Journal of Metals, Materials and Minerals. Vol.16 No. 1 pp. 13-19, 2006

Fractures of As-Cast Low-Carbon Steels in the Plasticity Degradation Area at High Testing Temperatures

Antonín ŠEVČÍK[1] and Jarmila ŠEVČÍKOVÁ[2]

[1] *Institute of Material Research SAS, Košice, Slovak Republic*
[2] *Dept. of Materials Science, Faculty of Metallurgy, Technical University, Košice, Slovak Republic*

Abstract

The work studies the fractures of low-carbon steels after tensile tests in a temperature interval from ca 1200°C, where a plasticity degradation takes place, up to the liquid phase formation temperature. Statistical analyses of the temperature dependence of the reduction of area were made with the aim to determine integral effects of the chemical composition on the plasticity course.

In the plasticity degradation temperature area, intercrystalline fractures occur, which have several morphologies, even within the same steel grade. Besides typical intercrystalline fractures, a fine-grained, interdendritic fracture often occurs. A specific case is the failure of steels in the $\gamma + \delta$ two-phase area, where fractures are formed at the phase interface. According to the failure type, fractures have been classified under 4 basic groups.

The fracture process and subsequently the plasticity degradation temperature and the plasticity course are determined by the intensity of decohesion of polygonal boundaries of austenite grains, or δ-ferrite grains, and of boundaries of interdendritic space. With increasing content of phosphorus, which segregates on the polygonal grain boundaries, the plasticity degradation start temperature decreases. At the highest testing temperatures, phosphorus degrades the cohesive strength of grain boundaries by formation of a liquid phase.

The slope of the temperature dependence of plasticity degradation and the temperature of plasticity degradation to a nearly non-measurable level, th5, are mainly determined by the Al/N ratio. An increase of the Al/N ratio, which was in the interval from 2.2 to 16 for the tested steels, shifts the th5 temperature to the right.

Introduction

During continuous casting of low-carbon steels, various surface defects are formed. The study of high-temperature properties of material and the subsequent modification of technology is one of the ways to eliminate them. The high-temperature properties of as-cast low-carbon steels are tested under various experimental conditions and using various testing equipment.(1-3) Tensile tests are often used, where the reduction of area is usually used as a criterion.

At medium testing temperatures, steel is in the austenitic area and the reduction of area is mostly high, even above 99%. At the highest testing temperatures, above ca 1200°C, plasticity decreases. To evaluate the degradation of high-temperature values, the literature uses such terms as a zero strength temperature and a zero plasticity temperature.(4) The zero strength temperature is a limit temperature, below which there are measurable ultimate strength values. As stated by.(4) this temperature corresponds to at least the 75% share of the solid phase in a material. The zero plasticity temperature is lower than the zero strength temperature, since measurable plasticity values only exist in a material without the presence of the liquid phase. Several authors state that phosphorus degrades the cohesive strength of grain boundaries and at the highest testing temperatures it forms liquid eutectics on grain boundaries.(5-6) The kinetics of phase transformations in the $\gamma + \delta$ area was studied in situ, using "laser scanning confocal microscopy", described in.(7-8) The results of these works can be applied in studying the morphology of fractures, formed in this area.

Regarding the chemical composition, low-carbon steels can be considered as a multi-component system, where, besides C, Mn, Si, P and S, trace contents of As, Sb, Sn, B or other elements occur. In steels produced using the scrap process, increased contents of certain elements occur, which may adversely influence the high-temperature properties, such as Cu.(3, 9) Fractures are a result of the grain boundary strength weakened by segregation processes, where several interaction phenomena occur.(10-11) In steels with low carbon contents, Transformation $\gamma \rightarrow \delta$ takes place and fractures are formed, in the dependence on the chemical composition of steels, in austenite, in the two-phase $\gamma + \delta$ area or in δ-ferrite.

