forming ferrous chloride ($FeCl_2$), H_2O, and H_2. The chemical reactions are as following [4].

$FeO + 2\ HCl = FeCl_2 + H2O$

$Fe_3O_4 + Fe + 8\ HCl = 4\ FeCl_2 + 4\ H_2O$

$Fe_2O_3 + Fe + 6\ HCl = 3\ FeCl_2 + 3\ H_2O$

$Fe + 2\ HCl = FeCl_2 + H_2$

Figure-1: Typical Black Patch Defect of Cold Rolled Sheet

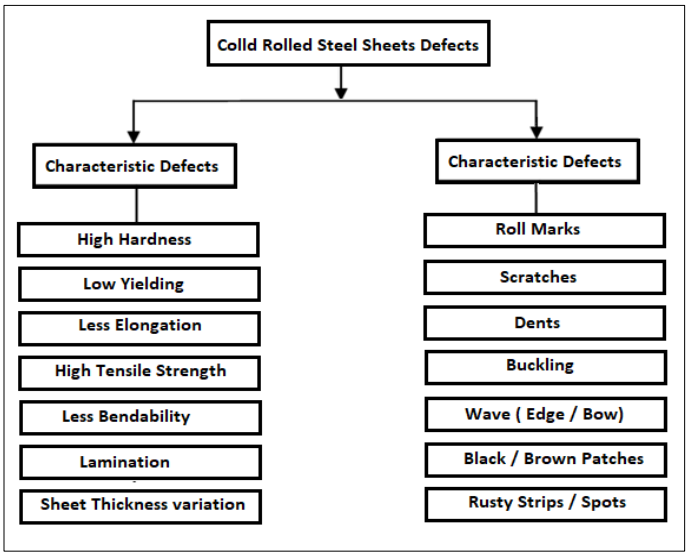


Figure-2: Some common defects of cold rolled steel sheets

These defects, as shown in Figure-2, are harmful from the quality perspective as the are not desired by the end customer. These defects create problem in mass production of manufacturing parts during the production. Every defects has its own nature, initiation point and initiation root cause. Similarly each defect can be minimized by focusing the individual parameters to root out the cause and eliminate it. Less the defects are more suitable is the sheet for the customers. A few defects are minor in their nature while many others including Black Patches are severe in its nature depending upon its quantum. Many dark spots on both surfaces of rolled sheets have been observed in the cold-rolling steel sheets during the manufacturing of sheets. Majorly in the shape of elongated patterns, varying in color from dark gray to black. This not only adversely affects the quality / physical appearance of the finished sheets, but also creates unfavorable conditions when coating the steel strip with zinc during galvanizing on the sheet. [5] For sheet metal used in automobile and manufacturing industries, cold rolled sheets over hot rolled sheets are preferred due to their excellent mechanical properties and surface finish. As A2-Wheeler, 3-Wheeler and Multi Wheeler cold rolled coils are utilized in manufacturing [6]. In cold rolling, metal working is done by passing sheets from roll Pairs. Coil proceeded for annealing is again processed for skin pass purpose which enhance yield point by cold reducing to less than 2% and eliminate fluting phenomenon during forming. Also it Improves and Imparts strip flatness, roughness and surface texture.

RESEARCH AND EXPERIMENTS

Process which is followed for the cold rolling of low Carbon grades and interstitial free carbon includes pickling, when talking about pickling, several factors are included in this particular process which are time, acid concentration, inhibitor and iron content. Sheets appearance is of major concern in this process. More importantly residual elements on sheet surface can produce surface defects at later stages. Second main station is Cold rolling mill where sheet thickness is reduced by altering the mechanical properties. Here controlling of emulsion parameters are of important in terms of rolling friction including tank temperature, agitation and Emulsion stability index. After initial rolling sheet undergoes for batch annealing, where major focus is flow of hydrogen gas and remaining quantities of residuals on coils surface. Finally sheets come to Cold rolling skin pass mills again here coils are skin passed in presence of skin pass emulsion. Producing the final product.

Emulsion stability does affect surface quality and increased content of Chlorides decreases the emulsion stability. Lubricity of emulsion deteriorates when particle size of emulsion increases. Stagnant emulsion's water evaporates with increasing time and temperature. Oxides and residual emulsion rests on the sheet surface. Moreover, the defected spot region was prone to face corrosion compare to clear area under the similar conditions. [7]. The increased Chloride content in terms of FeCl3 is not desirable on sheet surface. The water droplets in the rolling emulsion are very prone to react with Chlorides ion present on sheet surface to form oxide which are rare cause of black patch generation. The maximum allowable limit by the process manual is 3mg/m2.[8]

After thorough study, literature review and process Technical Specification manual study. The suggested remedies were put into real time scenario and experiments were done on different aspects to determine the defects trend. The experiments include

1- Microscopy analysis
2- Hardness testing
3- Spectroscopy
4- Scanning Electron Microscopy (SEM) Analysis
5- X-Rays Diffraction (XRD) Analysis
6- Residual chloride analysis.

