

Metallurgical assessment of two HSS rolls grades for Hot Strip Mill

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INTRODUCTION

On the basis on information coming from Japanese Hot Strip Mills (HSM) and relating to a new grade with very high performance, Marichal Ketin (MK) started the development of High Speed Steel (HSS) rolls in 1992.

In the late nineties, after many laboratory and industrial trials with different compositions, a grade called Kosmos was considered as the standard one. Since then more than 800 rolls have been delivered in 45 Hot Strip Mills with good results in performance and reliability in operation.

From metallurgical point of view, the behaviour of a roll in a mill may be linked to the various component of its microstructure. The main characteristic of the HSS structure is the presence of different kind of very hard eutectic carbides whose nature, shape, size and distribution, are the result of the alloying elements content as well as the solidification rate.

Chemical composition of Kosmos optimises the precipitation of three kinds of eutectic carbides: MC, M₂C, M₇C₃. Large amount of very hard MC eutectic carbides improve strongly the wear resistance, but too large amount can lead to an increase of the friction coefficient and even chattering. The hard M₂C eutectic carbides are also very wear resistant. However, when that carbide precipitates mainly in the flake morphology it makes the material very brittle and the resistance to mill incident may becomes very poor. The M₇C₃ eutectic carbide due to its high Chromium content may have a beneficial effect on the roll oxidation behaviour. However that carbide is rather soft compared to MC and M₂C.

When a roll grade is world-wide considered as common, each rollmaker try to improve the performance of its rolls either by using another manufacturing process or another chemical composition.

Four years ago MK decided to modify alloying elements content of Kosmos grade while keeping the vertical spin casting process, leading to a new improved grade called Aurora. About sixty rolls are now in operation in various HSM. When starting the development of Aurora grade prices of alloying elements were in the range between 6 to 7 €/kg. Thus the total cost of alloying elements appears to be the same for both grades.

The present work is intended to enhance alloying elements influence on microstructure and mechanical properties while comparing earlier Kosmos HSS grade with newly Aurora one towards various sophisticated examination techniques and specific mechanical tests.

An attempt will also be made to correlate laboratory results to the good behaviour of rolls in operation in the early stands of HSM.

EXPERIMENTAL PROCEDURES

Studied Materials

As for almost rolls material for HSS alloys, rolls characteristics are fixed by their composition, solidification rate and heat treatments. Many tool steels are described by their equivalent Tungsten (Weq) which equals $(2Mo+W)$. Generally speaking, it means that two atoms of Tungsten have a similar effect on the material as one atom of Molybdenum. Some publications describing structural investigation on tool steels with the same Weq and various Mo and W content have shown that a high Molybdenum content (1, 2, 3):

- Decrease the total amount of eutectic carbides and the alloying elements content of the metallic matrix is thus increased;
- Promote the MC eutectic carbide precipitation.

Figure 1 shows the relationship between Carbon content, Tungsten equivalent and morphology of the eutectic carbides.

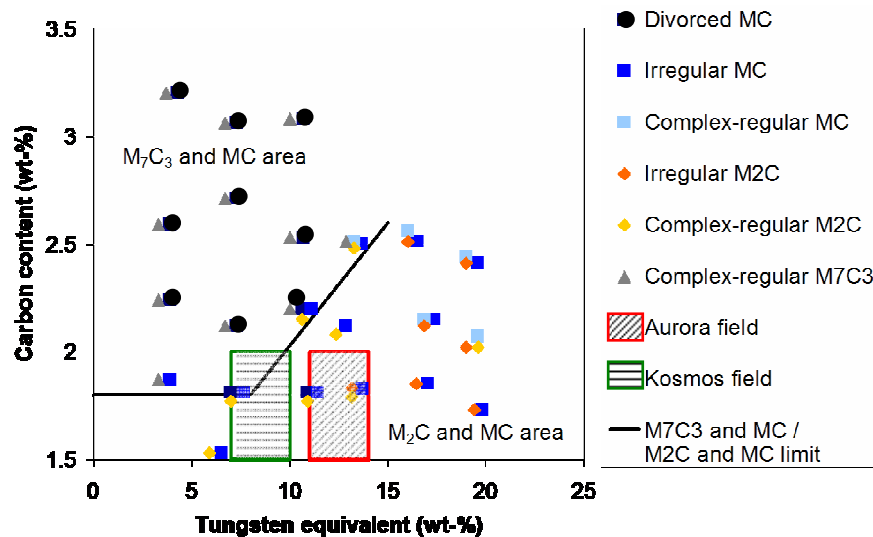


Figure 1- Influence of C and Weq contents on type and morphology of eutectic carbides (4)

Kosmos and Aurora composition are described in Table I.

