Scale development on steel during hot strip rolling

M. Graf, R. Kawalla

The paper presents a new method to describe and to predict the development of scale during metal forming processes, especially hot strip rolling processes. This is necessary because scale develops during all hot deformation processes over 600 °C and affects the mass loss of the raw weight as well as the surface quality of the semi-finished product. The main components of oxide scale at steel are wustite, magnetite, and hematite with various volume fractions. A challenge for the correct characterisation of the total scale layer is the consideration of the strong inhomogeneity with respect to the mechanical properties of each scale component. Owing to these differences, the deformation behaviour of the single oxide layers is diverse, too. As result of high deformation stresses, the oxide scale cracks, however low deformation degrees can be compensated. Due to the high hardness of the oxides, the fragments are pushed in the raw material and influence the grip conditions in the rolling gap and the material flow. Because of this the Institute of Metal Forming developed a new method to determine the temperature dependent properties of the scale layers, which are relevant for the arrangement of metal forming processes. The strategy was based on the use of pure powder of each scale element for the production of testing samples with different geometries (cylindrical and cubic samples) by compression and heat treatment steps.

Keywords: Oxide scale - Scale structure - Hot strip rolling - Deformation behaviour - Experimental simulation - Surface quality

INTRODUZIONE

For the simulation of hot rolled flat products the scale growing and its behaviour during rolling are very important, as scale developing on steel surfaces at temperature over 570 °C in oxidizing atmosphere. This influences the material flow due to the friction conditions in the rolling gap. Additionally, heat transfer and surface roughness are affected as well.

Simulated process chains often do not consider the surface quality. Especially the surface quality, however, strongly influences the achievable profit of the production line. Thus, it is important to predict the scale development exactly. At the same time the simulation should give a technologi-

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cal proposition for a good strip surface. During hot rolling processes three kinds of scale exist, which differ in the temporal and local evolution. Thereby, primary, secondary, and tertiary scale is formed on the surface within the hot rolling process chain, shown in Fig. 1.

Primary scale occurs during reheating of slaps and billets without deformation stress. Reheating needs long time in comparisons to the deformation and transport. That results in a massive scale growth up to 2 % of the sample weight for steel, which depends on the kind of reheating. It can be assumed that this first scale will be removed by the descaler in front of the first rolling mill. Therefore, this scale type is uninteresting in terms of the deformation behaviour and the description of the surface evolution as it is not involved in the deformation process. In contrast, the secondary and tertiary oxide scale must be analysed and considered because they grow between two deformation steps and are deformed in the mills. As a result of the significant differences in terms of the deformation behaviour of scale and substrate, the oxide layer cracks and the oxide fragments will be pressed in the metal matrix or the metal will be extruded between two single neighbouring oxide broken bits.

To understand the morphology of scale along a continuous rolling it is necessary to know the scale structure and the mechanical properties of the single scale components independent of each other. It is known that the scale develops according to a reaction kinetic growing law depending on the diffusion (thick layer) and the interface reactions

Fig. 1 - Kinds of scale during hot rolling

Fig. 1 - Tipologie di scaglia durante la laminazione a caldo
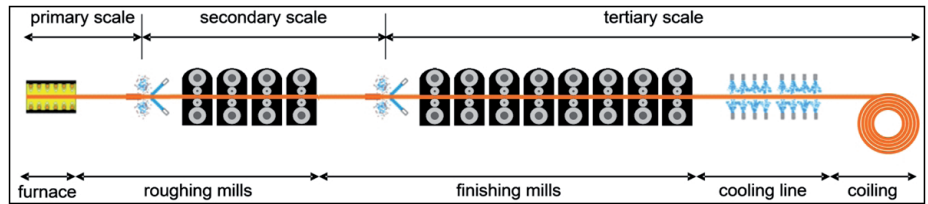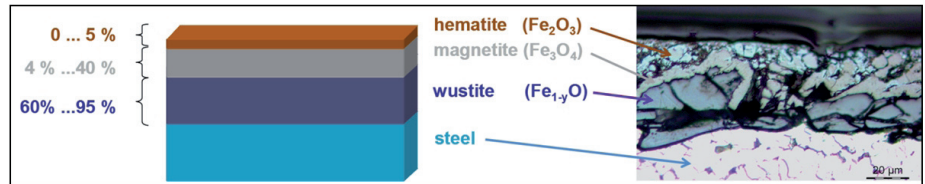