A sufficient number of coils were selected as samples for analysis of each test and the subsequent results were recorded to analyses the affect.

1- Stereo-microscopy:

A stereo microscope is an optical microscope that provides a three-dimensional view of a specimen. It is also known by other names such as dissecting microscope and stereo zoom microscope. A stereo microscope uses reflected light from the object. It magnifies at a low power hence ideal for amplifying opaque objects. Since it uses light that naturally reflects from the specimen, it is helpful to examine solid or thick samples.. Opaque objects like coins, fossils, mineral specimens, insects, flowers, etc. are visible under a dissecting microscope magnification.

Visual examination was done to examine the surface appearance before stereo-microscopy. Surface appeared with black patches not feel by hand any rough surface or debris. Defected sheet was analyzed under low magnification stereo Microscopy to visualize the surface irregularities. With slightly different viewing angle part with black shade and non-black shade area was examined along with pictorial record was maintained. Below Figure 3

showing images of defected surface on various magnifications,

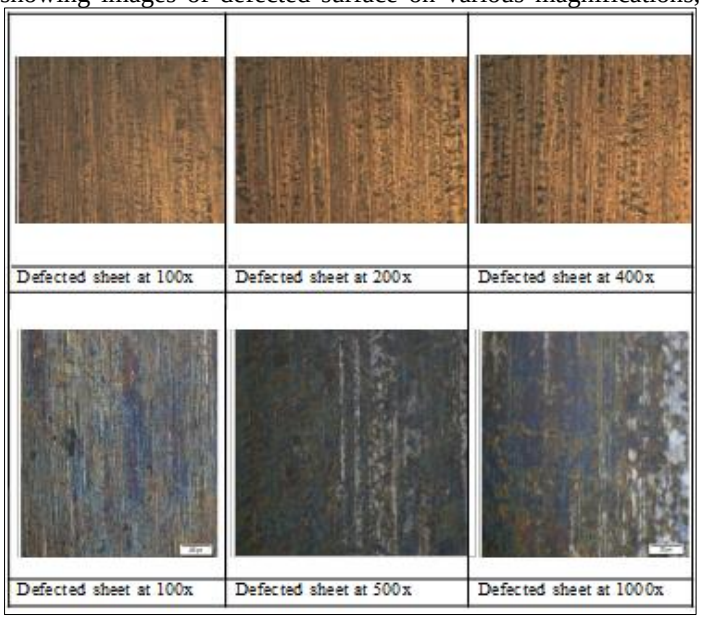
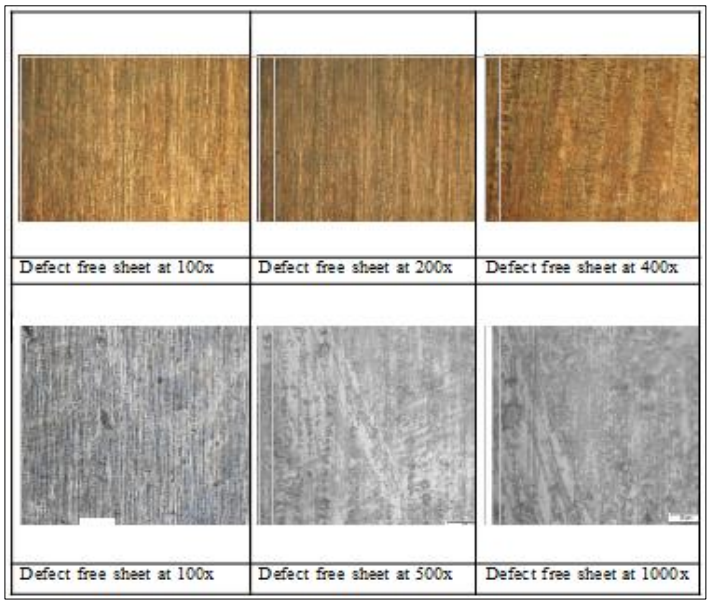


Figure-3: Metallographic images of defected sheet on different magnifications

Defect Free sheet samples results showed a clear surface in microscopic results. That indicates that this is not something just above the surface but also affected the sub-surface of the material as shown in Figure-4.Defected sheet surface was polished to 4um depth to investigate the depth of the impact left behind by the Black patch. That suggest that this defect has something defiantly to do with chemical reaction phenomenon. The Physical stickiness of the black patch would give a clear result of the surface upon microscopic examination after the 4um deep polishing.



magnification selected were as 100x, 500x and 1000x.

Figure-4: Metallographic images in defect free sheet on different magnifications

2- Hardness:

Hardness is the property of material to resist a penetration. To see effect of the formed Black layer on mechanical properties of the sheet, Micro-Vickers (Superficial) hardness testing was adopted on specified loads.

