

ROLE OF OXIDATION AND MICROSTRUCTURE CONSTITUENTS ON HIGH CHROMIUM STEEL WORK ROLL SURFACE DEGRADATION*

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Abstract

The study of oxidation behavior of High Chromium Steel material used for surface layer of work rolls was carried out. Specimens taken directly from the roll were subjected to thermal fatigue in the temperature range between room temperature and 500 °C up to 700 °C by using thermo-mechanical simulator Gleeble 1500D. Our observation was focused on the impact of oxidation as a principal mechanism of degradation of the roll's surface layer - mainly on the characteristics of oxidation propagation in relation to the test temperature and microstructure constituents characteristics of the material. Oxidation is considerably accelerated by certain characteristics of eutectic and primary carbides, i.e. their size (area), composition, morphology, orientation regarding to the water-cooled surface, etc. In the presented paper selected cases where emphasized oxidation takes place are investigated, analyzed and explained. Obtained results thus represent a contribution to explanation of conditions which lead to acceleration of oxidation behavior that in turn can lead to chipping or even early spalling of material from the roll surface layer.

Keywords: Rolls; High Chromium Steel; Oxidation; Thermal Fatigue.

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1 INTRODUCTION

Hot rolling is a bulk metal forming process for producing of strips, sheets, wire, rods, various profiles, etc. The central tool of hot rolling process are rolls made from different type of materials according to the position on a rolling mill. Rolls shells presented in this paper are made of High Chromium Steel material and are mounted in roughing stands. In real industrial practice several unexpected early failures of working rolls can occur whereas spalling from roll surface and breakdown of rolls are most frequent. Production costs due to unexpected failures of working rolls are considerably increased since this requires production stop and replacement of working rolls [1-8]. The process of unexpected spalling from rolls surface is not enough explained whereas it is assumed that this can be related to process of oxidation [9].

Hot working rolls are subjected to high cyclic thermal (up to 700 °C), mechanical between 100 MPa and 1GPa, tribological and chemical loads (oxidation). Removal of material from rolls that refers to usually small parts spalling leads to increasing of roughness. This can be attributed to connection of two cracks originating from cooled surface, abrasion and adhesion wear that was also confirmed in laboratory experimental works whereas rate of material removal lies in expected ranges. On other hand unexpected early spalling of increased sized parts leads to considerably change of surface topography and requires immediately replacement of rolls. In this context very valuable are research works which present the microstructure of premature failed rolls from industrial practice and are related to emphasized spalling [1-11]. Thus, Colàs et al [9] presented results of microstructure for early spalling removal of Hi-Cr rolls where extended oxidation processes by formation of loops are presented. Further the authors quote that unevenly oxidation rate between individual constituents generates pathways

where strength of materials was enormous decreased and at linking of two such pathways, i.e. isolating of healthy material with brittle (oxidized) layers, increase spalling removal from surface layer takes place. The authors also admit that reasons for such behaviour are not entirely explained. Furthermore, in laboratory testing of oxidation in humid atmosphere as well as in tribological and thermal fatigue testing [2-5,10-35] some results for slightly increased oxidation penetration behaviour were obtained [2,25] but no explanations where oxidation achieved enormous extend were reported. Oxidation behaviour is less understanding process at hot working rolls [5]. In general, it is assumed that unexpected early spalling is related to common influence of loads in relation to randomly presented unfavourable (uncomfortable) local characteristics so of microstructure as well as local chemical compositions of used roll material which would lead to emphasized oxidation as given in [9].

In this study thermal fatigue test of Hi-Cr steel roll was carried out whereas test samples were heated on maximal temperatures of 500, 600 and 700 °C followed by water cooling. Our observation was focused on selection of cases where oxidation processes and consequently growth of cracks reached enormous extension in comparison to usual observed oxidation behaviour in the testing. Obtained results identified conditions, i.e. combination of test temperatures, characteristic of microstructure for accelerated of oxidation behaviour that can lead to early spalling of material from roll surface layer.

2 EXPERIMENTS

2.1 Material, test and conditions for thermal fatigue investigation

Testing material was Hi-Cr steel for hot working rolls which chemical composition is given in Table 1; increased amount of C

(>1.5wt%), Cr (>12wt%), Mo (>1wt%) and Ni (>1.5wt%) is main characteristic. From the shell of centrifugally casted roll samples were cut out and machined while thermal fatigue tests were carried out on Gleeble 1500D thermomechanical simulator whereas test is described in detail in [36] (Figure 1a). The values of maximal test temperatures were 500, 600 and 700 °C, respectively, whereas the tests were interrupted at 200, 500, 1000 and 2500 cycles. The temperature of samples is measured in the middle of the sample's working surface. The samples were conductive heated (controlled by computer) (Figure 1b) to the maximal temperature in approximately 3 s and then rapidly internal cooled with water (Figure 2).