Fig. 2 - Volume fraction of layered scale components

Fig. 2 - Frazione di volume dei componenti di strati di scaglie

(thin layer). Hidaka et. al. was one of the first who identified the flow curves and the fracture toughness at pure steel samples. Their results were carried out in specific atmospheres focusing on the oxygen partial pressure in order to develop only one single ferrous oxide. Afterwards the specimens were tested in tensile test with a very slow strain rate ($2*10{-4}$ s-1) [1]. However the strain rates applying are not representative for hot deformation processes, especially for hot rolling. Furthermore oxide scale has similar behaviour to ceramic materials, which are very sensitive to tensile stresses in comparison to compression stresses [2]. Hence, the first measured and published flow curves constitute a good result and orientation. For the consideration of dominant compression stresses during rolling, however, compression tests are necessary. These allows a high strain rate and temperature combination.

**METHODE FOR IDENTIFICATION OF SCALE PROPERTIES**

An evaluation of a scale-covered steel sample is carried out by a light microscope. Thereby, the volume fraction of the oxides can be identified (Fig. 2).
This figure shows an inhomogeneity in the layer thickness

as well as in the resulting deformation properties. The diverse behaviour of the scale during deformation process is due to the different thickness of the particular scale layer and its internal structure. Additionally, there is also a relationship between these characteristics and temperature strain rate combination which will be shown in this paper.
The new experimental method is based on pure oxide powder of homogeneous stoichiometry as initial material, which is possible for hematite and magnetite (Fig. 3). Wustite has the chemical-specific properties that it is semi stable at room temperature. It is therefore essential to use a modified method to generate these sintered samples.
The sample geometries were produced into two different types as indicate in Fig. 3. This is important for test method requirements, e.g. compression and 3-point-bending-test. Then, a compression step in conventional presses with a force of approximately 50 kN were implemented. After demoulding the green bodies are sintered in different oxygen partial pressures depend on the material. The thermo-mechanical treatment is necessary to realise a material cohesion in the powder samples. Hematite can be sintered in electrical heated chamber furnaces, because the ambient pressure is sufficient for this material to protect the existential necessary oxygen partial pressure (red line in Fig.

Fig. 3 - Technology for Identification of Scale Properties [3]

Fig. 3 - Tecnologia per l'identificazione delle proprietà/caratteristiche della scaglia [3]
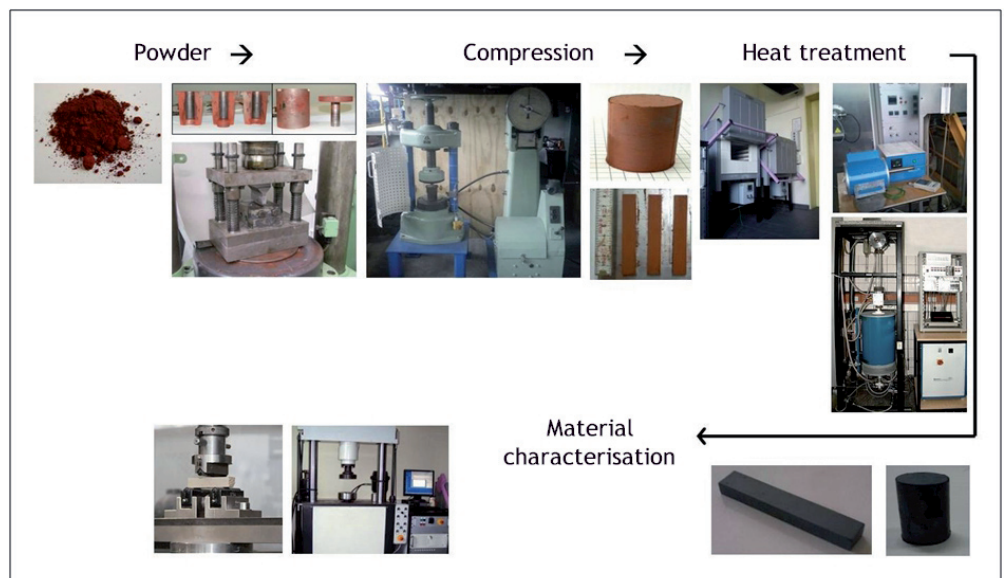
4). The technology for magentite is more complicated, because for chemical equilibrium the oxygen partial pressure increase with sintering temperature but it is lower as in normal furnace atmosphere. For realization of the correct pressure of 10-4 – 10-8 atm at 1200 °C (blue line in Fig. 4) a nitrogen air mixture is used. As has been noted, the heat treatment strategy for wustite is also different due to the stability of this at room temperature. Hence, magnetite powder is the initial material again for the wustite samples. As result of a special oxygen pressure value a phase transformation to wustite is enforced. A finale pressure for wustite of 10-9 – 10-12 atm at 1200 °C (upper green line in Fig. 4) can not be set by using a oxygen based gas mixture (very low oxygen concentration is required), therefore a auxiliary gas mixture on basis of hydrogen-water vapour is advisably (lower green line in Fig. 4) [4]. For the heat treatment of wustite a thermogravemetry was used [5], which realised an oxygen partial pressure of 10-10 atm by injecting a humidified argon hydrogen gas in the furnace.