Table-1: Hardness results of Black patch portions

Sheet No.	Hardness (HRB)
Sample Sheet-1- Defected	49
Sample Sheet-2- Defected	53
Sample Sheet-3- Defected	49
Sample Sheet-4- Defected	55
Sample Sheet-5- Defected	48
Sample Sheet-6- Defected	46
Sample Sheet-7- Defected	53
Sample Sheet-8- Defected	54
Sample Sheet-9- Defected	50
Sample Sheet-10- Defected	53
Sample Sheet-11- Defected	49
Sample Sheet-12- Defected	54
Average Hardness	**51**

The results of the defect free portions of the same sheet are not very far from the results obtained on the black patch portion.

Table-2: Hardness results of Clear portion of the black patch sheets.

Sheet No.	Hardness (HRB)
Sample Sheet-1- Defect Free	47
Sample Sheet-2- Defect Free	48
Sample Sheet-3- Defect Free	49
Sample Sheet-4- Defect Free	51
Sample Sheet-5- Defect Free	44
Sample Sheet-6- Defect Free	45
Sample Sheet-7- Defect Free	47
Sample Sheet-8- Defect Free	42
Sample Sheet-9- Defect Free	52
Sample Sheet-10- Defect Free	50
Sample Sheet-11- Defect Free	54

Sample Sheet-12- Defect Free	47
Average Hardness	**48**

There is an average of 3HRB difference in the defected and defect free portion. That indicates that this black patch is minor effective on the mechanical properties of the sheets.

3-Spectroscopy:

Spectroscopy is the process of finding out the chemical composition of the material. This techniques is used to determine the major elements on the steel surface of the defected sheets. This will help out to identify the possible chemical activity on the defected surface. Both surfaces (defected and defect Free) were analyzed to check the variant in elemental composition Aisha steel mills quality department laboratory. Sample sheets to be examined were tested by surface grinding at 60 grade paper. Average of five values were taken for examinations.

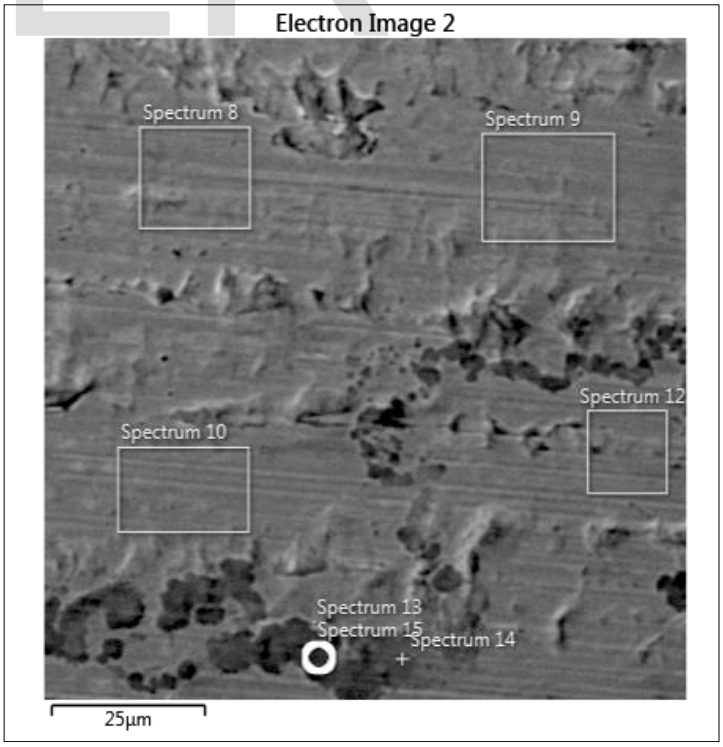
Table-3 Spectroscopic results of Defected and defect free Sheet samples

S. No	Element (%)	Defected sheet	Defect free sheet
1	C	0.076	0.084
2	Si	0.022	0.023
3	Mn	0.2	0.2
4	P	0.014	0.014
5	S	0.004	0.004
6	Cr	0.018	0.018
7	MO	<0.0010	<0.0010
8	Ni	<0.002	<0.002
9	Al	0.02	0.02
10	Co	0.006	0.005
11	Cu	0.013	0.012
12	Nb	0.009	0.011
13	Ti	<0.0010	<0.0010
14	V	0.002	0.002
15	W	<0.007	<0.007
16	Pb	<0.002	<0.002
17	Sn	0.001	0.001
18	Mg	<0.0010	<0.0010
19	As	0.012	0.015
20	Zr	<0.002	<0.002
21	Br	<0.002	<0.002
22	Ca	0.001	0.002
23	Ce	<0.002	0.002
24	S	0.005	0.005
25	Se	0.002	0.002
26	Te	0.001	0.001
27	Ta	<0.020	<0.020
28	B	0.001	0.001
29	La	<0.0005	0.007
30	N	0.019	0.029
31	Fe	99.5	99.5

Chemical composition of both defected and defect free portions have similar results. This indicates that there must be some non metallic oxide formation which can be further investigated through Scanning Electron Microscopy (SEM) and X-rays Diffraction (XRD) techniques. Main five elements of steel technology i.e. Carbon, sulfur, Manganese, Phosphorus and Silicon are in similar ranges. Additionally the over all impact of Fe Iron content remained the same i.e. 99.5%.

