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ASSESSMENT OF THE SUSCEPTIBILITY TO "HOT SHORTNESS" OF COPPER CONTAINING STEELS[1]

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Abstract

Environmental pressures are forcing an increase of use of highly contaminated steel scrap in the electric-arc furnace processing route. Copper is a particularly troublesome contaminant since it is not possible to remove it from the molten steel and hence, all the copper entering the furnace will be present in the final alloy. At hot-working temperatures, iron is oxidized preferentially at the external surface and liquid copper forms on austenite grain boundaries, causing cracking (or "hot shortness") in the steel. Within the bulk, copper segregates to MnS inclusions exacerbating the problem of segregation. On the positive side, the copper that remains dissolved in ferrite, leads to age hardening, an important improvement in mechanical properties, and also improves weathering corrosion resistance. Hence, there is significant commercial interest in capitalizing on the improved properties resulting from copper additions without encountering hot shortness during thermo-mechanical processing. Previous researchers have shown that for samples tested in an argon atmosphere, the presence of copper in steel only slightly impairs the hot-ductility in the temperature range 700-900°C because of the precipitation of copper sulphide. We contend that hot shortness actually results from the melting of segregated copper metal at the temperature of hot-deformation, causing cracking. Therefore, the best way to simulate the actual role of copper in causing the hot shortness is by conducting tests in air since iron will then be oxidized preferentially, thereby increasing the extent of copper segregation. However, it is not possible to accurately measure the reduction of area because excessive oxidation at the testing temperature produces highly spoiled fractures. Consequently, the frequently used technique of determining hot ductility in a hot-tensile test has serious limitations with respect to assessing hot shortness since an inert atmosphere is required to ensure reliable measurements at temperatures above the melting point of copper. Hence, the hot-tensile test is not an appropriate technique to assess hot shortness and alternative ways of assessing hot shortness will be discussed.

Key words: Hot shortness; Copper containing steels; Cracks; Continuous casting; Hot processing.

ACESSIBILIDADE DA SUSCETIBILIDADE DA FRAGILIDADE À QUENTE (*HOT SHORTNESS*) DOS AÇOS CONTENDO COBRE

Resumo

Pressões ambientais forçam o aumento de uso de sucatas altamente contaminadas na fabricação de aço em forno elétrico. O cobre é uma contaminação particularmente problemática, pois não se pode removê-lo do banho de aço liquido e, portanto, todo o cobre carregado será incorporado à liga. Nas temperaturas de processamento à quente do aço, o ferro é oxidado na superfície, preferencialmente ao cobre, durante o resfriamento, formando um filme de cobre líquido no contorno de grão austenítico, causando fragilidade a quente (*hot shortness*) do aço. Disperso no interior do aço, o cobre migra para o entorno das inclusões de MnS, tornando ainda pior o problema de segregação. Em seu lado positivo, o cobre que permanece dissolvido na ferrita, promove o fenômeno de envelhecimento por precipitação, uma melhoria importante nas propriedades mecânicas do aço, e também o aumento da resistência à corrosão atmosférica. Assim, há um interesse comercial importante em capitalizar os benefícios resultantes da adição de cobre, desde que não ocorra o fenômeno da fragilidade à quente no processamento termomecânico do aço. Pesquisadores anteriores mostram que, para amostras testadas ao ar, a presença de cobre causa apenas uma leve redução na ductilidade à quente, na faixa de temperatura de 700-900°C por causa da precipitação de sulfeto de cobre. Nós afirmamos que a fragilidade à quente realmente não pode ser simulada por ensaio de ductilidade, pois ela é resultado da fusão do cobre segregado na temperatura de deformação a quente a quente, causando a trinca. Portanto, a melhor forma de simular o papel real do cobre na fragilidade a quente é conduzindo ensaios ao ar, pois assim é produzida a oxidação preferencial do cobre e aumentando a quantidade de cobre segregado. Contudo, não é possível uma medição precisa da redução de área porque a oxidação excessiva na temperatura de ensaio produz uma fratura bastante danificada. Consequentemente, a técnica usualmente empregada para determinar a ductilidade a quente por ensaio de tração à quente apresenta sérias limitações no sentido de similar a fragilidade a quente, já que é necessária uma atmosfera inerte para assegurar uma medida de redução de área confiável. Desta forma, o ensaio de tração à quente não é uma técnica apropriada e formas alternativas para similar a fragilidade à quente e outros métodos de simulação são discutidos.

Palavras-chave: Fragilidade à quente; Aços contendo cobre; Trincas; Llingotamento contínuo; Processamento à quente.

