МАТЕРИАЛОВЕДЕНИЕ И ТЕРМИЧЕСКАЯ ОБРАБОТКА МЕТАЛЛОВ

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HOT SHORTNESS CRACKS FORMATION IN A LOW ALLOY STEEL: INVESTIGATION ON THE CRITICAL CONDITIONS

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Abstract. In this work the effect of the temperature on the growth of the shortness cracks and consequently on the mechanical properties of a 20Mn4 steel was studied. Uniaxial tensile tests were carried out on the thermo-mechanical simulator Gleeble 3800™ in a range of temperatures between 1000° and 1280°C. Heat treatments at different temperatures and holding times were performed in a tubular furnace.

After the tensile test and the heat treatments, the samples were characterised by secondary electron microscopy in order to evaluate the effect of microstructural evolution on the hot shortness behaviour. The copper agglomeration at the metal/scale interface is mainly responsible for the hot shortness phenomenon: for a characteristic range of temperature, a copper enriched liquid phase penetrates along grain boundaries promoting crack formation and hot shortness behaviour.

Keywords: Steel, heat treatments, hot-shortness, mechanical properties, microstructural evolution.

Introduction

Surface Hot Shortness (H-S) is caused by the enrichment of residual elements during oxidation that can give rise to a liquid phase, which subsequently weakens austenite grain boundaries. H-S is not a new problem; it has been known since the early 1900, and the topic arose again in the late 1950 and 1960, when the increasing of the electric arc furnace steel production has led a constant growth of copper amount in the final product. The results of the research showed that copper was the main element responsible of the formation of surface cracks or “surface cracking” [1]. At the end of the 70’s the main mechanism of the hot shortness was explained by the diffusion the copper in the steel during heat treatment [2].

The H-S behaviour is defined as the embrittlement of the metal in the field of the hot working, and it occurs mainly in steels containing traces of elements nobler than iron and with low melting point, in particular copper [3]. At temperatures higher than 400°C, the oxide scale of the steels is formed by three continuous and compact layers: hematite, magnetite and wustite. In these conditions the nobler alloying elements remain unoxidized and concentrate at the steel/oxide interface, where they form low melting zones, which are responsible of the so called heat embrittlement in the material structure.

In copper containing steels, oxidized at temperature higher than 900°C, large areas of Cu were found at the steel/oxide interface. The H-S in these steels is caused by the penetration of the liquid Cu into the cracks (Cu is completely liquid a 1100°C) at the surface or along grain boundaries and it depends strongly on the temperature, time, residual elements concentration and atmosphere [4, 5].

The characteristics and causes of the H-S phenomenon take different connotations: at lower temperatures the hot-shortness depends only on the rate and the intensity of the scale formation on the material surface, whereas at higher temperatures, the process of occlusion of Cu into the scale is more relevant, and the hot-shortness depends mainly on of copper content [6–9].

According to Salter, the main factors that influence the evolution of the H-S phenomenon, excluding the steel oxidation rate, are in order of importance: copper solubility in austenite, penetration of the molten copper phase along grain boundary (wettability), and temperature of diffusion of the copper enriched phase [9–12].

In this work, the operative conditions were selected for the realization of tests in order to determine the conditions which are useful to block the formation of copper rich phases and/or to minimize the phenomenon of copper enrichment at grain boundaries and. The tensile tests at elevated temperature were conducted in order to evaluate the effects of the microstructural changes on ductility characteristics of the investigated steel.

Experimental

The material studied in this work is a 20Mn4 steel commonly employed for structural application. It was supplied as round bar (60 mm diameter). The chemical composition of the steel is reported in Table 1.

The copper content in this steel is 0.33% wt%, but the effect of copper is increased by the presence of Sn, according to the Eq. 1 [13]:

$$\%Cu_{eq} = \%Cu_{real} + K \cdot \%Sn_{real} \quad (1)$$

where $\%Cu_{eq}$ is the "equivalent copper content" and K is a constant between 7 and 10. Assuming an intermediate value of K equal to 8.5, the equivalent copper value corresponds to 0.495 wt%. The presence of Sn diminish copper solubility in steel and thus it promotes the formation of dangerous enriched zones. Moreover, its presence produces the lowering of the melting point of the enriched copper phase, facilitating the penetration of the molten phase along the grain boundary.

The tensile tests were carried out on the thermo-mechanical simulator Gleeble 3800™ in a range of temperatures between 1000°C and 1280°C. The metal sample was heated up to the testing temperature by Joule effect at 10°C/s, held in temperature for 15 minutes, and then strained until fracture by applying a constant strain rate equal to 10^{-1} s^{-1}. After testing, the sample was left cooling in calm air. The ductility characteristic chosen as a reference in this study was the total elongation of the sample at fracture.