4-Scanning Electron Microscopy (SEM)

To generate an image scanning electron microscope (SEM) scans, over a surface, a focused electron beam. The electrons beam interacts to specimen, results in various signals by which information like topography and composition can be obtained. The human eye can differentiate two points 0.2 mm apart In sufficient light, without in addition to aided lenses. It is resolving power or resolution of the eye. Samples of Black Stains surface were examined under SEM technique to determine the elemental analysis of the surface matter as well as the surface analysis on closer level.



Picture-5: SEM Image Result of the Black Patch surface of sheet

Spectrum-14 and Spectrum-15 from the Figure-5 were deeply analyzed to determine the constituents. Spectrum results are available in Figure-6 and Figure-7. These graphs are helpful to find the root cause of the black layer formed on the defected steel sheet surface.

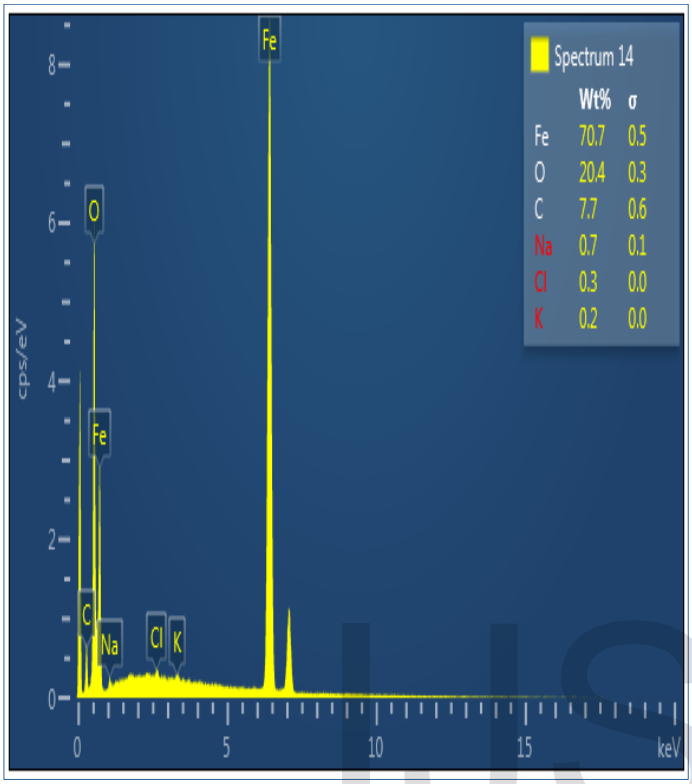


Figure-6: Spectrum-14 Results of the Black Patch surface sheet

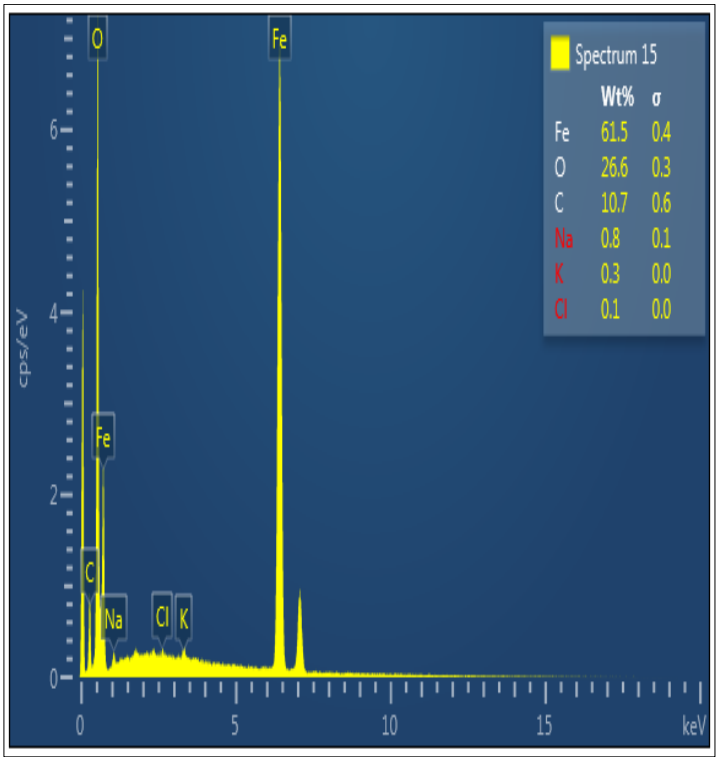


Figure 7: Spectrum-15 Results of the Black Patch surface sheet

Results suggests a highlighted group of elements like sodium, Potassium and Chlorine present at the surface having Black/ Brown Patch. To properly analyze the spectrum of defect free portion has to be taken into account.