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1 INTRODUCTION

The problem of "hot shortness" is gaining importance as recycling of steel products is becoming mandatory because of global environmental pressures. A large fraction of scrap is derived from the automobile and appliances industries and as the use of electronic devices is increased, the level of contamination of scrap is also elevated and as a consequence, recycling is becoming more difficult and expensive. Copper is a particularly troublesome contaminant since it is not possible to remove it from molten steel(1) and hence, all the copper entering the furnace will be present in the final alloy, causing cracking during the hot processing of copper containing steels, a phenomenon commonly referred to as 'hot shortness'. Traditionally, hot shortness has been attributed to the formation of a thin film of liquid copper that surrounds austenite grains in the steel, since copper is not oxidized because it is more noble than iron.(2) As a consequence of the presence of this thin film of liquid copper, cracks form during the hot processing the steel, and hence the copper content of scrap has to be strictly controlled thereby limiting the extent to which copper contaminated scrap can be recycled. The addition of nickel increases the solubility of copper in the austenite(3) and although nickel additions are expensive, this is traditionally the technique used to reduce the hot shortness problem.

On the positive side, copper that remains dissolved in ferrite, leads to age hardening, an important improvement in mechanical properties and increased weathering corrosion resistance.(4) Hence, there are significant commercial and environmental interests in capitalizing on the improved properties resulting from copper additions without encountering hot shortness during thermo-mechanical processing.

Traditionally, the susceptibility of steel to crack initiation and growth during the continuous casting of steel is assessed by measuring hot ductility in a tensile test conducted at the temperatures the steel experiences during continuous casting. Hot ductility of steel is usually presented as a curve of reduction in area plotted against the testing temperature and more often than not, a ductility trough is found in the temperature range between the Ae_3 and Ar_3 temperatures. The loss of ductility in this temperature range is attributed to the formation of a thin film of ferrite on pre-existing austenite grain boundaries.(5) Once this film of ferrite has formed, stress concentrates in the softer ferrite, leading to crack initiation and propagation along the grain boundaries and eventually to inter-granular fracture. Clearly, the measurement of hot ductility by hot tensile test is relatively easy to conduct, once it is easy to carry on and provides a reasonable simulation of the high temperature behaviour of steel so it is frequently used to design crack prevention approaches. Although good simulation for hot ductility, the test does not correctly predict the influence of copper on the behaviour of steel at hot rolling temperatures.(6) In commercial practice serious problems with hot shortness is found when steel containing appreciable amounts of copper is hot rolled. However, hot ductility tests conducted in an argon atmosphere (to control excessive oxidation during the test) on the same steels, show very little impairment of hot-ductility. When these same steel are tested in the as-cast condition and in an oxidizing atmosphere, some impairment of hot ductility is found but due to excessive oxidation in the fracture area, it is very difficult to measure to any degree of precision, the ductility at fracture. The inability of the traditional hot ductility test to predict the behaviour during hot deformation of copper containing steels indicates that crack formation during hot working in such steels is not a conventional hot ductility problem. Hence, hot shortness encountered in copper containing steels should be distinguished from decreased hot ductility typically observed in carbon steels; hot shortness results by a different mechanism and a different test should be used to simulate the behaviour of copper containing steel at hot working temperatures.

2 RESULTS

Forming part of a larger investigation conducted over the last seven years, on the hot shortness in steel, the current investigation concentrated on the influence of small additions of copper and nickel on hot ductility.

2.1 Experimental

Hot ductility tests have been carried out in a Gleeble machine using 10mm diameter and 100m long cylindrical samples. Samples were heated up to cast "*in situ*" with the melt protected by a crystal glass. After casting, the temperature was decreased at the indicated cooling rate, held 3 min in order to stabilize the temperature and strength to break. The hot ductility is measured by the reduction of area considering initial and final diameter of the sample.

SEM investigations have been carried out in the fracture and also in the longitudinal section; TEM investigations using carbon replicas also have been carried out in order to investigate the occurrence of precipitates.

Although there are limitations to the effectiveness of the hot ductility test to simulate the behaviour of steel at hot working temperatures, this test has often been used to simulate and predict the behaviour of steel, especially during the unbending operation in continuous casting. This test has therefore also been used in this study in an attempt to predict the influence of copper and nickel on hot ductility. Figure 1 shows that the addition of 0.5%Cu slightly impairs the hot ductility in tests carried out in an argon atmosphere. The addition of nickel to the copper containing steels results in full recovery of the deleterious effect of copper. In an attempt to explain these findings, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) have been conducted on samples following hot tensile tests.

