

# INFLUENCE OF MICRO-SEGREGATION IN PB-S-ALLOYED FREE MACHINING STEELS ON THE SURFACE QUALITY OF THE ROLLED WIRE-ROD

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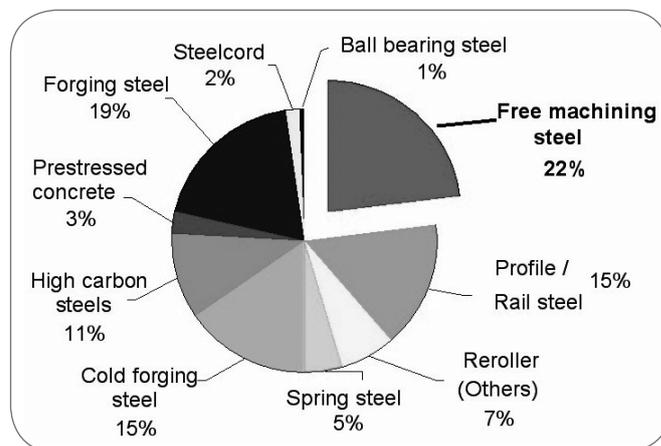
*Free machining steel billets were manufactured at the continuous casting machine. The manufactured billets did not exhibit any kind of surface defects but surface cracks and slivers appeared when the billets were rolled into wires and rods at the wire-rod mill. The defects on rolled wire-rod have been detected by a hot eddy current system. Further investigations in these defects with the help of microprobe analysis system and scanning electron microscope equipped with image analysis system revealed micro-segregation of the elements manganese, sulphur and phosphorous in the interdendritic zones.*

*Keywords: Pb-S Free machining steel, micro-segregation, surface cracks and slivers, interdendritic zones*

## INTRODUCTION

Mittal Steel Ruhrort GmbH has been an important steel making site in Germany for over 150 years. Today, it specializes in high-quality steel grades, which it supplies as continuous cast blooms or rolled billets. The company manufactures its products from 1.3 million tpa of hot metal following the process route of hot metal desulphurisation, LD Converter, secondary metallurgy comprising of RH and ladle degassers and two continuous casting machines with formats of 385 x 265 mm<sup>2</sup> and 130 mm<sup>2</sup>. An important constituent in the production plan of Mittal Steel is the free machining steel. Free machining steels alloyed with elements such as lead, bismuth, tellurium and selenium are produced both as blooms (385 x 265 mm<sup>2</sup>) and as billets (130 mm<sup>2</sup>). The yearly production of alloyed free machining steel is approx. 22% (Fig. 1) of the total production and is one of the core businesses of Mittal Steel Ruhrort GmbH. Free machining steel alloyed with lead and selenium are largely produced on the billet casting machine.

These products are rolled into wires and rods at Mittal Steel's Hochfeld subsidiary. The wire rod mill in Duisburg-Hochfeld is a three-strand rod mill that produces steel wire-rod in the size range of 5.5 to 21 mm from 2-ton billets. The



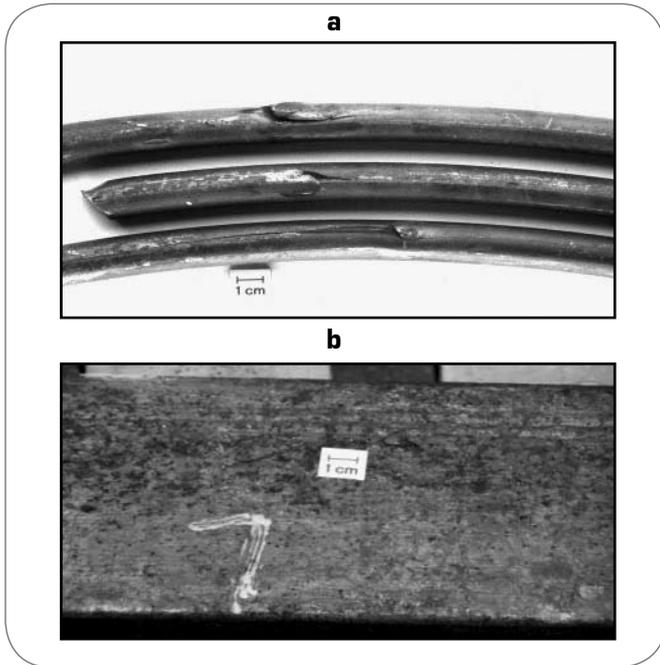
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Fig. 1

**Distribution of steel production at Mittal Steel Ruhrort GmbH, Duisburg in the year 2005.**

*Distribuzione della produzione di acciaio presso la Mittal Steel Ruhrort GmbH, Duisburg nel 2005.*

three-strand rod mill is a continuously processing rolling mill and consists of a pusher type furnace, roughing blocks and finishing blocks. Behind the finishing block, is an eddy current testing system [1] integrated into the three-strand rod mill for the continuous inspection of the surface of the hot rod. This is followed by Stellmor cooling, coiling and

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▲  
Fig. 2

(a) Slivers and cracks on the surface of the wire-rod. (b) Billet surface of free machining steel billet. (a) Scaglie e cricche sulla superficie della vergella. (b) Superficie della billetta di acciaio da lavorazione ad alta velocità.

conveyer station, storage and finally, shipment to the end customer.