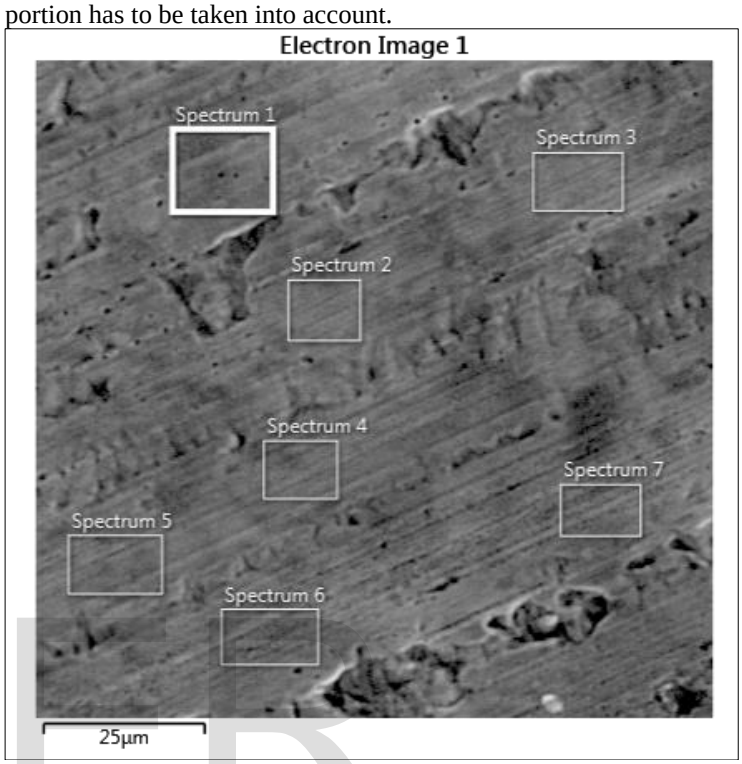


Figure-8: SEM Image Results of the defect free surface sheet

Spectrum-6 and Spectrum-7 from Figure9 were selected to investigate the results.

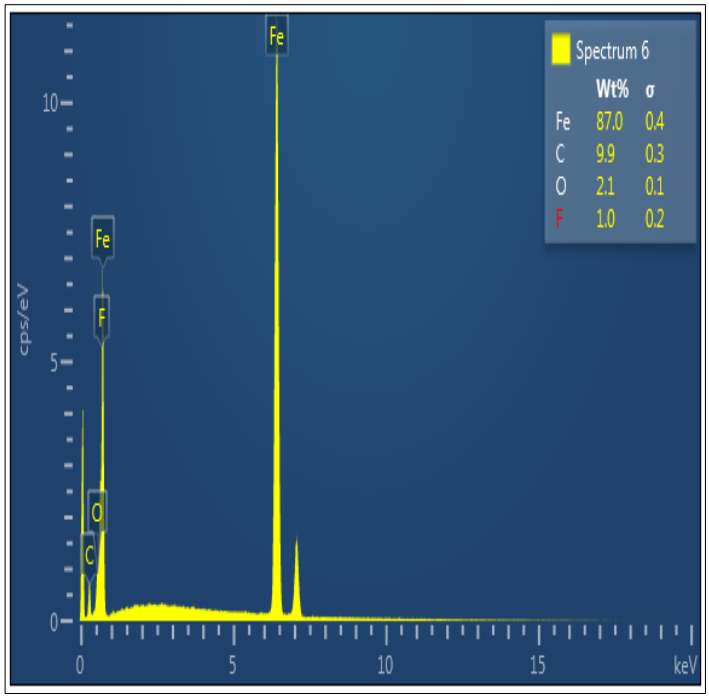


Figure-9: Spectrum-6 Results of the Clean surface sheet

Figure-9 specifies the spectrum-6 graphs of the clean surface of the steel sheet.