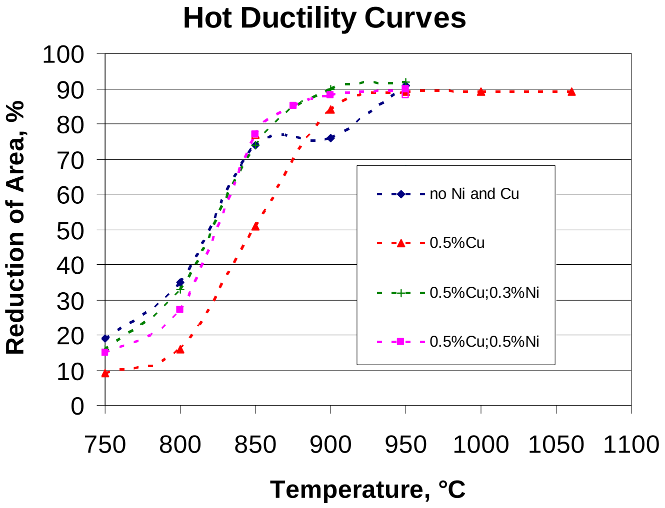

Figure 1- Hot ductility curves for C-Mn-Al steels tested under an argon atmosphere at a cooling rate of 25°C/min. Cu additions slightly impair hot ductility while Ni additions to the copper containing steel recovers hot ductility.(7)

For samples tested in argon, SEM analysis of the fracture surface shows that copper segregates to and surrounds inclusions, as shown in Figure 2.

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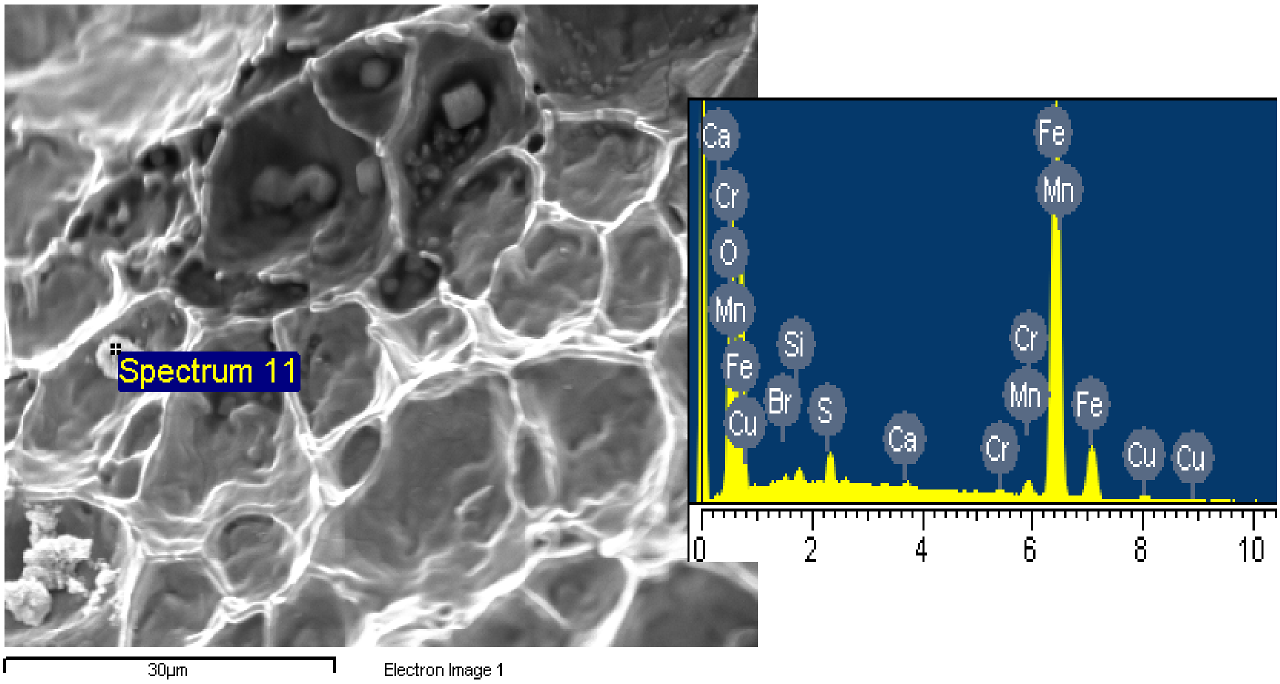

Figure 2 - SEM analysis on fractured samples showing Cu segregation surrounding Ca/MnS inclusion (magnification 1,600x) and the respective X-ray analysis. 0.5%Cu steel, cast *"in situ"* cooled at a rate of 25°C/min and tested at in argon at a temperature of 800°C – reduction in area 16%.(8)

Heavy concentration of copper around inclusions has been found in cross sections of the steel containing 0.5%Cu. Figure 3 shows details of copper segregation on an inclusion line and Figure 4 shows finer particles in the same steel in back scattering mode (Back Electrons Image).