High-temperature plasticity tests have a conventional nature and their results are, besides the chemical composition and the time-temperature history of material, also dependent on other factors, such as the loading method and the strain rate.(12) studied under laboratory conditions the susceptibility of low-carbon steels to the crack formation at the highest testing temperatures in relation to defects that are formed in a mould. Based on tests of high-temperature properties and calculations made using the finite element method with special software, they state that cracks in steels where the peritectic reaction takes place are formed at the strain values above 2%. For steels with very low carbon contents, the limit strain value is 1.5% and for steels with high carbon contents this value is 1%. Within the strain rate from 5 to 50 10-4 .s-1, selected for the experiments, its influence on results was not observed.

The presented work is mainly aimed at studying fractures formed in the plasticity degradation area at the highest testing temperatures, above ca 1200°C. The influence of the chemical composition and the structure on the decrease of plasticity in this area was evaluated using statistical regression analyses. The tests were made after constant heating conditions, at the strain rate of 4.7 10-2 .s-1 . The selected strain rate is relatively low and, in the authors' opinion, it should approach to real loading conditions of material during continuous casting of steels.

Material, Experimental Procedures

The experiments were made on a high-temperature tensile testing facility, which consisted of a high-frequency generator, a tensile testing machine and measuring with control equipment. The temperature-time heating regime was controlled by dedicated software. Specimens with the diameter of ca 6 mm were placed in silica glass tubes and were protected by the argon atmosphere. The testing regime consisted of ca 3.5-minute heating to 1350°C, 1-minute dwelling at this temperature, 1-minute heating or cooling to the test temperature and 1-minute dwelling at the test temperature. Dwelling at 1350°C was included in the test in order to model slabs heated in pusher-type furnaces. The tests were made on 22 commercial low-carbon steels made in basic oxygen furnaces and electric furnaces and on two steels with typical chemical compositions. The commercial steels had the carbon contents from 0.006 to 0.20%, Mn from 0.012 to 1.56%, Si from 0.007 to 0.432%, S and P from 0.003 to 0.015%. Among these steels, 5 steels were microalloyed with various combinations of V, Nb and Ti. The contents of the other available elements were in the following intervals [%]: Al <0.02; 0.06>, N <0.009; 0.012>, Cu <0.012; 0.2>, Cr <0.007; 0.070>, Ni <0.006; 0.070>, As <0.0024; 0.01395>, Sn <0.0007; 0.0061>, Sb <0.0011; 0.0043>, B <0.001; 0.0062>. For As, Sb and Sn, both heat and local chemical analyses were available; for the other elements, only heat analyses were available.

The reduction of area and the ultimate tensile strength were measured on the tested specimens. Fractures were studied using SEM and light microscopy on longitudinal sections of fracture areas.

Results

The results course of the reduction of area of all the tested steels is documented in (Figure 1a). Schematically, it can be depicted by designating characteristic points and temperature intervals according to (Figure 1b). The th temperature shows the start of plasticity degradation and the th5 temperature defines the decrease of plasticity to a nearly non-measurable level, i.e. the reduction of area of 5%. The th5 temperature was selected, because the quantification of the temperature at

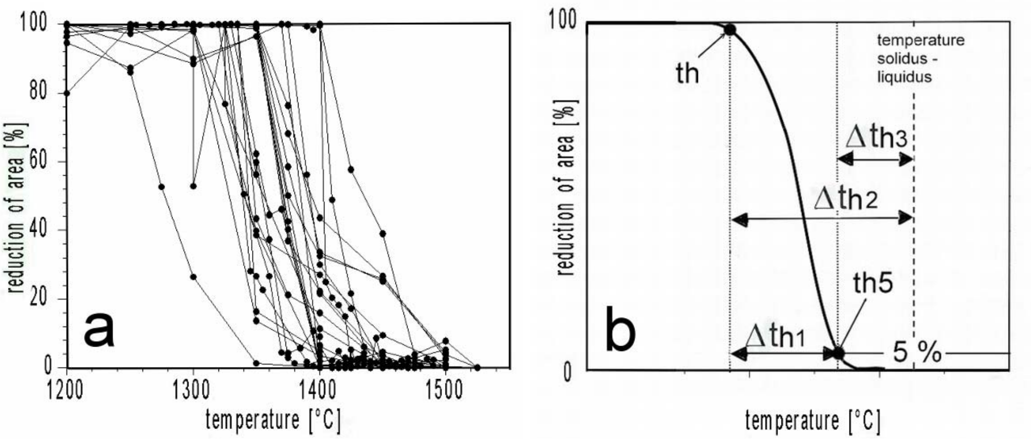

Figure 1. Course of reduction of area of tested steels at temperatures above 1200°C (a) and scheme of characteristic points and temperature intervals (b)

which the reduction of area falls to zero is rather inaccurate.

