CORRECT MANAGEMENT OF INCLUSIONAL DEFECT BASED ON A FAILURE ANALYSIS

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Abstract

The non-metallic inclusions are metallurgical defect always present within the steel bulk. Although high cleanliness levels can be reached by the most up dated techniques, their presence is not avoidable, so the users of steel have to learn a correct management of such defects. This problem has become even more significant with the use of resulphurised steels, in which the sulphur is added to cause the precipitation of an abundant MnS population, which makes the steel more workable by the cutting tools. However, the presence of a not proper inclusional population can forbid the implementation of certain technological processes to be applied, i.e. welding, hot working etc.. Moreover, the sulphide inclusions improve the workability, but they remain within the product also after the technological transformations and they can weaken the whole material, so their use has to be decided only after a proper choice of the technological route. Thus, some indications about this topic have been obtained on the basis of some significant cases on which a failure analysis has been applied.

Riassunto

Le inclusioni non metalliche sono un difetto sempre presente all'interno degli acciai, nonostante attraverso le tecniche siderurgiche più moderne si possano raggiungere elevati minimi livelli di presenza. Quindi gli utilizzatori di acciaio devono apprendere la corretta gestione di tali difetti. Tale problema è divenuto ancor più significativo a seguito dell'avvento degli acciai risolforati a lavorabilità migliorata, nei quali lo zolfo è stato aggiunto per provocare la precipitazione di una consistente popolazione di MnS, che rende questi acciai più lavorabili agli utensili da taglio. Comunque, la presenza di una popolazione inclusionale dalle caratteristiche non corrette in termini di dimensione media, distribuzione e forma, può impedire l'applicazione di particolari processi tecnologici, es: saldatura, deformazione plastica a caldo ecc.. Inoltre, i solfuri, se da una parte migliorano la lavorabilità, dall'altra rimangono all'interno del materiale anche a seguito del processo di trasformazione tecnologica e possono indebolire il materiale, quindi il loro utilizzo può essere effettuato solo in seguito alla scelta di una corretta procedura tecnologica. Alcune indicazioni circa questo aspetto sono state ottenute nel presente studio mediante l'analisi eseguita su alcuni casi di avaria particolarmente significativi.

INTRODUCTION

This paper aims to clarify the problems arising from inclusional compounds that with the other frequent defects, like segregational imperfections or defects in the metallic matrix of a steel product represent the most usual and not avoidable defect within the steel products.

The designers and the users think that steels are perfect, completely homogeneous and internally sound. The possibility to have an interruption of the matrix is normally excluded, although it certainly takes place.

The reality in industrial production is not able to avoid a certain number of imperfections and defects, more or less detrimental for the life of the produced piece.

Today it is possible to produce "clean steels", i.e. steel with an extremely low inclusion content. But the production cycles are very expensive and in consequence they are not affordable for generic uses.

In this paper, based also on experimental evidences founded on the failure analysis of some critical components, we will define the concepts of imperfection and defect and describe the origin of the most common defects as, in particular, the inclusions. At the end, we will present the most recent trends of the acceptance criteria based on the final application of the piece.

DEFINITION OF DEFECT

In the steel, the internal or surface discontinuities are geometrical irregularities or unwanted non-homogeneous areas.
These discontinuities are classified as imperfections when their dimensions are equal to, or minor of, the specified limit. They are classified as defects if their dimensions is above this limit.
It is true that some imperfections can originate failures even if they have a dimension defined as "acceptable" by the designer: for example they can act as notch [innesco] to create larger and more important defects. But it is also true that a lot of defects exist, with a much larger dimension than the limits defined as acceptable, and they do not create any consequence on the piece and can be easily tolerated.

Origin of the defects

The defects in the steel can originate in any step of the production cycle: melting, solidification, cooling, hot deformation, heat treatment, cold transformation, surface finishing.
Normally the defect forms during the melting and the solidification, while they are modified during the deformation processes. The defects caused by the first steps are internal or on the surface, but those generated towards the end of the production are mainly superficial; nevertheless important exceptions exist, especially related to the cold deformation phases.
The inclusions are probably the most known defect of the steel because they can be found in every material. They originate from different causes:

- external pieces of foreign bodies drawn into the steel by mechanical action or chemical erosion
- not controlled chemical reactions that should have happened and didn't or should not have happened and did.
- modification of the solubility of some elements in the liquid bath with the modification of the temperature.