For all heat treatments the finale temperature amounted 1200 °C, which was set with a heating rate of 3 K/min. This is slow enough to minimise the risk of thermal stresses, which can damage the sample and induce the material failure. After a holding time of two hours a likewise slow cooling process follows. In addition to get a material cohesion the aim was to generate a comparable structure in the sintered sample as well as in the real oxide scale in a rolling line (Fig. 5).
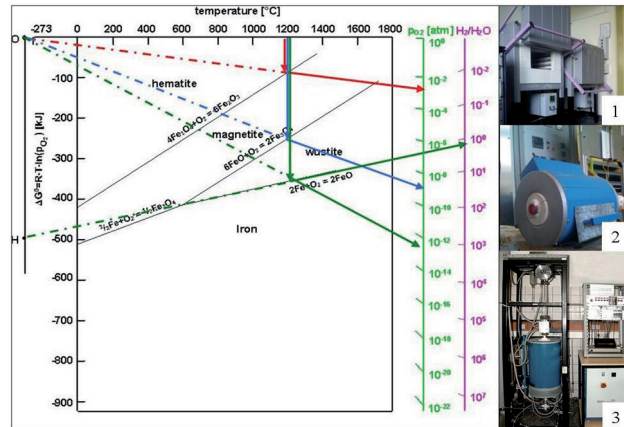

Fig. 4 - Modified Richardson-Ellingham-Diagram [4] and used furnaces (1: electrical chamber furnace; 2: tube furnace with nitrogen gas connection; 3: thermogravimetry [5])

Fig. 4 - Diagramma Richardson-Ellingham modificato [4] e forni utilizzati (1: forno elettrico a camera; 2: Forno tubolare con allacciamento per azoto gassoso; 3: termogravimetria [5])

