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UNDERSTANDING THE EFFECT OF DEOXIDATION REGIME ON THE FORMATION AND ARRANGEMENT OF SULPHIDE INCLUSIONS AND ON MECHANICAL PROPERTIES IN STEEL

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Non-metallic inclusions, deoxidation, aluminium, silicocalcium, oxidation level, grain boundary, steel, mechanical properties, sulphides.

ABSTRACT

This paper looks at the effect produced by oxygen on oxide, oxysulphide and sulphide inclusions and how they form in medium-carbon and low-alloy steels. The results of a laboratory study that looked at specimens made of steel 45, revealed a negative role of aluminium used as a deoxidizer, as it causes the sulphide inclusions arrange themselves at grain boundaries. In 38KhN3MFA steel specimens deoxidized with silicocalcium, the sulphide inclusions have better arrangement as they are located in the grain, due to the doping effect of the deoxidizer. The authors analyze how the level of oxidation (vacuum degassing) in and the type (basic or acidic) of the 38KhN3MFA steel can affect the mechanical properties of forgings for power engineering application.

Introduction

Modern steel making technology enables to produce high-quality steel products due to the application of secondary metallurgy processes [1].

At the same time, some research papers [2, 3] point out that the strive for ultralow concentrations of impurities in the melt does not always lead to enhanced performance of metal (and in some cases, this can even result in performance degradation). This can be attributed to the thermodynamics and kinetics behind the formation and growth of non-metallic inclusions when one of the reagents is lacking in the process. The mechanism behind this phenomenon is yet to be investigated. Researchers still need to understand how non-metallic inclusions form, arrange themselves and behave in a strong deoxidation environment or in vacuum.

What makes the need to thoroughly investigate how inclusions affect the structure and properties of steel ever so relevant is increasingly stricter requirements to the quality of parts produced from large ingots, and a general trend to produce heavier ingots. The latter leads to a greater unevenness in the distribution of inclusions in the metal and a poorer uniformity of its properties [4–6].

One of the main targets in improving the steel quality is to minimize the concentration of sulphur as the most harmful of the impurities. What gives relevance to this problem is the way sulphide inclusions form and behave during processing. The workpieces are not strong enough and can be easily deformed and longitudinally stretched during rolling, which affects the uniformity of properties within each particular workpiece [7]. How sulphide and oxysulphide inclusions form and grow in steel is tightly related to the presence of oxygen in metal. Depending on its concentration, inclusions of several types can emerge — from globular to film-type inclusions (classification by K. S. Sims and F. B. Dal), which absolutely affect the quality and properties of the final product.

The fact that deoxidation plays the key role in sulphide precipitation has been proved by numerous papers authored by both Russian and foreign researchers, and is now commonly recognized [8–10]. Modern steel refining techniques, which help bring the concentration of sulphur in metal down to 1–2 ppm and even more, should be considered a drastic method of tackling this problem.

However, as it was mentioned before [2, 3], the use of ultralow concentrations of one of the reagents without changing the concentrations of the others may shift the balance in the "matrix (metal) — inclusion" system, which may affect the quality of the workpiece. Moreover, the use of secondary metallurgy involves quite a cost, which can impact the cost of the steel and make it less competitive.

Another effective technique includes control of the oxidation level, which helps control the shape and the type of sulphide inclusions. This can help minimize their impact on the ductile properties of steel products [11, 12].

Earlier the authors found [13] that when a growing amount of aluminium (from 0.05% to 0.2%) and silicocalcium were used simultaneously for deoxidation of steel 20, the number of sulphides that formed in the specimens was inversely proportional to that of oxysulphides. The authors established the same relationship when they looked at the arrangement of sulphide inclusions in the grain and at the grain boundaries — the less inclusions there were in the grain, the more of them were found at the grain boundaries. The findings demonstrated the behaviour of sulphur and sulphide inclusions in low-carbon steels as the oxidation level of the melt dropped. The sulphides that form in this case are of film type, which is even less desirable.

Research Materials and Methods

To analyze the above processes that take place in medium-carbon structural steels, experiments were carried out that looked at how the deoxidation degree in steel 45 and 38KhN3MFA influence the morphology of sulphide and oxysulphide inclusions. The sulphur concentration in steel 45 was 0.03%, in 38KhN3MFA — 0.01%, respectively.