One of the most important and frequent defect is represented by the non-metallic inclusions, that in some cases are also intentionally produced with particular chemical composition, solidification and cooling pattern. An interesting case is constituted by the resulphurised or micro-resulphurised steel, in which the non-metallic inclusions, generally regarded as an usual defect, are developed to improve the cutting operations of the steels. A good calibration of this task could be an important example to understand the management of the defect in order to improve the desired mechanical characteristics and to avoid the detrimental effects that the non-metallic inclusions can generate too.

EXPERIMENTAL PROCEDURE

Four examples of the role of the non-metallic inclusions have been studied starting from the failure analysis developed on four different types of mechanical components which have shown a not proper behaviour during the exercise and the technological operations, like cutting, plastic deformation and welding.
The failure cases have occurred repeatedly in a great number of samples produced and worked in different conditions:

1. rupture along the extrusion axis in a resulphurised steel (table I) component during the drilling along the extrusion axis itself. This type of piece has been heated at 1200°C, hot extruded and then annealed to remove the residual tension and the hardening effect before the drilling operation (fig.1);
2. rupture of two types of steel samples (table II) produced by hot forging operations performed between 1200-1260°C. After forging the samples have been austenitized between 860°C-880°C, oil quenched and tempered between 540°-560°C. The geometrical shape of these two types of failed samples are characterized by axial-symmetry;
3. defects on the welding borders showing significant and not acceptable irregularities. The two steels involved in the welding are X14CrMoS 17 and X5CrNi 18 10.

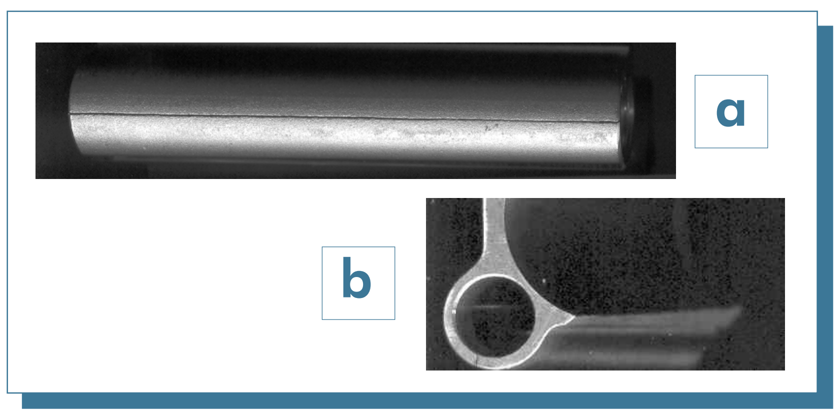

Fig. 1: Failed sample of the hot extruded steel with crack developed along the extrusion axis. Longitudinal view (a), transverse view with the hole produced by the drilling operation (X1)

Table I. Chemical composition of the hot extruded resulphurised steel (%wt)

%C	%Mn	%Si	%Cr	%Ni	%P	%S
0.31	0.68	0.5	12.32	0.21	0.019	0.06

TableII. Chemical composition of the two hot forged steels (%wt)

Steel	%C	%Mn	%Si	%P	%S	Ca (ppm)
A	**0.28**	**1.01**	**0.62**	**0.001**	**0.5**	**7**
B	**0.31**	**0.9**	**0.38**	**0.001**	**0.1**	**5**

On the hot extruded steel 10 tensile tests have been performed according to ASTM E8 in the not deformed state, in the deformed state, and in the annealed state after deformation. The specimens for these tests have been taken with their axis parallel to the extrusion one.
The study of the failed samples has implied to perform SEM analysis aided by EDS (Energy Dispersive Spectroscopy) for determining the chemical compositions of the non-metallic compounds involved in the fracture process. The optical microscope has been used to acquire the images studied by the image analyzer system to determine the ditribution, the shape and the volume fraction of the observed non-metallic compounds. On each of the hot extruded and forged samples eighty photos have been performed with a magnitude of X200 and the statistical determination of the specified parameters has been developed.
For a steel with the same composition of the forged ones five tests for the determination of K_{IC} have been performed according to ASTM E1820 with a classical notch compact specimen tested at the environment temperature (25°C).
The shape ratio has been determined by the approximation of the non-metallic inclusion with an ellipse whose major and minor axis have been determined. The anisotropy has been evaluated by the ratio between the major axis and the minor one.
In the forged sample a chemical etching has been operated to point out the stream-line of the plastic flow. The chemical etching has been performed by a chemical solution of 50%HCl and 50%H_2O in which the samples have been introduced for 17 minutes at 80°C.

