J. Innovative Eng. Nat. Sci., vol. 1, no. 1, pp. 15-24, 2021.
http://dx.doi.org/10.29228/JIENS.49913

JIENS
Journal of Innovative Engineering and Natural Science
(Yenilikçi Mühendislik ve Doğa Bilimleri Dergisi)
journal homepage: https://jiens.org

Original Paper

Hot shortness mechanism and heat treatment of Cu containing carbon steel

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ARTICLE INFO

Article history:
Received: 12 March 2021
Received in revised form: 5 May 2021
Accepted: 25 May 2021

Available online

Keywords:
Hot Shortness
Heat Treatment
Tramp Element
Steel

ABSTRACT

Hot shortness mechanism in low carbon steels is studied with 1.0% wt. Cu containing steel. Slabs were prepared with scrap rebar steel and Cu billets. Slab was smelted in induction furnace and casted in mould. Casted sample was investigated with optical microscopy and chemical analysis was done with optical emission spectroscopy. Slab was heated up to 1200 ^oC and annealed for 1 hour before the hot rolling process. Hot rolling process was carried out with hot rolling press capacity of 25 tonnes for height reduction of 60%. Hot rolled slab was investigated with optical microscopy and scanning electron microscopy (SEM) techniques to observe the segregation of Cu. Hot rolled samples were heat treated at 800 ^oC, 900 ^oC and 1000 ^oC for 1 and 4 hours. Samples were cooled at air and water quenched. All samples were observed with optical microscopy and selected samples were examined with SEM. Cu segregation did not occur in as-cast condition but after hot rolling process Cu segregation occurred. Heat treatment decreased the segregation degree of Cu yet Cu particles were observed in microstructures.

I. INTRODUCTION

Steel is the most produced metal worldwide [1]. High production amounts of steel oblige the recycling of steel. Recycling of the steel is mainly done with Electric Arc Furnace process [2]. The most common industrial practice of recycling the steel via using Electric Arc Furnace (EAF) is followed by continuous casting technique for semi-finished products. Quality of the re-produced steels by recycling is mainly depend on scrap quality [2, 3]. Chemical composition of the scrap directly affects end product in EAF. Ladle furnace operations of secondary metallurgy is insufficient for refinement some of the impurities in molten steel. Secondary metallurgy of recycled steel is based on oxidation of impurities then floating the oxides to slag. Impurities that have lower oxygen affinity than iron are unable to be refined from steel by oxidation. These elements are known as tramp elements in steel [4]. Tramp elements are; Cu, Sn, Zn, Pb, Bi, Sb, As, Ni, Cr, Mo and V [4]. Tramp elements effect steel properties in different ways. Formation of solid solution or precipitation may enhance steels mechanical performance while intergranular or surface segregation which originated from tramp elements effects steel performance in detrimental means [4, 8].

Hot shortness is segregation of tramp elements known as tramp element presence in recycled steels produced via Electric Arc Furnace (EAF) [9]. Recycling of steel decreases the carbon footprint nevertheless accumulation rate of tramp elements increase in recycled steels [10]. Increase in tramp element amounts in steel requires; dilution,

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refining operation, control of tramp element behaviour with alloying or heat treatment [3-7]. Dilution is decreasing copper content of steel by using cleaner scrap or different iron sources like Hot Briquette Iron (HBI) or Direct Reduced Iron (DRI). Ammoniac, Chloride and Sulphide based slags were studied for copper removal from steel. Results showed that decopperization is possible above 70% [13]. Using sulphur in steel slags increases decopperization but the effect of sulphur-based slag in end product was not studied. Tee and Fray suggested oxychlorination process to remove the copper from steel before smelting. Copper chlorination during the oxidation of iron is called oxychlorination. It is basically based on simultaneous oxidation and chlorination processes of iron and copper at 800 ºC. Oxidation of iron prevents copper condensation on iron oxide surfaces. Copper chloride is removed from the system in gas phase. Authors indicated that removing chlorined copper from steel is technically feasible [14]. Nickel alloying to steel increases both copper containing phases melting temperature and copper solution in austenite [15]. Increasing copper solubility in austenite prevents intergranular copper segregation and hot shortness.

Studies are focused on refining of copper or other tramp elements from steel in steelmaking process. On the other hand, effect of copper on mechanical or corrosion properties is studied to foresee possible difficulties in using tramp element containing scrap in steelmaking. This study is aimed to understand heat treatment effect on 1% Cu containing steel after hot shortness formation. Slabs are hot rolled and hot shortness is observed in steels with different amount of copper. Heat treatment process is applied after hot shortness in 1% Cu containing low carbon steel slab to investigate hot shortness mechanism.