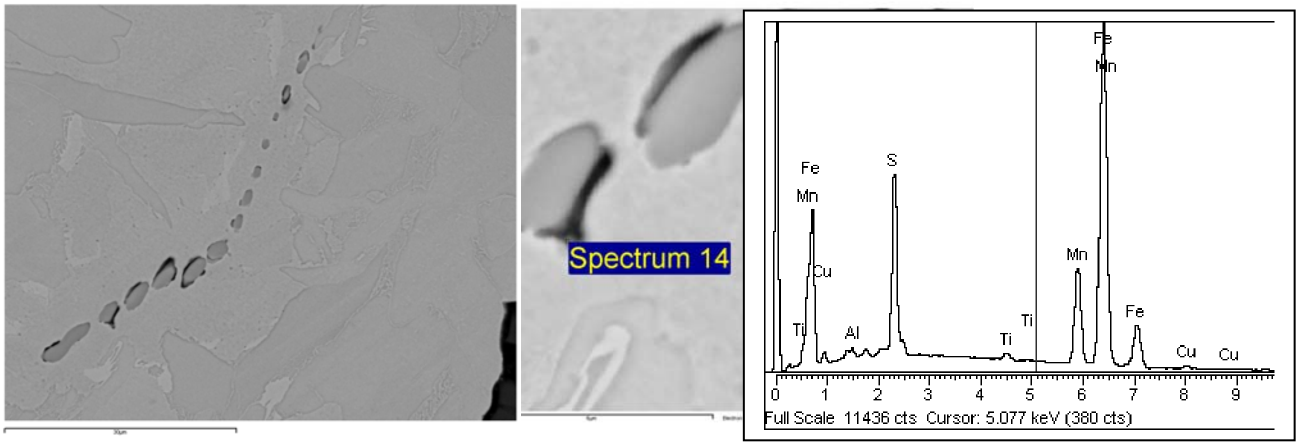

Figure 3 - SEM micrographs of the cross section of the 0.5%Cu containing steel. Cooling rate 200°C/min, test temperature 800°C. On the left, a line of MnS inclusions (magnification 1000x); in the centre, details of Cu segregation at the edges of MnS inclusion (magnification 5,000x); on right, the X-ray spectrum. Reduction in area 25%.(8)

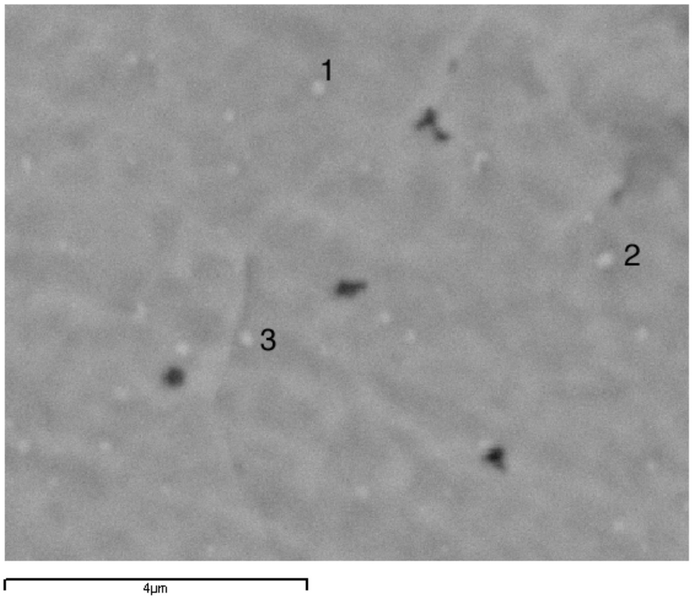

Figure 4 - Cu segregation around particles in the same steel as shown in Figure 2 (~15%Cu average in particles 1, 2 and 3) surrounding CaS/MnS particles in the matrix of the 0.5%Cu steel. Test temperature 800°C. Particle size 180 nm; cooling rate 25°C/min (SEM-BEI)(9). Magnification 10,000x.

TEM investigations have shown that copper also can precipitate as coarse CuS inside the matrix of the steel, as shown in the Figure 5.

