Development of New Materials Based on On-Site and Laboratory Evaluation Methods for Understanding Work Roll Surface Degradation

The Centre for Research in Metallurgy is now equipped with a series of laboratory and on-site methods for evaluating the surface degradation behavior of work roll material. These methods include classical mechanical testing and specially developed evaluation tests and procedures. This paper describes some examples of the application of these methods and the role they have played in a better understanding of the work roll surface degradation mechanisms and in the development of new materials.

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Gisèle Walmag, program leader (gisele.walmag@ crmgroup.be), and Sébastien Flament, project leader, CRM Group (sebastien.flament@crmgroup. be), Liège, Belgium; Jurgen Malbrancke, senior project leader, CRM Group (jurgen.malbrancke@ crmgroup.be), Zwijnaarde, Belgium; and Geneviève Moreas, program leader (genevieve. moreas@crmgroup.be), CRM Group, Liège, Belgium; and Mario Sinnaeve, Marichal Ketin, Liège, Belgium (quality.control@mkb.be) Alittle more than 30 years ago, Marichal Ketin (MK) initiated a long-term partnership with Centre for Research in Metallurgy (CRM) in order to support the Belgian roll foundry in future material developments. Several new work roll materials were investigated and developed during the last few decades based on this cooperation. Most of these grades are still standard grades in many hot strip mills in the world today.

Some outstanding results need to be mentioned within the context of new work roll grade development:

- The chrome steel grade, introduced in many roughing mills in the early 1980s, was one of the first grades to be investigated with CRM.^{1,2}
- In the early 1990s, based on their success in Japan, a huge research work started for the development of high-speed steel (HSS) work roll grades for the finishing stands. These developments helped MK to become one of the world's leading HSS work roll suppliers.^{3,4}
- Microalloyed indefinite chill cast iron (ICDP) followed as a research topic in the early 2000s until 2005, a period during which many hot strip mills switched from the conventional ICDP grade to the so-called "enhanced" type.

The work roll surface quality and its evolution during a

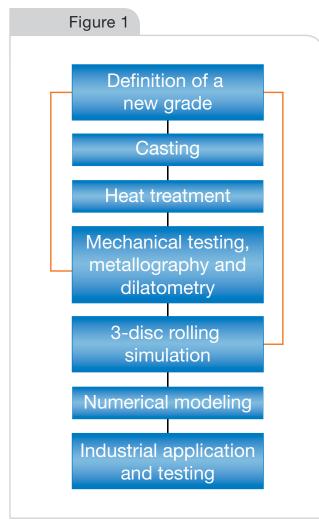
campaign play an increasing role on the strip surface quality, and some defects are not detected on the hot strip and appear only after galvanizing. Controlling, understanding and improving the work roll surface degradation are thus gaining increasing importance. Several research projects were initiated from 2003 to 2009 for filling the lack of knowledge in the field of work roll degradation in hot strip mills. The results of this work were an important milestone in the understanding of roll behavior and served as an input for new work roll developments, as well as for the improvement of the rolling process.

More recently, overcoming the limits of traditional centrifugal casting and chemistries has become the subject of an ongoing development project with CRM.

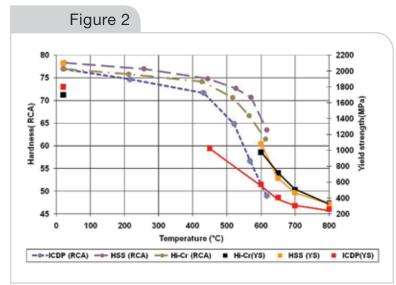
This paper will describe how CRM and MK jointly carry out their research activities in the field of work roll materials for hot rolling, together with the techniques used and specific application cases.

Development of New Work Roll Grades

New work roll grade development is based on a specifically defined methodology (Figure 1). As MK work roll grades are cast materials, this development requires casting laboratory samples while ensuring a microstructure similar to an industrial roll shell. Based on numerical modeling, a dedicated mold and casting procedure has been designed in



Methodology for new work roll grade development.