The fractographic analyses of the specimens tested above the th temperature show that fractures have several different morphologies. Based on SEM and light microscopy observations of longitudinal sections, the fractures can be classified according to their formation mechanism (Figure 2) under the following groups.

In specimens tested at the highest temperatures, fractures were often formed on molten boundaries of austenite or δ ferrite grains (Figure 3) – A type fractures.

The occurrence of variable morphologies was relatively frequent – B type fractures, which are documented, for example, in (Figure 4) These fractures, which are fine-grained form the microscopic point of view, are formed by separation in the original interdendritic spaces (Figure 2) The B type fractures were more frequent in steels with medium or higher carbon contents and in steels with higher Mn contents. Specimens with this fracture morphology had, in certain cases, the reduction of area as many as several tens of per cent. At highest test temperatures, the articulation of the B type fractures was not so marked.

The group C (Figure 2) includes typical intercrystalline fractures with smooth or less folded facets (Figure 5), which are formed on austenite or δ-ferrite grain boundaries.

The group D (Figure 6) includes fractures with relatively large facets, which are markedly folded by more or less parallel parts. The D type fractures were observed in steels with lower carbon contents in the austenite and δ-ferrite coexistence area. They are formed at the gamma-delta phase interface.

On fractures, several morphologies often occur simultaneously. In certain cases, the fracture can be described from the macroscopic point of view using one of the above-mentioned morphologies, but from the microscopic point of view, elements of a different morphology occur on the facets. Less frequently, facets of different morphologies occur on fractures simultaneously.

Metallurgical observations showed that in the described temperature area changes of the austenite or δ-ferrite grain size occurred. With a growing temperature, two opposite phenomena occur: certain grains grow as a result of the boundary movement and, at the same time, the formation and growth of new grains takes place. A maximum grain size is different for each steel grade. The maximums were from ca 1.0 to 2.5 mm at temperatures around 1380°C. This situation is documented in (Figure 7) for steel with 0.2% C, where the size of intercrystalline facets on the

the fracture of a specimen tested at 1400°C is much greater than at 1440°C.

The tested steels had various chemical compositions. It can be assumed that the high-temperature properties are influenced by several interaction phenomena of a diffusion nature, whose intensity depends on the temperature.(10-11)

To quantify the reduction of area at high temperatures, a multiple linear regression analysis was therefore used with the aim to determine basic integral effects of the chemical composition. The temperatures th, th5 and the temperature differences $\Delta th1$, $\Delta th2$, $\Delta th3$ were analysed. In the equations shown below, the contents of individual elements are given in % by weight. The probability of function relationships was min. 95% and p-values of dependent variables were less than 0.05.

It resulted from the statistical analyses that the th temperature is, as regarding the chemical composition, mainly dependent on the phosphorus content, $th = f(P^{-2})$, $r = 0.6959$. The correlation coefficient is relatively low, therefore we can assume that the plasticity degradation temperature is, besides the P content, also dependent on other factors.

The difference $\Delta th2$ is also dependent on the P and Mn contents, $\Delta th2 = (thteor – th) = f(P^{-2}, Mn^{0.5})$, $r = 0.7094$, where the Mn content may express its influence on the change of the solidus line position in the $Fe\text{-}Fe_3C$ diagram.

For $\Delta th1$ (th5 - th), a dependence on the phosphorus content and the Al/N ratio was found out, $\Delta th1 = (th5-th) = f(P,[Al/N]^{-2})$, $r = 0.6322$. Even though the correlation coefficient is relatively low, the p-values of members on the right side of the equation, 0.0047 or 0.0199, are favourable.