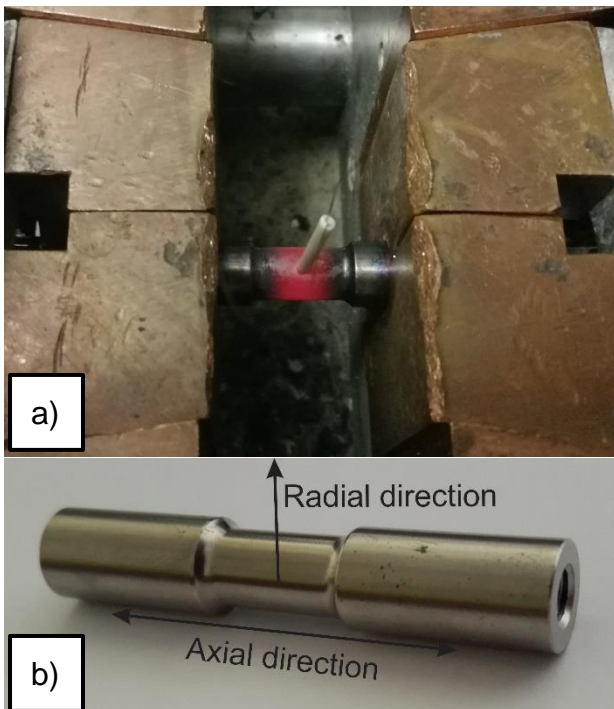


Figure 1. a) Setup of the specimen; b) Direction of investigation.

Thus, resembles the conditions prevailed on roll surfaces during hot rolling of steels can be achieved, i.e. conditions for emphasized oxidation were established, i.e. heating of surface layer up to temperature range 500 – 700 °C in time of its contact with hot roll-piece followed by water cooling to a minimum temperature of about 100 °C. Coolant inlet and outlet are

enabled through tubes on the sample sides.

Main characteristics of microstructure of used material regarding to initiation and growth of cracks, conditions for material spalling and crushing of carbides, occurrence of internal cracks, etc. as well as quantification about thermal fatigue resistance of test roll steel. i.e. crack density, average cracks length, maximal cracks length, areas with enclosed cracks were reported in [37] wherein conditions for emphasized oxidation behavior regarding microstructure characteristics and temperature were less researched.

Table 1. Material chemical composition in wt%

C	Si	Mn	Cr	Mo	V	Ni
1.50	0.50	0.60	11.00	1.00	0.20	1.50
-	-	-	-	-	-	-
2.00	1.00	1.20	13.00	2.00	0.50	2.00

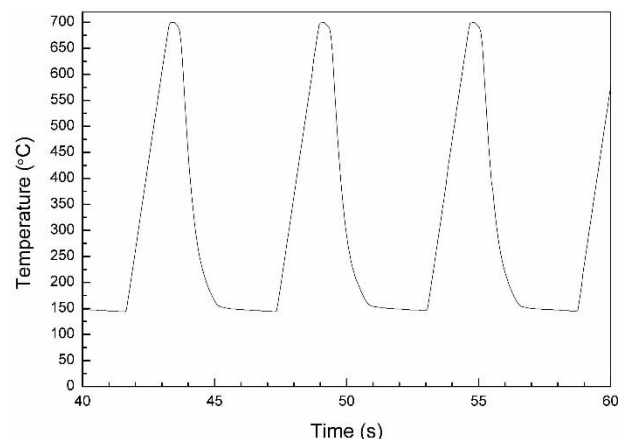


Figure 2. Time course of temperature for maximal test temperature of 700 °C.

2.2 Characterization of microstructure

Optical microscopy (Carl Zeiss AXIO Imager.A1m) and scanning electron microscopy (JEOL 5610, ZEISS and FEI NanoSEM) were employed for the observation of surfaces, microstructure, oxidation and cracks. SEM microscopy was combined with attached energy dispersive X-ray spectroscopy (EDX) for chemical mapping of microstructure. In order to obtain clear distinction regarding to chemical composition of primary and

eutectic carbides Murakami etching was applied.

Initial phase identification was performed with a conventional X-ray diffraction (XRD) measurements on a polished sample using a Bruker D8 Advance X-ray diffractometer with position sensitive detector (LynxEye EX) and Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$) in a combination with optical and electron microscopy. A continuous scanning mode was chosen for XRD with a rate of 0.01 s^{-1} over an angular width of $2\theta = 30^\circ - 120^\circ$ and a Ni filter was used to obtain a nearly monochromatic X-ray beam.