Hot hardness and hot compression properties of work roll materials.

order to achieve a cooling rate close to the cooling rate of a bi-metallic roll shell. The cooling rate after casting and solidification is also controlled for simulating the industrial roll cooling conditions.

A laboratory induction furnace enables the efficient alloying of various compositions using industrial ferroalloys. Heat treatments are carried out either in the laboratory or, more preferably, in the industrial furnace together with rolls.

Various evaluation laboratory methods are then used for characterizing the newly developed grades, including chemical analysis, metallography, mechanical testing and dilatometry. Specially developed techniques for the evaluation of the work roll materials are also frequently used, like 3-disc rolling simulator tests, hot oxidation tests and pilot mill trials.

Materials Characterization — Chemical analysis equipment is available in CRM for the chemical analysis of roll materials. Optical spectrometry is currently used by MK for evaluating the chemical composition of the alloy, but x-ray fluorescence (XRF) analysis is also frequently carried out for detecting and quantifying a possible pollution of the alloy by less common elements like Zn, Pb, Sn, Sb and As.

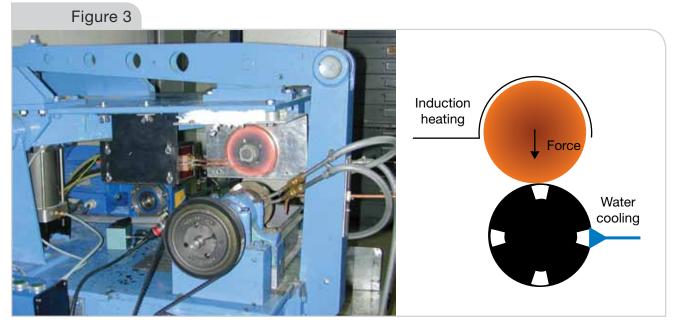
Metallography by both optical microscopy and scanning electron microscopy (SEM) are basic tools of evaluation when developing a new roll grade. Indeed, the distribution of the various carbides, as well as their shape and size, significantly influences material properties such as toughness and thermal fatigue resistance.

Mechanical Testing – Mechanical tests for roll materials characterization include tensile and compression testing, hot compression testing, fatigue and impact tests. Mechanical testing and hardness tests (from room temperature up to 625°C) are mainly

used to characterize newly developed roll grades. It is indeed a characteristic of work roll materials that their high-temperature mechanical properties exhibit a sharp variation within the temperature range at which the roll surface is heated during contact with the hot strip (Figure 2).

3-Disc Rolling Simulator — Laboratory tests combining the effects of fatigue, oxidation and wear are important for validating a roll material or for comparing various grades. For this purpose, CRM has developed a 3-disc wear test machine simulating the hot rolling of stands F1–F3.

The 3-disc rolling simulator consists of two or three discs enabling the simulation of the degradation of work or backup rolls in hot rolling. In the 2-disc configuration (Figure 3), the upper disc is heated by an induction coil enabling preheating of its surface up to 1,100°C. This upper disc simulates the strip. The lower disc simulates



3-disc rolling simulator (2-disc configuration).

the work roll. Four inserts of different roll grades (40 mm long) are placed in this disc.

In the 3-disc configuration (Figure 4), the upper and the lower discs simulate the work roll while the middle disc simulates the backup roll. As in the 2-disc configuration, a load can be applied on the upper disc and water (with the desired composition and temperature) can be sprayed. This trial enables the simulation of contact fatigue between the work roll and the backup roll.

Each disc rotation is controlled by its own motor and speed variator. A load is applied on the upper disc. The contact time of a specific zone of the discs depends on this load and on the disc speed. Watercooling is applied on the surface of the lower disc at a specific distance after the contact between the two discs, depending on the disc speed. The composition and temperature of the cooling water can be adjusted in order to simulate the best industrial conditions.



3-disc rolling simulator (3-disc configuration).