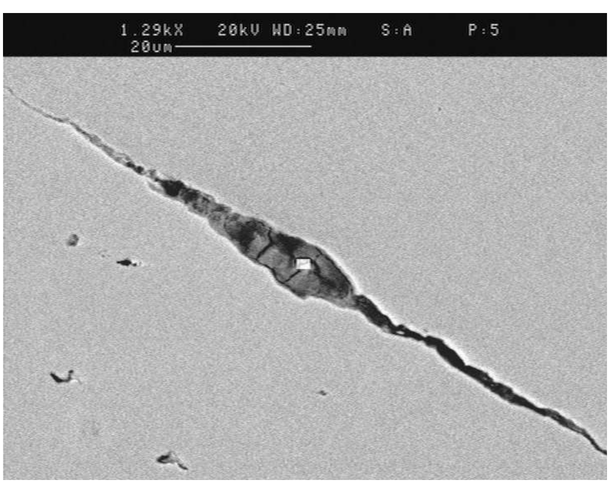

Fig. 2: Typical string of non-metallic inclusions in the extruded samples

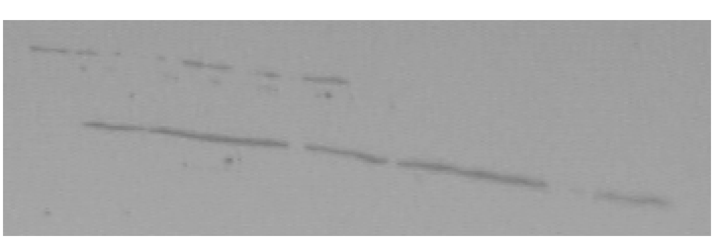

Fig. 3: Typical string of non-metallic inclusions in the extruded samples (X400)

RESULTS AND DISCUSSION

In all the failed samples the non-metallic inclusions have played an important role. The feature landscape of the inclusions of the plastically deformed samples is quite different.
In the hot extruded samples the inclusions have shown a significant anisotropy, because the shape factor defined just before has an average value of 11.2 and a standard deviation of 5.7, while in the not deformed annealed condition these parameters have the value of 2.4 and 0.95 respectively. The measured percentage volume fraction has an average value of 1.2% and a standard deviation of 0.3%. The population of the non-metallic inclusions is prevalently composed by MnS, nearly pure, actually only an average value of 4%wt (st.dev.1.7%wt) of Fe has been detected. The Ca is practically absent because it is present in an average quantity of 0.77%wt (st.dev.0.7%wt), so it can be concluded that there are not CaO and CaS within the non-metallic inclusions. The inclusions are not homogeneously distributed within the metal matrix, because they appear to be grouped like strips involving several aligned non-metallic inclusions (fig.2,fig.3) and they usually assume the form of aligned clusters grouping an average between 3 or 4 inclusions with some significant exceptions (fig.4). The average area of the inclusions is 83*$10^{-6}mm^2$ and shows an important standard deviation of 93*$10^{-6}mm^2$.

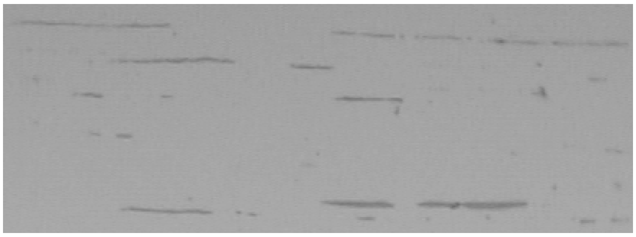

Fig. 4: Cluster with a number of non-metallic inclusions over the average value. Only a little number of these examples has been evidenced in the extruded samples (X400).