After the above two steels grades were produced in an induction furnace, they were poured in Æ50 mm and 60 mm high steel cylinders. The casting of steel 45 was combined with aluminium stream deoxidation in 3 different modes: without deoxidation, deoxidized with aluminium in the amounts of 0.7 kg/t and 1.5 kg/t. Two casting options were applied to the 38KhN3MFA steel: without deoxidation and deoxidized with silicocalcium in the amount of 0.25 kg/t. The resultant specimens were cut along the vertical axial plane and then polished. Non-pickled sections were used for non-metallic impurity analysis, which was based on the method L (per GOST 1778-70) at ×500 magnification. An ES series METAM RV microscope was used for the analysis. 3 levels (bottom, centre, top) were analyzed in the specimens for inclusions, i.e. sulphides, oxides and oxysulphides, with an error of 0.001.

Results and Discussion

The analysis showed that the non-metallic inclusions are relatively small and do not exceed 2-10 μm. Different deoxidation levels associate with different arrangement patterns. In the steel 45 specimens that were cast without deoxidation, relatively large inclusions were observed arranged mostly randomly at grain boundaries. When aluminium was added to the melt in the amount of 0.7 kg/t, the inclusions were more organized. And when the amount of the deoxidizer was increased to 1.5 kg/t, stringers emerged located mostly in the intergranular areas. **Fig. 1** shows how the main types of non-metallic inclusions are arranged along the height of the specimens depending on the deoxidation level.

The above data indicate that sulphide inclusions prevail in the metal and that non-deoxidized specimens contain most of them. Specimens deoxidized with aluminium in the amount of 0.7 kg/t contain less sulphides. And in the specimens deoxidized with aluminium in the amount of 1.5 kg/t, the concentration of sulphides goes down to match that of oxides and oxysulphides. We should note another typical feature: when the amount of the deoxidizer is raised from 0.7 to 1.5 kg/t, the levels of oxide and oxysulphide contamination in both cases are almost the same. This suggests that at the consumption rate exceeding 0.7 kg/t, aluminium does not contribute to the deoxidation process but just dissolves in the melt.

Fig. 2 shows a distribution of sulphides, oxides and oxysulphides along the height of the specimens depending on the amount of aluminium introduced in the melt.

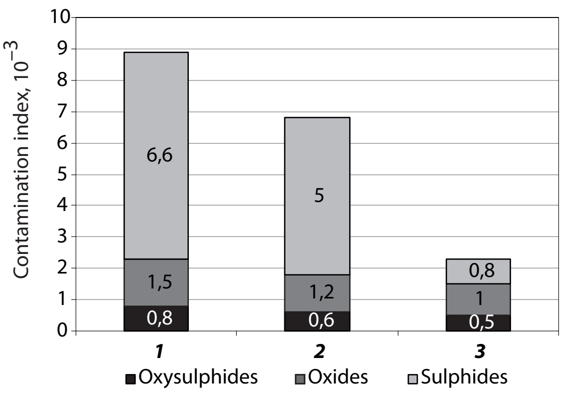

Fig. 1. Distribution of non-metallic inclusions in the specimens depending on the deoxidation level: *1* — without deoxidizer; *2* — 0.7 Al; *3* — 1.5 Al