The degradation evaluation is carried out by:

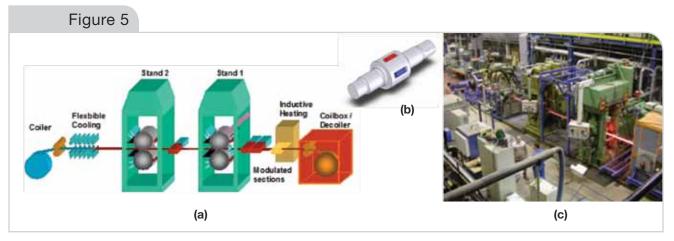
- Roughness and profile measurement.
- Glow discharge optical emission spectroscopy (GDOES) measurements.
- Optical microscopy (surface and cross-section).
- SÊM.

Pilot Trials — In order to assess roll material performance, a real hot rolling process can also be performed on the continuous hot rolling pilot line at CRM Ghent. The hot rolling pilot line enables the continuous rolling of "baby coils" (max. diameter 1,700 mm, max. width 200 mm, thickness 2–4 mm) preheated in a protective atmosphere.⁵

Figure 5 shows a photograph and the layout of the continuous hot rolling pilot line.

In order to deeply investigate the complex interactions in the roll gap, the stands are equipped with work roll cooling, skin cooling, lubrication and

additional measuring systems (i.e., laser speed, roll and strip surface inspection systems). To study different work roll grades, rolls are prepared with inserts of different roll materials (Figure 5). Each work roll contains five inserts. The dimensions of an insert are 175 x 75 x 30 mm³, while the work roll diameter is 400 mm and the table width is 350 mm. This approach gives a direct comparison during the complete hot rolling operation for the different grades because each insert is submitted to identical rolling conditions. When the procedure is used to simulate the rolling conditions in a specific stand, it is advised to have at least



Continuous hot rolling pilot line: (a) layout, (b) work roll with inserts and (c) photo of CRM hot rolling pilot plant.

one insert with a grade that is well known for that specific stand.

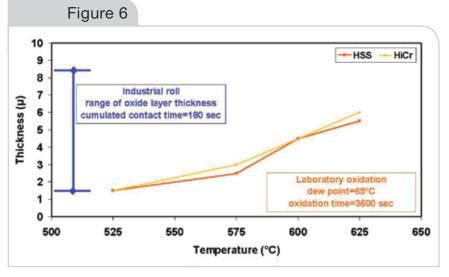
To obtain an accurate assessment of roll material performance, a wide range of measurements is performed, each with a specific objective. The different types of measurements can be divided into three groups:

- On-line measurements taken during the trial:
 - Rolling data (e.g., force, temperature, speed and motor current).
 - Work roll surface state with a rollscope (monitoring of the top roll surface).
 - Product surface quality with a stroboscopic on-line strip surface inspection system.
 - Exit strip speed measured by a laser speed measurement device.
 - Spindle torque measured with strain gauges (monitoring of the symmetric/asymmetric rolling behavior in roll gap).
- Measurements taken during a stop, between two trials or at the end of the procedure:
 - Photographic images.
 - Roughness measurement.
 - Profile measurement.
 - Microscopic analysis with the portable optical microscope.
- Destructive measurements performed after the removal of the inserts.
 - Optical microscopy.
 - SÉM.

Using work rolls prepared with different inserts in the continuous hot rolling pilot line offers the opportunity to compare different grades directly and under equal industrial conditions. All the measurements give a complete scope of the advantages that one grade may possess compared to another. It can be concluded that this procedure opens up many possibilities toward a better understanding of work roll behavior and the introduction of new work roll grades in lines.⁶

Hot Oxidation Tests — Oxidation during hot rolling has a noticeable effect on roll performance and product surface quality. A classical approach to oxidation consists in performing hot oxidation tests in humid atmosphere. The temperature range corresponding to the surface contact temperature of the roll (525– 700°C) is usually investigated with dew point from 40 to 90°C, simulating the humid air at the exit of the roll gap, and a duration from 1 to 100 hours.