The statistical analyses of th5 and $\Delta th3$ parameters (Figure 1b) using the chemical composition did not find out any unambiguous relationships, or found out relationships with a less accuracy or an unclear physical-metallurgical background.

Figure 2 Scheme of the fracture formation mechanism – metallografical section

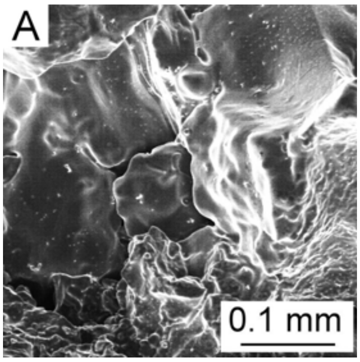

Figure 3 A type fracture – formed on molten boundaries of γ, or δ grains

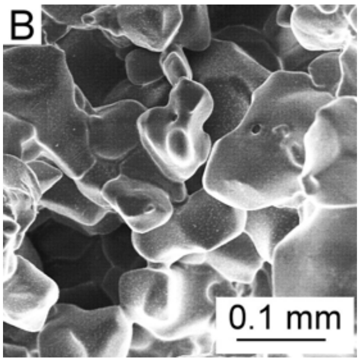

Figure 4 B type fracture – boundaries of the original dendritic space

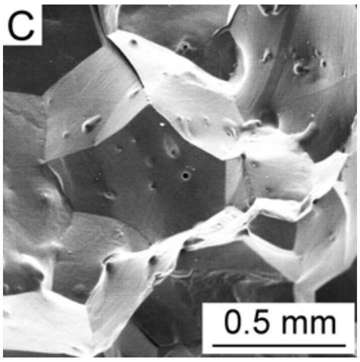

Figure 5 C type fracture – typical intercrystalline fracture on γ or δ boundaries

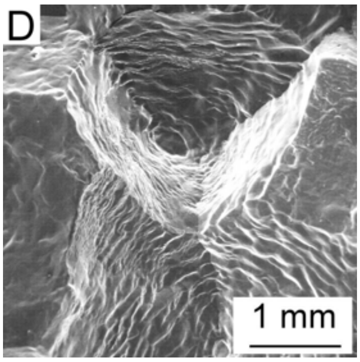

Figure 6 D type fracture – at the γ-δ interface

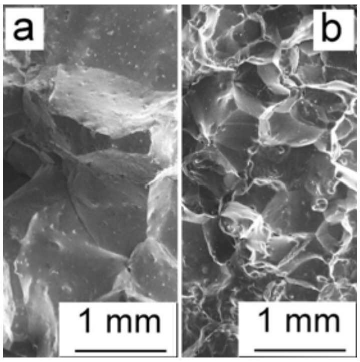

Figure 7 Intecrystalline fracture of steel with 0.02%C, testing temperature 1400°C (a) 1440°C (b)

Discussion

Based on observations of fractures using SEM, "the average fracture appearance" parameter, AFA [%], was determined. This parameter is a percentage estimate of the occurrence of B type fractures, documented in (Figure 4), in the temperature area from the th temperatures up to the molten phase occurrence temperatures. The AFA values ranged from 0 to 90%, with the arithmetic mean of 43.6%. Statistical analyses of the AFA parameter indicated its relation to the carbon content and the Al/N ratio, with a relatively good correlation coefficient:

$$AFA = 66.12 + 1.67\,C - 4.81\,(Al/N) = 0.8679 \quad (1)$$

The AFA parameter was statistically significant in the analyses of th5 and Δth3 (thteor-th5), th5 = f (A_{FA}^{-1}, $[Al/N]^{-1}$), r = 0.7330 and Δth3 = f (A_{FA}^{-1} $[Al/N]^{-1}$), r = 0.6667. An increase of the Al/N ratio, which ranged from 2.2 to 16 in the tested steels, shifts the th5 temperature to the right and narrows down the temperature interval of Δth3 (thteor-th5).