The wire-rod mill at Hochfeld has a definite input dimensional size (a maximum of 130x130 mm<sup>2</sup>). As a result of this fixed size, the blooms manufactured at the bloom casting machine are rolled into billets of the above mentioned dimensions at Ruhrort and then transported to the wire-rod mill. Since the direct cast billets (130 mm<sup>2</sup>) satisfy the required dimensions, they are transported directly without further processing.

## DESCRIPTION OF THE PHENOMENA

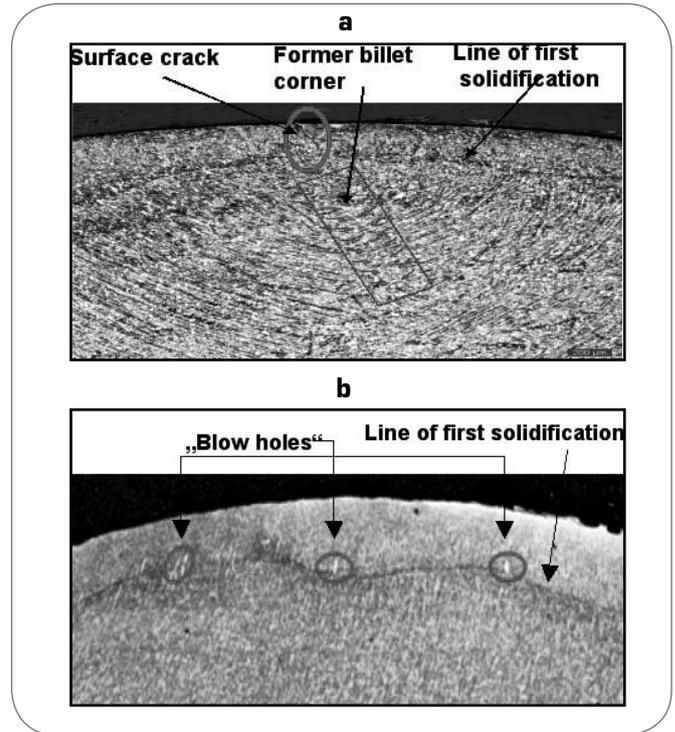
Surface cracks and slivers (Fig. 2 (a)) appeared on the wire-rod when free machining steel billets were rolled into wires and rods at the wire-rod mill. Investigations made on the billet corner showed neither transverse nor longitudinal corner cracks. Also, presence of surface cracks and deep oscillation marks was not observed (Fig. 2 (b)).

Trial No.	Steel grade	Parameters of Casting			
		[O]	Casting velocity	Mould powder	Cooling rate
1	LFC steel	70-80 ppm	3.2 m/min	Acidic	1600 l/min
2	FC steel	40-50 ppm	3.2 m/min	Acidic	1600 l/min

The acidic mould powder employed had a basicity of 0.69.

▲  
Tab. 1

Description of the first set of trials carried out at the billet-casting machine. Descrizione della prima serie di prove effettuate presso la macchina di colata.



▲  
Fig. 3

(a) A surface crack associated with a former billet corner and the line of first solidification. (b) Blowholes in and around the line of first solidification. (a) Una cricca di superficie associata al preesistente angolo della billetta e al linea di prima solidificazione. (b) Soffiature all'interno e intorno la linea di prima solidificazione.

Simultaneous investigations using an appropriate etching method made in the regions of slivers and cracks that appeared on the wire rod showed that, in most cases, the points of initiation of slivers and surface cracks were the former corners of the billet (Fig. 3 (a)). Further investigations showed that there was a concentration of the so-called blow holes around the line of first solidification. Fig. 3 (b) shows the so-called blow holes in and around the line of first solidification.

## EXPERIMENTAL INVESTIGATIONS

On the basis of the results of the initial investigations, the former billet corner and the line of first solidification associated with blowholes became the heart of the investigations performed at Mittal Steel Ruhrort GmbH. Two sets of trials had been planned and carried out in order to find a solution to the above mentioned phenomena. The following is a description of the first set of trials carried out at the continuous casting machine. Two different grades of free machining steel, one with lead (LFC steel) and the other without (FC steel), were cast at the billet casting machine and then skew-rolled at the bloom rolling mill



▲  
Fig. 4

**Descrizione della prima serie di prove effettuate presso la macchina di colata.**

*Cricche agli angoli sulla billetta in rotoli dopo la prima serie di prove.*

from 130 mm<sup>2</sup> to 100 mm<sup>2</sup>. The reason for using the process of skew rolling was to create a similar stress situation on the corners of the billet as in the wire-rod mill. The casting parameters that were employed during the first set of trials have been given in Tab. 1.