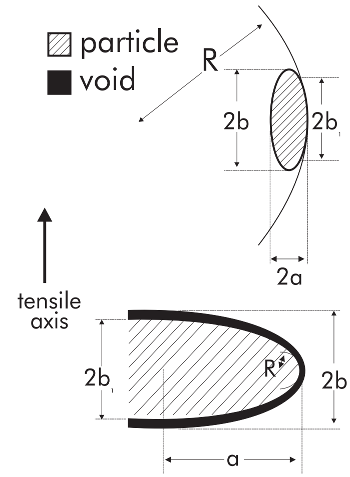

Fig.5 Layout of the decohesion between the non-metallic inclusion and the metal matrix under a stress in longitudinal and transverse direction with respect to the major axis of the inclusion itself (a, b_1 are the longitudinal axis and transverse one with respect to the tensile axis respectively; b is the width of the nucleated void longitudinal to the tensile axis, R is the frontal radius of curvature (b^2/a))

According to the classification proposed by Gladman [1] the inclusions can be considered in this state as holes, because they can be regarded as discontinuities in the metal matrix. The string shape of the clusters is probably due to the elongation of an original inclusion fractured during the plastic deformation of the metal matrix. As this behaviour has been observed, it can be concluded that the MnS observed belongs to the II types [2]. From several studies [3,4,5] on the mechanism of the ductile fracture the decohesion occurring at the interface between the non-metallic inclusions and the metal matrix (fig.5) has been found to be the phenomenon ruling the fracture process. The growth of such voids once nucleated is highly dependent on the strain state imposed. The rate of void elongation is increased with respect to the longitudinal strain.
The void width is not sensible to the strain in a plane tensile test, but it can decrease under biaxial compressions like that occurring during the hot extrusion. On the other hand, the ultimate failure can occur when the adjacent voids link by shear. The strain to fracture produced by void coalescence generated by inclusion-matrix decohesion depends on the inclusion parameters:

$$\varepsilon_t = \varepsilon_0 + 0.5\left[\frac{\frac{F^2}{f^2}+\frac{k}{r^2}}{1+\frac{k}{r^2}}\right]$$

where
ε_t true strain to fracture
ε_0 strain for the initial void decohesion
F constant related to critical volume fraction of voids for catastrophic failure
f initial volume fraction of the non-metallic inclusions
k stain void factor (about 2)
r average shape factor of the inclusions

The *F* constant for the hot extruded steel has been determined on the basis of the tensile strength performed on the specimen of the not deformed and deformed-annealed samples (table III, table IV) tested on the direction parallel to the deformation one.
On the basis of these results and under the hypothesis that the strain for the initial void decohesion has a not significant value so that it can be disregarded, the F value has been determined to vary between 0.0134-0.0135.
But if the measured values are inserted taking into account the transverse direction (in which the shape ratio assumes the value of 0.09) then the ductility resources of the material has nearly completely eliminated, because is reduced to 1.07% against the 19.6% proper of the longitudinal stress. The drilling tool during the perforation induces a strength just in the transverse direction with respect to the elongation of the non-metallic

Table III. Properties of the steel in the not deformed state

R(MPa)	$R_{p0.2}$(MPa)	%A
315	270	16

Table IV. Properties of the steel in the deformed-annealed state

R(MPa)	$R_{p0.2}$(MPa)	%A
320	290	11

TABLE V. AVERAGE CHEMICAL COMPOSITION OF THE NON-METALLIC INCLUSIONS OBSERVED IN THE FORGED SAMPLES (STEEL A AND STEEL B) (%MOLAR)

Steel	%Mn	%S	%Fe	% other elements
A	**37.7**	**35.6**	**15.2**	**11.5**
B	**43.2**	**44.1**	**7.8**	**5.2**