	C	Cr	Weq	Mo/(Mo+W)	V
KOSMOS	1.5/2.0	5.0/7.0	7.0/10.0	0.6/0.7	4.0/6.0
AURORA	1.5/2.0	3.0/5.0	11.0/14.0	0.9/1.0	4.0/6.0

Table I - Composition of Kosmos and Aurora (%-wt)

The main differences between both grades are:

- Aurora contains less Chromium than Kosmos. The purpose is to decrease the amount of Chromium carbide (M_7C_3);
- The Weq and the Mo/(Mo+W) ratio are both higher for Aurora than Kosmos.

We notice in figure 1 that such compositions lead to a shift of Aurora field from the M_7C_3 -MC area to the M_2C -MC area.

Samples analysed in this study correspond to real industrial conditions for work roll used in the early finishing stands of Hot Strip Mill with a diameter in the 700/800 millimetres range. The rolls were manufactured by centrifugal vertical spin casting process. Pieces are

cut off from the roll shell in the as-delivered state to obtain samples corresponding to different depths of the working layer. They were used for microstructure characterisation and mechanical testing.

Heat treatments

HSS rolls are heat treated in order to get a hardness of 78/83 Sh.C. Such hardness is commonly used in the early stands of HSM because it gives the best compromise between wear and mill incidents resistance.

For Kosmos, the high hardness of the as-cast microstructure containing only austenite and martensite allows us to get the required hardness by tempering only. As concern Aurora, its lower Chromium content induces a soft as-cast structure containing austenite and bainite. An austenitisation followed by air quenching have to be performed before the tempering treatments to get a hardness in the 78/83 range.

Microstructure characterisation methods and mechanical tests

The characterisation of the microstructure was done using various techniques such as Differential Thermal Analysis (DTA) to study the solidification sequence, Scanning Electron Microscopy (SEM) to determine the analysis of carbides, and image analysis to quantify the volume fraction of carbides and the grain size.

Experimental DTA conditions were heating from room temperature up to 1620°C with a 5°C/min rate. This rate was chosen in order to be as close as possible of industrial conditions. The upper limit of 1620°C is set by our DTA device.

Some correlations were also made with related mechanical properties such as compression tests and hot hardness. 12 and 10 compression tests were performed until failure respectively for Kosmos and Aurora grades.

The use of DTA technique for phase transformations enhancement

DTA technique was used to investigate the solidification sequence and especially the carbides precipitation. DTA is a technique in which the sample is heated (or cooled) following a temperature schedule and which can detect any endothermic or exothermic type transformation. Any phase change leads to variations in the sample temperature. The difference in temperature between the sample and the programmed temperature is monitored against time. With the DTA method, any transformation even the small ones, can be detected (fusion, solidification, decomposition...).

During DTA trial a phase transformation appears on the thermogram as a single peak at a given temperature (figure 2a). If two transformations phase occur at temperatures ranges closed one to another, the two resulting peaks can overlay (figure 2b). The occurrence of overlapping peaks is frequently observed in high alloyed steels and particularly during fusion and carbides dissolution.

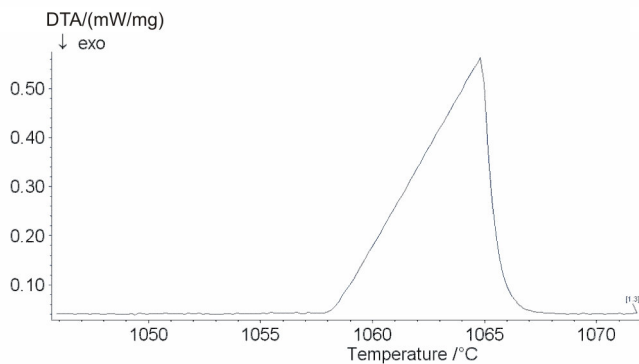


Figure 2a – DTA signal with one peak of transformation

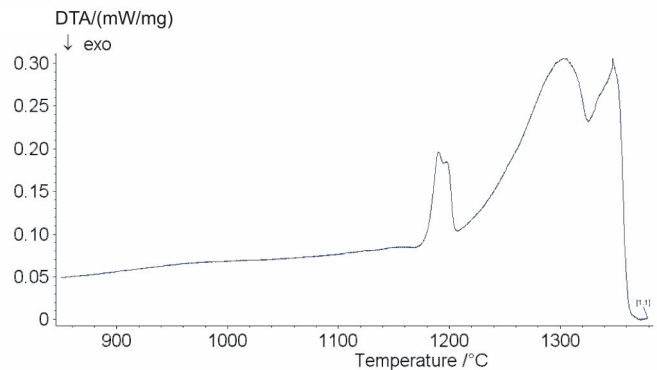


Figure 2b – DTA signal with peaks overlapping

RESULTS

Microstructure

The Kosmos and Aurora microstructure consists of a matrix containing the products of austenite decomposition (tempered martensite) with precipitated globular secondary carbides and eutectic carbides distributed both in the interdendritic or intercellular region (Fig. 3a to 3c and Fig. 4a to 4c) (10)

The nature and morphology of the eutectic carbides are influenced by solidification rate and chemical composition. In conventional High Speed Steels:

- MC carbide dissolves mainly Vanadium;
- M₂C carbides are rich in Molybdenum and Tungsten and can dissolve some Chromium;
- M₇C₃ are rich in Chromium.