II. EXPERIMENTAL METHOD

This study aimed to understand and explain hot shortness behaviour in carbon steels. Carbon steel is smelted from scrap rebar in induction smelting furnace. Smelted steel scrap was alloyed with extra copper for artificial tramp element observation. Prepared steel composition was casted in resin moulds that shaped as slab with measurement of 20x500x200 mm. Chemical composition of casted slab was determined with optic emission spectroscopy. Microstructure analysis of as-cast sample was done with Olympus X-70 microscope, after metallographic sample preparation and etching with 3% Nital solution. Slab was hot rolled after annealing at 1200 ºC for 1 hour. The slab was hot rolled for 60% height reduction. Hot rolled slab was analyzed with both optical and electron microscopy. Heat treatment samples were collected from hot rolled slab. Heat treatments after hot shortness were done for 800, 900 and 1000 ºC for 1 and 4 hours with air cooling and water quenching. Heat treated samples were prepared for metallographic observation. Heat treated samples were etched with 10 ml FeCl3- 100 ml H2O etchant to reveal possible Cu segregations in samples.

III. RESULTS AND DISCUSSIONS

Chemical analysis of casted sample is given in Table 1. It is seen in Table 1 steel has composition of plain carbon steel except copper content. Copper content is desired to be 1.00 %wt. however result has deviation from desired composition.

Table 1. Chemical analysis of steel casting

Elements	C	Mn	Si	Cr	Ni	Cu
%	0.279	0.45	0.43	0.10	0.09	0.90

Casted sample is metallographically evaluated with optical microscopy. Result showed that there is no copper segregation in sample in as-cast situation. Optical microscopy image of casting is given in Figure 1. Optical microscopy showed that as-cast metallographic situation of casting has dendritical structure of casting.

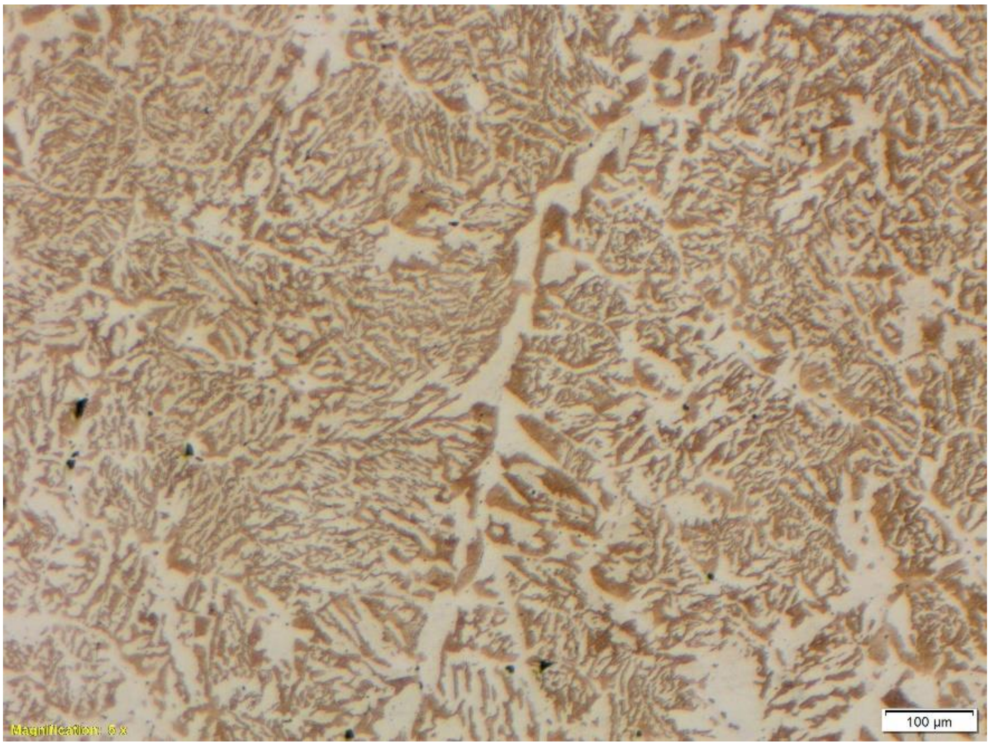

Figure 1. General view of slab in as-cast condition with optical microscopy

Hot rolled slab height is reduced by 60% in 15 passes. Mill scale on annealed slabs are cracked by mechanical force before hot rolling nevertheless new mill scale formed afterwards hot rolling. Macro cracks on slab could be seen on slab. Photograph of hot rolled slab is given in Figure 2. Edge cracks and heavy mill scale formation can be seen clearly.