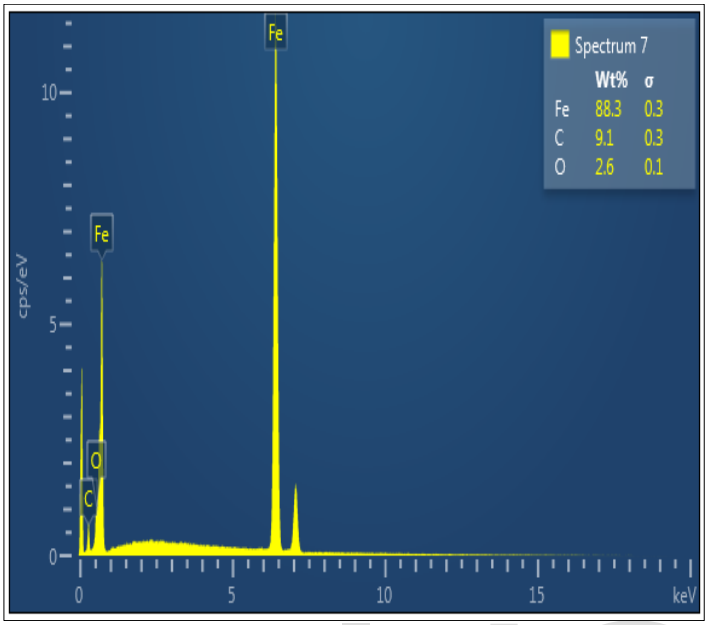


Figure-10: Spectrum-7 Results of the Clean surface sheet

If we compare both the spectrum-6 and Spectrum-7 as shown in Figure-9 and Figure-10 of clean surface and Spectrum-14 and Spectrum-15 of the defected potion of the sheet as shown in Figure-6 and Figure-7, there comes a clear idea of presence of additional elements of Sodium, Potassium and Chlorine. Therefore, Oxides of Chlorine and potassium can be the possible material provider of the Black Patch defect.

5- Residual Chloride Test

To identify the Chloride content present on the sheet surface, we opted a chloride determination test. This test provides the amount of chloride left at the sheet surface. After the analysis of SEM results, it was necessary to determine the chloride content of the defected surface and the defect free surface. This comparison will give us the clear affirmation of the SEM results.

It is to be considered that during pickling of the sheets before rolling, the Chloride can be gained by the sheet surface from the HCL acid liqueur which is used to clean the rusting of the sheet surface. We decided to randomly test a number of sheet sample from the pickled coils to determine the chloride content of the sheets ready for the rolling process.

Table-4-Residual chlorides on pickled coil appeared with defected surface

Sheet No	Residual Chloride (mg/m2)
Sheet Sample-1	8.2
Sheet Sample-2	16
Sheet Sample-3	13
Sheet Sample-4	8.9
Sheet Sample-5	4.2
Sheet Sample-6	4.3
Sheet Sample-7	16
Sheet Sample-8	3.9
Sheet Sample-9	7.8
Sheet Sample-10	6.6
Sheet Sample-11	8.1
Sheet Sample-12	3.5
Sheet Sample-13	7
Sheet Sample-14	7.2
Sheet Sample-15	8.3
Average	**8.2**

Residual oil contents on the sheets which have no Black Patch defects depicts the below mentioned results.

Table-5-Residual chlorides on pickled coil appeared with defect free surface

Sheet No.	Residual Chloride (mg/m2)
Sheet Sample-1	2.7
Sheet Sample-2	2.5
Sheet Sample-3	3.1
Sheet Sample-4	2.9
Sheet Sample-5	4.2
Sheet Sample-6	2.5
Sheet Sample-7	2.9
Sheet Sample-8	2.8
Sheet Sample-9	2.4
Sheet Sample-10	2.1
Sheet Sample-11	2.3
Sheet Sample-12	2.5
Sheet Sample-13	2.4
Sheet Sample-14	2.8
Sheet Sample-15	2.6
Sheet Sample-16	2.7
Sheet Sample-17	2.3
Sheet Sample-18	3.2
Sheet Sample-19	6.5
Sheet Sample-20	3.8

Sheet Sample-21	2.1
Sheet Sample-22	2.6
Average Chlorides	**2.9**

A clear difference of chloride content between the defected and defect free samples suggests that the lower concentration of chloride content leads to the defect elimination. The active chloride content must reflect some chloride ions in composition analysis to support these trends.

6- X-Rays Diffraction (XRD) Analysis

X-Ray Diffraction, frequently abbreviated as XRD, termed as non-destructive test method used to evaluate the structure of crystalline materials. XRD analysis, by way of the study of the crystal structure, is utilized to find the crystalline phases exist in a material and thereby reveal chemical compositional information. Both the Defected and defect free sheets were checked and analyzed.

Surface with defect was analyzed under XRD to evaluate the component of sheet surface, for defected sheet results were found as;

