Use of Microscopy for Identification of Complex MC, M₂C, M₇C₃, M₆C and M₂₃C₆ Carbides in High Speed Steels

Abstract

High-speed steels (HSS) rolls are used in front finishing stands of hot strip mills. Good wear resistance and hardness at high temperature are defining features of HSS. Many types of carbides are present in these alloys, each having a different effect upon the final properties of HSS. As a result, nature, morphology and amount of these carbides are factors of important concern. Identification and characterization of HSS carbides are realized with optical microscopy combined with electron microscopy.

Introduction

Raw material is manufactured by vertical spin casting in the industrial conditions. This process leads to a bimetallic roll, with shell material (HSS) different from the core (Ferritic nodular graphite iron). The average composition of shell material is given in Tab. 1. After casting, the roll is air-cooled, and double-tempered. Blocks are cut out of the shell part. Sampled samples are then obtained from these blocks, and they are polished and etched, each sample showing the working surface of the roll at variable deepness within the shell material. SEM analyses are carried out to determine the nature of each type of carbide, with a great emphasis on EDX mapping. After electron microscopy, samples are examined using Groesbeck etching, which allows rapid optical identification of carbides by coloring them, leaving the matrix unetched. The grain size and the volume fraction of leading carbides are roughly assessed by image analysis.

Results of Microscopy Analysis

Shell microstructure is composed of tempered martensitic matrix containing a network of carbides mostly located at grains boundaries. Grain size increases with decreasing cooling rate from the shell to the core (Fig. 1).

Colored etching allows rapid optical identification of carbides, after determining their nature by the means of SEM/EDX. This appears to be of interest for image analysis prospects. Groesbeck reagent (KMnO₄) colors differently each type of carbide, as shown in Fig. 2 and Tab. 2.

Five types of carbides can be found after SEM/EDX analysis (Fig. 3): MC, M₇C₃, M₆C, and M₂₃C₆. MC, M₇C₃, M₆C, M₁₇C₆ types are eutectic carbides, which means that they precipitate from the liquid. MC is V-rich and its morphology is often globular M₇C are Mo/W-rich, with an acicular shape (cluster of rod-like particles). M₆C are Cr-rich, and they are located at grains boundaries and distributed as a continuous network. M₆C are Fe/Mo-rich, and they appear to be located only near the surface edge of the shell material (higher cooling rates), whereas others carbide types are present on the entire deepness of the shell. M₂₃C₆ are Cr-rich fine secondary carbides fully distributed inside the matrix. Hence, M₂₃C₆ appear during tempering.

Complex carbides cluster give an idea on the precipitation sequence of carbides. MCs that are located at the top of cluster branches appear to be the first eutectic carbide to precipitate. As the solidification goes ahead, the V content of the residual liquid is lowered, and that allows others carbides types such as M₁₇C₆, to precipitate in the cluster centre (Fig. 4).

Discussion and Conclusions

Overall distribution, nature and carbide size directly affect rolls mechanical properties. In fact, additional mechanical tests are achieved using samples coming from the shell. For a given HSS composition, various types of carbides are present in the microstructure, depending on the cooling rate and the heat treatment performed after casting. It is quite possible to determine the precipitation sequence of carbides while analyzing a mixed carbide cluster. Eutectic carbides precipitate mostly at grains boundaries, while secondary carbides are fully distributed inside the matrix. Vanadium forms very hard MC eutectic carbides mainly inside grains, improving hardness and wear resistance. High content of Cr causes formation of M₁₇C₆ eutectic carbides mainly at grain boundaries, improving hardness and preventing ox-

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>W</th>
<th>P</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.52</td>
<td>0.11</td>
<td>1.02</td>
<td>1.02</td>
<td>5.08</td>
<td>2.05</td>
<td>3.05</td>
<td>1.04</td>
<td>&lt;0.005</td>
<td>75/85</td>
<td></td>
</tr>
</tbody>
</table>

Tab. 1: Average composition of raw material (% weight)

<table>
<thead>
<tr>
<th>Etching with KMnO₄</th>
<th>MC</th>
<th>M₇C</th>
<th>M₁₇C₃</th>
<th>M₆C</th>
<th>M₂₃C₆</th>
</tr>
</thead>
<tbody>
<tr>
<td>Etching with KMnO₄</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Tab. 2: Color of HSS carbides after KMnO₄ etching (Groesbeck reagent)

Keywords
carbide cluster, alloys, electron microscopy, optical microscopy

48 • G.I.T. Imaging & Microscopy 2/2005
dation phenomenon. Both Mo and W lead to the formation of $\text{M}_2\text{C}$ eutectic carbides, which lower the secondary hardening effect during tempering. $\text{M}_2\text{C}_3$ are very fine secondary carbides, which precipitate in the matrix during tempering at high temperatures. This second hardening effect seems to improve the ultimate tensile strength in temperatures around $600^\circ\text{C}$.

Acknowledgments

SEM micrographs have been obtained using ULg CATp devices. Raw material is supplied by Marichal Ketin, Rolling mill rolls manufacturer located in Liege (Belgium).

References

[1] Quasi-quantitative description of $\text{M}_2\text{C}$ and $\text{M}_6\text{C}$ $\text{M}_2\text{C}$ carbides in high speed steel rolls. J. De Colbert, L. Paris, J. Lecomte-Beckers, Y. Boeze, H. Gibert, J. T. Choufahng — Proceedings of ASMIF-3 International Conference, Brno, Czech Republic 2001 (pp 710 to 717)


Short CVs

Prof. Jacqueline Lecomte-Beckers has been graduated as Civil Engineer (Physics, 1978) in the University of Liege. She obtained her Ph.D. in the University of Liege in 1985. Currently she is head of the „Materiaux Metalliques Speciaux“ unit in the Materials Science (ASMA Department) of ULg. Her research topics are related to Metallography, Phase transformation, Thermophysical properties, Metallic Materials, and Microscopy.

Dr. Jerome Choufahng Chuiudjang has been graduated as Civil Engineer (Automatics and Computer-Integrated Manufacturing, 1998) in the University of Liege. Currently he is a research engineer in the Materiaux Metalliques Speciaux unit.

Jacqueline Lecomte-Beckers

Jerome Choufahng Chuiudjang

Institut de Mecanique et Génie Civil, ASMA
Département ASMA — Materiaux Metalliques Speciaux
Bâtiment 852/3 — Chemin des chevreulx, 1
4000 Liége (Sart Tilman) — Belgium
Tel. +32 4 366 91 62 — Fax. +32 4 366 91 13
jacqueline.lecomte@ulg.ac.be · jerome.choufahng@ulg.ac.be

www.ulg.ac.be/metaux

Fig. 1: Carbides network at grains boundaries. Increasing of the grain size towards the depthness. 1(a) 5 mm depth – 1(b) 50 mm depth

Fig. 2: Cluster of complex carbides after Giesebeck reagent etching. Variation in the carbide color depends on the holding time while etching. 2a (20 sec etching) and 2b (5 sec etching)

Fig. 3: Carbides cluster (SEM Image with SE detector, left) and related EDX mapping (right)

Fig. 4: Complex carbides cluster (SEM Image with BSE detector) related to the precipitation sequence while freezing