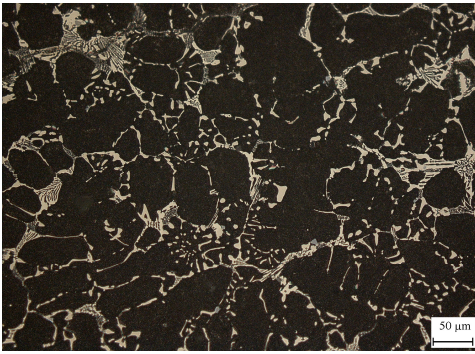


Figure 3a – Deep etching of the matrix leading carbides unetched (Kosmos grade)

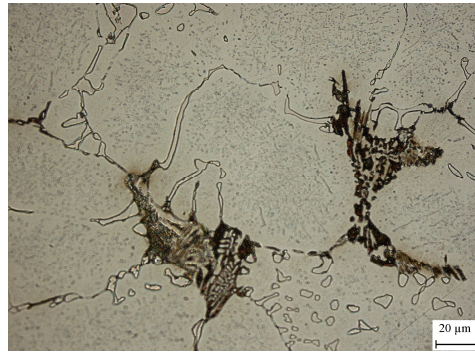


Figure 3b – M₇C₃ (Dark and Fishbone like) and MC (Light and globular) carbides (Murakami etching, Kosmos grade)

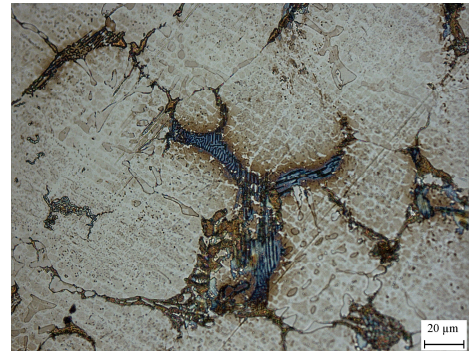


Figure 3c – M₇C₃ (Dark to blue) carbides at grain boundaries (Groesbeck etching, Kosmos grade)

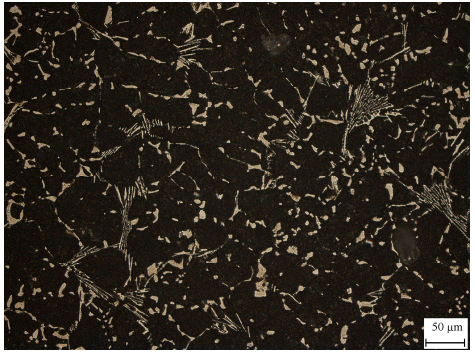


Figure 4a – Deep etching of the matrix leading carbides unetched (Aurora grade)

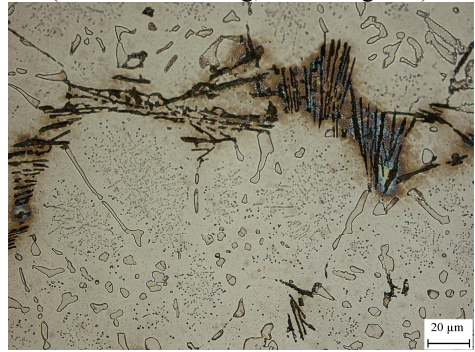


Figure 4b – Acicular M₂C (dark) and globular MC carbides (Murakami etching, Aurora grade)



Figure 4c – M₂C (Dark to blue) carbides at grain boundaries (Groesbeck etching, Aurora grade)

SEM/EDX to determine matrix and carbides nature

The identification of carbides in Kosmos and Aurora was made by electron microscopy (SEM) combined with EDAX microanalyses. Both figures 5 and 6 show carbides distribution and morphology in Kosmos and Aurora grades. Three types of carbides were identified in both grades: MC, M₂C, M₂₃C₆ (figures 4 and 5). In Kosmos, carbide M₇C₃ was found in addition (figure 5) (6). MC, M₂C, M₇C₃ are eutectic carbides which precipitate from the liquid while M₂₃C₆ are very fine secondary carbides that precipitate in the solid state and which are fully dispersed within the matrix .