The relationship between the plasticity degradation temperature th and the AFA parameter has a good accuracy:

$$th = 1442.45 - 8.87\,A_{FA}^{\,0.5} + 4440.67\,P^{-2} - 361.60\,N^{-1} \quad r = 0.8230 \quad (2)$$

Thus, statistical analyses indicate that the plasticity degradation start temperature, or the temperature interval thteor – th, is mainly influenced by the phosphorus content. Alternatively, the th temperature can be expressed, with a better correlation coefficient, as a function of AFA, P and N.

The th5 temperature, which was used to evaluate the decrease of the reduction of area to a nearly non-measurable level, and certain further analysed parameters, are dependent on AFA and the Al/N ratio. The influence of phosphorus on the plasticity degradation temperature interval was not found out. The AFA parameter values increase with increasing the C content and with decreasing t Al/N ratio. The dissociation of AlN takes place at temperatures above 1100-1150°C.(13) while the analysed parameters occur within the temperature area above 1290°C, where the existence of AlN is unlikely.(13-14) The effect of the Al/N ratio in this case may be connected with the mutual affinity of Al and N atoms. The fracture process may also be influenced by the oxygen content in steels; however, its analyses were not available. The assumed effect of the microalloying elements V, Ti and Nb was not found out in the statistical analyses.

Based on the carried out experiments, we can suggest a hypothesis arising from an assumption of the existence of a dual structure in as-cast low-carbon steels at high temperatures. The structure is formed by polyedric austenite or δ-ferrite grains, whose cohesive strength is weakened by diffusion of impurities, mainly of phosphorus. In addition, a structure exists based on the original dendritic structure of steel, and the cohesive strength of these parts is primarily determined by the nitrogen and aluminium contents. In particular cases, a fracture occurs on the boundaries of parts with a lower cohesive strength. Phosphorus has a significant effect on the plasticity degradation start temperature and its effect is also applied at the highest testing temperatures by degradation of the cohesive strength of grain boundaries as a result of formation of the liquid phase.

The slope of the temperature dependence of the plasticity degradation, and particularly the temperature of plasticity decrease to a nearly non-measurable level, are mainly determined by the carbon and aluminium contents. The dendritic structure is determined by the chemical composition of steel.(15) and the cooling rate.(15-16) while the effects of individual elements are nonlinear and interactive.(4)

This discussion of the plasticity degradation issue was based on the results of metallographic and statistical analyses. The determination of characteristic variables (Figure 1b) is influenced by several errors.(17) The regression equations shown above only apply to the intervals of variables in the database and to the particular testing conditions. Relatively lower correlation values in certain equations may be due to errors occurred during determining the variables, as well as due to imperfect function relationships. The equations describe integral effects of the chemical composition and the structure on the characteristic plasticity degradation temperatures. A more detailed study of this issue could be carried out on model materials.

In practice, various defects are formed on the slab surface. Besides long cracks, which are mainly formed as a result of stress due to a non-uniform temperature field on the slab circumference, the weakening of primary casting grains by several mechanisms is a frequent cause of defect formation.(18) An example of a crack that was formed on the boundaries of primary casting grains, weakened by fine particles, residues from secondary metallurgy, is documented in (Figure 8). The identification of an interdendritic crack is unambiguous (Figure 9). However, if we take into account the described variability of the morphology of fractures formed at high temperatures, the identification of the causes of formation of cracks is, in general, difficult.