In order to determine the conditions of temperature, which are useful to minimize the phenomenon of copper enrichment at grain boundaries and to block the formation of copper rich phases, samples of steels underwent heat treatments in a tubular furnace for 60 min in a temperature range of 1000–1230°C in air and in atmosphere with a lower amount of oxygen (15% and 10%) (Table 2).

The treatment temperature was reached by a heating rate of 10 °C/min. The samples were cooled in air.

After heat treatments, the cross-section of specimens was included in epoxy resin and polished by metallographic procedures and then characterized by a Cambridge Stereoscan 440 SEM equipped with a Philips PV800 EDAX probe.

Table 1

Chemical composition of 20Mn4 steel (wt%)

C	Mn	S	P	Si	Cr	Ni	Cu	Sn	N	Mo
0.2104	1.044	0.0259	0.0118	0.1699	0.0776	0.0732	0.329	0.0195	0.00113	0.0283

Table 2

Conditions of the heat treatments.

	1000°C	1050°C	1100°C	1140°C	1180°C	1230°C
Air, 60 min	x	x	x	x	x	x
10% O_2 60 min		x	x	x	x	x
15% O_2 60 min		x	x	x		

Results and discussion

A preliminary characterization of the round bar of the 20Mn4steel was performed before the tensile test and the heat treatments to examine the copper distribution in the structure. In the central areas of the bar, manganese sulphides were found, while copper segregation was not detected. Near the surface of the round bar, small particles of the copper enriched phase were observed at grain boundary. The average size of these agglomerates was in the order of the micron, and their depth from the surface was about 15 μm (Fig. 1). This agglomeration of copper is the consequence of the previous hot working, to which the steel has been subjected to obtain the final shape.

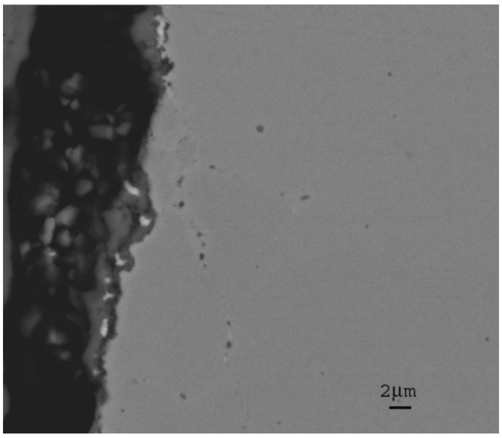

Fig. 1. SEM-BSE image of the section of initial round bar (interface scale-substrate)

Therefore, the samples for all experiments were obtained from the central area of the round bar, where no segregation of copper was detected.

Uniaxial tensile tests

Samples of steel underwent to tensile test at temperature between 1000°C and 1280°C. The photos of the strained samples are shown in Fig. 2, where it is evident the different amount of post-necking deformation exhibited by the steel at different testing temperatures.

In Fig. 3 the graph of the total elongation of the samples at fracture as a function of the testing temperature is reported: the effect of liquid embrittlement due to presence of copper is well exhibited. As a matter of fact, the steel ductility is drastically reduced at temperatures higher than 1000°C and lower than 1230°C, confirming that above the copper melting temperature the copper enriched phase, which grows at the interface metal/scale, promoted the formation of surface cracks and embrittled the steel structure. At higher temperatures, the oxidation rate is so high that the copper enriched phase is encapsulated on the scale and the metal exhibits higher ductility. However, it was expected higher ductility of the sample heated at 1230°C, but tensile tests were made with higher heating rate which involves a lower sample oxidation. In these conditions the temperature range where the hot shortness is exhibited is shifted at higher temperature.

The SEM-BSE images of the section of the samples submitted to tensile test at the different temperatures are reported in Fig. 4. As expected, at the lowest temperature (1000°C) the sample does not exhibit agglomeration of copper at the interface metal/scale. Increasing the temperature (1140°C and 1230°C) a copper enriched phase (lighter zone) at the interface scale/metal and along grain boundary was detected. Moreover, in the sample tested at 1140°C many intergranular surface cracks, due to the penetration of the liquid along grain boundaries combined with the oxidation of the steel, were observed [4]. At the highest temperature (1280°C) globules of copper were found in the scale.

