Düzce University
Journal of Science & Technology

Research Article

Microstructural Characterization of Hot Shortness in Steels

Selçuk Yeşiltepe

Department of Mechanical Engineering, Osmaniye Korkut Ata University,TURKEY
selcukyesiltepe@osmaniye.edu.tr
DOI: 10.29130/dubited.1373828

ABSTRACT

Hot shortness in steels is a macro crack on the steel surface especially on the slab edges of hot rolled steels produced by recycling. Hot shortness in steels is characterized by intergranular cracking of steel on the surface during hot rolling process. The hot shortness damage can be seen by naked eye due to macro cracks on steel surface, after the hot rolling of slab. Although macro cracks can occur due to various parameters such as excessive force or deformed rolls, hot shortness is directly linked to residual elements in the steel composition. Most common and effective residual element for hot shortness is copper. Copper in steel originates from the used scrap in Electric Arc Furnace (EAF) steelmaking. Decreased primary sources and environmental concerns with increased scrap output in the world make the recycling of steel inevitable.

Copper segregates in the austenite grain boundaries during the annealing of steel prior to hot rolling. Liquified copper film decreases the grain cohesion between austenite grains and results in intergranular cracking by the force applied during the rolling. Detection of copper in microstructure is vital to understand mechanism of hot shortness. Common knowledge in the literature of hot shortness suggests that hot shortness mechanism consists of oxidation-segregation-decreased grain cohesion-crack route. In this study, detailed microstructure images of steel surfaces are discussed with optical and scanning electron microscopy examinations. Examined samples of hot shortness are collected from scientific experiments and industrial practice examples. Helpful techniques and etching agents for copper revelation in microstructure are evaluated and explained in this paper.

Keywords: *Hot shortness, Microstructure, Optical microscopy, Electron microscopy*.

Çeliklerde Sıcak Yırtılmanın Mikroyapısal Analizi

ÖZET

Çeliklerde sıcak yırtılma çelik yüzeyinde oluşan makro çatlakların özellikle slabın kenar bölgelerinde görüldüğü ve sıcak haddelemeye özgü geri dönüştürülmüş malzemelerde ortaya çıkan bir hasar çeşididir. Sıcak yırtılma, sıcak haddeleme esnasında oluşan taneler arası kırılmanın oluşması ile karakterize bir hasardır. Sıcak yırtılma sıcak haddeleme sonrasında gözle görülebilen makro çatlakların oluşumudur. Çelik yüzeyinde sıcak haddeleme sırasında görülebilen makro çatlaklar aşırı haddeleme kuvveti, merdanelerin çarpıklığı gibi farklı nedenlerle meydana gelebilmesine karşılık sıcak yırtılma direkt olarak çelikte bulunan kalıntı elementlerle ilişkilidir. Bakır çeliklerde en çok rastlanan ve sıcak yırtılmada en etkin rolü oynayan elementtir. Elektrik ark fırınında (EAF) kullanılan hurda çelikteki bakırın kaynağıdır. Azalan birincil kaynaklar, çevresel endişeler ve artan hurda miktarları çeliğin geri dönüştürülmesini kaçınılmaz hale getirmiştir.

Ulusal Elektron. Mikroskopi Kongresi (EMK26)
Received: 10/10/2023, Revised: 12/11/2013, Accepted: 21/11/2023

Bakır sıcak haddeleme öncesindeki tavlama sırasında östenit tane sınırlarında ayrışmaktadır. Sıvı bakır filminin tane sınırlarındaki varlığı taneler arası kohezyonu zayıflatarak sıcak haddeleme sırasında uygulanan kuvvetle birlikte taneler arası kırılmaya neden olmaktadır. Bakırın mikroyapıda tespit edilmesi sıcak haddeleme sırasındaki davranışının anlaşılması için elzemdir. Konu üzerine yazındaki ortak kanı sıcak yırtılma mekanizmasının oksitlenme-ayrışma-taneler arası kohezyonun zayıflaması-çatlama şeklinde olduğunu söylemektedir. Bu çalışmada sıcak yırtılma hasarına uğrayan çeliklerin detaylı mikroyapı görüntüleri optik ve taramalı elektron mikroskopları ile incelenmiştir. İncelenen numuneler bilimsel araştırmada kullanılan ve endüstriyel üretimden alınan sıcak yırtılma örnekleridir. Mikroyapı incelemesinde kullanılan ve bakırın davranışının incelenmesini sağlayan yararlı teknikler ve dağlayıcı ajanlar bu çalışmada açıklanmıştır.
Anahtar Kelimeler: *Sıcak yırtılma, Mikroyapı, Optik mikroskop, Elektron mikroskobu.*