One example of the hot rolling process for flat products should illustrate it. Along the rolling line secondary scale is formed with a porosity of 20 % volume fraction. Therefore, through sintering an equally porosity must be produced in
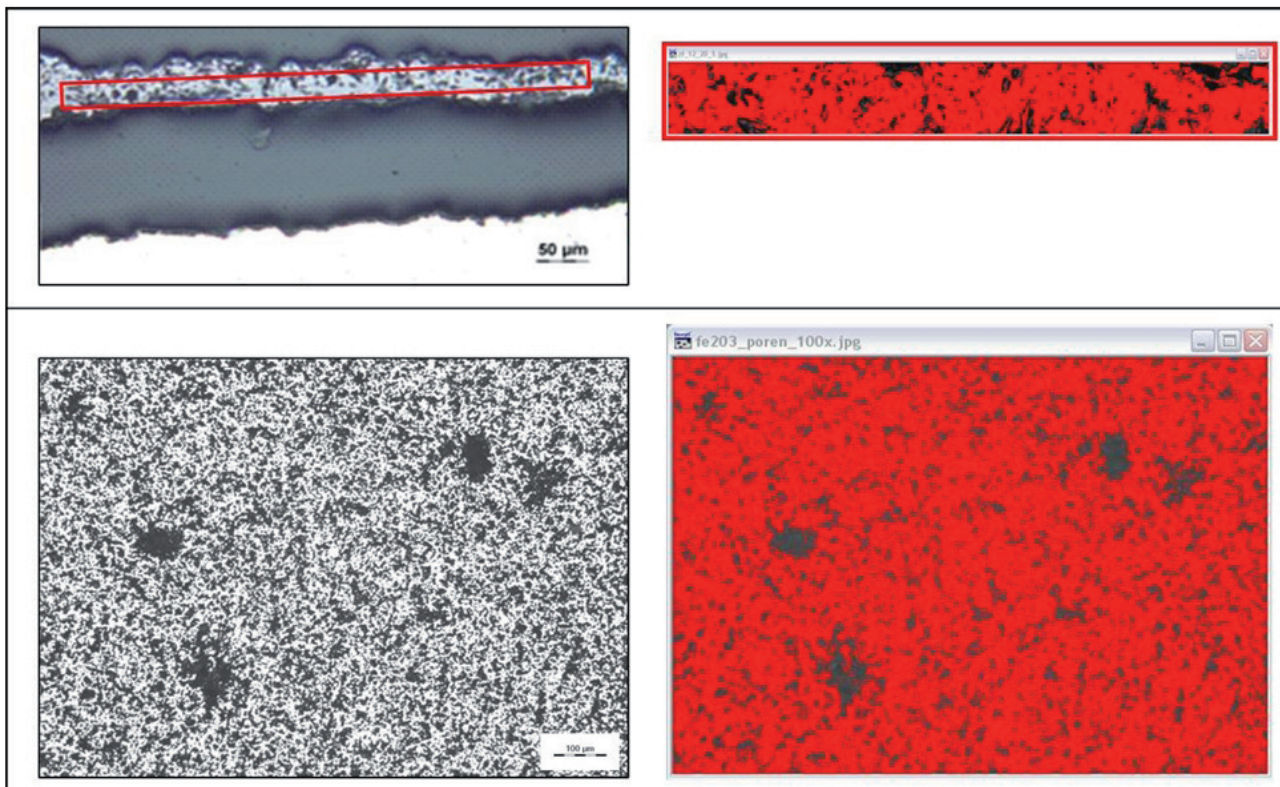

Fig. 5 - Structure of secondary scale in front of first deformation (1200 °C and 10 s oxidation time; upper pictures) and of sintered samples (lower pictures)

Fig. 5 - Struttura di scaglia secondaria conseguente alla prima deformazione (1200 °C e con tempo di ossidazione di 10 s; in alto) e di campioni sinterizzati (in basso)

the pellets to describe the scale behaviour of secondary scale properly. Two different mechanical testing methods were performed (Fig. 3) - the cylindrical samples were tested in the hot deformation system to measuring flow curves at different temperatures and strain rates. On the other hand the cubic samples were analysed at 3-piont bending equipment or characterisation of the critical stress intensity factor of the scale composites.

DEFORMATION BEHAVIOUR OF OXIDE SCALE

Experimental Simulation

For numerical simulation it is essential to provide the temperature dependent and oxide-specific material parameters. Previously, the determination method for ferrous oxide mechanical properties was introduced. This method allows the calculation of flow curves with measured force-way-relationships up to a deformation degree $\phi=0.3$ and a strain rate of maximum 10^{s-1} (Fig. 6).

The deformation degree for fracture is dependent on oxide phase as shown in Fig. 6. Wustite achieve a maximum deformation degree of $\phi=0.27$ in dependence of temperature and strain rate. Therefore the biggest oxide phase can compensate the highest deformation. By comparison, hematite and magnetite samples break at $\phi=0.1$. Hematite is the hardest phase and cracks earlier but can endure higher flow stresses. All identified flow stresses are 50 MPa higher as pointed out from the literature. The reason is contributed to the different experimental simulation (tensile test vs. compression test) and the initial material for the tests. In the previous literature, the influence of substrate steel sample must be considered, because the steel can not transfer completely in an oxide phase.

The determination of the fraction toughness was done in accordance to DIN ISO EN 15732 for ceramics. This is possible as the characteristics of the different sintered oxide sample are similar to ceramics with regard to mechanical and thermophysical properties. The tests were realized at room temperature and under normal atmosphere. During the short contact between roller and material decrease the material temperature very strong less than 300 °C. Fig. 7 shows the fracture analysis of the 3 x 4 mm² sample which was prepare with three indents of Vickers cone as crack initiation.

The calculated stress intensity factor did not show so high differences between oxide phases, as in the case of all other determined properties. With increasing the oxygen content the stress intensity factor increase too. However, this reflected and confirmed the same correlation of the other oxide-specific characteristics.