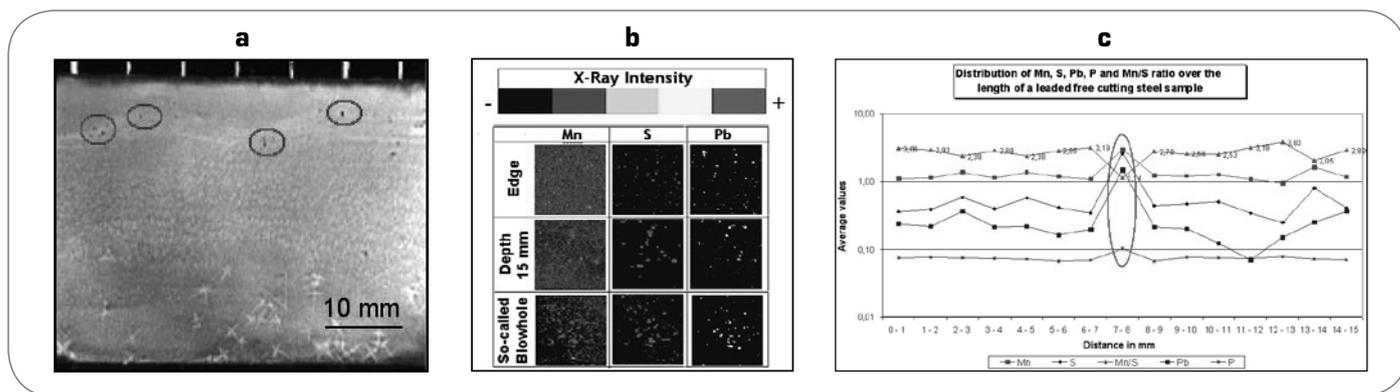
After rolling at the bloom rolling mill, cracks appeared on the corners of the free machining steel billets. In the other case, i.e., unleaded free machining steel, no cracks were ob-

served. Fig. 4 shows the corner cracks that appeared after the first set of trials. This result was identical to the result of rolling of leaded free machining steel at the wire rod mill. Samples (with and without cracks) had been collected and the microstructure was investigated with the help of micro probe analysis system and a scanning electron microscope equipped with an image analysis system. Also, the primary and secondary structure investigations with the help of appropriate etching methods were carried out on these samples. The results of these investigations will be mentioned under results and discussion.

On the basis of the results of the first set of trials, a second set of trials was carried out at the billet continuous casting machine. Samples had been collected and subjected to image- and microprobe analyses to determine the extent of micro-segregation of manganese, lead and phosphorous. The casting parameters employed in the second set of trials have been tabulated in Tab. 2.

## RESULTS AND DISCUSSION

As mentioned in the above section, samples from the first set of trials were taken from the rolled billets and were sent for microprobe analysis measurements. Further, samples from



▲  
Fig. 5

**(a) so-called blow holes (red circles) near the solidification line; (b) Microprobe mapping of elements Mn, S and Pb; (c) Concentration profiles of the elements Mn, S, Pb and P.**

*5 (a) cosiddette soffiature (cerchiate di rosso) in prossimità della linea di solidificazione (b) mappatura alla microsonda degli elementi Mn, S e Pb; (c) Profili delle concentrazioni di Mn, S e Pb.*

the second set of trials had been investigated with the help of scanning electron microscope to determine the extent of segregation of Mn, S and P.

The results of the first set of trials showed that the so-called blow holes that are located in and around the line of first solidification and the line of first solidification itself (which sometimes reaches the surface) could be responsible for the weakening of the microstructure at that particular point. This weakening of microstructure could be in turn respon-

sible for the origination of cracks during rolling. It appears that the billet corners are more sensitive to the applied forces in comparison to that of the sides. The above mentioned points will be discussed in detail below.

### So-called blow holes in and around the solidification line

In Fig. 5(a), the so-called blow holes in and around the solidification line have been illustrated. It can be observed that the solidification line is located at a distance of 4 – 8 mm below the surface and the blow holes lie