3 RESULTS

3.1 Relevant microstructure properties of Hi-Cr steel

From Figure 3a can be seen that microstructure is composed from a primary, eutectic (Figure 3b) and secondary carbides embedded in a tempered martensite matrix. Eutectic carbides are in general formed as lamellae, lenticular and globular shape while primary carbides solidified at grain boundaries are elongated. Further primary carbides are longer ($50 \mu\text{m}$) and thicker (usually above $10 \mu\text{m}$) in comparison to eutectic carbides which length amounts below $40 \mu\text{m}$ while their thickness is usually below $10 \mu\text{m}$. Usually all three shapes of eutectic carbides were observed as shown in Figure 3a. Moreover, observing detail depicted in Figure 3b it can be seen that outer area of eutectic carbides exhibits thicker carbides with increased continuity compared to the internal area of eutectic carbide clusters. Measured thickness of carbides in outer area is in range $3\text{--}10 \mu\text{m}$. Size of a spheroidal carbides in the matrix were measured between 200 nm and 700 nm , while lenticular carbides exhibited approximate size of $400 \text{ nm} \times 1000 \text{ nm}$. The material was indented to determine Vickers hardness. Vickers hardness amounted $601 \pm 10 \text{ HV}_{10}$. The microstructure was identified (using EDX)

as a mixture of martensite and several types of carbides. Carbides detected in the sample were hexagonal Mo based M₂C and hexagonal Cr based M₇C₃ carbides. Further, in matrix along primary carbides, i.e. inside band width of $4\text{--}5 \mu\text{m}$ (see Figure 2a), as well as in eutectics (matrix) decreased content for Cr was observed, i.e. in range $4\text{--}5 \text{ wt\%}$. On other hand content of Cr in matrix, i.e. center of grain, is in range $7\text{--}9 \text{ wt\%}$. Cr content in primary carbides is in range $42\text{--}50 \text{ wt\%}$ while in eutectic carbides 50 wt\% . These values rapidly fall in range of $4\text{--}5.5 \text{ wt\%}$ in vicinity, i.e. narrow band of width of about $4 \mu\text{m}$, along carbides and increase again in matrix area, i.e. in distance of several μm from carbides. In matrix of eutectic also decreased contents for Cr, i.e. $4\text{--}5.5 \text{ wt\%}$, were obtained.

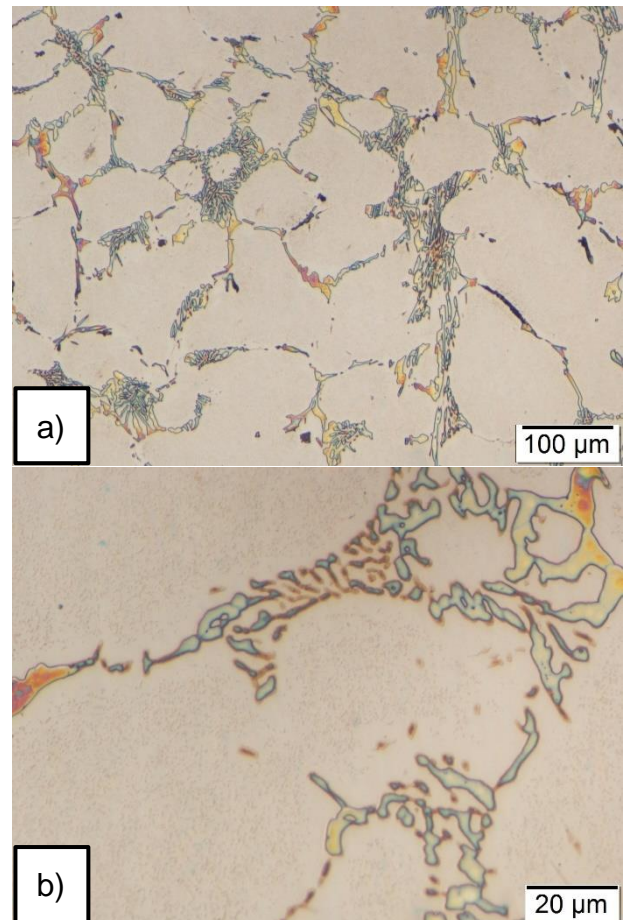


Figure 3. Initial microstructure; a) visible presence of primary and eutectic carbides; b) eutectic carbide.

3.2 Spalling of small parts from cooled surface

Removal of material from rolls that refers to small parts spalling leads to increasing of roughness. This can be attributed to crushing of carbides, to connection of two small cracks which originate from cooled surface (see Figure 4a-b) as well as to abrasion and adhesion wear whereas rate of material removal lies in expected ranges; this was also confirmed in laboratory experimental works [4-5,9-15,36]. Above mentioned connection of cracks usually takes carbides pathways; i.e. growth of cracks is related to cracking of carbides and their oxidation on spots of cracks occurrence.