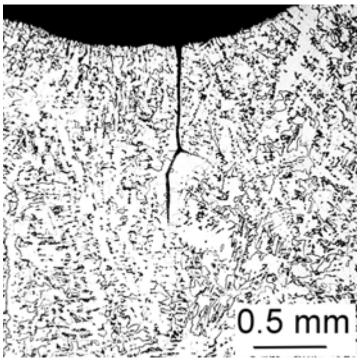

Figure 8. The crack on the slab surface was formed on the boundaries of primary casting grains

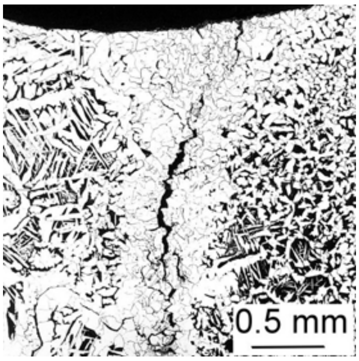

Figure 9 The crack on the slab surface has an interdendritic nature

Conclusions

Based on the carried out experiments and their analyses, the following main pieces of knowledge can be formulated:

1. At the testing temperatures from ca 1200°C up to the melting temperature, fractures with several morphologies are formed in the plasticity degradation area. According to their mechanism, the fractures were classified under 4 basic groups:

A – Fractures on melted boundaries of gamma or delta grains

B – Interdendritc fractures, formed by separation on the boundaries of the original dendritic spaces

C – Typical intercrystalline fractures on the boundaries of polyedric gamma or delta grains, whose cohesive strength is weakened by impurities

D – Fractures formed at the $\gamma - \delta$ interface

2. The plasticity degradation start temperature, or the width of the thteor-th temperature interval, are mainly influenced by the phosphorus content. The correlation coefficient is relatively low, therefore we can assume that the plasticity degradation temperature is, besides the P content, also dependent on other factors. Besides the effect of phosphorus on the plasticity degradation start temperature, this effect is also applied at the highest testing temperatures by degradation of the cohesive strength of grain boundaries as a result of formation of the liquid phase.

3. The temperature of plasticity degradation to a nearly non-measurable level, or the temperature interval of decrease of the reduction of area from 100% to 5%, are mainly determined by the nitrogen and aluminium contents. Increasing the Al/N ratio shifts the th5 temperature and narrows down the temperature interval Δth3 (thteor-th5).

Acknowledgements

This work was supported by the Slovak Grant Agency for Science VEGA, Project No. 2/4174/04

References

(1) Mintz, B. 1999, *ISIJ Int.* **39** : 833 – 855.

(2) Sricharoenchai, P.Nagasaki, C. and Kihara, J. 1992. *ISIJ Int.* **32** : 1102 – 1109.

(3) Nagasaki, C. and Kihara, J. 1997. *ISIJ Int.* **37** : 523 – 530.

(4) Won, Y. M. and Thomas, B. G. 2001. *Met. Trans.* A. **32A** : 1755 – 1768.

(5) Gao, Y. and Sorimachi, K. 1995. *ISIJ Int.* **35** : 914 - 919.

(6) Yoshida, N., Umezawa, O. and Nagai, K. 2003. *ISIJ Int.* **43** : 348 - 357.

(7) Yin,H., EMI, T. and Shibata, H. 1999. *Acta Mater.* **47** : 1523 – 1535.

(8) Phelan, D. and Dippenaar, R. 2004. *ISIJ Int.* **44** : 414 – 421

(9) Matsuoka H, et al. 1997. *ISIJ Int.* **37** : 255 – 262.

(10) Mazanec, K. 1988. *Kovové Materiály*. **26** : 5 – 50.

(11) Grabke, H.J. 1989. *ISIJ Int.* **29** : 529-538.

(12) Yu, C.H., Suzuki, M.and Shibata, H. 1996. *ISIJ Int.* **36** : Supplement S159 - S162.

(13) Gao, Y. and Baker, T. N. 1998. *ISIJ Int.* **38** : 744 – 751.

(14) Easterling K 1992. *Physical Metallurgy of Welding*. London , Butterworths : 122

(15) Yoshida, N., Umezawa, O.and Nagai, K. 2004 *ISIJ Int.* **44** : 547 – 555.

(16) Ode, M. et al. 2001. *ISIJ Int.* **41** : 345 – 349.

(17) Ševčík, A. and Ševčíková, J. 2005. *Acta Metallurgica Slovaca.* **11** : 36 – 44.

(18) Ševčík, A. and Ševčíková, J. 2005. *Transactions of the Universities of Košice* **No 1** : 48 – 53.