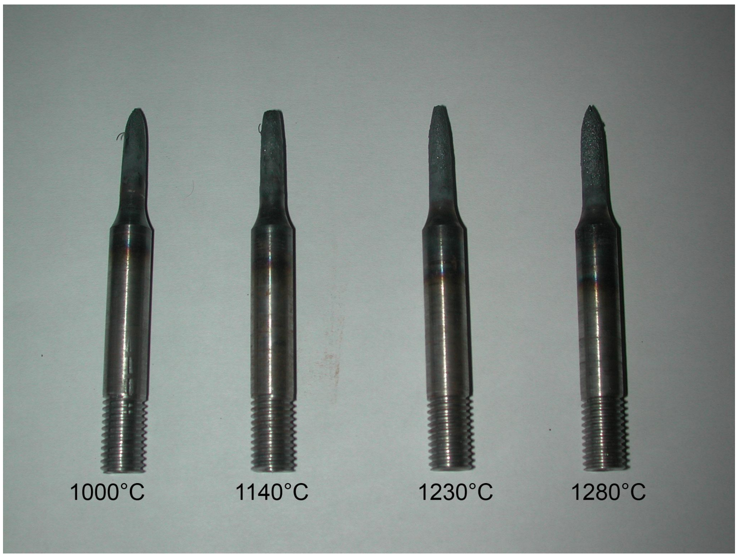

Fig. 2. Image of samples after mechanical tensile test at different temperatures

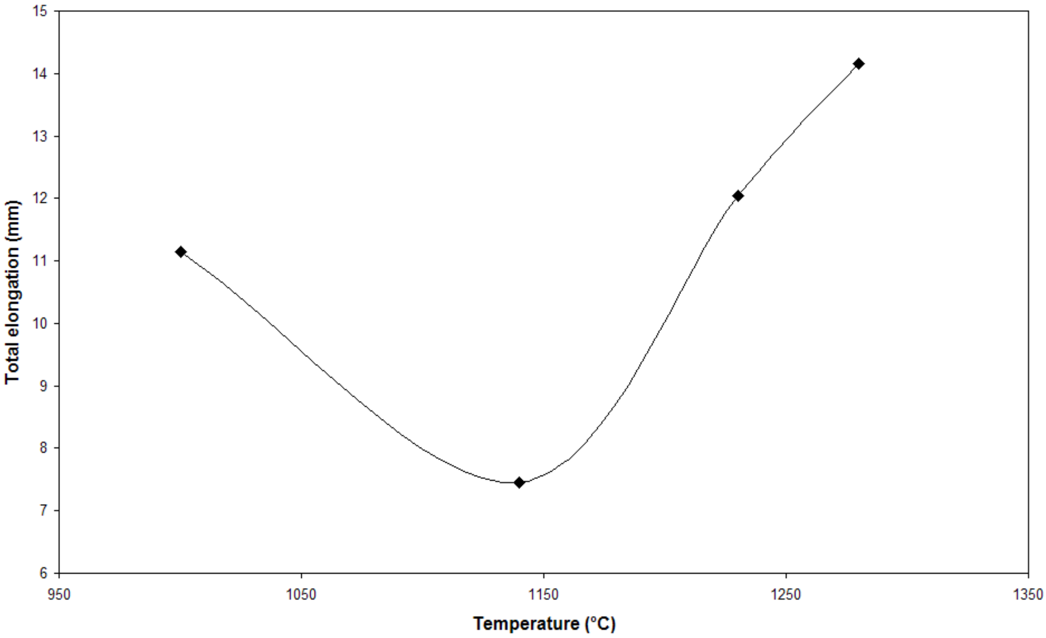

Fig. 3. Graph of the total elongation of the samples at fracture as a function of the testing temperature