As a result of the Vickers indentations for the fracture toughness is it possible to determine the hardness through the measurable diagonals of the indentation area (Fig. 8). Therefore the calculated hardness of hematite at room temperature is over 900 HV10 and for magnetite between 700 until 600 HV10, which is higher as the basic material and the value range is approximately the same as ceramics. Wustite has the lowest hardness of 400 HV10.
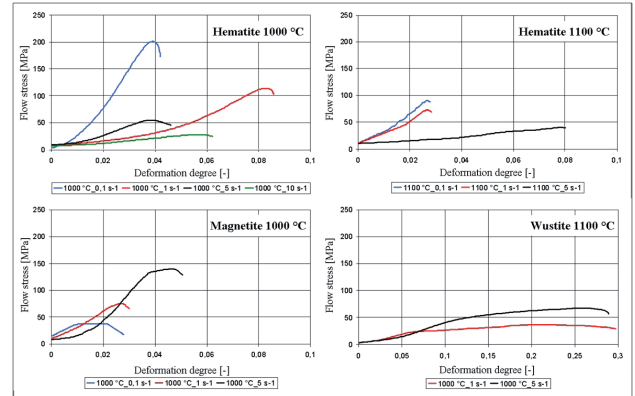

Fig. 6 - Flow curves (upper: influence of testing temperature, bottom: effect of iron oxid)

Fig. 6 - Curve di flusso (in alto: influenza della temperatura di prova, in basso: effetto del tipo di ossido di ferro)
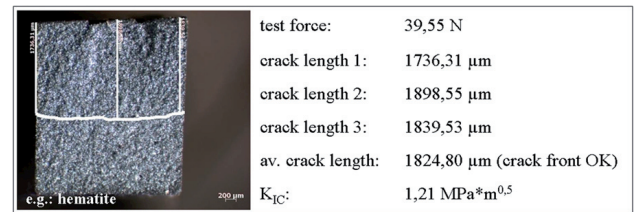

Fig. 7 - Fracture surface of hematite sample visualized by light microscopy

Fig. 7 - Superficie di frattura del campione di ematite osservato al microscopio ottico