Figure 2. Hot rolled slab surface

Optical microscopy result of hot rolled slab is given in Figure 3. Segregation of copper can be seen in optical microscopy image. Segregated copper has settled in grain boundaries of steel. Rolling process has manipulated copper through the rolling direction.

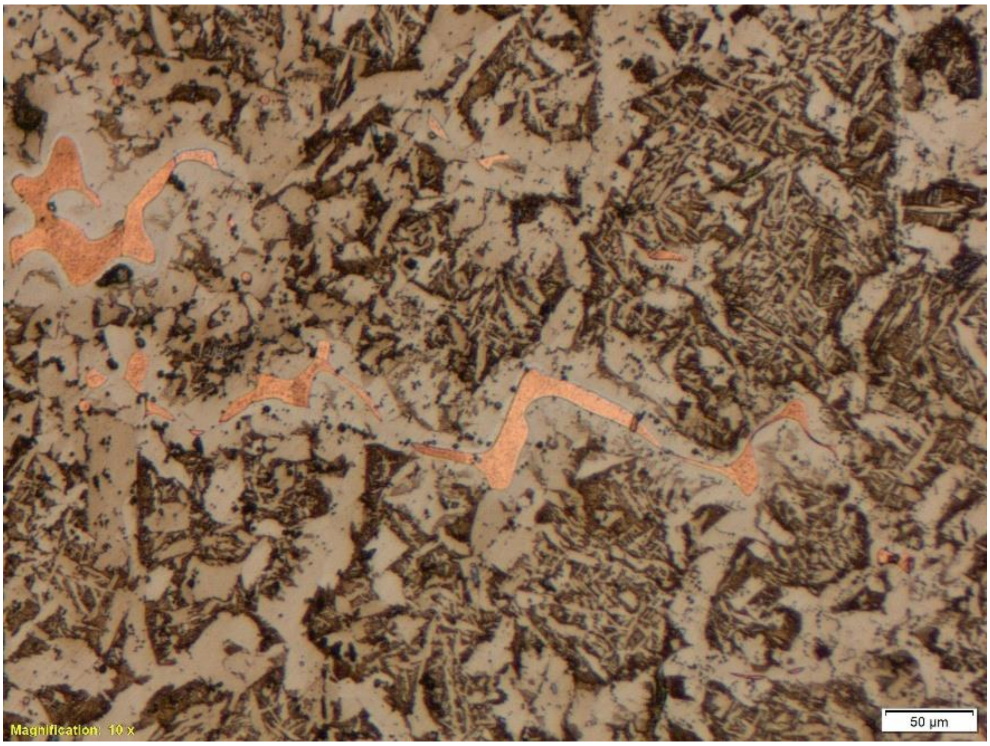

Figure 3. Optical microscopy image of hot rolled slab with 10x magnification

Excessive copper segregation in crack propagation points are revealed. Optical microscopy images of cracks of hot rolled slabs are given in Figure 4 and 5, respectively.