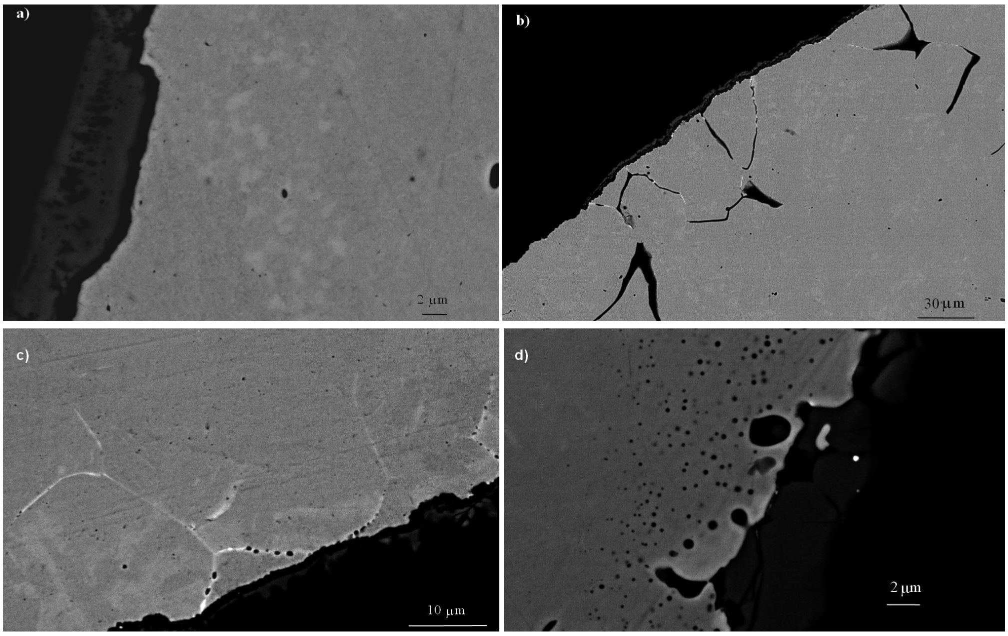

Fig. 4. BSE-SEM images of the section of the samples after mechanical tests at a) 1000°C; b) 1140°C; c) 1230°C; d) 1280°C

Therefore, after the tensile test at elevate temperatures, it is possible to conclude that the hot shortness behaviour in this steel is more pronounced at 1140°C.

For a better understanding of the effect of temperature on the segregation of copper at the surface of this steel, heat treatments at different temperatures, were carried out in a tubular furnace.

Effect of the temperature

The first tests was carried out at 900°C and 1000°C, at which temperatures is expected the copper enriched phase remains solid, because this temperature is just lower than the melting point of the copper. As a matter of fact, the SEM analysis revealed that the globules of copper enriched phase were localised inside the scale and distributed parallel to the interface metal/oxide (Fig. 5, Fig. 6). At 900°C was possible to detect the presence of only some small globules in the scale, while at 1000°C their presence was more pronounced and with higher dimension. In both cases it was not found penetration of copper into the material, since copper enriched phase remains solid during the length time of the treatment. Therefore, the treatments carried out at lower temperatures than the one of enriched phase liquefaction would not produce H-S, and thus do not represent a dangerous condition for the steel hot working, in agreement with the results of tensile tests. In fact, the embrittlement under hot working is closely linked to the penetration of the molten phase along the grain boundary and to the formation of aggregates of lens situated at the interface between the metal and the scale, which may transform into molten phase during the treatment. As long as the globules remain occluded in the oxide scale and separated from the metal, the metal is protected by the embrittlement.

In Fig. 7, the section SEM-BSE images of the samples surface treated at 1050°C, 1100°C and 1230°C for 60 min are reported. It was observed that after the heat treatment at low temperatures, 1050° and 1100°C, a planar interface metal/scale has developed, while at 1230°C the interface is characterized by a noticeable roughness [14]. In the first case, the copper enriched phase is distributed along the interface and at grain boundary, and it is constituted mostly by pure copper. Moreover, at 1050°C the copper enriched zones was predominant near the oxidized interface whereas increasing the temperature at 1100°C the agglomeration of copper is more pronounced at the grain boundaries. This behaviour is always inter granular; because the copper rich phase at this temperatures is molten [15] and it wets the austenitic grain boundary. In fact, it was observed a penetration along the boundaries due to the high diffusivity of the iron in the molten phase of copper, in a similar way as it happens in the alloys FeCu [16]. At 1230°C the interface metal/scale was irregular and copper accumulation near the interface was not detected: the particles of copper enriched phase were occluded inside the scale. These occlusions were also constituted by a significant amount of nickel (Fig. 7d). At this temperature the oxidation rate was higher, therefore the oxidised interface was able to occlude the copper enriched phase into the scale before the melting was completed. In addition to the formation of an external scale, the steel was subjected to an internal oxidation behaviour. This behaviour is characterized by the development of a 1–2 µm globular FeO particles along the austenitic grain boundaries.