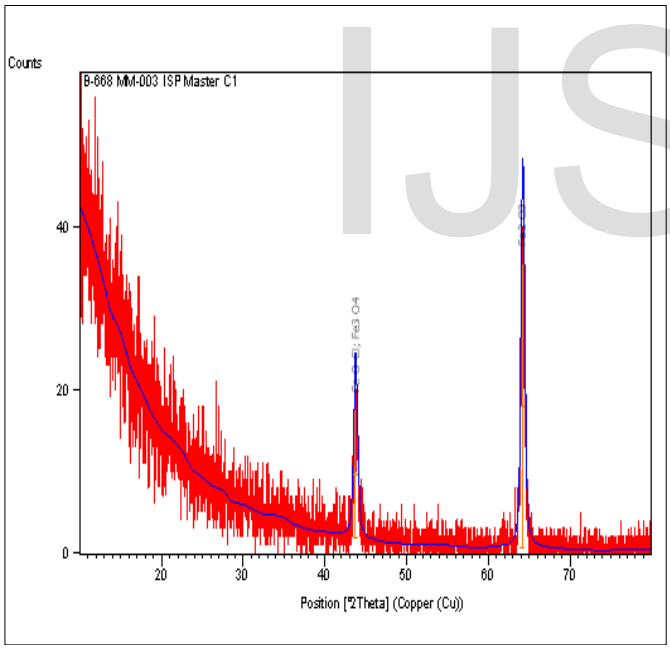


Figure-11: XRD Spectrum of Black Patch portion

Tab;le-6: Peak and pattern list of defected portion

Peak List					
Pos.[°2Th.]	**Height [cts]**	**FWHMLeft[°2Th.]**	**d-spacing [Å]**	**Rel. Int. [%]**	
43.72807	**15.657**	**0.566784**	**2.07016**	**44.9**	
64.17795	**34.868**	**0.495936**	**1.45122**	**100**	

Pattern List					
Visible Ref. Code	**Score**	**Compound Name**	**Displ.[°2Th]**	**Scale Fac.**	**Formula**
01-074-1369	**1**	**Iron Oxide Chloride**	**0**	**0**	**Fe OCl**
00-003-0863	**0**	**Iron Oxide**	**0**	**0**	**Fe3O4**
00-005-0637	**2**	**Iron Oxide**	**0**	**0**	**Fe2O3**

Compared with the above results Shown in Figure-11 and Table-6, the defect free portion of the sheet was also tested and results were obtained as defined in Figure-12 and Table-7;

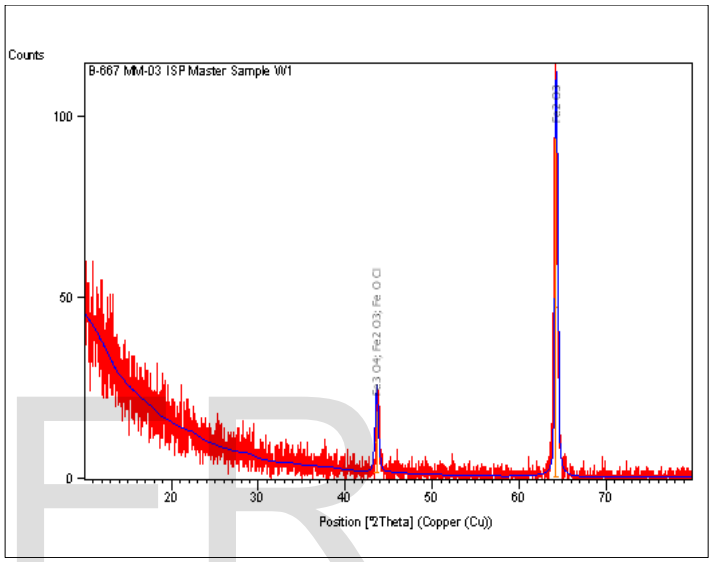


Figure-12: XRD Spectrum of Defect Free portion

Table-7: Peak and pattern list of defect free portion

Peak List					
Pos.[°2Th.]	**Height [cts]**	**FWHMLeft[°2Th.]**	**d-spacing [Å]**	**Rel. Int. [%]**	
43.59659	**16.87996**	**0.425088**	**2.07609**	**18.02**	
64.26965	**93.68073**	**0.283392**	**1.44937**	**100**	

Pattern List					
Visible Ref. Code	**Score**	**Compound Name**	**Displ.[°2Th]**	**Scale Fac.**	**Chem. Formula**
01-075-0449	**1**	**Iron Oxide**	**0**	**0**	**Fe3 O4**
00-039-1346	**0**	**Iron Oxide**	**0**	**0**	**Fe2 O3**
01-074-1369	**0**	**Iron Oxide Chloride**	**0**	**0**	**Fe O Cl**

A clear indication of FOCl compounds is visible in the spectrum of the defected portion. This indicates the possible activation of chloride ions carried by the sheet surface from the pickling or HCL cleaning process.

DISCUSSION

The black Patch defect at a sight looks like an aesthetic defect which can be neglected considering the application of the steel sheets. However, the phenomenon leading to generation of the black patch is not as simple as it looks apparently. To analyze the root cause of Black patch, Different dimensions were examined and results were deeply analyzed in a correlative manner to find out the initiation basics of the Black patch. Initially Microscopic examinations of the cold rolled steel sheet containing a black patch was performed to identify any significant change in surface appearance of the steel sheets. Additionally Metallurgical microscopic technique was also used to determine any significant difference at micro structure level. Microscopy and Metallurgical microscopic analysis showed surface appearance defected and non-defected surface to a magnification up to 1000x. No such variance observed between the two analysis. Both sheets containing Black patch and a clean surface sheets shown almost similar patterns of the surface. This negates an idea of any structural change of a phase transformation phenomenon to occur at the patch covered with Black stains. This confirms initially that the defects is of superficial nature and does not affect the characteristics property of the sheet. This was further assured by the mechanical properties tests of the both clean and defected sheet samples.