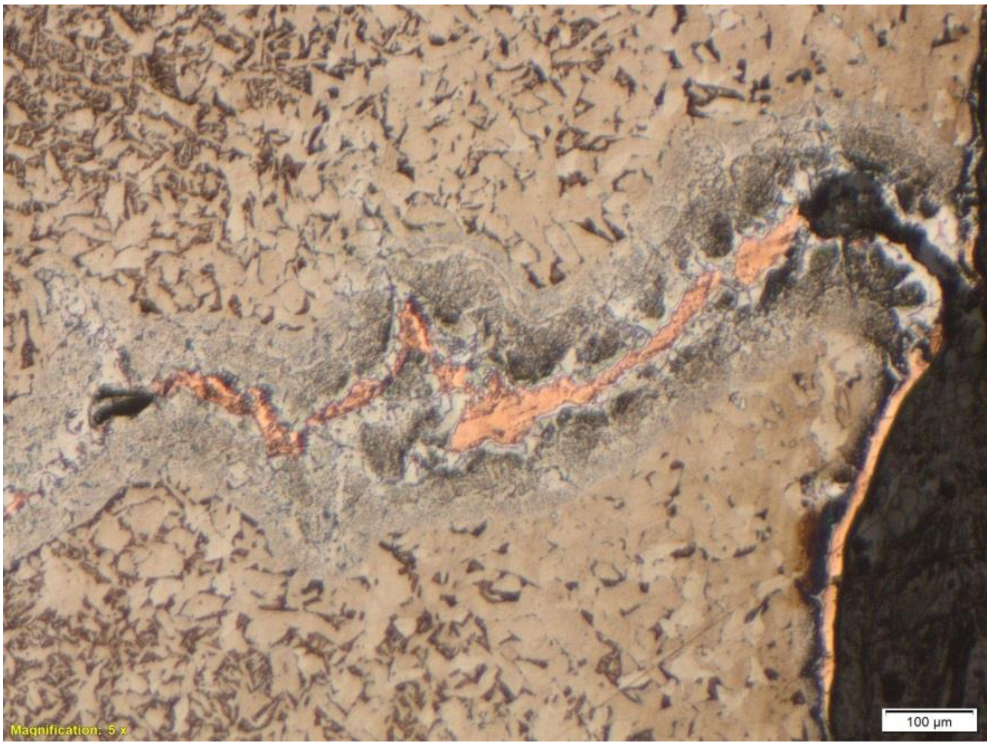

Figure 4. Crack propagation point with copper segregation, 5x magnification

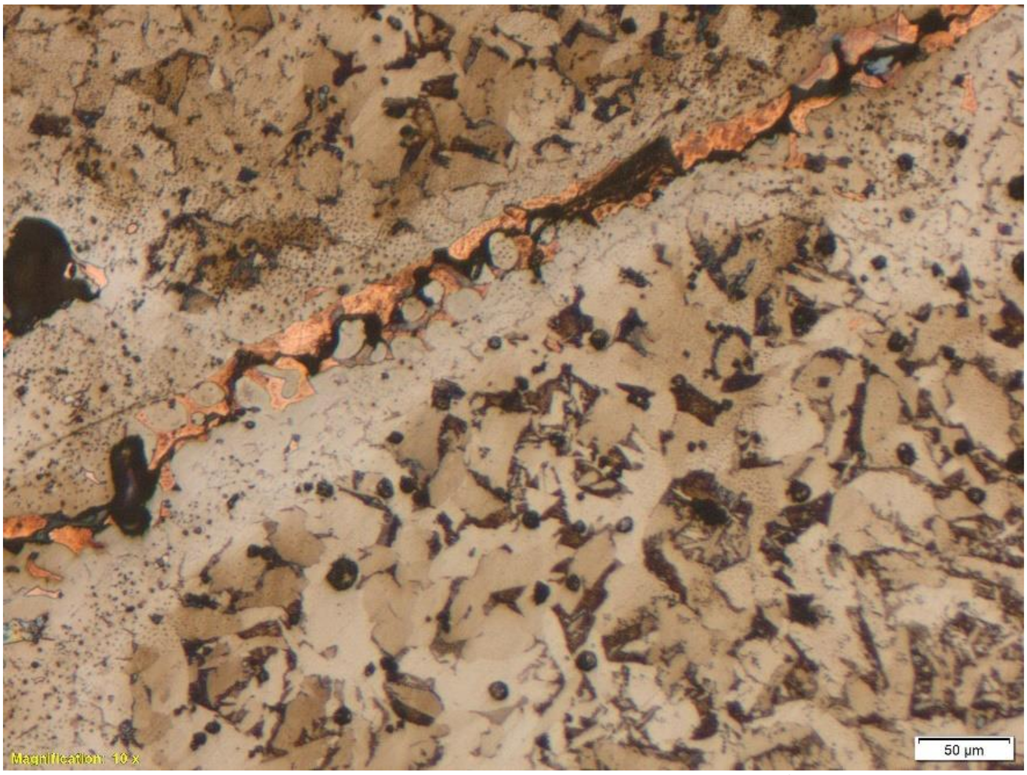

Figure 5. Crack propagation point with copper segregation, 10x magnification

SEM imaging and EDS analysis of crack starting points were done. Results showed that copper is segregated in crack propagation region with intergranular characteristic. Result is given in Figure 6 for SEM image and EDS analysis is given Table 2. EDS points 1 and 2 are chloride containing corrosion products which are result of etching with ferric chloride. EDS point 3 is copper segregation in crack propagation region.