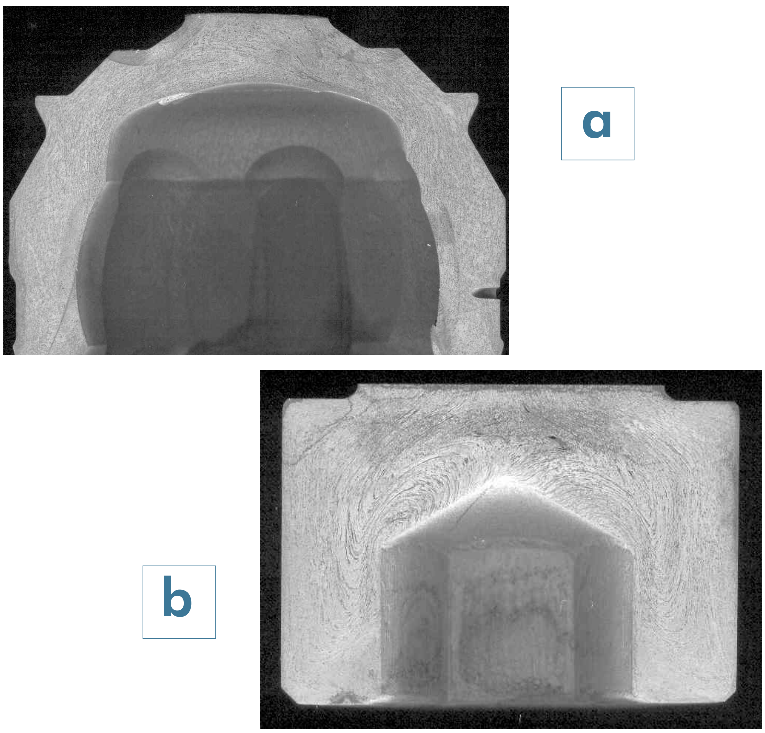

Fig. 5: Streamlines flow of the forged samples for component (A) and component (B)

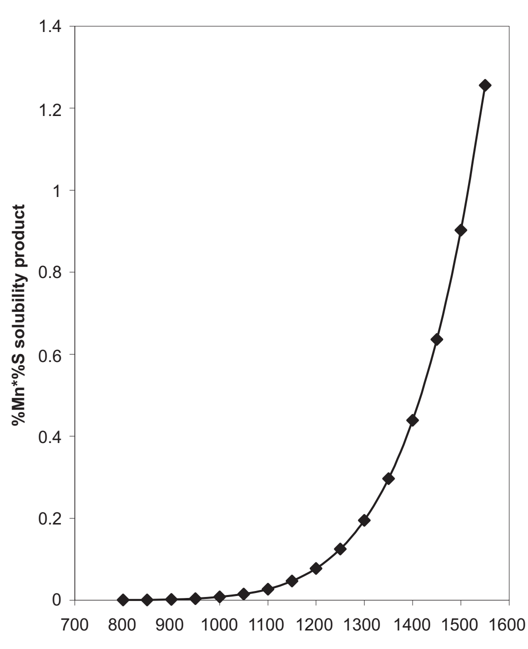

Fig. 6: Variation of the solubility product of MnS

inclusions and this can bring the material to a fast fracture process, as the ductility resources of the material have been nearly completely removed.

The chemical composition of the non-metallic inclusions observed in the forged samples have revealed that the population is completely constituted by MnS (table V).

But a main difference with the previously observed samples can be found in the shape ratio which has an average value of 2.2 and a standard deviation of 0.8, so that the anisotropy related to the non-metallic inclusions is not significant as in the previous case. This is really peculiar, because the streamlines analysis has pointed out a great plastic deformation flow (fig.5) which should induce a great anisotropy on MnS inclusions, actually on the basis of the nearly completely absence of CaS any hardening effect on the inclusion structure should be observed. The inclusions of two samples have a similar average size of the area: $16*10^{-6}mm^2$ (st.dev. $11*10^{-6}mm^2$) and $14*10^{-6}mm^2$ (st.dev. $12*10^{-6}mm^2$). On the contrary, the average number of inclusions composing a cluster is higher and in the range between 8 and 9.

The more little size and a more round shape of the inclusions of the forged samples can be a clue of the occurence of another important process related to the solubility of the MnS within the austenite and a new precipitation during the following coolings processes in a form of string composed by a greater number of non-metallic inclusions featured by a more little size. Similar processes have been described also by other authors [6,7] but with respect to other and not re-sulphurised materials with an inclusion population of a more little size than that observed in this study, however nowadays this represents the most conceiving explanation of the observed phenomenon. During the re-heating and austenitizing treatment the solubility product of MnS increases (fig.6)

$\%Mn\ \%S = 1/K$ where $K = 9281/T + 5.19$ [8]

and so part of the precipitated MnS can dissolve again in the form of Mn and S.

The dissolved Mn and S have not a great diffusivity within the solid state (in the order of $10^{-10}\ m^2s^{-1}$ [9]) so after the cooling the atoms of Mn and S can meet again and precipitate in the form of different and more little inclusions with a not significant anisotropy.