Mechanical properties of steel can be altered from the remaining sheet portion if the defect has a sever nature. Moving to the mechanical properties, hardness was taken into consideration to check if there is any significant change in the hardness of the defected portion of the sheets. Hardness of the different sheet samples with Black patch on surface and clean surface were tested on Micro vickers hardens tester. The results were almost alike. Hardness, taken from Micro Vickers hardness tester, of defected sheets and non-defected sheets resulted variance no more than 3 HRB that is average of 48 HRB of defected stained sheet whereas average 51 HRB of Defect free sheet. The closeness of the hardness range specifies that black stains defect does not impact on mechanical properties of the steel sheets. The table-1 and table-2 shows hardness values of both defected and defect free surface hardness taken on Micro Vickers hardness testes.

Any possible change in chemical composition of the sheet surface was taken into consideration. The change in composition of steel alloys at surface my result the service failure of the sheets. The spectroscopy of both detect free and defected steel surface were performed. Likewise hardness results, spectroscopic analysis of both steel sheet surfaces gave 99.5 % Iron, see table-3, in both the results along with Carbon, Manganese, Silicon, Phosphorous and Sulfur with almost similar composition.

Residual chloride content on steel sheet surface after pickling process has a significant role in Black stains generation. To analyze this, a number of steel sheets samples were taken and Residual chloride content was calculated. These tests were performed on both defected and defect free steels sheets surfaces. From data analysis, it was found that coil which were observed with defect of black patch were containing higher residual chloride on pickled surface average of 8.2g/m2 on pickled coil appeared with black patch defect, whereas 2.9 g/m2 on pickled surface found with no defects. table-4 and table-5 specifies the residual chloride values of steel sheets taken under consideration. The 8.2g/m2 is on a higher side which indicates the chlorides ion presence is enough to initiate a reaction leading to the black patch. The threshold or reaction equilibrium is an essential point to generate any reaction. The higher chloride content suggests that the reaction equilibrium has changed and the reaction has occurred that ended up with the generation of the products that has resulted Black stains on steel sheets surface.

SEM analysis is a deep probe into the Black patch generation. The elemental characterization of the sheet surface helped to find out any possible activity happened on sheet surface due to the process parameterize analysis of both defect free and defected steel sheet surface were taken into consideration. Moving forward towards the SEM analysis of Blackish Stains surface, the Spectrum 14 and 15 have elements a few different from those found on clean surface. The presence of Chloride ion is of special importance. This is the same chloride taken up by the sheet surface from the pickling tanks due to improper rinsing or cleaning of acid liquor. The presence of chloride shows the blackish stains are there as a result of a chemical reaction supported by the chloride ions leading to the formation of FeOCl and Fe3O4 along with the organic solvents at a higher temperature of 350-400 ^{o}C during annealing process of the sheet .

The SEM analysis of Clean surface and Black Patch surface has clear indication of difference of elements present on the sheet surface. Spectrum 2, Spectrum 6 and Spectrum 7 of Clean surface sheets shows the presence of Iron-Fe, Carbon-C and Oxygen-O. This indicate that the surface has maximum Iron content (88%) with a few carbon (2%) and 2% by weight oxygen. XRD results are confirming the presence of FeOCl on defected sheet along with Fe3O4.

CONCLUSION AND RECOMMENDATIONS

Black Patch defect appearing on cold rolled steel sheet resulting in decrement of prime quality, was analyzed. As per the result no bulk properties in term of Hardness, changed after appearance of this defect. Moving to the surface appearance it appears as patches random in direction but with no debris on surface. Several factors are considered during this probe which includes residual chloride on pickling surface.

As per data analysis coils with this defect, possess high residual chloride on sheet surface after pickling process. This loss of chlorides on sheet surface has major influence on the Black patch formation process by containing the Black Patch reaction.

Moreover, by wet chemical analysis, it was found that sheet with patch defect possess high amount of Fe in ppm where as compared to sheet with defect free surface. SEM analysis evident presence of Cl element on defected sheet as well as XRD traces of Iron Oxy Chloride (FeOCl) on defected sheet also Fe2O3 compound. Complete removal of Cl content and oxide layers after pickling is recommended for elimination of the defect.

It is recommended that the Rinsing (Water Cleaning) of the coils must be taken with care to reduce the residual chloride on pickled surface. The control of chloride ion concentration should be less than 3mg / L. To achieve this, wringer rolls at the end of all Acid concentration tank should be cleaned properly to avoid the strip surface residual chlorine ions.

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