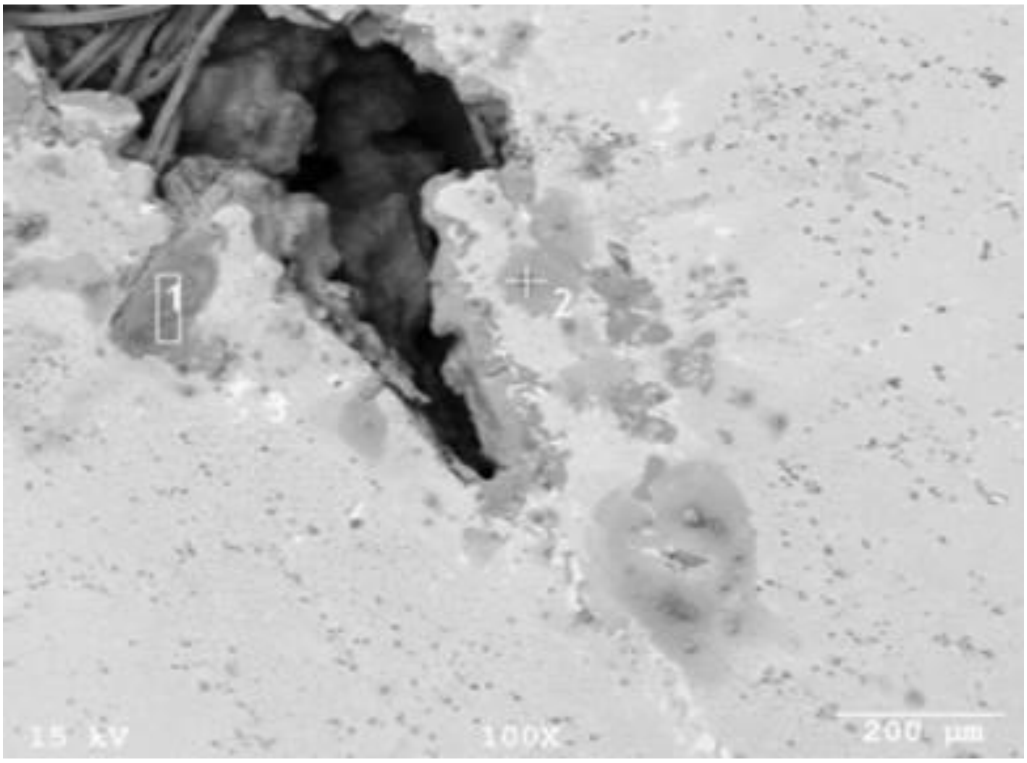

Figure 6. SEM image of crack in casting

Table 2. EDS results

EDS Point	Fe%, wt.	Si%, wt.	O%, wt.	Cl%, wt.	Cu%,wt.
1	70.91	2.39	21.20	5.50	-
2	79.89	0.65	12.00	7.46	-
3	7.07	0.80	1.36	-	90.77

Heat Treatment Results

Heat treatment of samples was done in electric resistance furnace. Samples were placed in furnace then heated to desired temperature. Samples were annealed for 1 and 4 hours, cooling of samples were done with air cooling and water quenching. Heat treated samples were metallographically examined. Surface region of samples observed with 10x magnification inner matrix of samples observed with 20x magnification.

1 hour annealed and air-cooled sample microstructure is given in Figure 7. Upper microstructures indicate surface region while lower microstructures inner regions. Annealing temperature increase does not have vital effect on copper segregations in 1 hour. Copper segregation decreases in annealing process, yet full recovery could not obtain. Inner matrix has only particulate copper segregates in ferrite regions.