However, the effect is not less dangerous, because these adjacent non-metallic compounds constitute a string in which the coalescence of the nucleated voids is very easy and fast after the first growth. As the non-metallic inclusions are really close, the process of growth of dimples generated by the development of the voids is decreased. Because the part of energy spent for the growth of dimples is greater than that spent in the process by the coalescence process, this mechanism can facilitate the development of the overall process leading to the formation of a greater and not sustainable defect. The sustainability of a defect is clearly related to the K_{IC} for the material. For the treated material the K_{IC} tests have pointed out an average value of this parameter equal to $57MPam^{1/2}$. Under the hypothesis of a mean stress of 270MPa:

$$K_{IC}=\delta\beta\sqrt{\pi a}$$

where

σ is the applied stress

β is the shape factor (for this type of inclusion is roughly 1)

a is the defect size

the steel can sustain an overall defect of 0.014m. Because the mean length of the string is 32.5mm only 443 elongated inclusion clusters in which the dimples have coarsed can be enough to produce the unstable propagation of the fracture within the components. As the average volume fraction of non-metallic inclusion for the two steels is 1.8% (st.dev.0.09) for the steel A and 1.94% (st.dev.0.3) for the steel B, there is a great possibility that the unstable propagation of the fracture can occur. Actually, considering the clusters like ellipses with the found average length in a volume of only 6*10-5m3, it is possible to estimate that at least 793-855 string shaped clusters can be theoretically present. This means that only half of the estimated amount can produce the unstable propagation of the crack. It is true that some inclusions are not oriented in a favorable direction to interact with the applied stresses and some inclusions could not be present in the form of clusters. On the other hand, the performed measures have pointed out that 75% and 69% for component A and component B, respectively, are the ratio of the inclusions present in the form of string cluster and this appears certainly as a dangerous condition, to which can be ascribed the happened and repeated failure events occurred to these components.

In the welding observed the non-metallic inclusions forbid a good heat exchanging and their removing promoted by the flow of metal can cause an unacceptable defect due to a great irregularity of the welding border (fig.7). This phenomenon can be produced mainly by very great non-metallic inclusions (with one of the dimension >25μm), otherwise the thermal shielding developed by the non-metallic material of the non-metallic inclusion cannot develop its insulating effect. If the heat flow is considered:

$$Q=2k_{inc}(\Delta T)/(\Delta r_{inc})$$

where

Q the heat flow

k_{inc} thermal conductivity of the non-metallic inclusion

ΔT thermal difference between the metal pool and the metal border

Drinc inclusion dimension

it will be possible to estimate the effect of the presence of a non-metallic inclusion of large size. The non-metallic inclusions with a higher size can produce a decreased heat exchange fifteen times more little than that exchanged in the absence of the insulating inclusion. This effect is amplified by the more little thermal conductivity of the non-metallic material that composes these compounds, based on SiO2 and MgO, which can be evaluated to be between 4-10$Wm^{-1}K^{-1}$ [10]. Actually the evidenced defects cannot be ascribed to the MnS which has an average size of 50μm^2 (st.dev.3 μm^2) with a maximum linear size of 8.9 μm. Although the decreasing of the thermal exchange, the removal of the non-metallic inclusion operated by the convective flow in the metal pool [11,12,13] can rapidly remove the non-metallic inclusions, eliminate the thermal insulation and permit the adjacent liquid to compensate the void volume left by the removal of the inclusion itself. In presence of a great non-metallic inclusion the adjacent liquid cannot fill the pool, because the thermal insulation can avoid the heat input needed to melt a sufficient quantity of steel able to fill the significant volume made free by the inclusions. These inclusions are usually originated from exogenous factors (powder covering the tundish, pieces of entrapped refractory etc.). On the other hand, the dimensions of such inclusions are not acceptable also because they can constitute the originating factor of a possible fatigue fracture. They can be avoided by a better control of the parameters of the production process.

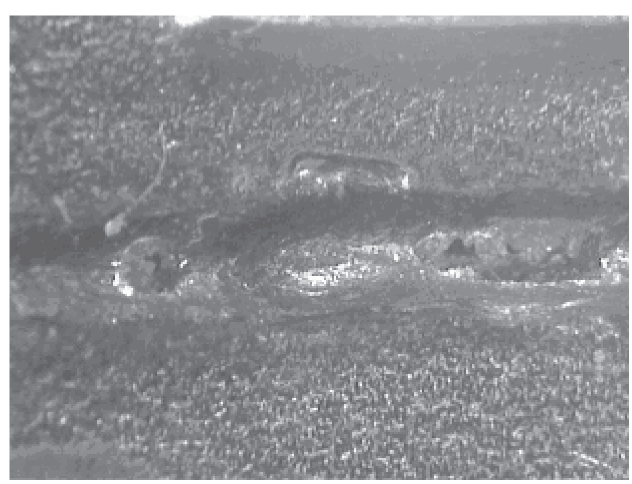

Fig. 7: Defects on the welding border for a not complete penetration due to the former presence of large non-metallic inclusions whose trace can be noted in the central upper part of the welding border