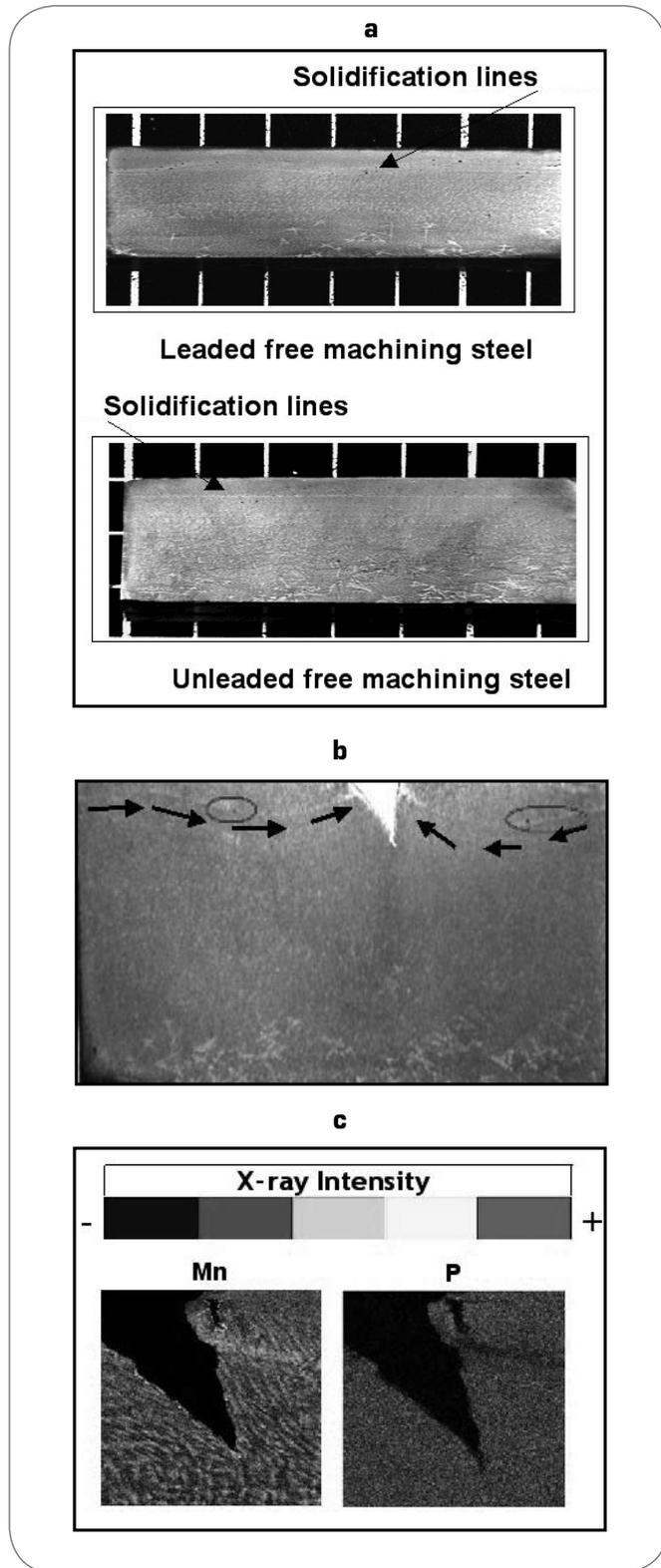
Trial No.	Steel grade	Parameters of Casting			
		[O]	Casting velocity	Mould powder	Cooling rate
1	LFC steel	40-50 ppm	3.2 m/min	Basic	1600 l/min
2	LFC steel	40-50 ppm	2.8 m/min	Basic	1600 l/min
3	LFC steel	40-50 ppm	3.2 m/min	Basic	1400 l/min

*The basic mould powder employed had a basicity of 1.08.*

▲  
Tab. 2

**Casting parameters of the second set of trials.**

*Parametri di colata della seconda serie di prove.*



▲  
Fig. 6

**(a) Comparison between leaded and unleaded free machining steel (b) Solidification line reaches the surface (c) Microprobe mapping of the elements manganese & phosphorous.**

*(a) Confronto fra acciai da lavorazione ad alta velocità al piombo e senza piombo (b) linea di solidificazione che raggiunge la superficie (c) mappatura alla microsonda degli elementi manganese e fosforo.*

along the solidification line. The following is an explanation for Fig. 5 (b). A corner sample, with an approximate length of 20 mm has been investigated with the help of microprobe analyzer. This length was chosen so that the so-called blow holes lie within the sample. The microprobe mappings of the elements manganese, sulfur and lead have been illustrated in this figure. It can be observed that the position of manganese and sulfur overlap and the element lead is oriented at the edges of MnS inclusions. In the areas at the edge, the sulfides are fine in size and more in number. This can be seen in the first row of the Fig. 5 (b). An enrichment of all the three elements has been observed in a blowhole. It can also be observed that the MnS inclusions are coarser in size and less in number inside the blowhole. These great differences of the above mentioned elements probably relates to a variation of heat withdrawal in the mould. Interesting to observe is that the size of the MnS inclusions inside the blowhole is comparable to the size of the inclusions present at a distance of 15 mm from the surface although the blow holes are located at just 4 or 5 mm below the surface. The above phenomenon could probably be related to a local perturbation of heat withdrawal.