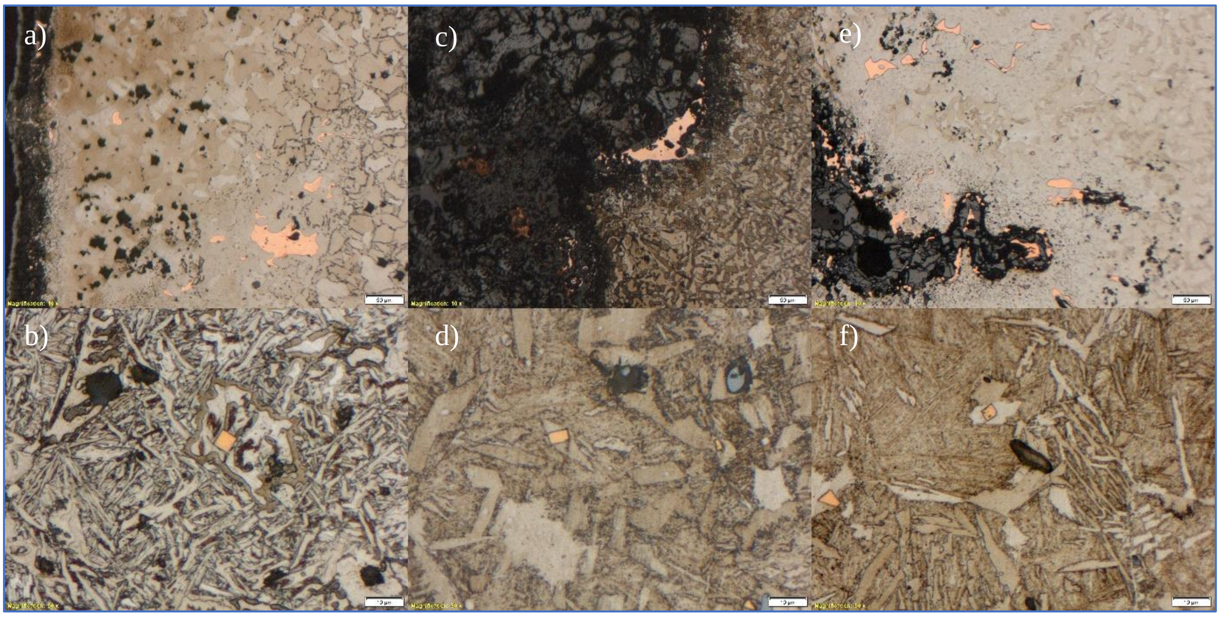

Figure 7. Heat treatment microstructure of casting 1 (Annealing Time: 1 hour, Cooling: Air Cooling, Temperature: 800 (a,b), 900 (c,d), 1000 (e,f) ºC).

1 hour annealed and water quenched sample microstructure is given in Figure 8. Upper microstructures indicate surface region while lower microstructures inner regions. Heavy copper segregation can be observed in samples. Copper segregates on austenite grain boundaries at elevated temperatures and water quench keeps high temperature structure of samples. Inner matrix has similar results as air cooled samples. Particulate copper segregation can be seen in matrix. General microstructure of 800 and 900 ºC annealed samples are ferrite – pearlite while 1000 ºC annealed sample is martensitic.

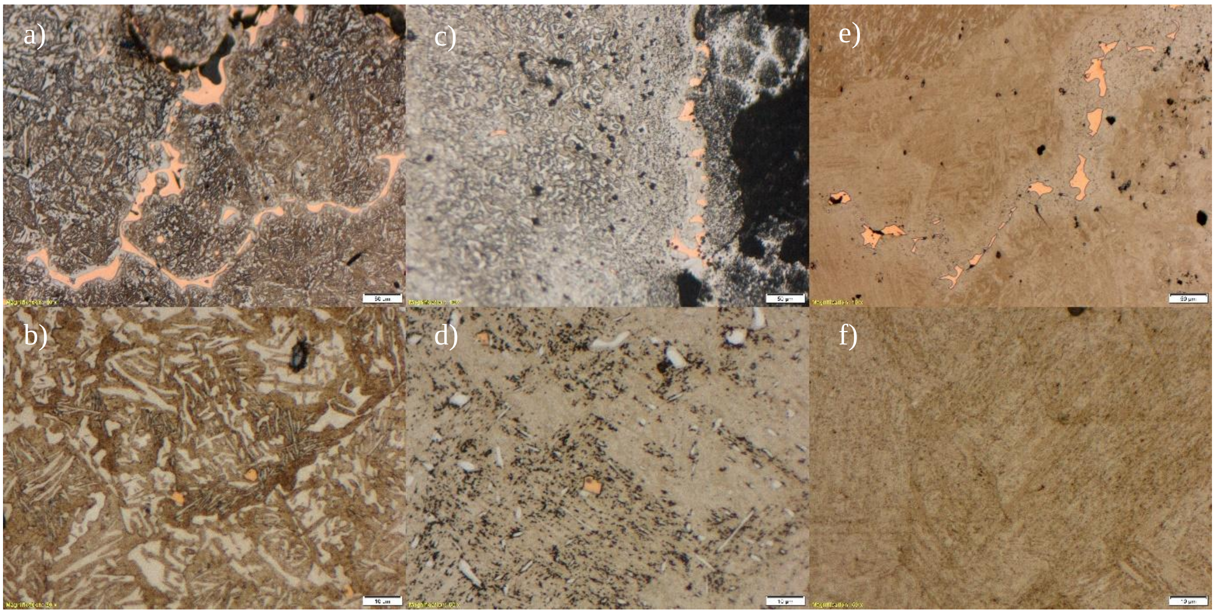

Figure 8. Heat treatment microstructure of casting 1 (Annealing Time: 1 hour, Cooling: Water Quench, Temperature: 800 (a,b), 900 (c,d), 1000 (e,f) ºC).