CONCLUSIONS

According to the introduction of this paper and to the results of the experimental procedures, it is evident that the possibility to perfectly define the permissible limits of the defects contained in the steel (in particular the inclusion content) could be a good solution in order to avoid praecox failures of the pieces.
On the other hand, it is almost impossible to manage the form, the dimension and the location of each single inclusion (otherwise they would not be regarded as defects): their distribution is regulated by the statistic laws as well as by the process parameters.
The modern plants for steel production and transformation work more and more automatically: the process parameters are solely controlled and the results are used for the adjustment of the process itself. Each anomalous modification of the parameter results in an automatic rejection of the material produced in the "out-of-control" condition; the results of the examination of this material are also used to improve the process.
This is not enough: even if the number of the controlled parameters is increased and all the parameters are optimised, one cannot be sure to detect all the potential defects introduced by the process itself and, of course, by the previous processes.
The results of the control are not completely reliable, both because the measure are uncertain and because the processes themselves are variable.
In the following table a list of the more common controls is presented for some siderurgical products together with the type of defect that can be detected.
Each of the above mentioned controls have several limitations: for example the ultrasonic control is not normally able to detect the defects just below the "skin" of the material, while the eddy current controls are not able to control the bar ends (50 mm each side), to detect the cracks that have been "welded" by the hot rolling and to detect the "short" defects (machine with rotating probes) or long defects (machine with magnetisation coils).

Table VI. Typical control and defects for long products

Type of control	Defects that can be detected
Product: billets	
destructive control of specimen taken in particular zones	segregations, inclusions, internal defects, cracks
Baumann test	sulphur segregations
acid attack	internal cracks, segregations
blue fracture test	macro-inclusion condition
ultrasonic control	macro-inclusion, internal defect, internal cracks
visual control	only the larger defects and cracks
automatic control	limited possibilities to detect the defects
Product: hot rolled or cold finished bars	
cut of samples and laboratory examination	some defects of raw material: cracks, segregation, decarburization
ultrasonic control	macro-inclusions, internal holes
eddy current	surface defects

The possibility to perform a "good" control on a semi-finished product depends on the type and dimension of the defect that has to be detected, on the technological limits of the control machine and on the possibility to separate the defective part (for example it is easy for a bar, but not for a coil).
A clear definition of the acceptability threshold allows to limit the rejections to the really defective pieces, i.e. those originating problems for the specific application.
Too tight and not differentiated thresholds increase the transformation and control costs without any advantages for the final product.
Only a few standards define the type and the dimensions of the permitted defects. In such cases, they refer to other standards cataloguing the defects and their evaluation criteria.
More commonly, the standards require that the defects "detrimental" to the final use must be avoided: this is obviously a poor definition, generally not able to help the producers.
Moreover, the standards do not allow any mistake in the control: no defect above the specified threshold is permitted. This would imply perfect process control together with perfect control machines: as above described, this is technologically impossible.
Only in the last period some European standards [14] permitted a certain percentage of pieces with defects above the specified limits (for example, the standard for bright bars in relation to the surface cracks). This is an important step in order to consider the real technological limits.
Some standards limit the inclusion content (for example the European standard for case-hardening steels [15]), but almost no standard specifies the limit for macro-inclusion content.
More generally, the control of the internal soundness is left to the agreement at the enquiry and order; but for the bars no reference standard exists for the ultrasonic control with automatic machines (the most widely used).
The control of the process and the control of the semi-finished products cannot eliminate the defects completely: some defects are not detected and, in consequence, cannot be eliminated.
The situation is even worse for the inclusions, where the distribution and the evolution of the defects in the metallic matrix, as explained in the previous part of this paper, is difficult to evaluate because it depends upon several process parameters.

In order to approach the "zero defect" philosophy it seems necessary to control, under certain circumstances, also some peculiar characteristics of the final products. This kind of control is extremely expensive, is not always effective for the inclusions, and cannot be performed by the intermediate transformers on the semi-finished products.

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