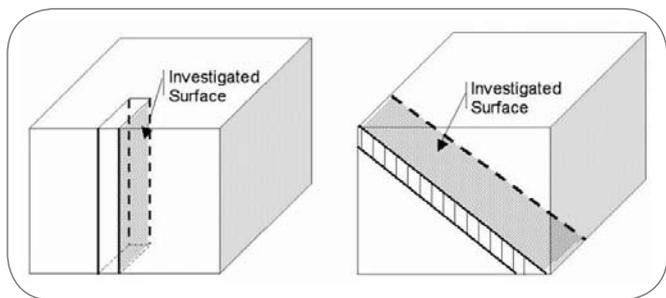
In Fig. 5 (c), concentration profiles of the elements manganese, sulfur, phosphorous and lead, across the sample have been illustrated. The region marked in red shows the concentration of the elements in a so-called blow hole. It can be observed that there exists an enrichment of the elements inside a blow hole. It is known that the interdendritic segregation behavior is influenced by the local solidification time [2]. A local perturbation of heat withdrawal in a billet corner leads to a difference (sometimes increase) in interdendritic spacing and therefore the elements in front of the solidification front i.e., the elements between the dendrite arms may have more time to agglomerate before this region solidifies. A supersaturation of sulfur leads to the precipitation of sulfur. Manganese dissolved in the matrix reacts with sulfur to form MnS. This reaction occurs at very high temperatures (around 1600°C). The so combined MnS inclusions probably have the possibility to accept and accommodate the liquid drop shaped lead at their edges. This possibility of association of lead with MnS has been shown in Fig. 5 and Fig. 9.

### Penetration of the line of first solidification to the surface

A second possibility for the origination of surface cracks and slivers is when the line of first solidification reaches the surface of the billet. A comparison between the solidification lines of the leaded and unleaded free machining steel samples has been shown in Fig. 6 (a). In the case of leaded free machining steel, the line of first solidification is wavy in nature and therefore sometimes reaches the surface (Fig. 6 (b)). In the other case, the line of first solidification of the unleaded free machining steel sample is relatively horizontal (Fig. 6 (a)). Relatively coarse MnS inclusions have been found at the point of crack initiation. Just a few micrometers away, in the areas near the cracks, fine sulfides have been found. It has also been observed that there is a sudden change in microstructure at the line of first solidification which can be associated to a perturbation in local heat withdrawal. Also, manganese enrichment and phosphorous depletion have been found in the area of the line of first solidification (Fig. 6 (c)).

### Corner and sides of the billets

A perturbation in the local heat withdrawal could also be caused by a tangential deviation of the billet inside the



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Fig. 7

**Schematic illustration of the longitudinal & transverse cross-sections.**

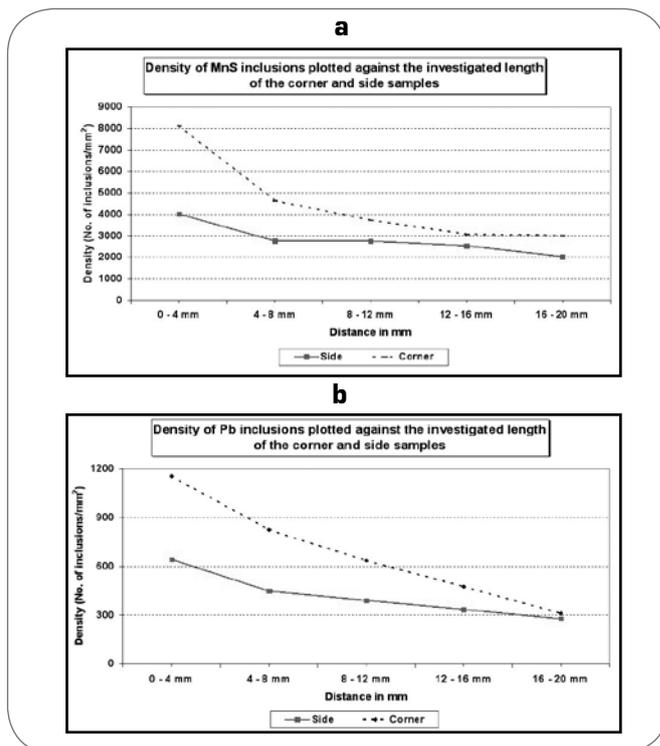
*Illustrazione schematica delle sezioni longitudinale e trasversale.*

mould which results in the deviation in the billet's final dimensions. A tangential deviation could be due to the distortion of the mould. The extent of distortion of the mould could be in turn influenced by the mould age. To eliminate the possibility of such an influence, the rhomboidity of the billet was verified. The results of the investigation showed no negative influence of the mould age on the billets cross section.