4 hours annealed and air-cooled samples are examined for microstructure with optical microscopy. Results of investigation are given in Figure 9 with 10x magnified photographs above and 50x photographs in bottom. 10x magnified photographs are collected from surface – mill scale intersection while 50x photographs show inner matrix. Decarburized region in surface has large portion of copper segregation. In 900 ºC annealed sample some of copper segregation can be seen in mill scale. Inner matrix investigation showed that particulate copper segregations are in ferritic regions likewise to 1 hour annealed samples.

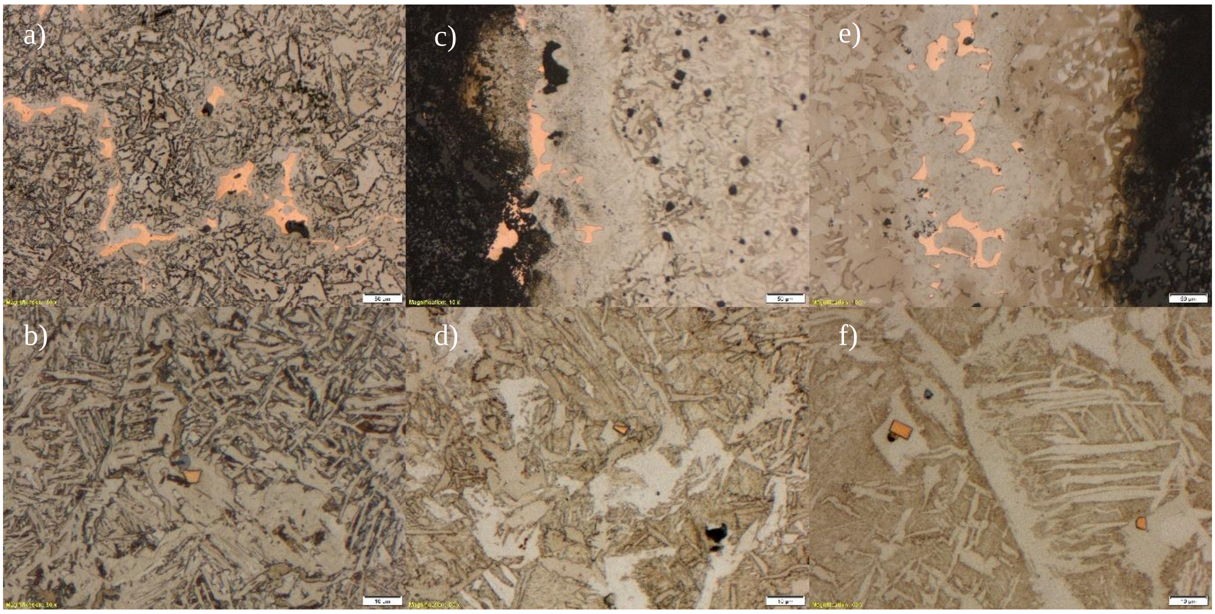

Figure 9. Heat treatment microstructure of casting 1 (Annealing Time: 4 hours, Cooling: Air Cooling, Temperature: 800 (a,b), 900 (c,d), 1000 (e,f) ºC).

4 hours annealed and water quenched sample microstructures are given in Figure 10. Photographs are given in order of 10x above and 50x bottom likewise previous figures. Decrease of copper segregation areas and segregation intensity can be seen in surface observations. Copper segregation intensity is decreased with increasing

temperature. Martensitic transformation can be seen in 900 and 1000 ºC annealed then quenched samples. Particulate copper is found in central microstructure. Copper is segregated in ferrite phase in the sample which annealed at 800 ºC while at 900 and 1000 ºC samples copper segregation is occurred without any selectivity.