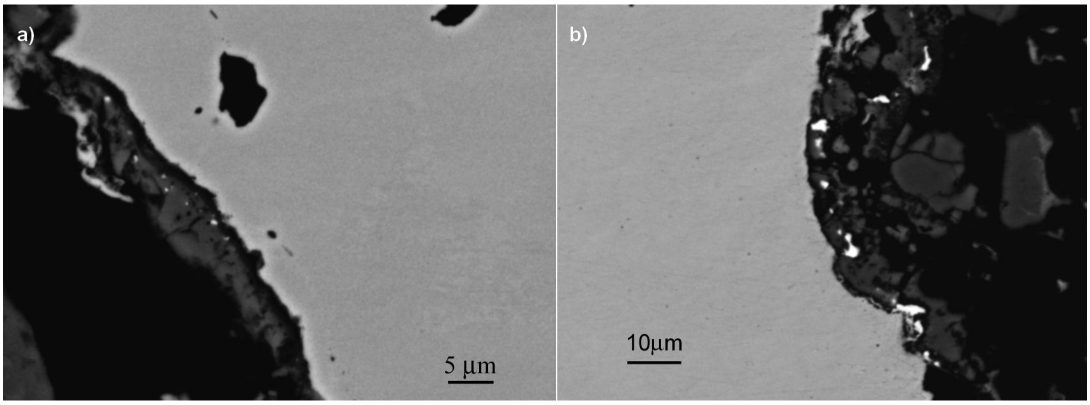

Fig. 5. BSE-SEM image of the section of the sample processed for 1h, in atmosphere with 20% oxygen, at a) 900°C and b) 1000°C

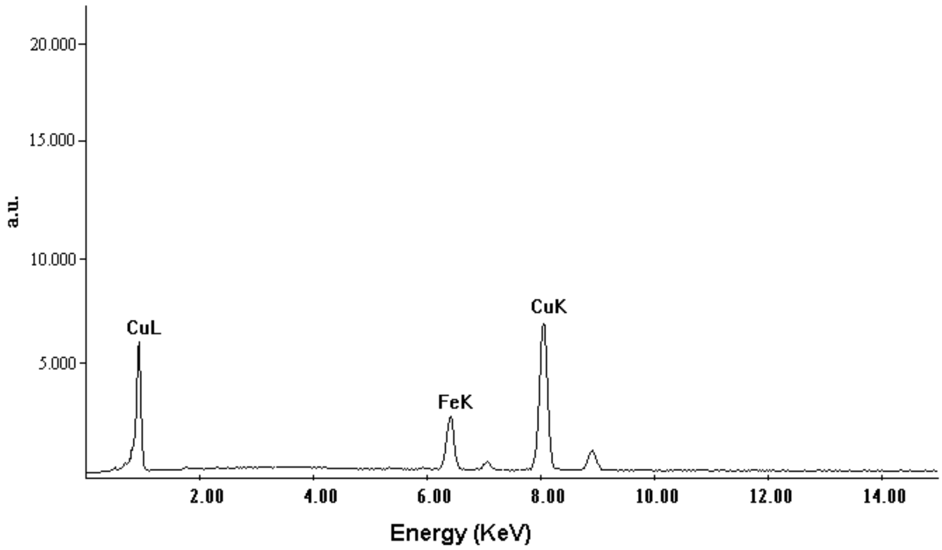

Fig. 6. EDS analysis in correspondence of the white globules in the scale of the sample treated at 1000°C for 1h

At temperature between 1050°C and 1100°C, the formation of small dihedral angles (θ) was observed between the austenitic solid phase and the molten phase, near the triple point.

The value of the dihedral angle θ is function both of the surface tension between the phases and of the wettability between the molten phase and the surrounding solid phases, which induces the so called “intergranular” penetration. [17] .This value can be determined by the equation (2)

$$\frac{1}{2}\cdot\frac{\gamma_{Fe-Fe}}{\gamma_{Fe-Cu}}=\cos\frac{\theta}{2} \qquad (2)$$

where the surface free energy of the austenite grain boundary is denoted by γ_{Fe-Fe} and the surface free energy of the austenite-molten phase boundary is denoted by γ_{Fe-Cu}. The shape of the molten phase adjusts itself such that the surface energy of the system is minimized.

The maximum of H-S is observed for temperatures where the value of θ approached zero, which is the value for complete wettability of the austenitic grain by the liquid copper enriched phase. Moreover, at these temperatures it was observed the highest copper enrichment at the interface metal/scale [9].

Surface tension plays an important role in the H-S embrittlement of steel. The value of the equilibrium dihedral angle depends on the temperatures of treatment, and the minimum value is reached when maximum amount and maximum penetration of the enriched phase along the austenitic grain boundary occur. When combination of maximum amount of molten phase with the deeper penetration of the molten phase along the grain boundaries is obtained, the maximum H-S effect takes place.

The treatments made at lower temperatures of 1050° and 1100°C exhibited the formation of the dihedral angles between the molten phase and the austenite; the penetration of the molten phase appears evident at both temperatures; the molten phase enclosed completely the first grain beneath the surface.