	Fe$_2$O$_3$	Fe$_3$O$_4$	FeO
stress intensity factor mode I [MPa*√m]	1,2	1,6	2,2

Tab. 1 - Calculated average stress intensity factor

Tab. 1 – Fattore medio della intensità di tensione calcolato

Rolling Process

In order to investigate the oxide scale behaviour during hot rolling process a heated sample with an input scale layer thickness of 300 μm was deformed. A thermographic camera is positioned at the roll gap exit to obtain immediately information about the surface condition after the roller pair is moved apart. Scale has the peculiarities (oxide compound and therefore similar to a ceramic or as insulators), another heat radiation behaviour, than the base material. Cracks are coloured differently. Because the emissivity of the scale is greater than that of the steel and the focus of the thermography was directed onto the steel, the measurement result of the scale is at lower temperatures than that of the base material. Therefore, the steel or the cracks in the scale appear "warmer" in the documentation. In a comparison with the real rolling sample,
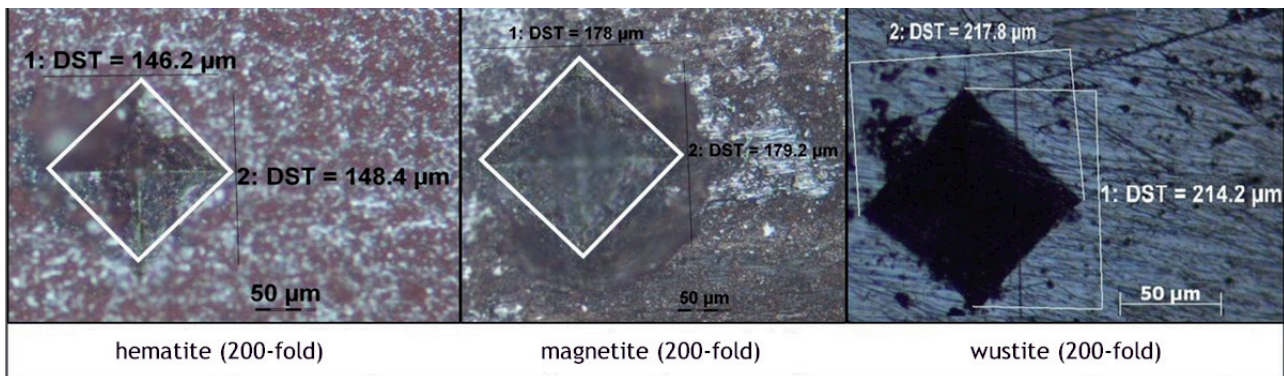
Fig. 8 - Dimension of Vickers-indents
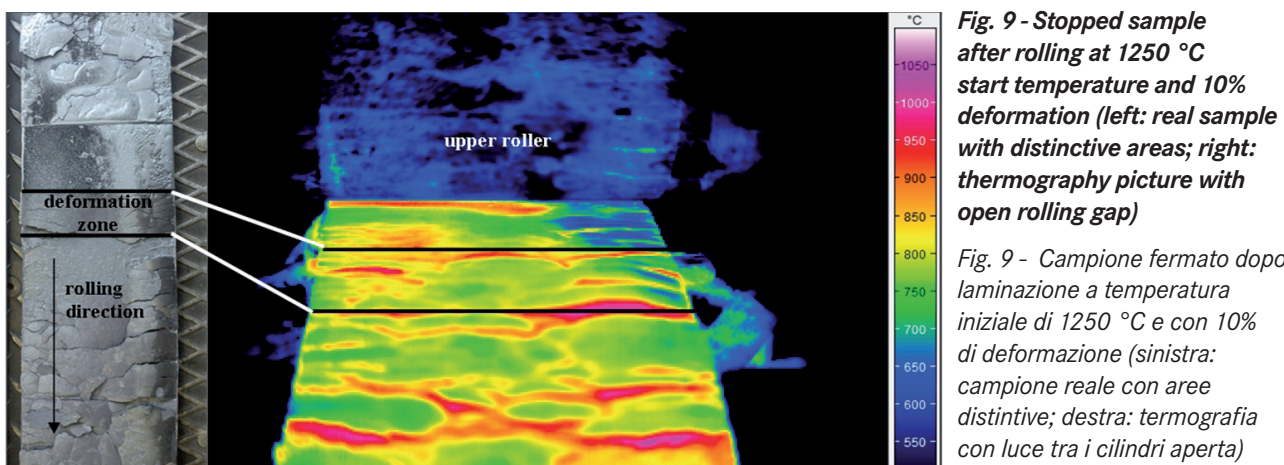*Fig. 8 - Dimensione delle impronte Vickers*

Fig. 9 - Stopped sample after rolling at 1250 °C start temperature and 10% deformation (left: real sample with distinctive areas; right: thermography picture with open rolling gap)

Fig. 9 - Campione fermato dopo laminazione a temperatura iniziale di 1250 °C e con 10% di deformazione (sinistra: campione reale con aree distintive; destra: termografia con luce tra i cilindri aperta)
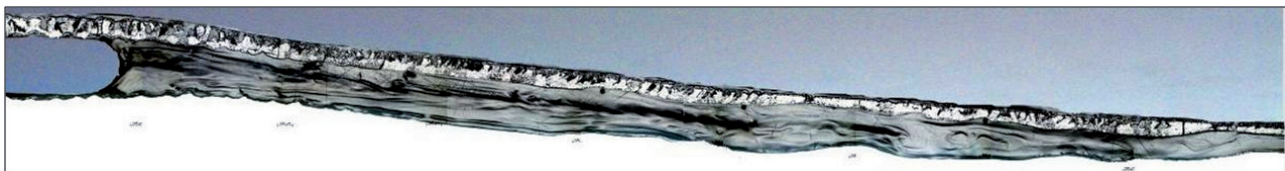

Fig. 10 - Scale layer as longitudinal cut from the centre of the specimen at 1250 °C and a maximum of 10% deformation (magnification: 25x)