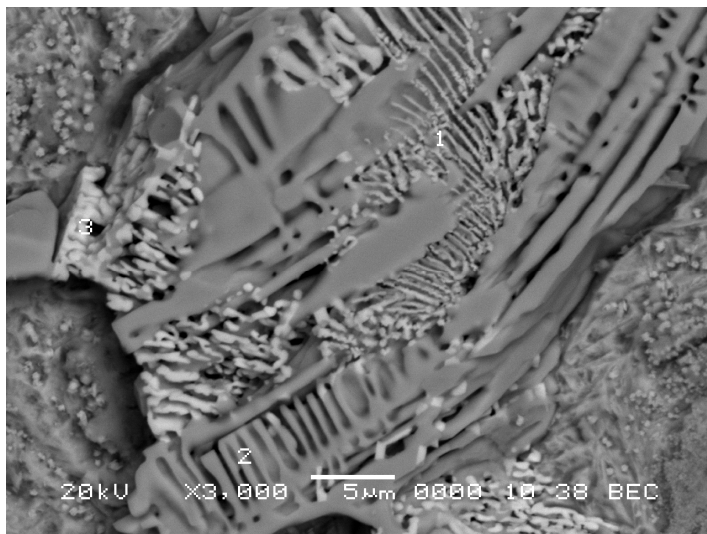


Figure 5 – SEM image on a complex carbide form Kosmos grade in the as-cast conditions

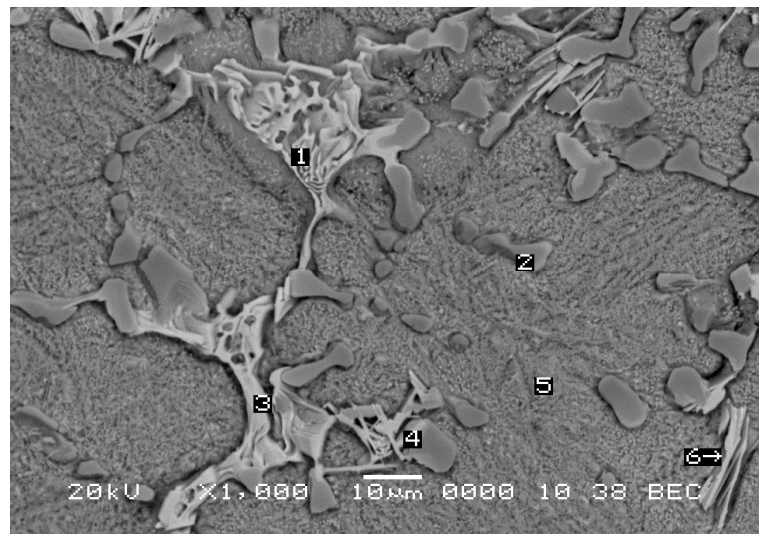


Figure 6 – SEM image on a complex carbide form Aurora grade in the as-cast conditions

Grade	Phases	Mo	V	Cr	W	Fe	C	Weq	Weq/Cr
Kosmos	MC	4,69	38,14	5,30	2,68	3,21	45,94	12,28	2,43
	M ₇ C ₃	4,53	4,92	21,98	1,06	26,98	40,53	10,13	0,47
	M ₆ C	13,09	6,84	15,49	3,52	13,71	47,43	29,69	2,01
	Martensite	3,16	8,29	5,14	1,41	38,19	43,81	7,73	1,50
Aurora	M ₂ C	18,46	9,78	10,03	0,24	9,05	52,06	37,16	3,83
	MC	4,72	47,56	2,52	0,20	2,09	42,96	9,64	3,87
	Martensite	5,02	5,14	4,79	0,06	40,82	42,30	10,09	2,31

Tableau III: Average chemical composition (ZAF, -wt%) of carbides and martensite of Kosmos and Aurora