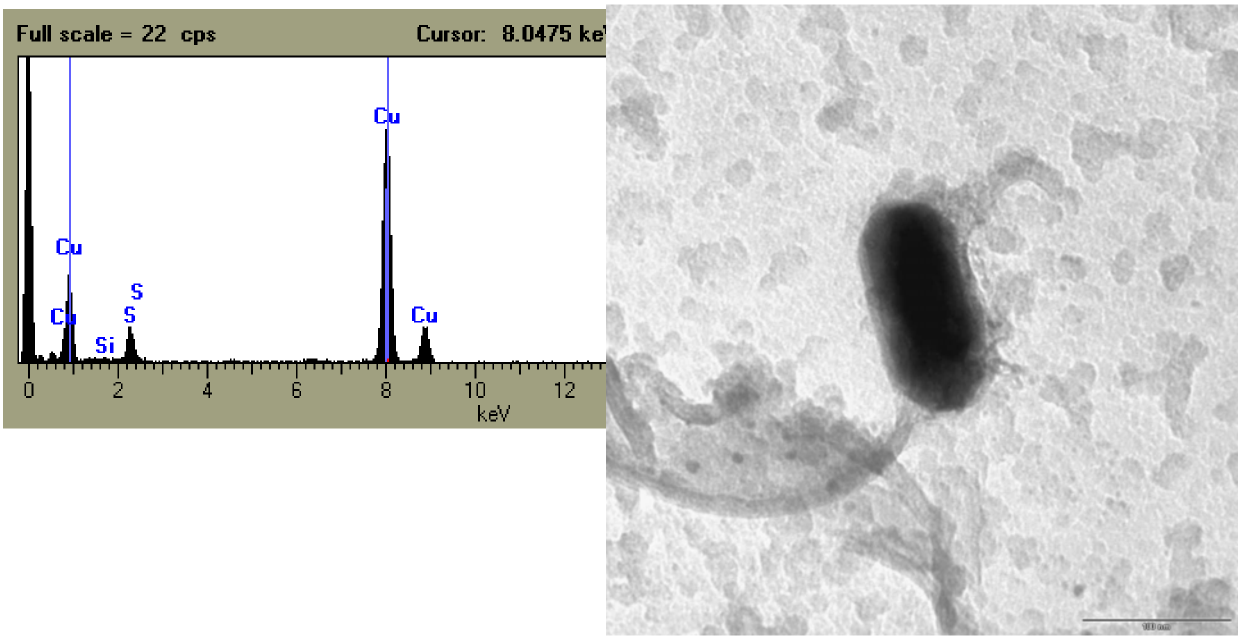

Figure 5 - Coarse CuS precipitate found in 0.5%Cu steel. Cooling rate 100°C/min, test temperature 850°C. Reduction in area 44%(10). The CuS precipitate size is about 100nm. Magnification 200,000x. Note: Although the holding grid for the carbon replica was made of copper, it can be concluded that the precipitate is CuS because no other element was identified.

The influence of nickel on copper segregation was investigated in the steels containing 0.3%Ni and 0.5%Ni following hot tensile tests in an argon atmosphere but SEM investigations could not reveal any segregation of nickel in the vicinity of inclusions. A typical example is shown in Figure 6. In both the steels, containing 0.3%Ni and 0.5%Ni, significant built up of copper was observed in the vicinity of inclusions but no evidence was found of an increase in nickel concentration in these areas.

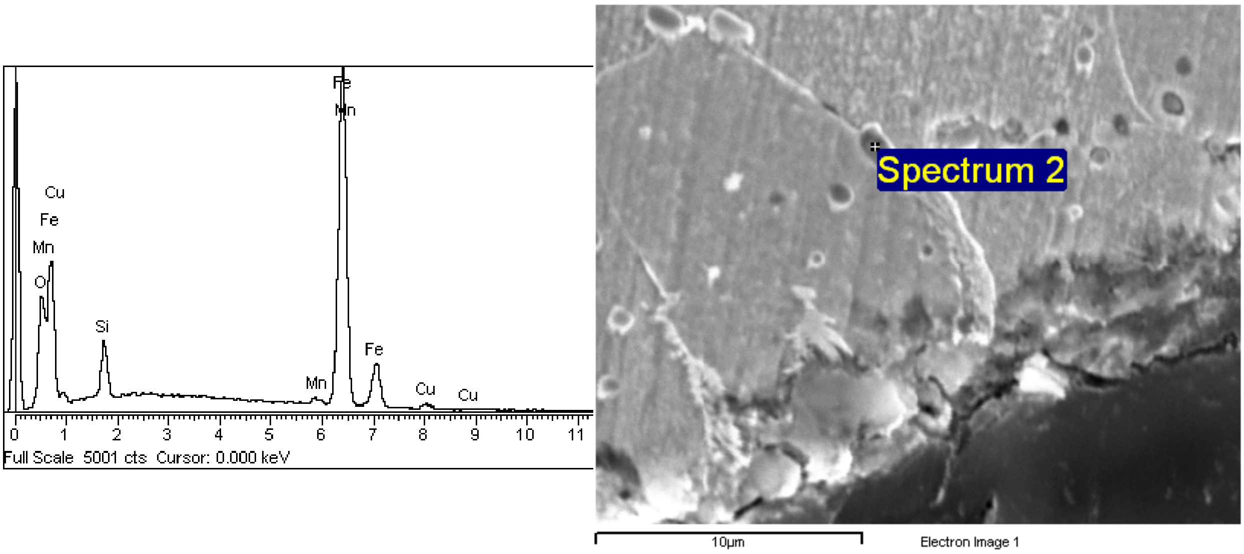

Figure 6 - Segregation of Cu (~2.5%Cu; no Ni) at inclusions in the 0.5%Cu-0.3%Ni steel. Hot tensile test conducted in argon; test temperature 850°C,(8,9) magnification 3,000x

Although the influence of copper and nickel on hot ductility could not be assessed due to excessive oxidation of the specimen as explained above, scanning electron microscopy of the copper and nickel containing steels following hot tensile testing in air revealed interesting results. In the highly oxidized region near the surface nickel as well as copper segregated to inclusions as shown in Figure 7. It actually appears as if nickel alloyed with copper.