Fig. 10 - Strato di scaglia tagliato longitudinalmente dal centro del provino a 1250 °C e con deformazione massima del 10% (ingrandimento: 25x)

these distinctive areas can be found again (see Fig. 9).
In Fig. 9, the distinctive sections are clearly visible within the deformation zone.
Using the continuous plotting of the forces and torques during this rolling experiments the flow stress could be roughly determined by the approach of force and torque demand by reverse calculation and compared with the experimental simulation results. This allowed additional the proof that the total scale layer can withstand up to a certain degree of deformation, and only then begins to break down.
For a detailed analysis of the rolling tests with only one pass, regarding the forming of scale in the complete roll gap, the specific characteristics of the oxide layer in the strip centre were determined by light microscopy. This was necessary because the scaling behaviour could be only partially taken into account under real process conditions on the simulation devices or just extrapolated.
If the temperature depend fracture deformation degree is exceeded the whole scale layer cracks partially or fully. The cracks start at the outside layer and growth along the thickness. Near the entry of roll gap are the lower deformations as well at the exit, why fine cracks are at the beginning of the rolling process which widen with rising of deformation. So it is logical that the crack tendency increase along the rolling gap in direction of exit. At the end of rolling gap or with greater whole deformation result more scale fragments which are pushed in the metal matrix.
For a whole deformation of 10 % with regard to the thickness of the raw material and a furnace temperature of 1250 °C are no cracks in the scale layer in the middle of the strip (Fig. 10).
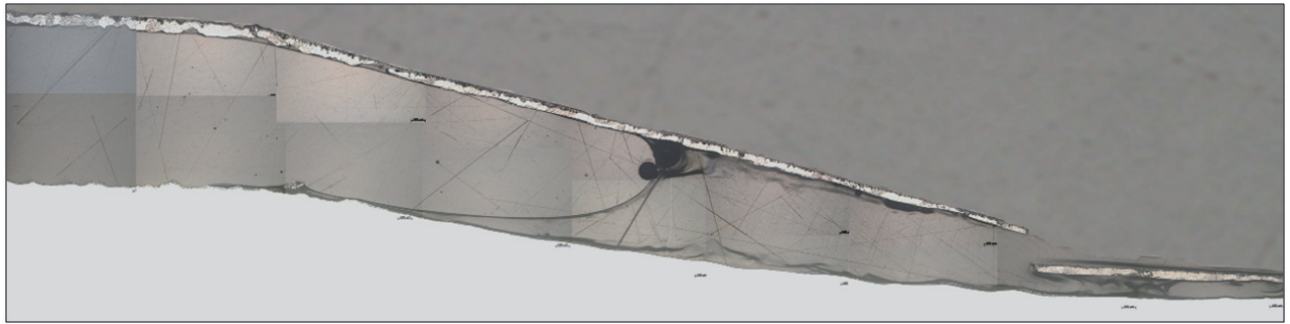The first crack in the oxide layer can identified after the rol-

Fig. 11 - Scale layer as longitudinal cut from the centre of the specimen at 1250 °C and a maximum of 20% deformation (magnification: 25x)

Fig. 11 - Strato di scaglia tagliato longitudinalmente dal centro del provino a 1250 °C e con deformazione massima del 20% (ingrandimento: 25x)
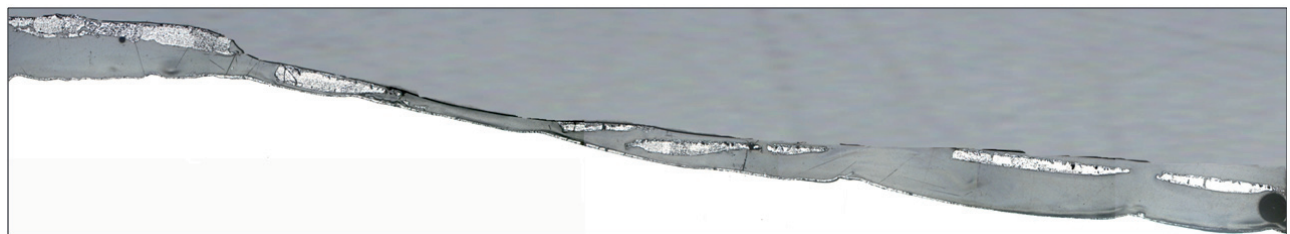

Fig. 12 - Scale layer as longitudinal cut from the centre of the specimen at 1250 °C and a maximum of 50% deformation (magnification: 25x)

Fig. 12 - Strato di scaglia tagliato longitudinalmente dal centro del provino a 1250 °C e con deformazione massima del 50% (ingrandimento: 25x)

ling gap exit but can be assumed that the crack develops as result of the quenching process (thermal stresses) or the crack must be reduced to the roughness of the basic material. This rolling experiment shows the change of the porosity inside the oxide scale as result of the compression process. The starting scale was very porous and after the deformation is the layer compacted and the pores were eliminated. The deformation degree of scale and steel is similar. So it can reflect that the maximum flow stress is not reached which can verified with the measured rolling force refer to the geometry conditions in the rolling gap.