At 1230°C, the copper enriched phase is instead enclosed in the scale, and shows a globular morphology. There were no signs of penetration along the grain edge. As discussed above, at higher temperatures the high diffusion rate of the copper allowed the migration of copper inside material preventing the formation of the enriched zones near the interface; the part of copper which is not able to diffuse inside of the material is quickly enclosed in the oxide scale. Because the wettability between the oxide and the molten phase is very low, copper and nickel globules appear.

Treatment at intermediate temperature between 1100°C and 1230°C were carried out. At 1140°C, Fig 8a, the molten phase forms a semi continuous layer along the interface with a lens morphology, as exhibited in the samples treated at 1100°C. However, a deeper penetration of the molten phase (about 50 μm) was exhibited along the grain boundary than that detectable at 1100°C and an early stage of entrapment in the inner zone of the oxide scale was observed. The samples treated at 1180°C, shown in Fig. 8b, exhibited a reduction of copper enriched zones, either near the interface or in intergranular zone and the presence of globules of copper occluded in the scale. Moreover an increase of the irregularity of the interface metal/scale, due to the higher metal oxidation rate, was observed, with a structure more similar to that one observed at 1230°C. Therefore, the structure obtained at 1140°C was the more prone to induce H-S behaviour, while at higher temperature the increased oxidation rate combined with the higher diffusion rate of the copper allowed the migration of copper inside material and gradually reduces the formation of the enriched zones near the interface, which are the main responsible of H-S behaviour.

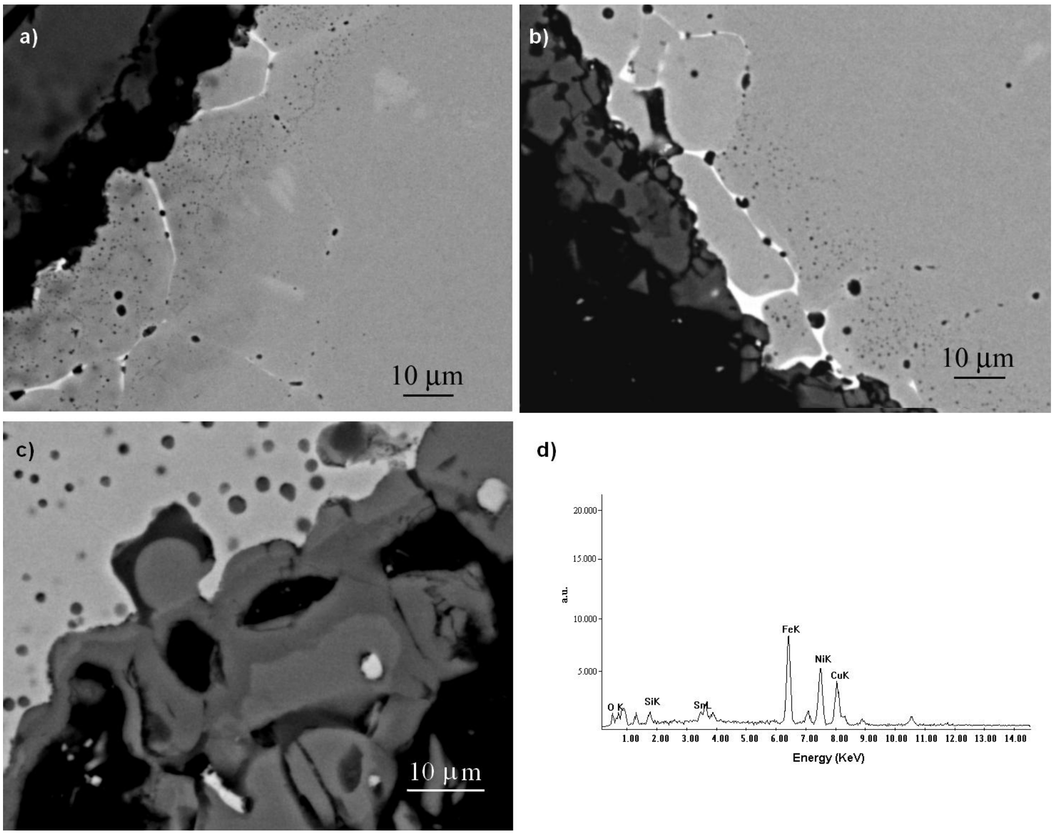

Fig. 7. BSE-SEM images of the section of the samples processed at different temperatures for 1 h: a) 1050°C; b) 1100°C; c) 1230°C; d) EDS analysis in correspondence of the white globules in the scale of the sample treated at 1230°C for 1h