Conversely, in areas within the bulk specimen, far away from the oxidized surface, only copper segregation was observed and nickel does not appear to have segregated and alloyed with copper. Hence, the SEM micrographs taken in these areas, of which Figure 8 is a typical example, show no segregation of nickel, similar to the findings of the samples tested under an argon atmosphere.

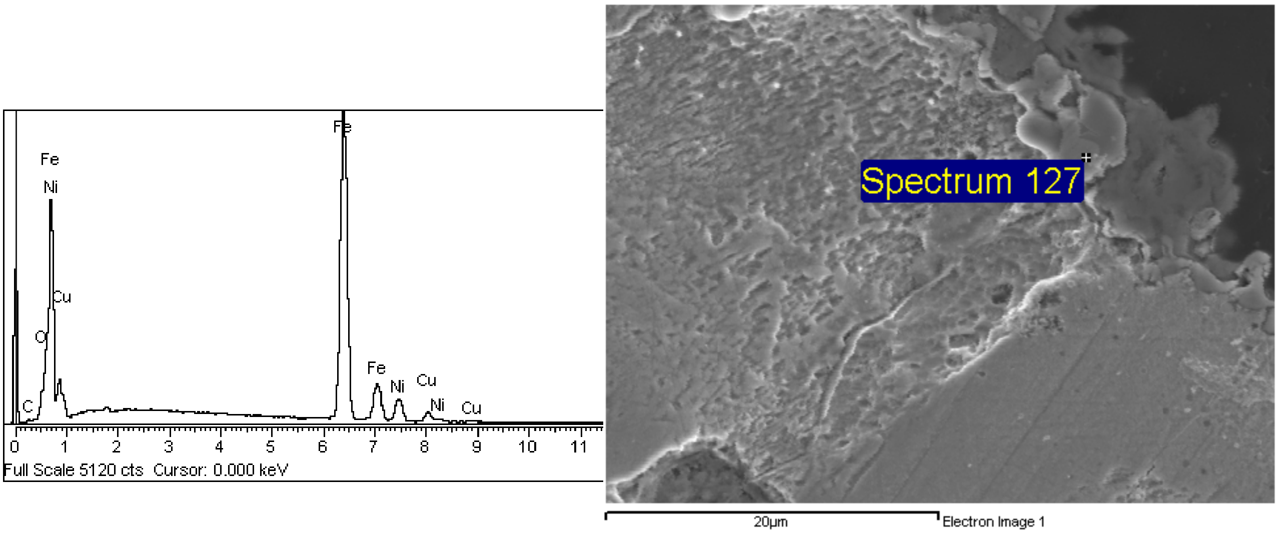

Figure 7 - Segregation (~5.8%Cu, 10.5%Ni) in the 0.5%Cu-0.5%Ni steel, tested in air, test temperature 800°C.(8) Magnification 2,100x