Oxidation kinetics as a function of the material composition and heat treatment can thus be evaluated. However, comparison with the oxide thickness observed on industrial roll samples has indicated that this method of static oxidation test tends



Comparison between oxidation kinetics in hot oxidation test and industrial oxide thickness.

to underestimate the oxidation kinetics (Figure 6). It has led to the development of a combined oxidationcorrosion test, which simulates the thermo-chemical cycle of a roll surface. Specimens are heated at 600° C/2 seconds in a vertical furnace and cooled by dipping in water. Different water compositions can be tested.

These simulations have shown a cumulative effect of oxidation at high temperatures and corrosion in water on the oxide layer thickness, as well as the influence of the cooling water composition on roll materials' oxidation and degradation.

Numerical Modeling and Dilatometry — When a new work roll grade has been developed, thermomechanical modeling by field emission microscopy (FEM) is used for defining the heat treatment parameters in terms of heating and cooling rate in order to achieve the satisfactory stress state both in the shell and at the core-shell bonding zone. Physical and mechanical properties of the new grades are required for this modeling together with dilatometric curves.

Dilatometric measurements are also frequently used for defining the specific phase transformation temperature for each grade.

Degradation Mechanisms

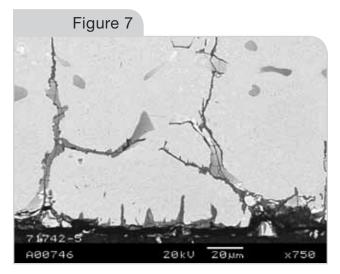
The degradation mechanism of work rolls in a hot strip mill (HSM) is a combination of three concomitant and interrelated mechanisms:⁷

- Thermal fatigue and contact fatigue.
- Oxidation-corrosion.
- Wear.

By combining these three mechanisms, roughness increase, peeling and banding can occur. These degradation mechanisms are dependent of the roll material (its thermal expansion coefficient, thermal conductivity, hot yield strength and low-cycle fatigue (LCF) at low temperature, roll composition, and heat treatment) but also on the rolling parameters (strip and roll temperature, reduction ratio, contact time, strip oxide thickness and roll cooling).

In order to have a better understanding of work roll degradation mechanisms and to improve work roll resistance toward degradation, CRM, with MK, is using several in-lab evaluation methods and also onsite techniques, including portable optical microscopy, portable Vickers hardness tester and rollscope.

Microscopy and Hardness — Metallographic examinations of roll surface on samples from rolls at the end of their life, or after an incident leading to roll breakage, are of prime importance for understanding the degradation mechanisms of a work roll surface. Figure 7 illustrates an example of metallographic examinations of roll samples in cross-sections, enabling the identification of the degradation mechanisms.



SEM cross-section of a roll sample.

On-site hardness measurements are also possible. MK is equipped with a portable Vickers hardness tester capable of performing 30 kg and 50 kg Vickers hardness tests on-site.

Surface analyses techniques like GDOES and timeof-flight secondary ion mass spectrometry (ToF-SIMS) often help in complementing the metallographic examinations. GDOES enables an in-depth profiling of the various elements constituting the roll material, together with the elements from the cooling water incorporated in the oxide layer. ToF-SIMS is used for detailing the information obtained by GDOES, as it enables a more local, in-depth profiling and an identification of the ions and their molecular form (oxide, nitride, etc.).

CRM has developed on-site metallography (nondestructive testing) for observing roll surfaces with a portable optical microscope without sampling the roll. It enables examination of the roll surface between two rolling campaigns. The microscope is installed on a specially designed holding system dependent on the size of the roll. Most often, the two rolls are dismantled and the microscope is placed on them (Figure 8a), but occasionally when the examination needs to be carried out during a campaign without dismantling the pair of rolls, the microscope is fixed vertically (Figure 8b). The examinations are usually carried out in the roll shop. The microscope is equipped with a digital camera and software enabling the microscope to compute a clearly focused image using a series of images of a surface at varying levels of focus. This feature is of particular interest for examining the rough surface of a degraded work roll at the end of a campaign.