I. INTRODUCTION

Electric Arc Furnace (EAF) steelmaking offers low carbon emissions in steelmaking technology and enables recycling of end-life steel products. [1] Although EAF offers advantageous production process it requires good scrap quality to produce high quality steel products. Scrap steel may contain large amounts of unwanted impurities for desired steel composition. [2] Cu, Sn, As, Sb and Pb are known as tramp elements because in conventional steelmaking refinement of these elements is not possible. [3] These elements, mainly Cu, cause hot shortness problem in hot rolling of steel. [4]

Hot shortness is the crack formation on steel surface during hot rolling process due to Cu in the composition. [5] Oxidation of steel surface causes Cu segregation on steel surface since iron oxides, wustite and magnetite, do not have Cu solubility to form solid solution. [2] Segregated Cu containing phase with low melting temperature elements follows grain boundaries of steel and further penetrates the steel surface. [6] Liquid phase between austenite decreases grain cohesion and causes intergranular cracking of steel during hot rolling. [6]

In this study microstructural characterization methods for hot shortness in steels evaluated. Scientific and industrial examples are investigated with microscopical techniques such as optical and electron microscopy.

II. Material and Method

Steels with hot shortness problem were selected and microstructural characterization applied to specimens. Samples were mounted in bakalite moulds and grinding and polishing applied. Polishing of samples were done with 1 micron Al2O3 suspension. Etching of samples were executed with 3% nitric acid containing Nital and 100 ml FeCl3 containing ethanol solution of 200 ml for scientific samples and FeCl3 containing ethanol solution for industrial samples. Chemical composition of steels were determined with Optic Emission Spectroscopy. SEM characterization of microstructure was done with Back Scattered Electron mode. EDS of samples were done to examine the chemical composition of desired areas.

III. Results and Discussion

Optical Emission Spectroscopy results of samples were given in Table 1. Scientific research samples had higher copper content to easily detect the copper segregation and behaviour during the hot rolling, while industrial example had lower amounts of copper to prevent hot shortness.

Table 1. Chemical composition of steels.

Sample	C%	Si%	Mn%	Ni%	Cu%	Sn%
Scientific 1	0.279	0.43	0.45	0.090	0.90	-
Scientific 2	0.263	0.47	0.47	0.090	1.48	-
Industrial	0.026	0.018	0.138	0.088	0.32	0.012

Microstructural view of Scientific 2 sample was given in Figure 1 for both Nital and FeCl3 based etchants. Both microstructural view were taken with same magnification of X50 and close areas to understand etchant effect on optical microscopy results. Nital etched view did not show any copper related segregation while FeCl3 based etchant revealed Cu segregation with natural Cu colour. FeCl3 based etchant was proved to be the best solution for copper segregation research hence the copper has more corrosion resistance and etching of copper with Nital was ineffective. Steel microstructure was revealed with Nital, yet copper was not etched and invisible in optical microscopy.