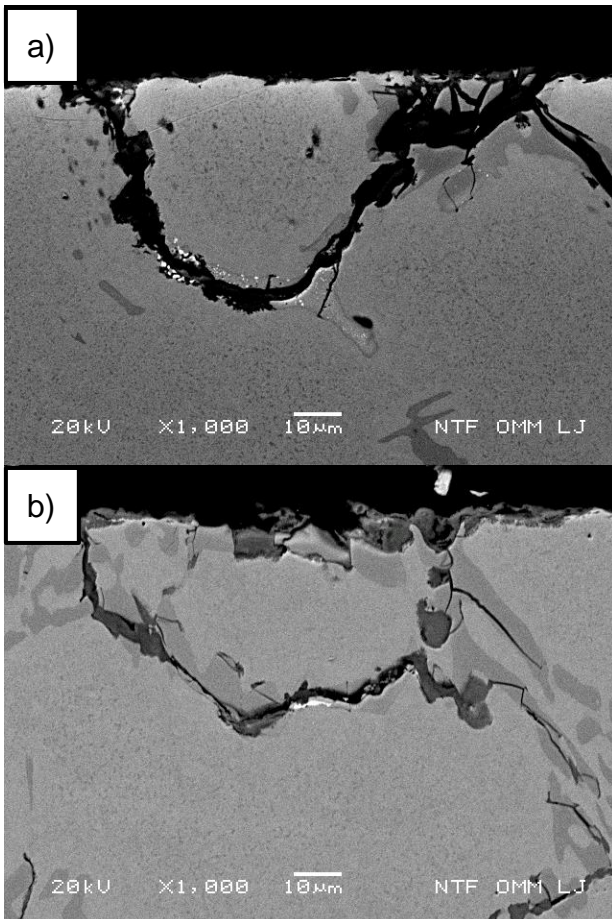


Figure 4. Small part spalling; crushing of carbides, cracks growth using carbides pathways and connection (linking) of cracks leading to small parts spalling (a and b).

3.3 Cracking and oxidation of primary carbides and eutectics at cooled surface

Both, i.e. thermal stresses at cooled surface that leads to cracking of carbides as well as oxidation rate increase with increase of testing temperature. Thus, primary (Figures. 4a-b) as well as eutectic carbides precipitated at cooled surface or close to cooled surface (Figure 5a-b) crack is first step of their degradation during thermal fatigue. The process of oxidation usually follows the process of cracking of carbides. The process of cracking predominately refers to carbides and in lower degree also matrix material between eutectic carbides; thus, cracks represent ways for entrance of oxygen in eutectic internal, i.e. conditions for accelerated oxidation of eutectics (Figure 5c, 700 °C, 2500 cycles) are established. Extent of oxidation increases with test temperature as well as number of thermal cycles. Thus, on Figures 5a-d are presented typical images of oxidized eutectics at test temperature of 700 °C after 200 cycles (Figure 5a), 1000 cycles (Figure 5d) and 2500 (Figure 5b-c) cycles. Here should be emphasized that increased size of carbides in eutectics are more prone to cracking that due to formation of new free surface in cracked carbides also accelerates oxidation. Thus, the oxidation that takes place on cracked carbides usually follows their initial crack path. Furthermore, from Figure 5d is visible that oxidation rate of matrix material in eutectics is increased in comparison to eutectic carbides; in this case no emphasized cracking of eutectic carbides is visible whereas oxidation process takes place by preferential oxidation of eutectic matrix. Thus, oxidation rate of eutectic matrix is increased and can also be attributed to lower content of Cr in eutectic matrix, i.e. in range 4.5–6 wt%; as mentioned above in matrix material these values are in range 7-9% wt. Oxidation of eutectics without previously cracking of eutectic carbides also indicate on their

accelerated oxidation regardless to their orientation, i.e. also in axial direction. This is relevant at linking of parallel cracks which grow in perpendicular direction, i.e. originate from cooled surface.