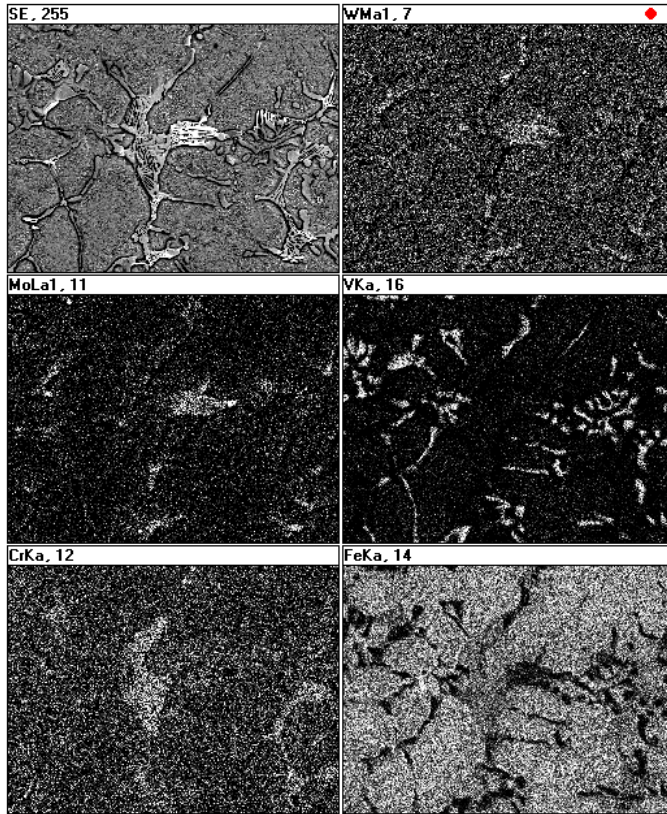


Figure 7 – EDX mapping on carbides of Kosmos grade

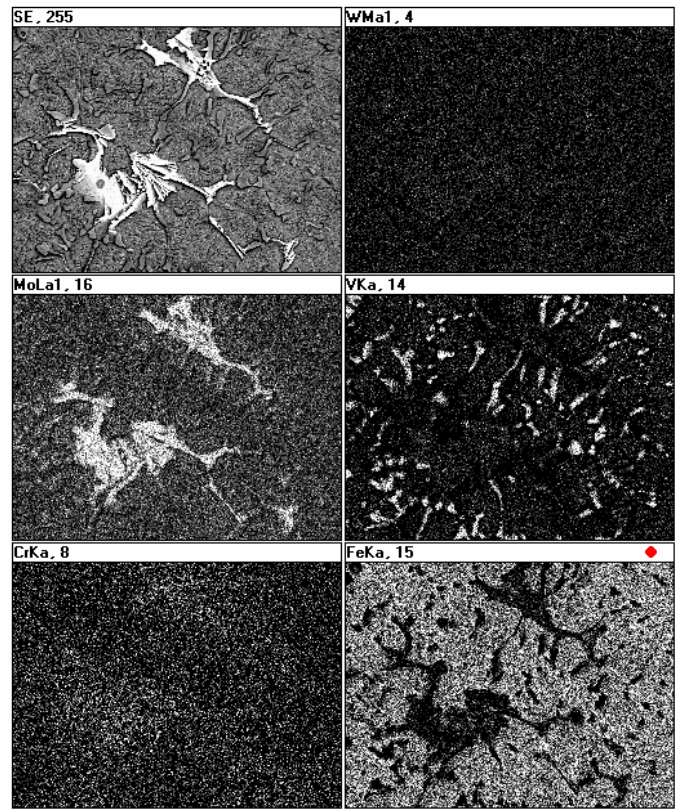


Figure 8 – EDX mapping on carbides of Aurora grade

Table IV compares the nature and morphology of carbides of Kosmos and Aurora to the litterarure.

Nature and morphology of carbides from SEM (Figures 3 to 7)	KOSMOS	AURORA
	1.5/2.0% C	1.5/2.0% C
Nature and morphology of carbides for similar composition with lower V content (4)	7.0/10.0% Weq	11.0/14.0% Weq
	Divorced MC	Divorced MC
	Irregular M ₂ C	Irregular M ₂ C
	Complex-regular M ₇ C ₃	Divorced MC
	Complex-regular MC	Complex-regular MC
	Complex-regular M ₂ C	Irregular M ₂ C
	Complex-regular M ₇ C ₃	Complex-regular M ₂ C

Table IV – Nature and morphology of carbides

The difference in carbides morphology between our results and those given by Boccalini is due to lower V content. (4) The results of ZAF quantification for MC, M₂C, M₇C₃ and the matrix towards EDX microanalyses are shown in Table III. Three parameters have been used:

- Weq (2Mo+W) of the matrix which is related to the solid solution hardening effect;

- Eq.Cr (Cr+1.5Si+Mo) of the matrix which gives the alphas power;
- Weq/Cr of eutectic carbides which is the parameter we propose to characterize the hardening effect of alloying elements in MC and M₂C eutectic carbides.

When comparing the nature and composition of carbides and matrix of Aurora and Kosmos, we observe for Aurora that:

- there are no M₇C₃ which can be explained by the decrease of Chromium content;
- the carbides MC and M₂C are:
 - richer in Molybdenum;
 - poorer in Chromium and Tungsten;
- the parameter Weq/Cr is higher;
- the matrix is richer in Molybdenum and its Weq is higher;
- the parameter Eq.Cr is higher which explains the existence of the peritectic transformation in Aurora.

Table V gives a summary of nature, composition, morphology, distribution of eutectic and secondary carbides in Aurora and Kosmos.

	Composition (EDX)		SEM Characterisation Distribution and morphology	
	KOSMOS	AURORA	KOSMOS	AURORA
MC	V rich (Mo, W, Cr)	V rich (Mo, Cr)	Divorced and idiomorphic	
M ₂ C	Mo, W rich (V, Cr)	Mo rich (V, Cr)	Irregular and platelike	
M ₇ C ₃	Cr rich (V, Mo, Fe)	-	Complex-regular Fan-shaped	-
M ₂₃ C ₆	V rich	V rich	Regular and globular	

Table V – Characterisation of Kosmos and Aurora carbides from SEM/EDX observations

DTA results from melting

Different peaks were observed during heating mode of DTA trials on Kosmos and Aurora grades (Figure 9), each one corresponding to a phase transformation. Depending on the heat release or absorption of the phase transformation, there is respectively dissolution of the phase involved or a precipitation of a new phase directly from one or a combination of several previous phases. Peaks 1 and 4 (Fig. 9) that are related to endothermic reactions involved precipitations of new phases while all other peaks are exothermic ones leading to phases dissolution.