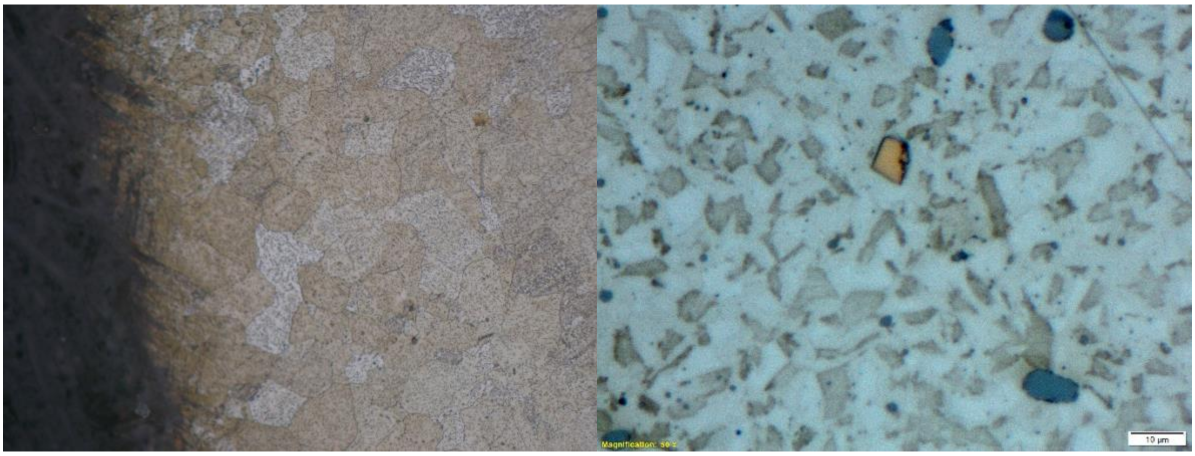

Figure 1*. Microstructural view of Scientific 2 (Nital on the left, FeCl3 on the right).*

Scientific 1 sample was further investigated under optical microscopy with FeCl3 based etchant. Crack propagation points of material were studied. Results showed that, cracks were filled by liquid copper during hot rolling. Cracks were completely filled with copper and solidified copper could be seen in optical microscopy. Microscopical view of Scientific 1 sample was given in Figure 2. Crack path showed intergranular characteristics of cracking.

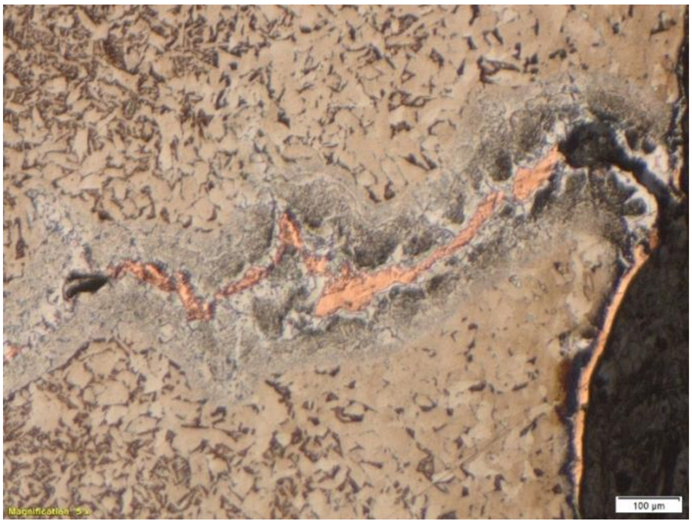

Figure 2. *Microstructural view of Scientific 1 etched with $FeCl_3$ based etchant.*

Industrial sample was investigated with FeCl3 based etchant on optical microscopy. Sample was showing hot shortness properties on macro scale. Investigations revealed segregation phase around the grain boundaries of steel. Yellow colour was used to create contrast in grain boundaries. Intergranular segregation and intergranular cracks were observed. Segregated phase showed characteristics of liquified-solidified phase. Optical microscopy view of sample was given in Figure 3.