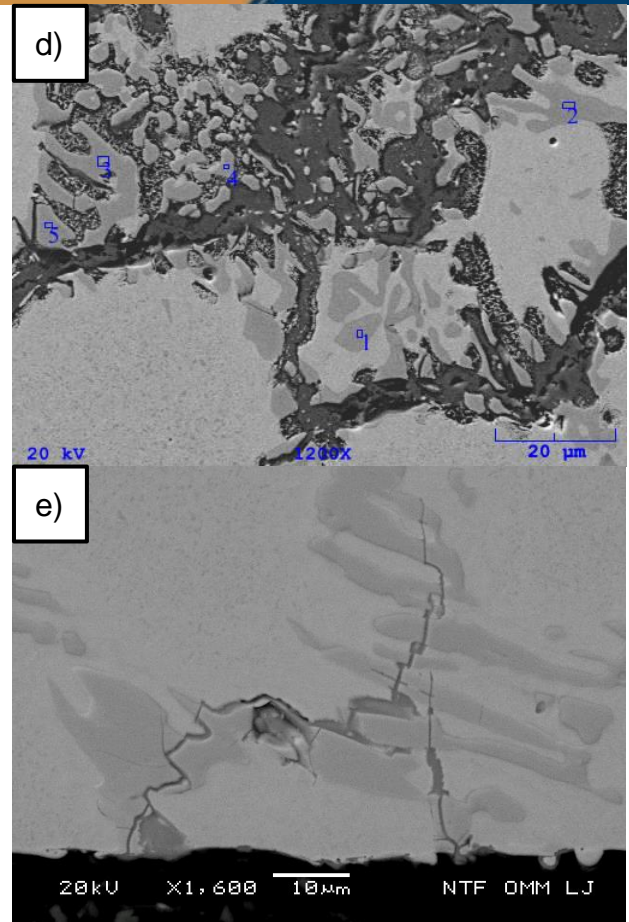
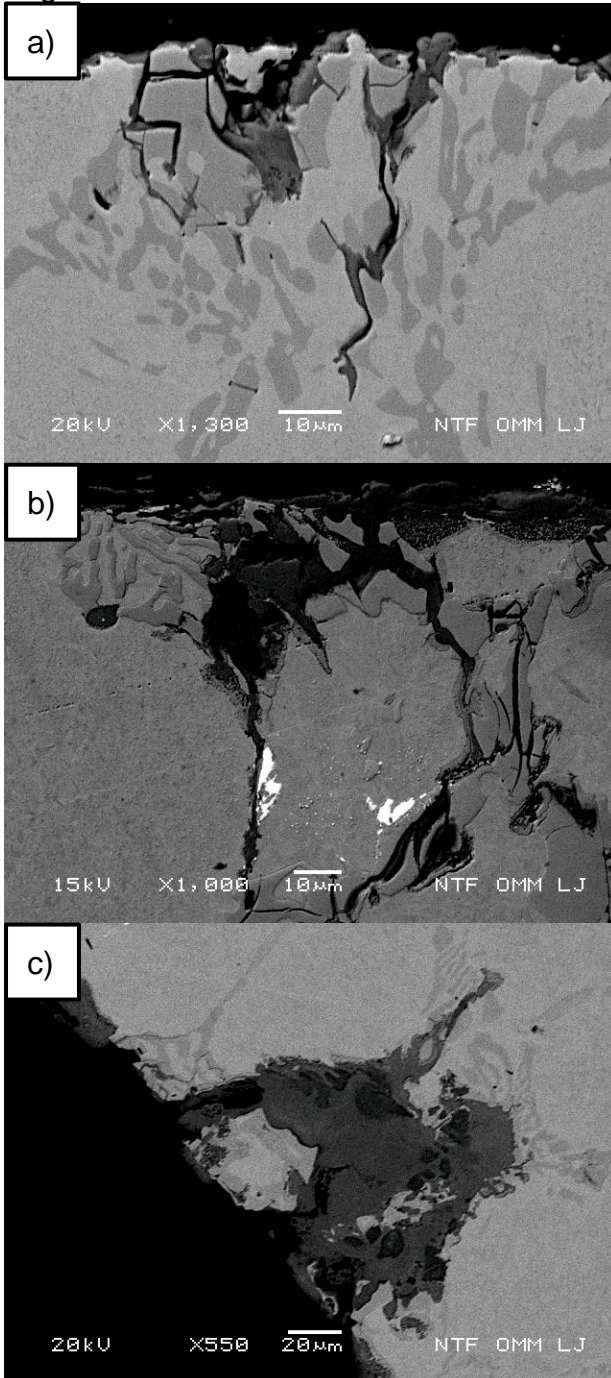


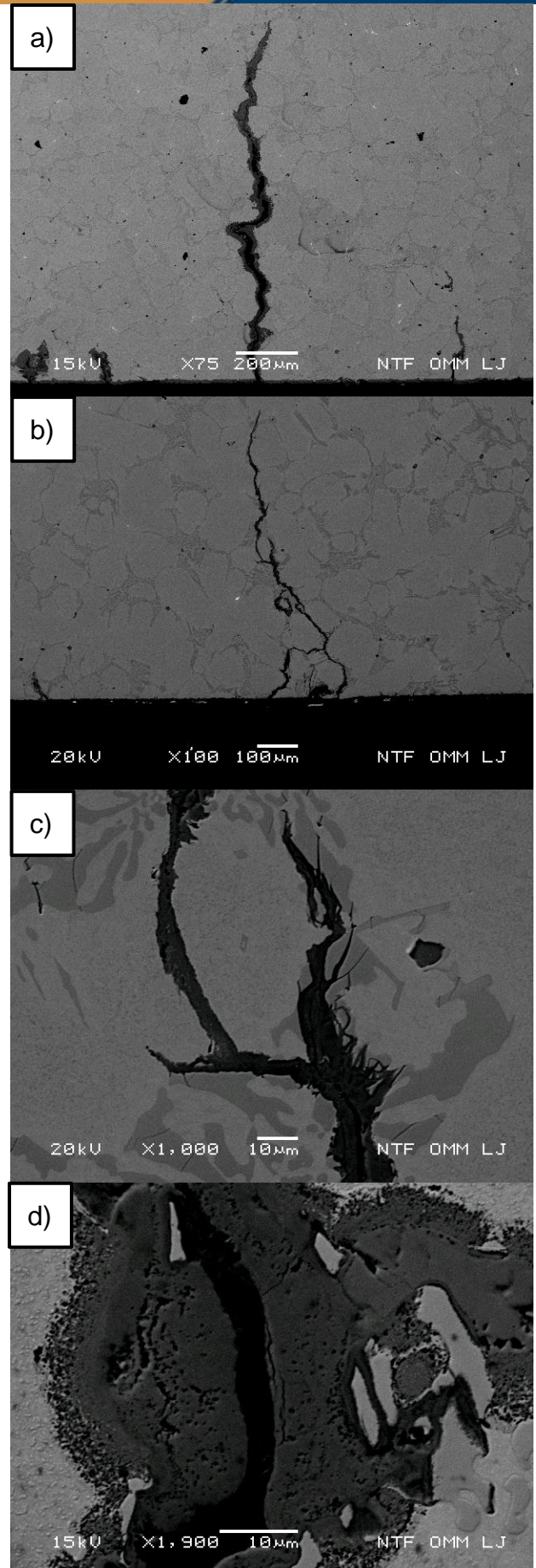
Figure 5. Cracking and oxidation of eutectics; a) at 700 °C and at 200 cycles; b) and c) at 700 °C and 2500 cycles; d) at 700 °C and 1000 cycles and e) at 500 °C and 1000 cycles.