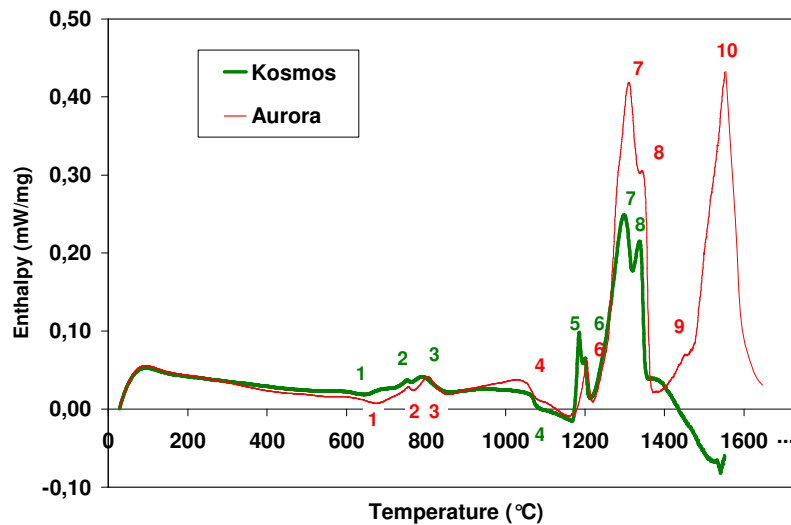


Figure 9 – DTA curve during heating

Table VI compare the melting behaviour of Kosmos and Aurora grades towards the determination of the reaction kinetics maximum that is given by a temperature.

Peak Number	KOSMOS		AURORA	
	Reaction kinetics maximum (°C)	Nature of the phase transformation	Reaction kinetics maximum (°C)	Nature of the phase transformation
1	650	M ₂₃ C ₆ precipitation in martensite	680	M ₂₃ C ₆ precipitation in martensite
2	755	Reverse eutectoid (Martensite → γ)	760	Reverse eutectoid (Martensite → γ)
3	800	M ₂₃ C ₆ dissolution in γ	810	M ₂₃ C ₆ dissolution in γ
4	1085	Peritectoid (δ res → γ)	1085	Reverse peritectoid (δ res → γ)
5	1185	Reverse eutectic 1 (γ + M ₇ C ₃ → L)	---	---
6	1200	Reverse eutectic 2 (γ + M ₂ C → L)	1205	Reverse eutectic 2 (γ + M ₂ C → L)
7	1300	Reverse eutectic 3 (γ + MC → L)	1315	Reverse eutectic 3 (γ + MC → L)
8	1340	Direct melting of γ (γ → L)	1345	Direct melting of γ (γ → L)
9	---	---	1460	Reverse peritectic (γ → δ + L)
10	---	---	1550	Direct melting of δ (δ → L)

Table VI - Temperatures of phase transformations in Kosmos and Aurora during heating

Many observations infer from Table VI and figure 9.

- Peak 1 is related to the secondary hardening effect consisting in the precipitation of fine and widespread secondary carbides of M₂₃C₆ type, directly in the martensite matrix. These secondary carbides are dissolved later in the solid solution gamma (peak 3).
- Peak 2 gives the austenitisation temperature with the complete transformation of martensite into austenite (γ).
- Peak 4 involves an endothermic reaction as there is an inversion in the heating curve slope.
- Peaks 5, 6 and 7 are related to reverse eutectic reactions that involved direction melting of eutectic carbides. Contrary to Kosmos grade, there are no M₇C₃ carbides in Aurora (lack of peak 5).
- Peak 8 which is related to the direct melting of γ involved the completion of the melting in Kosmos grade while it remains uncompleted on Aurora grade.
- In fact, there are two additional peak on Aurora heating curve which correspond respectively to the formation of intrinsic δ from previous γ (peak 9) following by the direct melting of intrinsic δ which also indicate the end of melting process.

Volume fraction towards image analysis

The total volume fraction of eutectic carbides and the volume fraction of each of them depend mainly on the chemical composition, the effect of the cooling rate being less significant [4]. The total volume fraction of eutectic carbides in High Speed Steels for Rolling mill rolls ranges from 9% to more than 15%, which is one of their main characteristics [4].

Figure 10 gives the volume fraction of the MC, M₇C₃ and M₂C eutectic carbides in Kosmos and Aurora as obtained in our image analysis study. The amount of MC and M₂C is higher in Aurora (8.5%) than in Kosmos (6.9%). But as there are no M₇C₃ the total volume fraction of eutectic carbides is higher in Kosmos (14.3%) than in Aurora (8.5%).