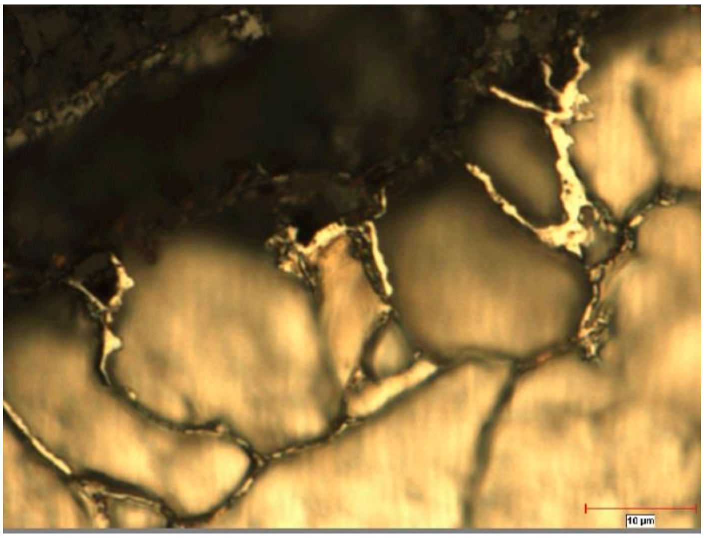

Figure 3. *Industrial sample microstructural view of hot shortness.*

Samples were investigated with SEM-EDS techniques to validate optical microscopy views of copper segregation. Back Scattered Electron mode was used during investigations. Back Scattered Electron Imaging had an advantage in SEM characterization of hot shortness because of Cu atomic mass is higher than Fe. Atomic mass difference causes Cu to be seen brighter in imaging and SEM thus EDS areas can be selected easily. SEM image of Industrial sample was given in Figure 4. SEM image was taken from a near region of Figure 3 to validate optic microscopy result. Image showed bright regions around grain boundaries similar to optic microscopy. Point EDS was applied to specimen for characterization of these phases. Results showed that the bright areas were rich in Cu and a segregated phase from steel grains. EDS results are given in Table 2. Copper segregation was found and nickel was found to accompany copper in segregated phase. Unlimited solid solution capacity of copper and nickel was unable to form a segregated phase together. However it was a possible solution offered in literature to decrease hot shortness susceptibility of steel since nickel increases the melting temperature of copper rich segregate.

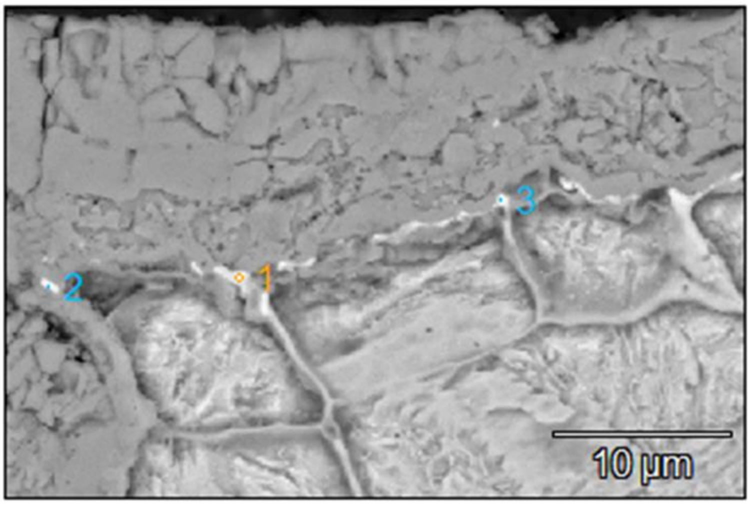

Figure 4. *SEM image of Industrial sample with EDS points.*

Table 2. *EDS point analysis results.*

Point	Cu%	Ni%	Fe%
1	59.91	10.44	29.65
2	30.41	6.92	62.67
3	9.65	1.40	88.95

Industrial sample was further investigated and EDS mapping technique was applied. Results showed that segregation was complete and Cu/Fe boundaries did not show cross-over. SEM image of EDS mapped area and EDS mapping results were given in Figure 5 and Figure 6 respectively.