MnS- and Pb distribution for the side and corner samples (longitudinal and transverse cross sections respectively) of the second set of trials (Tab. 2) have been determined by means of SEM / EDX. In Fig. 7, the samples and the investigated sections have been illustrated.

The results of these investigations have been shown in Fig. 8 (a) and (b).

The dotted line represents the MnS density (number of MnS inclusions/mm<sup>2</sup>) of the corner sample and the solid line represents the MnS density (number of MnS inclusions/mm<sup>2</sup>) of the side sample. It can be observed that the density of MnS inclusions at the corner is higher than that of the side and also the density decreases with increase in distance from the surface (side sample) and the edge (corner sample). It can also be observed that the slope of the density line of lead is identical to that of MnS. From this result, it can be deduced that lead solidifies in combination with MnS. This



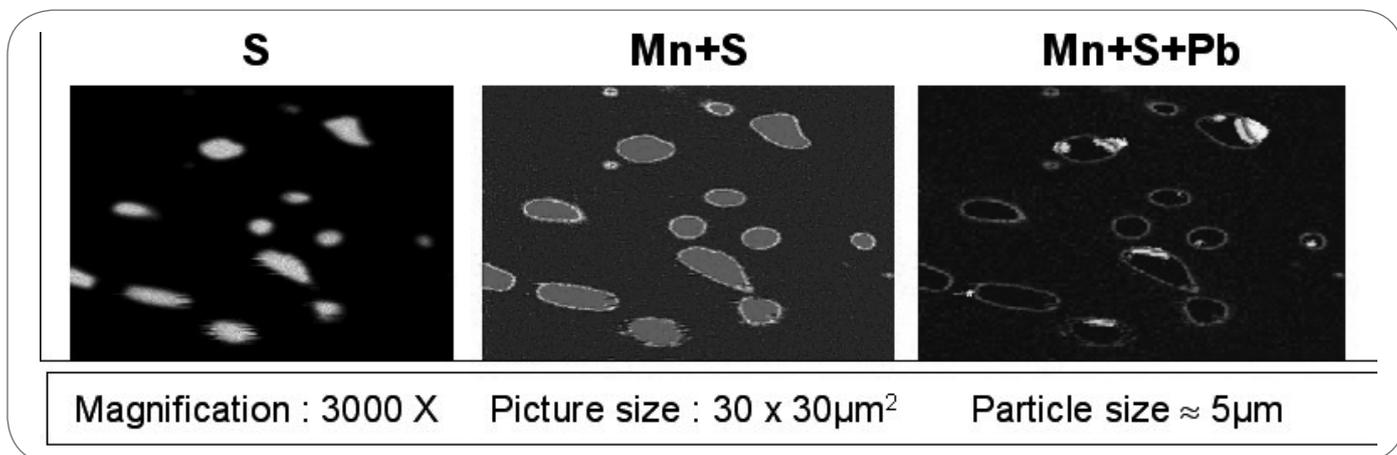
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Fig. 8

**(a) Investigation results of the distribution of MnS inclusions for corner and side of the billet. (b) Investigation results of the distribution of Pb particles for corner and side of the billet.**

*(a) Risultati dell'analisi della distribuzione delle inclusioni di MnS per angoli e lati della billetta. (b) Risultati dell'analisi della distribuzione delle particelle di Pb per angoli e lati della billetta.*

assumption was confirmed with the help of the micro probe image in Fig. 9.

Further, it is known that the precipitation behavior of particles and the secondary dendrite arm spacing are influenced by the heat withdrawal [4]. The precipitation behavior of MnS and Pb has been already illustrated in Fig. 8 (a) and (b).