A complete set of metallographic preparation equipment has also been adapted to industrial conditions. CRM is now able to do a complete on-site metallographic examination. It includes the direct surface analysis with the portable optical microscope, but polishing and etching before examinations is also feasible.



On-site microscopy with a portable microscope: (a) microscope on the roll and (b) microscope fixed vertically.

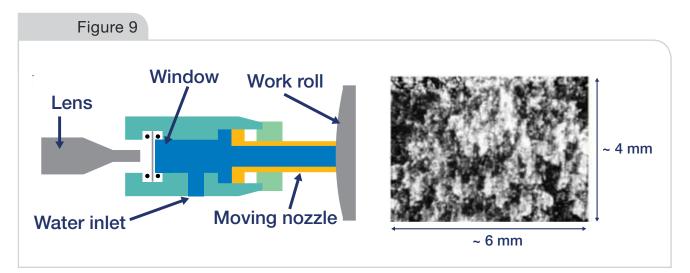
Rollscope — To visualize and analyze the degradation of rolls inside roughing and finishing mills, the rollscope sensor has been developed by CRM. This industrial sensor grabs images of the surface on-line and in real time in a 6-mm-wide field of view. To work in the harsh environment of roughing and finishing mills, the developed principle consists of the creation of a column of water between the lens and the surface using a moving nozzle (Figure 9). This column of water acts as an optical fiber and mainly as a protection of the window against dust and vapor. The result is a high-quality image of the surface.

The sensor synchronization enables the rollscope to grab images at random, incremental or fixed positions on the roll. This allows the rollscope to follow the surface evolution of a fixed position and also to realize a complete map of the roll when coupled with a translation system. The rollscope is used to monitor the work roll surface evolution, to analyze the roll degradation kinetics and to evaluate work roll performance. The monitoring of the roll surface aspect is essential for:

- Rollmakers, in order to optimize their roll grades.
- Hot strip mill plants, in order to:
 - Increase the lifetime of their rolls.
 - Prevent rolling problems and surface defects on the product directly linked to the roll surface characteristics.

Specific Applications

Table 1 summarizes the different techniques previously mentioned and their field of application (new material development or degradation mechanism). Some techniques (e.g., metallography, wear tests, pilot mill trials) are used for both applications.



Rollscope schematic (left) and image grabbed by the rollscope (right).

This paper will describe some specific cases where the combination of several techniques has helped to explain material behavior.

Optimization of Carbides Distribution — The new grade development procedure described in Figure 1 has been applied for optimizing the carbide distribution in an HSS grade. The examination by SEM clearly shows the more homogeneous and finer distribution of the different types of carbides observed in a new HSS grade when carbide nucleating elements are added (Figure 10).

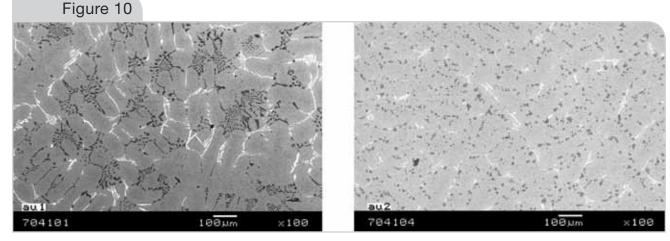
Comparison of an Industrial Roll With 3-Disc Rolling Simulator Samples — In order to verify the adequacy of the 3-disc test machine for simulating hot rolling, comparison exams have been performed on inserts after 3-disc rolling simulator trials and on industrial end-of-life rolls.