Cracking of eutectic carbides and oxidation rate decreases with lowering of temperature. Thus, on Figure 5e (500 °C and 1000 cycles) process of cracking and commencement of oxidation is visible and when comparing with Figure 5a (700 °C and 200 cycles) despite higher number of cycles both processes are decreased.

3.4 Growth of cracks using predominately primary carbides and matrix, and eutectic carbides pathways

On figures 6a and 6b two cracks are presented whereas the length of crack shown on Figure 6a is considerably lower in comparison to crack shown in Figure 6b. This is attributed to different pathways of growth of cracks.

Thus, crack on Figure 6a took at its growth at primary and eutectic carbides as well as matrix material. Growth of cracks through matrix (Figure 6c) is decreased since toughness of matrix is increased regarding to carbides. Consequently, also length of the crack is lower in comparison to the crack shown on Figure 6b which took predominately eutectic carbides (Figure 6d) pathways. This indicates that oxidation in eutectics decisive contribute to growth of crack. Namely eutectics areas are increased in comparison to primary carbides areas.



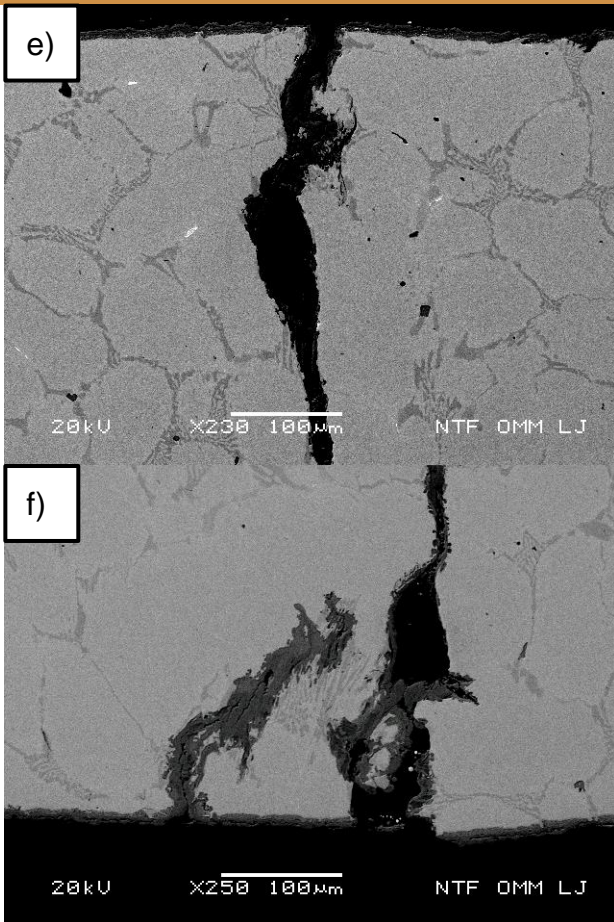
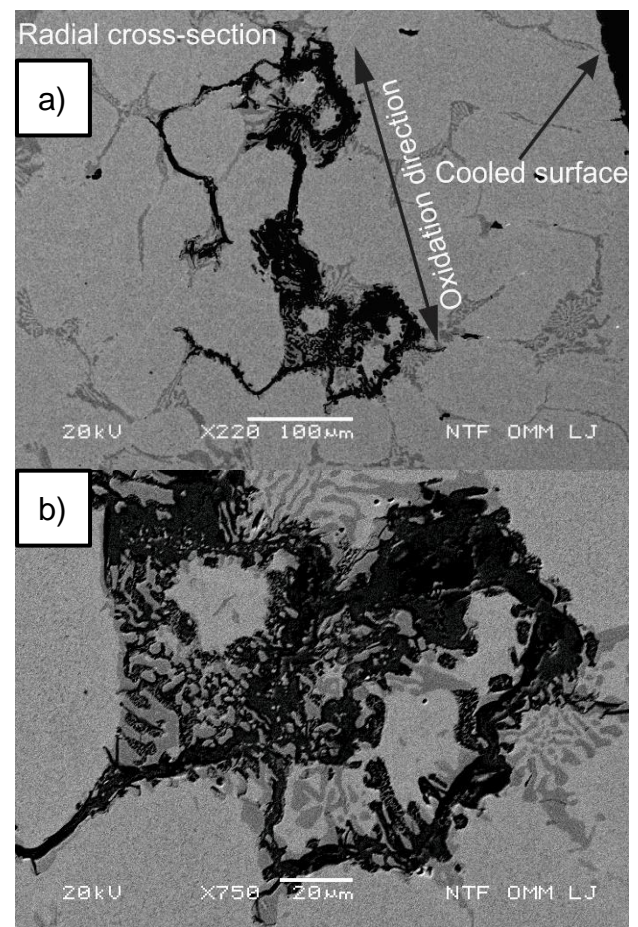