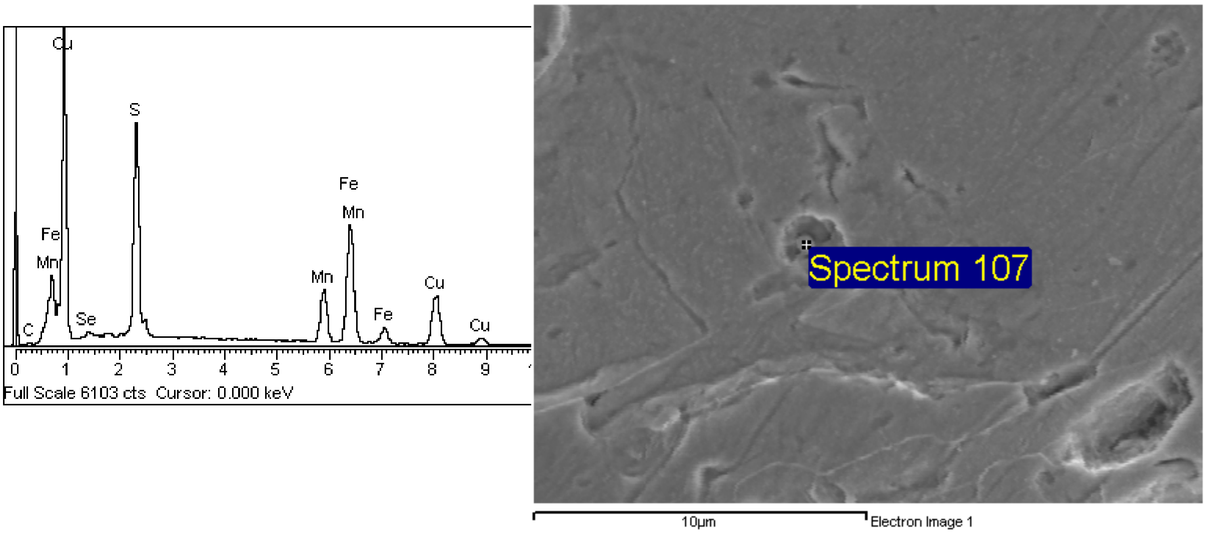

Figure 8 -Segregation (36%Cu; no Ni) in 0.5%Cu-0.5%Ni steel, tested in air; test temperature 800°C.(8) Magnification 4,200x.

3 DISCUSSION

3.1 Simulation of Hot Ductility and Hot Shortness

Clearly current results shows that hot ductility and hot shortness are very different so must be the simulation of the problem and its analyze.

3.1.1 Hot ductility

Hot ductility tensile testing remains a popular laboratory tool to assess the susceptibility to cracking at the operating temperatures during the continuous casting of steel. One of the objections to the validity of such testing is the fact that re-heated specimens are generally evaluated whereas in practice, it is a cast structure that is prone to cracking. In an attempt to make hot tensile testing more industrially relevant, the gauge length of the tensile specimen is often melted and resolidified in-situ. This approach was also taken in this study and although there are valid objections to the use of such tests to assess or simulate cracking during continuous casting, it remains one of the best tests for the simulation of crack formation and growth. The likelihood that cracks will occur during unbending is related to the existence of a ductility trough between the Ae_3 and Ar_3 temperatures in the hot tensile test. The significant decrease of ductility in this temperature range is attributed to stress and consequently strain concentration in the thin film of ferrite that forms on austenite grain boundaries during cooling from the austenite phase. As a general rule, the width of the trough is determined by the composition of the steel, which affects the Ae_3 temperature and the cooling rate, which determines the Ar_3 temperature. The depth of the trough is indicative of the presence of precipitates, which exacerbates crack growth that eventually leads to failure. In this respect, fine precipitates are worse than coarse precipitates. The likelihood that a crack will form during the unbending operation in continuous casting is assumed to be low when the hot ductility as measured by the reduction in area, is greater than 40%(5).

The reduction in area measured in hot tensile tests on samples tested in an argon atmosphere display the classical ductility trough as shown in Figure 1. It is not clear why the 0.5%Cu steel shows a deeper ductility trough than the copper-free steel. The only experimental evidence at our disposal points to the possibility that CuS precipitates by themselves or copper segregation to MnS may contribute to the reduced ductility in this steel. Although there is a suspicious that the role of Ni is by reducing volume fraction of precipitates, it is not clear either why addition of 0.3 to 0.5%Ni to the copper containing steel recovers the hot ductility in samples tested in argon.

3.1.2 Hot shortness

The hot shortness is a completely different phenomenon. It is based on the fact that residuals like copper present in steels are nobler than iron so iron is preferably oxidized which increase the concentration of copper nearby to the oxidized surface of the steel. That segregation of copper has lower melting point than the steel, so it can be liquid at the processing temperature. Obviously that is a condition very favourable for cracking. It is then clear that oxidizing is an important condition to simulate the problem of hot shortness.

However, recent investigations(8,9) show that effects of copper and nickel are not exactly like reported by previous works(3) investigating the hot shortness.

Current results show that the problem of the hot shortness can be more serious because rather than being homogeneously dissolved in the bulk of the steel, copper can also segregates surrounding the inclusions present in the steel as shown in Figures, 2-4. That find is very important since the segregation makes the build up of concentration of copper much faster and higher.

Unfortunately the segregation of copper surrounding inclusions has been found not to affect the hot ductility on tests carried out in argon, so no information can be taken from that on the hot ductility curves.

Addition of nickel recovers the small impairment of hot ductility caused by copper in steels as shown in the Figure 1. Although not clear, it is reported that is because nickel can prevent the precipitation of CuS.

By the side of the hot shortness the effect is opposite. On the occurrence of oxidation, the copper segregation can play a decisive role in causing the cracks by forming the liquid film of copper.

Here the effect of nickel in preventing the cracks of hot shortness is again completely different from previous work(3). Current work shows that nickel effect is by alloying with copper which delays the problem because of the higher melting point of the alloy. Deep oxidization is fundamental once the effect of nickel on the hot shortness is visible only in presence of oxygen, figure 7. However, the effect of nickel cannot be assessed in the hot ductility test carried out even in air, if the examination is far away from the oxidization, figure 8.