For the industrial roll, several examinations have been carried out:

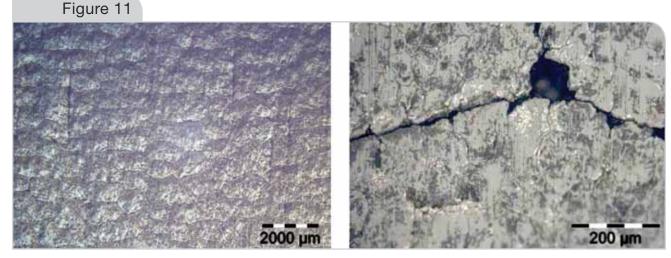
Table 1

Summary of the Evaluating Methods and Their Applications

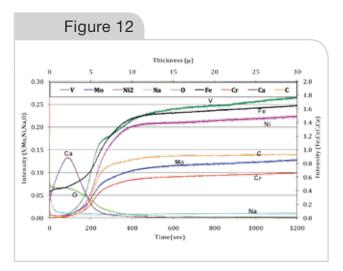
	New materials developed	Degradation mechanisms
OES	•	
GDOES		•
OM, SEM, SIMS	•	•
Mechanical testing	•	
Roughness measurements		•
Dilatometry	•	
Numerical modeling	•	
3-disc rolling simulator		•
Pilot line	•	•
Rollscope		•
Portable microscope		•
Portable hardness tester		•



Influence of nucleating agent on carbide distribution in an HSS grade.



On-site optical microscopy on a finished mill roll.



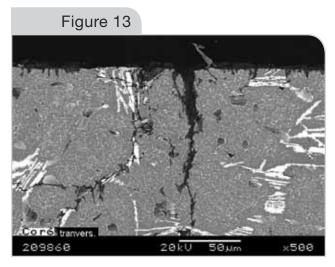
In-depth composition of the surface of an industrial finishing mill roll (HSS F1 bottom) by GDOES.

- On-site optical microscopy (Figure 11).
- GDOES (Figure 12).
- SEM on cross-section (Figure 13).

On-site optical microscopy is a non-destructive method enabling examination of rolls of various diameters. These examinations enable the observation of the surface degradation, i.e., roll oxidation, cracking pattern, carbides and defects (holes). These surface observations are of primary importance to understand the degradation mechanism.

GDOES gives information about the oxide layer formed on the roll (its thickness and its composition). In this case (Figure 12), an oxide layer of about 7 μ m is observed, constituted of iron oxide enriched with calcium originating from the cooling water.

SEM examination of a cross-section shows the thermal cracks propagating preferentially along the carbide network, while the surface and the crack edges have been oxidized by the cooling water.

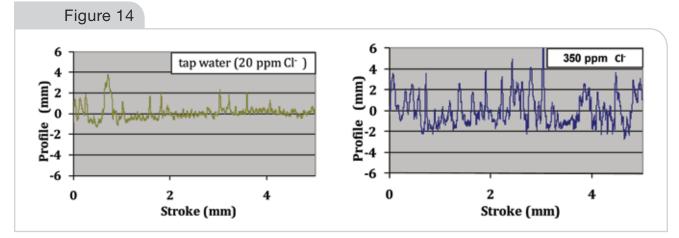


SEM examination of the cross-section of an HSS industrial finishing mill roll (cracks and oxide layer).

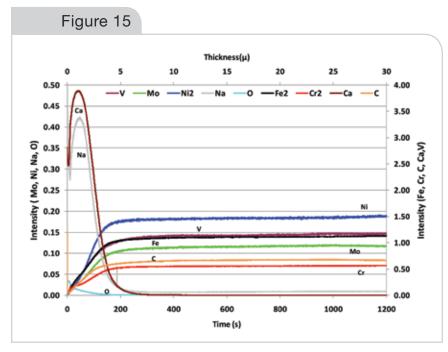
3-disc rolling simulator trials have been performed with various water compositions based on industrial data. Roughness and profile measurement are efficient methods for characterizing roll degradation. Figure 14 illustrates profile measurement on HSS inserts after 3-disc trials. It can be observed that cooling water composition influences the work roll surface degradation.

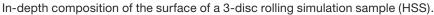
Figure 15 illustrates a GDOES in-depth analysis on an HSS insert after one of those tests. Although the oxide layer is thinner than that which is observed on an industrial roll, the composition profile is very similar between both graphs illustrated in Figures 12 and 15. It was expected to form a thinner oxide layer during a 3-disc rolling simulation trial compared to an industrial campaign, as those laboratory trials last 2,000 cycles versus 20,000 cycles for an industrial roll.