Figure 6. Growth of cracks; a) using eutectic and primary carbides as well as matrix material; b) with detail of using matrix material; c) using predominately eutectic carbides; d) detail oxidized eutectic carbides at $T=700\text{ }^{\circ}\text{C}$ and 2500 cycles; e) and f) accelerated growth of cracks.

Thus, in case oxidation due to increasing volume oxidized material also increases stresses at crack tip that accelerate growth of cracks (Figure 6e-f); on both figures is clear visible elastic deformation of spot around oxidized eutectic, i.e. lift of material from original level. In case that several eutectics in radial direction are successively precipitated this led to accelerated crack growth and consequently to longer crack that in case of oxidation of eutectic perpendicular to crack growth leads to spalling of increased size of material from surface layer.

3.5 Spalling of medium and increased size areas on base of crack growth and oxidation

Accelerated oxidation of eutectics can promote also crack growth in axial or other directions, i.e. in directions of usual crack growth which are almost perpendicular to cooled surface. Thus, on Figure 7a on radial cross-section of test specimen oxidation growth through eutectic in direction almost parallel to cooling surface is visible. On detail on Figure 7b is visible again that oxidation of matrix material is increased in comparison to carbides as well as that cracking of eutectic carbides is decreased.



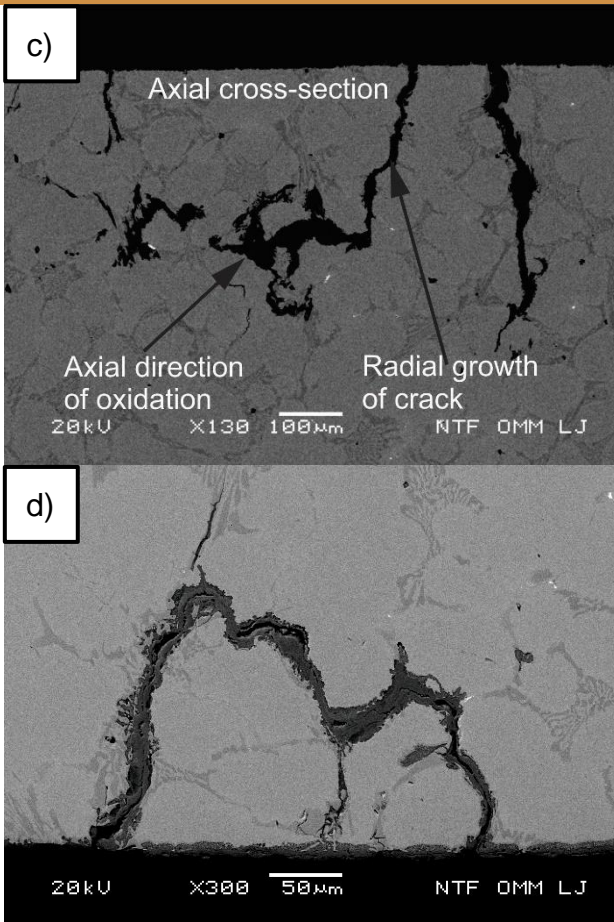
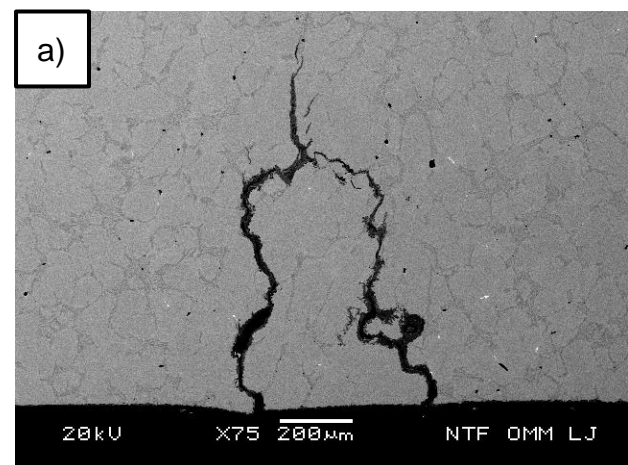


Figure 7. Oxidation of eutectics in direction perpendicular to crack growth on: a) radial cross-section; b) with detail of oxidation; c) linking of crack with oxidized area which direction is perpendicular to crack growth and d) linking of oxidized area of eutectics at $T=700\text{ }^{\circ}\text{C}$ and 2500 cycles.