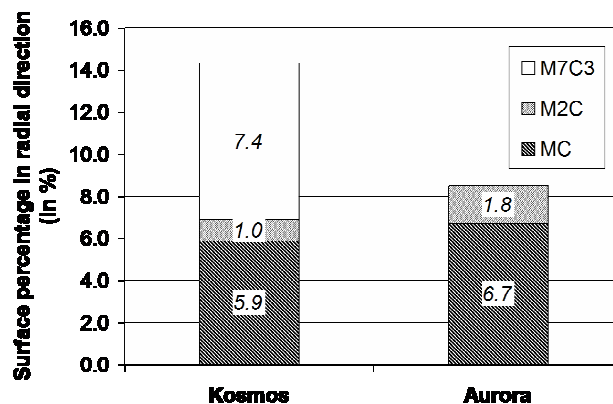


Figure 10 – Surface percentage of carbides in radial direction for Kosmos and Aurora

The amount of M₇C₃ eutectic carbides decreases when the Chromium content decreases. These carbides are rich in Iron, Chromium and other alphas elements. The decreasing amount of M₇C₃ is replaced by other carbides which contain mainly Vanadium, Molybdenum and Tungsten.

Mechanical tests results and industrial performances

Figures 11 and 12 indicate the properties of Aurora compared to Kosmos for compression test and hot hardness.

The Ultimate Tensile Stress (UTS) is higher in Aurora (3203 N/mm²) than in Kosmos (2440 N/mm²).

Another main characteristic of Aurora compared to Kosmos is its high hardness level at temperature in the 500/600°C range (figure 12). This is in good connection with all the observations. The matrix in Aurora is more resistant because its Weq is double (12) comparing to Weq in Kosmos (Table III). Aurora grade contains less carbides amount than Kosmos grade but these carbides are harder: there is no soft carbides such as M₇C₃. Moreover, the MC and M₂C carbides quantity is increasing and they are harder because they are enriched in Weq and poorer in Cr as described by the parameter Weq/Cr (Table III).

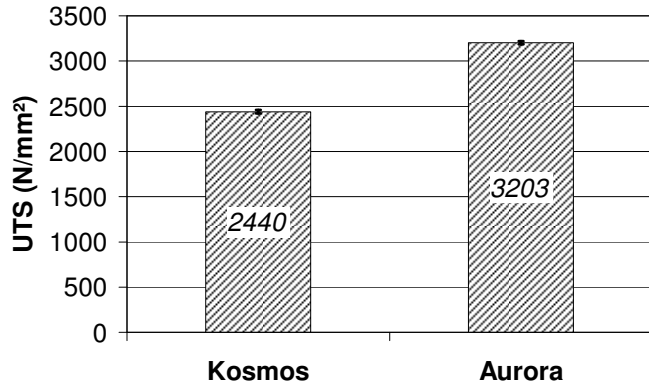


Figure 11 – UTS for compression tests on Kosmos and Aurora

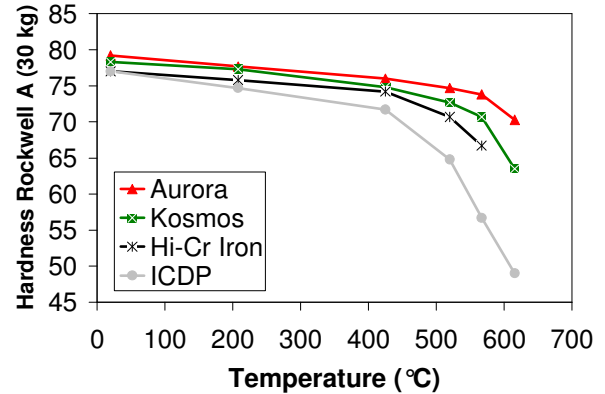


Figure 12 – Hot hardness on various high alloyed steels

Figure 13 compares for both grades the performance in stand 2 of a CSP (Compact Strip Mill). The outputs are expressed in kilometres of tons rolled in the stand per millimetre of stock removal. The performance of Aurora is 14% higher for Aurora (2606 km/mm) compared to Kosmos (2282 km/mm).

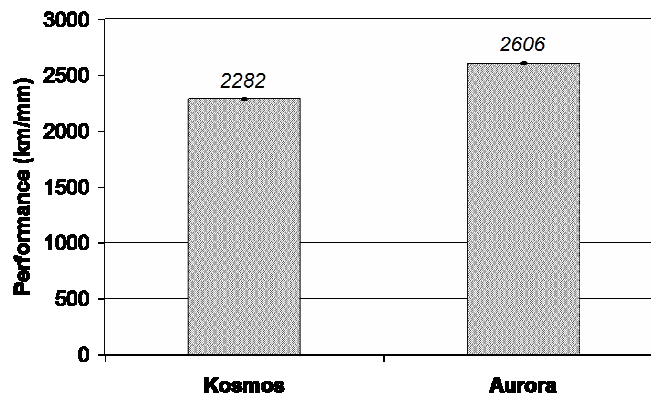


Figure 13 – Performance in stand 2 of a CSP

DISCUSSION

In addition to the inversion of the heating curve slope for peak 4 there is also an endothermic reaction. This thermodynamical behaviour of both Kosmos and Aurora grades is probably related to the presence of residual δ ferrite remaining from the uncompleted peritectic reaction after casting. In fact, evidence of such residual δ ferrite presence were founded in alloys of Fe-Cr-C-X similar to Kosmos and Aurora grades while cooling from the liquid, as there was a staircase in the related cooling DTA curve in the range of 1300 to 1050 °C (5).