The hot ductility test is important to simulate the likelihood of cracking on the hot processing of steel, when the basis of the hot ductility can apply and the oxidization is not a very important variable to be considered. Clearly very limited information concerning to the simulation of hot shortness can be collected by the hot ductility measured by test carried out in argon.

When the occurrence of crack is related to the hot shortness, such test is rather ineffective, because no accurate measurement of reduction of area is possible in samples tested in air, so there is no sense in hot tensile testing in open air. Basically, the same information could be obtained by a simple hot test carried out in air without any strain, followed by microscopy analysis. Quantitative measurements of cracks in a hot compressive test carried out in air could be an useful, easier and simpler alternative option for a closest simulation of the hot shortness. The hot tensile test is then clearly ineffective on simulating the problem.

4 CONCLUSIONS

It is clear that the basis of the hot ductility in assessing the likelihood of crack in hot processing of steels is completely different compared to the analysis of the hot shortness, so both of the problems cannot investigated by the same simulation of hot tensile test. Some conclusions can then be taken from the current work:

1 - The segregation of copper surrounding inclusions is very important in investigating the hot shortness. It can be taken from simple SEM microscopic analysis of the steel;

2 – The single reduction of area values taken from the hot ductility tensile test does not represent any reliable data to be used in the prevention of the hot shortness in Cu containing steels;

3 – Addition of nickel is effective in preventing the hot shortness. However, attention must be taken once its effect in recovering the hot ductility on the hot tensile test has no relationship with its effect in delaying the formation of the liquid film of copper by producing an alloy with copper of higher temperature;

4 – Reduction of inclusions could be an effective way to reduce the possibility of hot shortness once less segregation sites are present in the steel. A patent in this novel theory was recently required;

5 – Hot compressive test carried out in air could be an easier and cheaper alternative simulation of assessment of the hot shortness. That is to be investigated by the authors.

REFERENCES

1 HARLEY, A. J. ESTBURN, P. and LEECE, N. Steelworks Control of Residuals. In: Residuals Additives and Materials Properties, The Royal Society, London, UK *Phil. Transactions A* **295,** (1980) 45-55.

2 MELFORD, D. A.: The influence of residual and trace elements on hot shortness and high temperature embrittlement. In: Residuals Additives and Materials Properties, The Royal Society, London, UK *Phil. Transactions A* **295,** (1980) 89-103

3 FISHER, G. L.: The Effect of Nickel on the High-temperature Oxidation Characteristics of Copper-Bearing Steels, J. Iron Steel Inst., **207** (1969) 7, 1010–1016.

4 VAYNMAN, S.; FINE, M. E. and BHAT, S. P. High Strength Copper Steel for Rail Tank Cars to Bridges. Copper Applications in Metallurgy of Copper and Copper Alloys, 2006.

5 MINTZ, B., YUE, S. and JONAS, J. J.: Hot ductility of steels and its relationship to the problem of transverse cracking during continuous casting. INTERNATIONAL MATERIALS REVIEWS, 1991, **36**, 187-217.

6 MINTZ, B., ABUSHOSHA, R. and CROWTHER, D. N.: Influence of small additions of copper and nickel on hot ductility of steels MATERIALS SCIENCE AND TECHNOLOGY - May 1995 vol. 11.

7 MINTZ, B.; COMINELI, O. and KARJALAINEN, L. P. The Influence of Ni on the Hot Ductility of C-Mn-Al, Cu Containing Steels as a Way of preventing "Hot Shortness". In: 59th ANNUAL CONFERENCE OF ASSOCIAÇÃO BRASILEIRA DE METALURGIA E MATERIAIS, 2004, São Paulo. São Paulo: ABM, 2004. ref. 3638.

8 JARDIM, J. C.; COMINELI, O. and DIPPENAAR, R. Investigations on the Influence of Nickel on the “hot shortness” of C-Mn-Al, Cu containing steels. In: 45th ROLLING SEMINAR - Associação Brasileira de Metalurgia e Materiais, 2008, Porto de Galinhas. Pernambuco: ABM, 2008.

9 COMINELI, O.; KARJALAINEN, L. P. and DIPPENAAR, R. The Influence of Inclusions on the “hot shortness” of Cu Containing Steels. In: 62nd ANNUAL CONFERENCE OF ASSOCIAÇÃO BRASILEIRA DE METALURGIA E MATERIAIS, 2007, Vitória. Espírito Santo: ABM, 2007. ref. 11999.

10 COMINELI, O.; TULING, A.; MINTZ, B. and KARJALAINEN, L. P. The Influence of a Small Ti Addition on the hot ductility of Cu Containing Steels. In: 62nd ANNUAL CONFERENCE OF ASSOCIAÇÃO BRASILEIRA DE METALURGIA E MATERIAIS, 2007, Vitória. Espírito Santo: ABM, 2007.