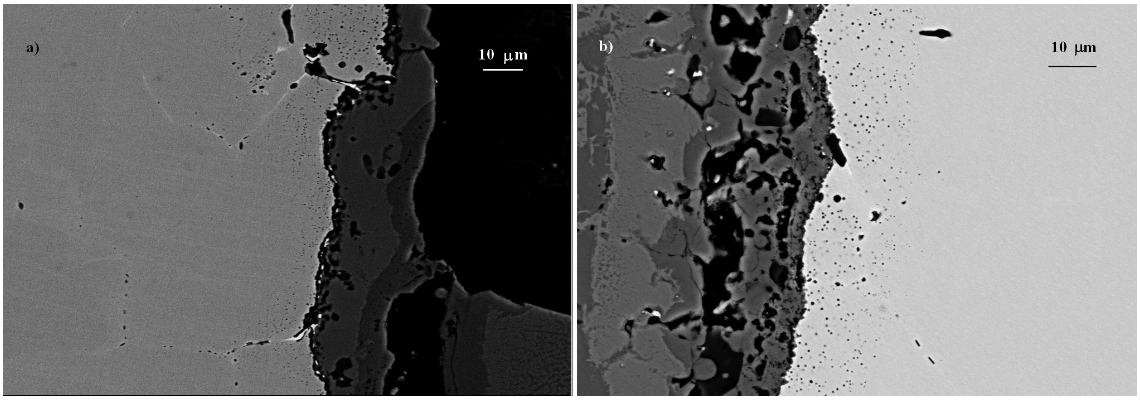

Fig. 8. BSE-SEM images of the section of the sample heat treated for 1h at a) 1140°C and b) 1180°C

Effect of oxygen concentration in treatments atmosphere

The heat treatments in atmosphere with a lower amount of oxygen content (15% and 10%) were carried out at 1050, 1100, 1140, 1180 °C. The reduction of oxygen concentration in the atmosphere produces a reduction of oxidizing rate, with the subsequent reduction of the copper enriched phase penetration. In the sample treated at 1140°C the copper phase developed at the interface, in a globular or lens morphology, but its penetration along the grain boundary is extremely reduced when atmosphere contains 15% of O_2, and it is negligible at 10% of O_2 (Fig. 9 a, b). From the results of the characterization of the treated samples at lower oxygen content it is possible to assert that when the oxygen content in the atmosphere is reduced the evolution of the molten phase in the material is ascribable at those observed at lower temperature. This is due to a reduced oxidation rate, which depletes the enrichment of copper at the interface and, as a consequence, the penetration of the molten phase along the grain boundaries. Therefore the H-S behaviour was reduced at lower oxygen content in atmosphere.

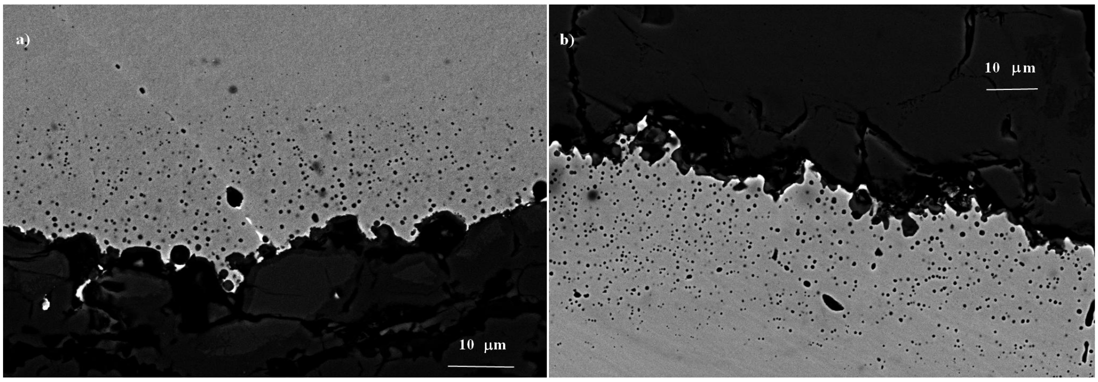

Fig. 9. BSE-SEM images of the sample treated at 1140°C for 1h in atmosphere with a) 15% oxygen and b) 10% of oxygen

Conclusions

The effect of heating temperature on the embrittlement of a 20Mn4 steel when subjected to hot working was investigated in this work

The presence of copper enriched phases near the surface, especially at the metal/scale interface and at grain boundaries is due to the oxidation process which produces the formation of the surface oxide scale and the penetration along the grain boundaries of the oxide into the steel.