▲  
Fig. 9

**Microprobe mapping of Mn, S and Pb.**

*Mappatura alla microsonda di Mn, S e Pb.*

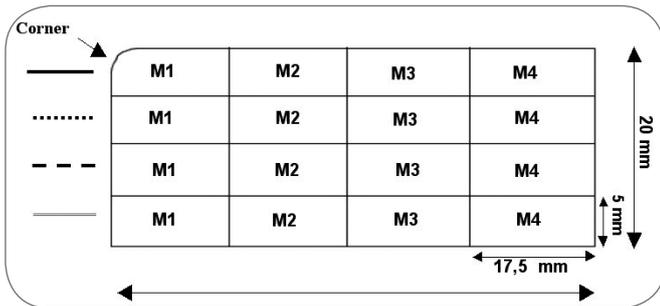


Fig. 10

**Schematic illustration of the sample for SDAS-measurements.**

*Illustrazione schematica del campione per le misurazioni SDAS.*

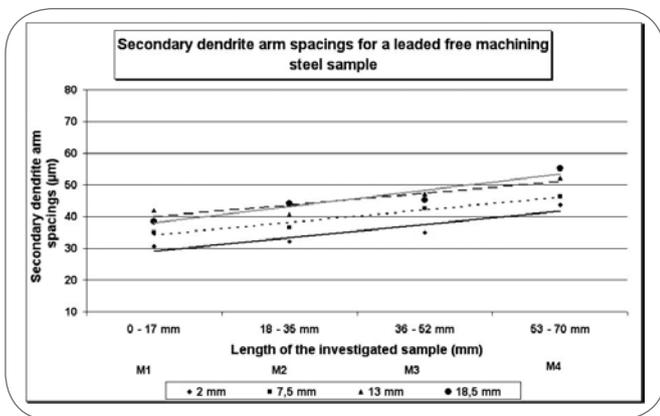


Fig. 11

**Illustration of the measured SDAS.**

*Illustrazione del SDAS misurato.*

A higher heat withdrawal at the corners than at the sides has been confirmed by measurements of the dendrite arm spacing [5],[6]. With the help of light microscopy, secondary dendrite arm spacing (SDAS) were measured. The procedure of the measurement has been illustrated in Fig. 10. The chosen patterns of the lines in Fig. 10 are identical to those of the lines in Fig. 11. The measured trend of the SDAS showed that the heat withdrawal varied with increase in thickness of the solidified strand shell. Less heat has been transported per unit time. The measured SDAS reconfirmed that a higher heat withdrawal was experienced at the corners.

To verify whether a relation between the measured SDAS and MnS density for different heat withdrawals exists, two samples, which were cast with different casting velocities, had been investigated. The samples were from the trial number 1 and 2 in Tab. 2. The cross sections of the samples were like those of the samples in Fig. 7. MnS densities, followed by SDAS were measured on these samples. The results have been shown in fig. 12. It can be observed that there exists a definite relationship between SDAS, MnS density and distance from the surface, all being a function of the heat withdrawal.

The measured SDAS trend is identical to the calculated SDAS using the mathematical model from Hamadou [7]. It is normally expected that the SDAS for the investigated sample cast with a casting velocity of 3,2 [m/min] would be lower in compari-

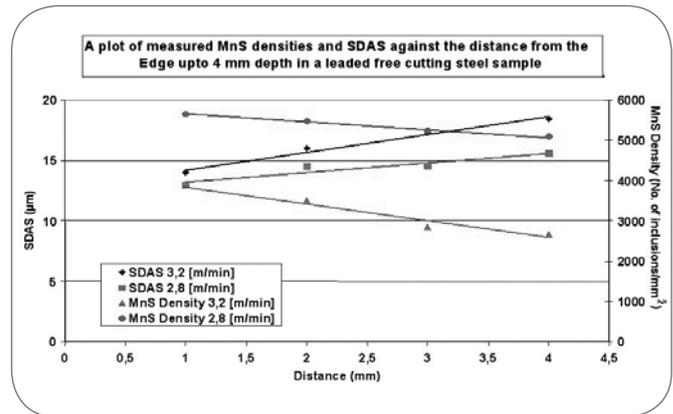


Fig. 12

**SDAS [ $\mu\text{m}$ ] and MnS densities [No. of inclusions/ $\text{mm}^2$ ] for samples taken from billets cast with different casting velocities.**

*Densità di SDAS [ $\mu\text{m}$ ] e MnS densities [No. di inclusioni/ $\text{mm}^2$ ] per campioni presi da billette colate a diverse velocità di colata.*

son to that of the sample cast with 2.8 [m/min]. In co-relation to the above, a higher MnS density is expected in the former case. The inverse has been observed in Fig. 12. A probable explanation to the above observed phenomena could be a movement of the strand (horizontal in this case) inside the mould perpendicular to the casting direction. This relative movement of the strand is independent of the casting velocity and thereby can lead to a variation in the local heat withdrawal as shown in Fig 13. The MnS densities of all the four corners of the same cross section have been shown in this figure.

It can be observed that the density of MnS inclusions have different levels in all the four investigated corners of the billet cross section, especially in the initial 4 mm (distance from the edge). It can be concluded that the heat withdrawal or the amount of heat transported had been different in the four corners. This could be a possible explanation for the unusual re-

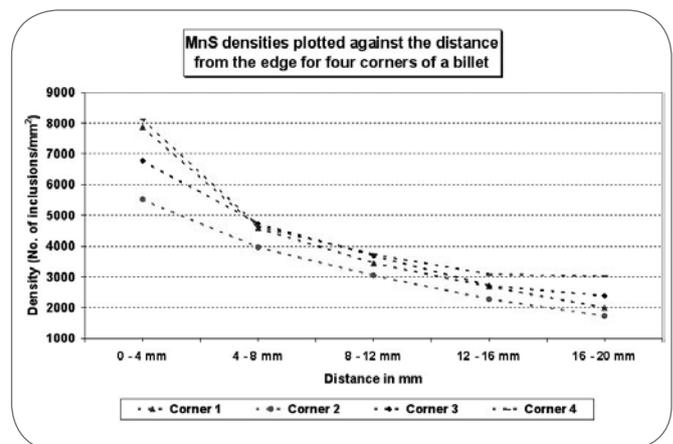


Fig. 13

**Density distribution in all the four corners of the same billet cross-sections.**

*Distribuzione della densità ai quattro angoli della stessa sezione trasversale della billetta.*

sults shown in Fig. 12. On the basis of these results and taking into consideration the slivers that arouse on the rolled wire-rod (associated with former corners of the billets), it must be determined whether these slivers could be intrinsically influenced. Further investigations have to be performed in order to determine this probable influence.