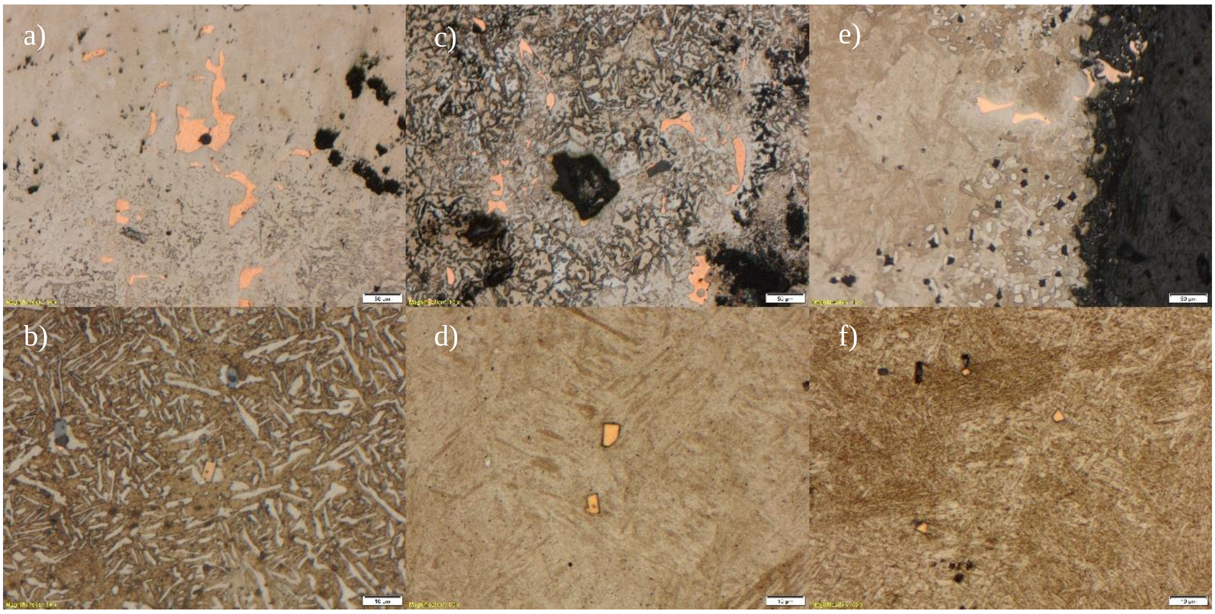

Figure 10. Heat treatment microstructure of casting 1 (Annealing Time: 4 hour, Cooling: Water Quench, Temperature: 800 (a,b), 900 (c,d), 1000 (e,f) ºC).

IV. CONCLUSIONS

Heat treatment process in ambient conditions applied to steel with high tramp element content. Results have showed that copper segregation due to hot shortness cannot be prevented in heat treatment process. Ambient conditions in heat treatment process supports further copper segregation thus forming solid solution of copper in austenite could not be achieved under these circumstances. Further conclusions can be drawn from this study as;

1. Hot rolling process in tramp element containing steels causes macro cracks with liquid embrittlement due to liquified copper on austenite grain boundaries. Copper enrichment in steel occurs with selective oxidation of iron on steel surface. General copper segregation is observed in crack propagation regions under the steel surface.
2. Heat treatment of steel samples retarded copper segregation to some extent. 1 hour annealed samples with air cooling have better results than water quenched samples. Slow cooling rate promotes diffusion of copper into austenite grains. Nevertheless, copper segregation could not be completely dissolved in austenite.
3. Increasing the annealing time to 4 hours did not decrease copper segregation to minimal degree. Segregated copper diffused to inner parts of steel following austenite grain boundaries. Oxidation of steel in high temperature continues due to annealing in ambient conditions. Protective annealing conditions could be studied to restrain further oxidation of steel. Oxidation of steel causes segregation of copper in steel scale.

4. Decarburization of surface increases ferrite – austenite transformation temperature on surface. Austenite has higher copper solubility than ferrite thus surface copper segregation occurs on surface with two reasons; selective oxidation of iron on surface, decarburization and retarded austenite transformation. Decarburization and oxidation of steel must be prevented to increase copper solubility and restraining further copper segregation.

Copper segregation mechanism in hot shortness can be explained by 4 steps. Oxidation of Fe on the steel surface, metallic Cu segregation in oxide scale, melting and diffusion of Cu to steel surface and grain boundaries, mechanic force on steel that causes macro cracks due to decreased grain boundary cohesion because of Cu segregation. Heat treatment in oxidative environment causes continuous oxidation and decarburization of steel. Decarburization of steel increases austenite transformation temperature and oxidation promotes Cu segregation. These results explain ineffective heat treatment on Cu segregation.

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