On Figure 7c is visible linking of crack which grew perpendicular to cooled surface with eutectic oxidized area whereas oxidation direction is parallel to cooled surface. On left side of the figure is visible second eutectic oxidized area while in direction to cooled surface small crack can be observed. With further increasing of heating - cooling cycle linking of all mentioned areas in Figures 7c would take place consequently also spalling of particle of increased size, i.e. ca 0.6 mm in length and ca 0.5 mm in height. On Figure 7d medium sized particle, i.e. 0.3 mm in length and 0.2 mm in height, before spalling is visible whereas almost entire oxidized area was eutectic area.

On Figure 8a-b increased size of particles close to spalling are visible where two parallel cracks growing perpendicular to cooled surface were linked. Increased width of oxidized crack means also oxidation of eutectic through which grew the cracks. On Figure 8a both parallel cracks (distance between cracks about 500 µm) in depth of about 800 µm change the direction of growth, i.e. from perpendicular to almost axial direction whereas both follow eutectic pathways and link together. Crack enclosed area on Figure 8b is slightly increased in comparison to one on Figure 8a, i.e. width between parallel cracks is almost 800 µm while axial oxidation (linking) occurs in depth of almost 1100 µm. Here is worth to mention that with increasing of depth, i.e. distance from cooled surface, the values of thermal stresses decrease that consequently increases the role of oxidation. Thus, high sized oxidized eutectic areas can also in deeper levels considerably accelerate the growth of cracks regardless the orientation direction of eutectics.



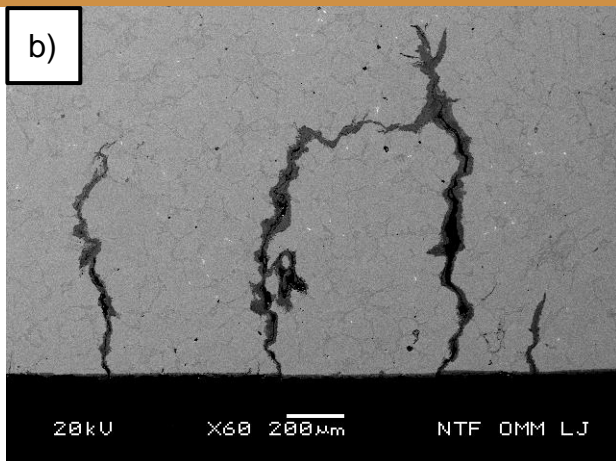


Figure 8. Cracks enclosed increased areas with emphasized oxidation in axial direction (a and b), T=700 °C, 2500 cycles

4 CONCLUSION

Explanation of early spalling of particles from roll surface layer (Hi-Cr steel) was focus of this work. This was carried out by thermal fatigue testing whereas test samples were heated to maximal temperature in range 500–700 °C following by internal water cooling of samples. Thus, conditions for study of oxidation behavior were established. Characterization of microstructure and oxidation behavior on test samples using electron and light microscopy was carried out. From obtained results following conclusions can be derived:

- Different eutectic types, i.e. spheroidal, lamellae and stick, was found in microstructure. Orientation of carbides is versatile that in combination with oxidation behavior and thermal stresses influences on growth of cracks.
- Depletion of Cr content in eutectic matrix material as well as in matrix material in shape of narrow band along primary and eutectic carbides exists, i.e. around 4-5.5wt% in comparison to content in matrix, i.e. around 7-9wt%.
- Lower content of Cr in eutectic matrix is base for emphasized oxidation eutectics.
- Favorable oxidation of eutectics on surface layer is additionally related to cracking of eutectic carbides as well as to

lower content of Cr in eutectic matrix. Oxidation of eutectics bellow surface is also related cracking of eutectic carbides as well as formation of cracks originating from surface to eutectic that enables entrance (diffusion) of oxygen. Thicker carbides in eutectic are more prone to cracking.

- Oxidation of eutectic is temperature, number of thermal cycles and chemical dependent. Higher test temperatures, number of thermal cycles and lower content of Cr in matrix increases oxidation rate.
- Oxidation rate of matrix in eutectics is higher in comparison to oxidation rate of eutectic carbides.
- Direction of oxidation is determined by orientation of eutectics, i.e. regardless the thermal stresses occur. This behavior in case of oxidation growth approximately perpendicular to growth of cracks originating from cooled surface enables linking of cracks (at least two) at their growth in radial direction.
- Oxidation of eutectics in axial direction in deeper levels below cooled surface in combination with perpendicular direction of cracks growth using predominately eutectic and primary carbides pathways leads to linking of oxidized areas and consequently to early spalling of enclosed areas from surface layer.
- Producing of Hi-Cr steel so with lower share as well as decreased size of eutectics is recommendable. Additionally, chemical element which decrease oxidation rate of eutectic matrix should be added. Mo could fulfil this task.

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