The increased whole deformation up to 20 % and 1250 °C increases the risk for crack developments in direction to rolling gap exit. But the first crack was located after the exit. This is the proof that the fracture deformation degree is not achieved during rolling. But the reason for the crack can be an overlay of the different elongation behaviour of steel and scale as well the thermal problem during quenching. In total the scale compensate the deformation plastic which means that the pores can reduced without to damage the scale layer (Fig. 11).

The adjusted whole high reduction of 20 % corresponds to a deformation degree of $\phi \approx 0.1$ for the scale layer at one side of the sample, because the symmetric must consider. This proves that the measured fracture deformation degree at this temperature range is not achieved for all three scale components. At the scale surface were founded small cracks which are not through the whole scale layer. Under consideration of rolling force and the geometry ratio the maximum calculated flow stress was 40 MPa during rolling, which is not the maximum of the flow curves (kf=50 MPa). The rolling speed was 0,3 m/s and it is comparable with a strain rate of approximately 3 s-1. As results of the inhomogenous temperature profile and the overlayed stresses (thermal, tensile and compression stresses) over the strip cross section the cracks are formed near the strip edges.

An additionally increasing of deformation degree (Fig. 12) or the decreasing temperature will eventually caused the crack intensity of the scale layer. So the fragment amounts increase along the rolling gap.

CONCLUSION

This article presented a method for the identification of single scale properties independent of the raw material. So it is possible to describe these material parameters with suitable models. Additionally, the determined scale properties could validated with strip rolling experiments under industrial relations.

The experimental results show that the scale keeps resisting to deformation without cracks dependent on temperature. This follows from the fact that pores / defects can

eliminate as well the flow stresses of each oxide layer.

As consequence of the detailed characterization of the inhomogeneous multilayer scale a better characterisation of the scale behaviour within a hot rolling process can be regarded to the physical and material properties. These analysed material parameters are required as input values in the numerical simulation systems. It allows a more practical simulation of the hot strip rolling process, which is coupled with a temperature calculation. The oxidation process influences the surface quality of metallic materials and therefore its changes the grip conditions and the material flow in the forming tool. With the results is it possible to design the processes to achieve the best material surface (without any cracks in the whole oxide layer) along the hot but also for the following cold deformation process chain. In addition of the better surface quality of the semi-finished product, the wear of the dies can be estimated and controlled.

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Sviluppo di scaglia durante laminazione a caldo di nastri di acciaio

Parole chiave: Trattamenti termici - Laminazione - Deformazioni plastiche

Il lavoro presenta un nuovo metodo per descrivere e prevedere lo sviluppo di scaglia durante i processi di formatura del metallo, in particolare nei processi di laminazione a caldo di nastri. Questo metodo è necessario perché la scaglia si sviluppa durante tutti i processi di deformazione a caldo superiori a 600° C e influisce sulla perdita di massa così come sulla qualità superficiale del prodotto semilavorato. I componenti principali dell' ossido nella scaglia sull' acciaio sono wüstite, magnetite, e ematite in diverse proporzioni.

Una sfida per la corretta caratterizzazione dello strato totale di scaglia è basata sulla forte disomogeneità riguardo alle proprietà meccaniche di ciascun componente della scaglia. A causa di queste differenze, anche il comportamento a deformazione dei singoli strati di ossido è diverso. Come risultato di forti sollecitazioni di deformazione, la scaglia di ossido si cricca, tuttavia bassi gradi di deformazione possono essere compensati. A causa della elevata durezza degli ossidi, i frammenti vengono conficcati nell'acciaio ed influiscono sulle condizioni di grippaggio nella laminazione e sul fluire del materiale. Per questo motivo l'Institute of Metal Forming ha sviluppato un nuovo metodo per determinare le proprietà, dipendenti dalla temperatura, degli strati di scaglia, che sono rilevanti per l'impostazione dei processi di formatura del metallo. Il metodo si basa sull'uso di polvere pura di ogni componente della scaglia per la realizzazione di provini con diverse geometrie (cilindrici e cubici) mediante passaggi di compressione e di trattamento termico.