## SUMMARY

Two different sets of trials were carried out at the billet continuous casting machine to identify the defect causing phenomena. The first set of trials indicate that there are mainly three reasons for the weakening of the microstructure thereby leading to the origination of surface cracks and slivers on the wire-rod. They are: the presence of a blowhole, a condition where in the line of first solidification reaches the surface and a former corner of the billet. It seems that a billet corner is more sensitive to the applied forces than the side as transverse cracks were found after the rolling process at the bloom rolling mill. The results of the performed investigations showed that this phenomena could be caused by a local variation of heat withdrawal. It has also been shown that the lead particles preferred to be associated with MnS inclusions independent of the distribution of the MnS inclusions. A plausible explanation for phosphorous enrichment in the so-called "Blow hole" could not be yet found.

The results of the second set of trials showed that there exists a close relationship between the measured SDAS, measured MnS densities and the heat withdrawal in the mould. It could also be shown that the highest amount of heat withdrawal exists in the corners but temporary variation in the heat withdrawal inside the mould probably arouse due to the movement of the strand (horizontal in this case) inside the mould perpendicular to the casting direction. On the basis of the results and taking into consideration the surface defects and slivers on the rolled wire-rod (associated with former billet

corners), it must be determined whether these slivers could be intrinsically influenced. Further investigations have to be performed in order to determine this probable influence. It can be deduced from the performed investigations that the elements manganese, phosphorous and sulfur, which segregate on a microscopic scale, have an influence on the surface quality of the rolled wire rod.

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## ABSTRACT

### **INFLUENZA DELLA MICRO-SEGREGAZIONE NEGLI ACCIAI DA LAVORAZIONE AD ALTA VELOCITA' LEGATI AL PB SULLA QUALITÀ SUPERFICIALE DELLA VERGELLA**

**PAROLE CHIAVE:** acciaio - difetti - colata continua - qualità

Il presente lavoro riporta i risultati di uno studio condotto su billette in acciaio da lavorazione ad alta velocità prodotte mediante colata continua. Nelle billette prodotte non si è osservato alcun tipo di difetto superficiale. Tuttavia si sono verificate cricche superficiali e scaglie quando le billette sono state trasformate in fili e vergelle. I difetti sulla vergella in rotoli sono stati rilevati mediante sistema a correnti parassite a caldo (hot eddy current system). Ulteriori studi di questi difetti, con l'ausilio del sistema di analisi microsonda (microprobe analysis system) e di un microscopio elettronico a scansione dotato di sistema di analisi dell'immagine, hanno rivelato la micro-segregazione di elementi di manganese, zolfo e fosforo nelle zone interdendritiche.

Per identificare il fenomeno responsabile dei difetti sono state effettuate due serie di prove sulla billetta alla macchina di colata continua. La prima serie di prove ha indicato che vi sono tre motivi responsabili dell'indebolimento

della microstruttura che hanno portato di conseguenza all'insorgenza di cricche di superficie e scaglie sulla vergella. Questi sono: la presenza di una soffiatura, la condizione in cui la linea di prima solidificazione raggiunge la superficie e uno spigolo precedente della billetta. Sembrerebbe che un angolo di billetta sia più sensibile alle forze applicate rispetto al lato, nei casi in cui si sono riscontrate cricche trasversali dopo il processo di arrotolamento al laminatoio di blumi. I risultati delle indagini effettuate hanno dimostrato che questo fenomeno potrebbe essere causato da una variazione locale del ritiro termico. È stato anche dimostrato che le particelle di piombo preferiscono associarsi con le inclusioni di MnS indipendentemente dalla distribuzione delle inclusioni di MnS. Una spiegazione plausibile per l'arricchimento di fosforo nella "soffiatura" non è però ancora stato trovato. I risultati della seconda serie di studi hanno dimostrato che esiste una stretta relazione tra la spaziatura tra i bracci delle dendriti secondarie (SDAS), la densità delle inclusioni di MnS e il ritiro termico nello stampo. Si è inoltre potuto dimostrare che vi è una maggiore quantità di ritiro termico agli angoli.

Dalle indagini svolte si può dedurre che manganese, zolfo e fosforo segregati su scala microscopica, hanno un'influenza sulla qualità superficiale della vergella in rotoli.