The lack of eutectic M₇C₃ carbide in Aurora grade is probably due to its weaker chromium content while comparing to Kosmos grade one. In fact Cr is the major element of M₇C₃ carbide and even if there is some chromium in the chemical composition of Aurora grade, most of Cr is consumed early in the solidification sequence for the precipitation of eutectic MC and M₂C carbides.

The reverse peritectic reaction is the transformation that occurs during the heating, starting from a solid austenite phase and leading to a mixture of solid delta ferrite and a liquid phase. Such a reaction was founded on Aurora (peak 9, fig. 9) leading to the formation of

the so-called intrinsic δ , as the crystallisation behaviour of Aurora grade involved a cross inside delta ferrite area. The presence of such a delta ferrite area at the end of heating is probably due to the alpha stabilizing elements (Si, Mo, Cr) mostly contained in Aurora. In fact Aurora had a high content of Mo.

As peak 10 of Aurora with its apparent maximum at 1620°C is closed to the upper limit of DTA device, further investigations could be performed especially with a slow heating rate in order to reduce the upper shift of phase transformation ranges.

The presence of “intrinsic” delta ferrite in Aurora and not in Kosmos is probably due to the presence of higher Molybdenum content in Aurora.

Tough M_6C carbides were observed on SEM micrograph (Fig. 5, Kosmos grade), these carbides appear with a high rate of cooling, near the external shell on the first 5 mm (5, 6, 10). Deep inside the shell there are no more M_6C . That the reason why no quantification had been performed for M_6C carbides.

During melting of Kosmos and Aurora grades two types of delta ferrite were observed:

- the “*residual*” *delta ferrite* founded on both Kosmos and Aurora, which remains from the previous casting as the incompleteness of peritectic reaction and which is destabilised around 1000°C while heating,
- the “*intrinsic*” *delta ferrite* which is related to the reverse peritectic reaction ($L + \gamma \rightarrow \delta$) occurring while entering into delta ferrite area at the end of the melting. This reaction was only founded on Aurora grade.

Because of its destabilisation above 1000°C, residual ferrite seems to have a minor influence on service conditions of Aurora and Kosmos grades.

In Kosmos and Aurora grade, the eutectic carbides have the same morphology:

- the divorced MC are characterised by an idiomorphic morphology as isolated massive crystals (figure 4 and 5);
 - the irregular M_2C are characterised by a platelike morphology as an acicular shape (cluster of rod-like particles) (figure 4 and 5);
- The complex-regular M_7C_3 presents in Kosmos are characterised by a complex fan-shaped distributed as a continuous network (figure 4).

CONCLUSION

An overview of the development of high Speed Steel during the last century shows that it was strongly connected to the price and availability of alloying elements such as: Molybdenum, Tungsten, Vanadium and Niobium.

It is clear that the strong increase of the price of alloying elements since 2004, mainly Molybdenum (10x) affects the manufacturing cost of the different HSS grades. The cost of the alloying elements is higher for Aurora than for Kosmos. Due to that difference, Aurora will have to show very good performance and reliability to compete with Kosmos.

The Aurora grade presents big differences compared to Kosmos grade:

- there are no eutectic carbides M_7C_3 ;
- a decrease of the total amount of carbides but an increase of MC and M_2C 's carbides quantity;
- harder MC and M_2C carbides with higher parameter Weq/Cr ;
- a more alloyed and more resistant matrix with higher Weq ;
- a high hardness level in the temperature range of 500 to 600°C;
- a higher UTS.

The decrease of chromium and the increase of Weq content in High Speed Steel allow obtaining a more resistant matrix and harder eutectic carbides. The matrix is more resistant due to higher Weq content. The eutectic carbides present in the structure are harder due to the fact that the amount of soft carbides such as M_7C_3 is small or equal to zero and that the amount of hard carbides such as MC or M_2C is higher. Moreover, these carbides are harder due to the higher content of Weq and decreasing content of Cr. These observations could explain the better mechanical properties of grades containing low Chromium and high Weq content.

The Aurora grade gives in operation better performance than Kosmos. That result is in good agreement with the mechanical as well as metallurgical properties described in the present study. The higher manufacturing cost of Aurora compared to Kosmos may be compensated by their better performance and properties.

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