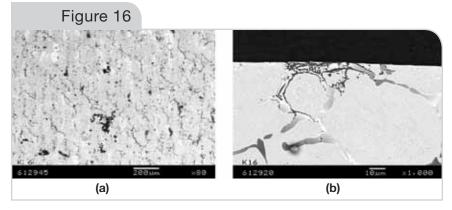
Figure 16 illustrates the surface aspect and a crosssection of an HSS insert after a laboratory trial. These examinations have been done by SEM. This micrograph shows internal oxidation-corrosion along the carbides network similar to that observed on industrial rolls.



Influence of chloride content on roughness profile after 3-disc rolling simulation (HSS).



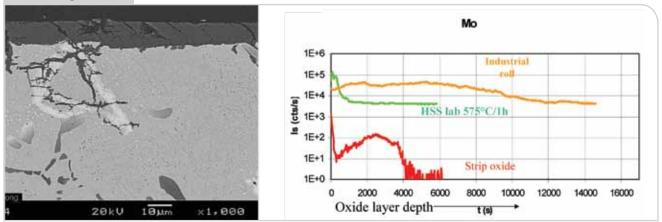




SEM image after 2D trial: (a) surface image and (b) cross-section.

Identification of a Roll Oxide Layer Origin – Some industrial rolls are covered with an oxide layer (Figure 17). It is often debated if this oxide is strip oxide sticking on the roll or roll oxide. To clarify this debate, SIMS examinations have been performed on an industrial roll covered with oxide and on strip oxide. These examinations have excluded the possibility that the oxide could be strip oxide. The molybdenum content in the industrial roll oxide is much higher than the molybdenum content of strip oxide. Besides, an HSS sample has been oxidized in the laboratory, and SIMS measurements have also been done on it. The molybdenum content of the oxide layer on the oxidized laboratory sample is at the same level as the molybdenum in the industrial roll oxide.

Rollscope in a Finishing Mill — The rollscope has been successfully used several times in finishing mills and rouging mills. In the case illustrated here, the rollscope has been implemented in a finishing mill (stand F2). Figure 18 illustrates the surface evolution of a HiCr work roll surface during a campaign. Images have been acquired at five positions over the width of the roll. The images presented have been



Oxide layer on an industrial HSS roll (left) and its SIMS analysis (right).

Figure 17

taken at the beginning of the campaign, $^{1}/4$ campaign, midcampaign, $^{3}/4$ campaign and at the end of the campaign. Due to rolling conditions, the surface degradation can vary over the width of the roll. The rolling condition optimization and an accurate evaluation of the roll performance thus require a full-width inspection.

Regarding the large number of acquired images, an automatic image-processing algorithm has been integrated, defining two discriminating parameters related to the work roll surface degradation.⁸

Conclusions

MK is collaborating with CRM on a long-term basis for developing new roll grades, improving the roll manufacturing process and achieving a better understanding of work roll degradation.

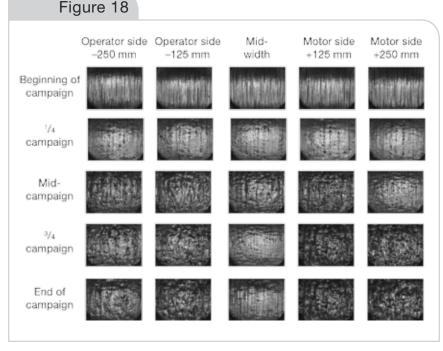
CRM is equipped for a detailed characterization of roll materials and roll degradation in the laboratory. Techniques are continuously developed for characterizing roll degradation on-site (portable optical microscope, portable Vickers hardness tester, full metallography set and portable spectrometer, and rollscope).

This combination of techniques is an efficient tool for developing higher-performing roll grades, as well as understanding work roll degradation and its relation to rolling process parameters.

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Evolution of the work roll surface during a campaign.

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