The mechanical tests carried out on the thermomechanical simulator Gleeble 3800™ evidenced a lack of ductility of the steel for temperature higher than 1000°C and lower than 1280°C, due to the formation of a layer of copper enriched phase at the metal/scale interface and to its penetration along the grain boundaries.

From the heat treatments in furnace for a time of 1h, it resulted that the copper-rich phase forms at the interface metal/scale at temperature in the range of temperature between 1000°C and 1230°C. At temperature up to 1000°C the copper enriched phase remained on solid state and therefore is entrapped into the oxide scale in the form of globules well distributed on the oxide scale. In these conditions the steel show high ductility and the hot shortness behaviour is not verified. At temperature in the range between 1050–1180°C the copper enriched phase melts and grows at the interface metal/scale and forms a semi-continuous layer. This molten phase penetrates along the grain boundaries into the metal, inducing the formation of globules inside the steel which embrittled the structure and could promote the formation of surface cracks. For temperatures above 1230°C, the oxidation rate is so high to enclose entirely the surface grains and entraps the copper enriched phase. In these conditions the presence of molten copper enriched zones, inside the material structure is strongly reduced and the steel has not been affected by hot shortness.

A reduction in the oxygen content in the atmosphere depleted the oxidation rate and the enrichment of copper at the interface and, consequently, the penetration of the molten phase along the grain boundaries. Therefore, in order to limit the impact of hot shortness, the suggested hot working temperature for this steel is over 1230°C. An alternative may be to operate at temperatures lower than 1230°C by reducing the oxygen amount in the atmosphere.

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ИНФОРМАЦИЯ О СТАТЬЕ НА РУССКОМ ЯЗЫКЕ

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ОБРАЗОВАНИЕ ГОРЯЧИХ ТРЕЩИН В НИЗКОЛЕГИРОВАННОЙ СТАЛИ: ИССЛЕДОВАНИЕ КРИТИЧЕСКИХ РЕЖИМОВ

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Аннотация. В работе изучено влияние температуры на рост хрупких трещин и, соответственно, на изменение механических свойств стали марки 20Mn4. На термомеханической моделирующей установке Gleeble 3800™ проведены испытания на одноосное растяжение в диапазоне температур от 1000 до 1280°С. Термические обработки при разных температурах и времени выдержки осуществлялись в трубчатой печи.

После проведенных испытаний на растяжение и термических обработок образцы были проанализированы при помощи растровой электронной микроскопии для оценки влияния эволюции микроструктуры на протекание горячеломкости. Явление горячеломкости в основном обусловливает скопление меди на поверхности раздела металл-окалина: в характерном диапазоне температуры жидкая фаза, обогащенная медью, проникает по всем границам зерен, способствуя образованию трещин и проявлению горячеломкости.

Ключевые слова: сталь, термическая обработка, горячеломкость, механические свойства, эволюция микроструктуры.

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BAINITE STEEL: STRUCTURE AND WORK HARDENING

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Abstract. Using the methods of transmission electron diffraction microscopy, a quantitative evolution analysis of defective and carbide subsystems of medium-carbon steel with a bainite structure under a compression strain up to 36% has been performed. A quantitative analysis of carbon redistribution has been carried out, as well as the dependence established of the concentration of carbon atoms arranged in a crystal lattice of α- and γ-iron on structural defects in cementite particles lying in a number of bainite plates and intra-phase boundaries, and on the degree of deformation.

It has been demonstrated that scalar dislocation density, material volume with deformation twins, a number of stress concentrators, the amplitude of crystal lattice curvature-torsion, the disorientation degree of fragments are increased with the growth of the degree of deformation and average longitudinal fragment sizes are decreased. The long-range stress fields have been estimated. The possible causes of the different stages of parameter changes of the carbide phase and dislocation substructure with deformation have been discussed.

Strengthening mechanisms with the boundaries of the plates and fragments, scalar dislocation density, long-range stress fields, and cementite particles, the interstitial atoms have been estimated. It has been shown that the largest contribution to the amount of work hardening of the steel examined leads to substructural hardening (hardening due to long-range internal stress fields and structure fragmentation) and solid-solution hardening, due to the introduction of carbon atoms into the crystal lattice of the ferrite.

It has been suggested that the cause of softening of steel with a bainite structure at high (over 15%) degrees of deformation is the activation of the process of deformation fine-scale twinning.

Keywords: hardening, bainite, deformation, cementite, dislocation substructure, mechanisms, steel.