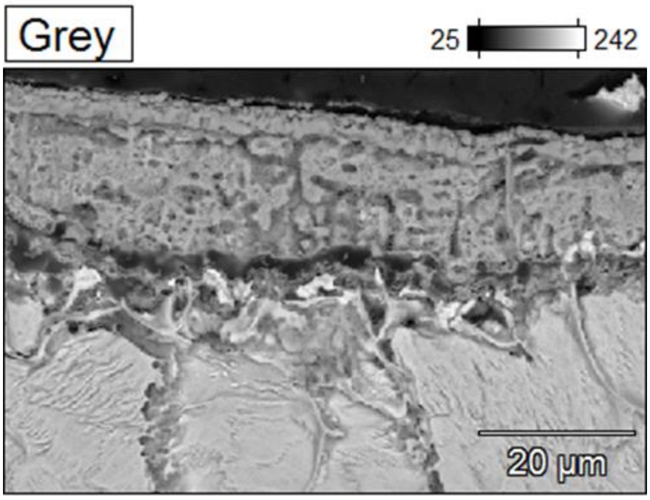

Figure 5. *SEM image of EDS mapped area.*

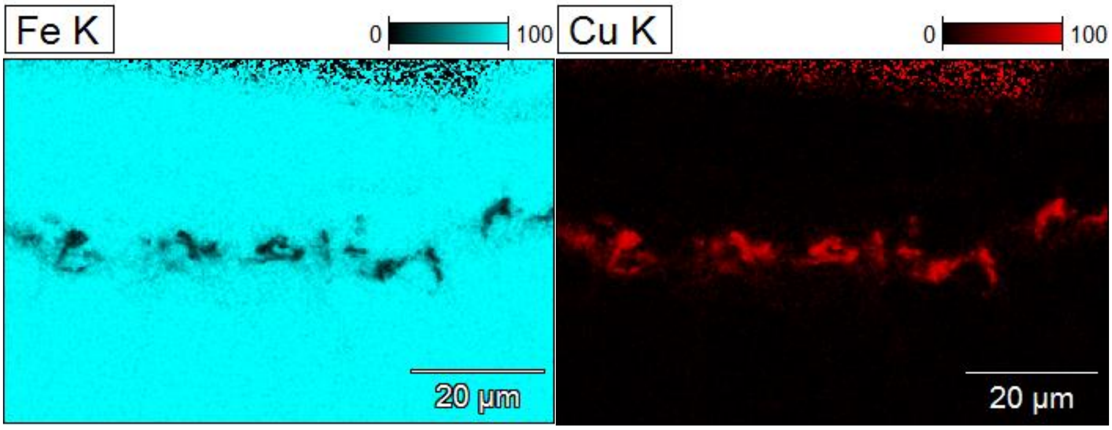

Figure 6. *EDS mapping results.*

IV. CONCLUSION

Microstructural analysis' of samples were done to understand hot shortness mechanism in steels for both scientific and industrial examples of hot shortness. Nital solution for etching was found ineffective in detect the copper segregation in samples. Copper is more noble than Iron by means of etching and FeCl3 based etchant was able to reveal the copper segregation in samples. Back Scattered Electron mode in SEM imaging was used to acquire microstructure of material and effectively created a contrast between steel grains and copper due to atomic mass difference. Understanding of mechanism and microstructural view of hot shortness is vital to prevent occurrence of this problem.

ACKNOWLEDGEMENTS: This work is supported by MMK Metallurgy Türkiye company financially. Author wants to thank Dr. Özgür Karakaş and MMK Metallurgy Quality Control Department for their support.

V. REFERENCES

[1] A. C. Ahindra Ghosh, Ironmaking and Steelmaking Theory and Practice, New Delhi: PHI Learning Private Limited, 2008.

[2] N. K. K. K. Norio Imai, «Effect of Cu and Ni on Hot Workability of Hot-rolled Mild Steel,» ISIJ International, cilt 37, no. 3, pp. 224-231, 1997.

[3] E. Stephenson, «Effect of Recycling on Residuals, Processing, and Properties of Carbon and Low-Alloy Steels,» Metallurgical Transactions A, cilt 14A, no. March, pp. 343-353, 1983.

[4] E. S. J. N. S. S. Lan Yin, «The Effects of Nickel/Tin Ratio on Cu Induced Surface Hot Shortness in Fe,» Oxidation of Metals, no. 76, pp. 367-383, 2011.

[5] M. H. K. Kunishige, «SURFACE HOT-SHORTNESS OF STEELS INDUCED BY A SMALL AMOUNT OF COPPER AND TIN FROM SCRAP STEELS AND ITS SUPPRESSION METHODS,» Materials Science Forum, Cilt %1 / %2539-543, pp. 4113-4118, 2007.

[6] Y. U. K. K. Y. T. S. H. K. O. Akihiro Takemura, «Surface Hot Shortness of Copper Containing Steel in a Compact Strip Production Process,» Material Transactions, cilt 52, no. 10, pp. 1905-1911, 2011.

[7] N. K. K. K. Norio Imai, «Effect of Cu. Snand Ni on Hot WorkabiIity of Hot-rolled Mild Steel,» ISIJ International, cilt 37, no. 3, pp. 217-223, 1997.