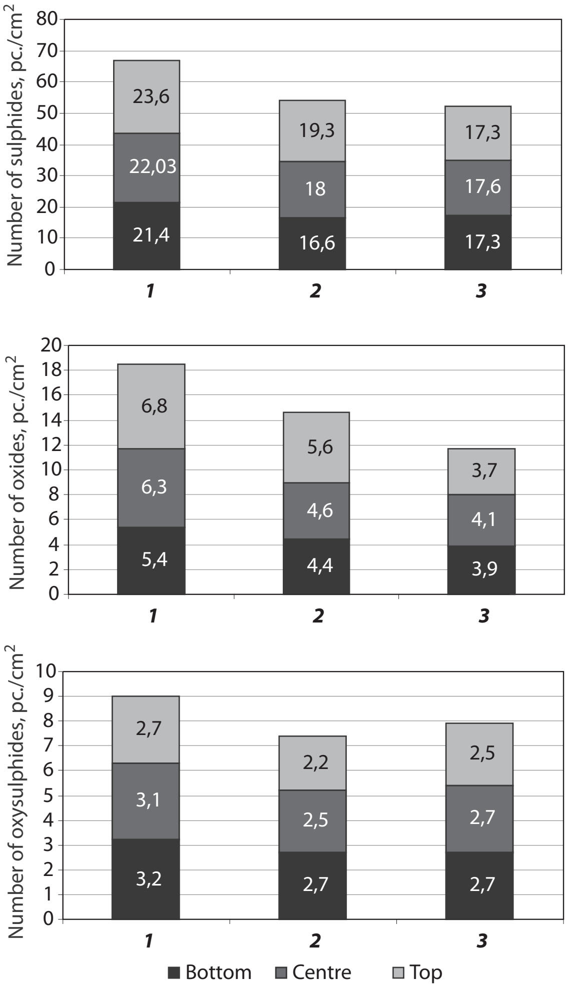

Fig. 2. Distribution of sulphide, oxide and oxysulphide inclusions in steel 45 specimens depending on the deoxidation level

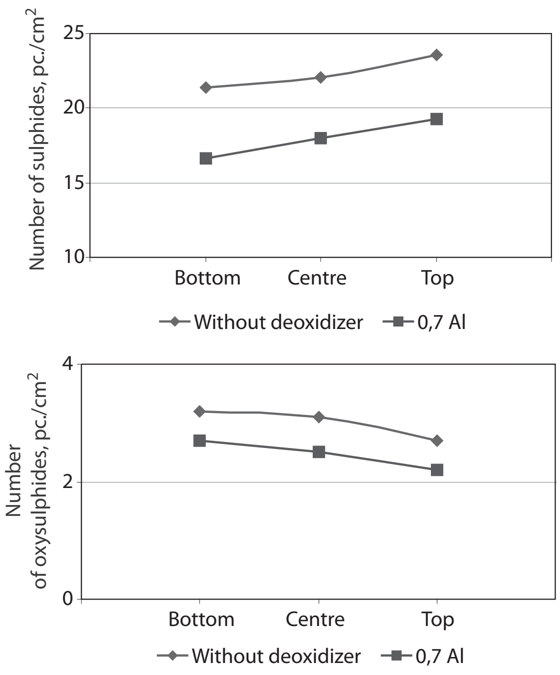

Fig. 3. Distribution of sulphides and oxysulphides along the specimens' height in non-deoxidized and deoxidized steel 45

The obtained data suggest that deoxidation with aluminium up to 0.7 kg/t result in fewer inclusions in the specimens, whereas the introduction of aluminium in the amount of 1.5 kg/t leads to a slight increase in the sulphide and oxysulphide concentrations. The sulphide and oxide inclusions slightly increase along the specimens' height while the number of oxysulphides goes down. **Fig. 3** shows how sulphides and oxysulphides tend to distribute along the height in non-deoxidized and oxidized (0.7 kg/t) steel.

One can see that the concentration of sulphides along the specimens' height is inversely proportional to that of oxysulphides. It indicates that it is possible to regulate the composition and shape of sulphide inclusions so that there were more oxysulphides and less "pure" sulphides, which tend to precipitate at grain boundaries. Such non-metallic inclusion distribution pattern is described in the paper [14], in which 3.3-ton ingots from 100Cr6 (ShKh 15) steel were analyzed. The findings show that the top layers have maximum segregation with the highest concentration of sulphide and high-alumina inclusions. A cluster of such inclusions was also found in the mother solution in interdendritic cells, which might be attributed to a high surface tension typical of alumina. The active role of aluminium in drawing the non-metallic inclusions to grain boundaries is confirmed by the findings of the paper [15], which demonstrates that in an industrial environment, when steel is deoxidized with aluminium in the amount of 0.8 kg/t, more than 80% of resultant inclusions are located at grain boundaries. **Fig. 4** shows a distribution of non-metallic inclusions in the 38KhN3MFA steel specimens deoxidized with silicocalcium.

What differentiates the resultant steel is a relatively low concentration of sulphur (<0.01%). That is why the non-metallic inclusions in it only include two types:

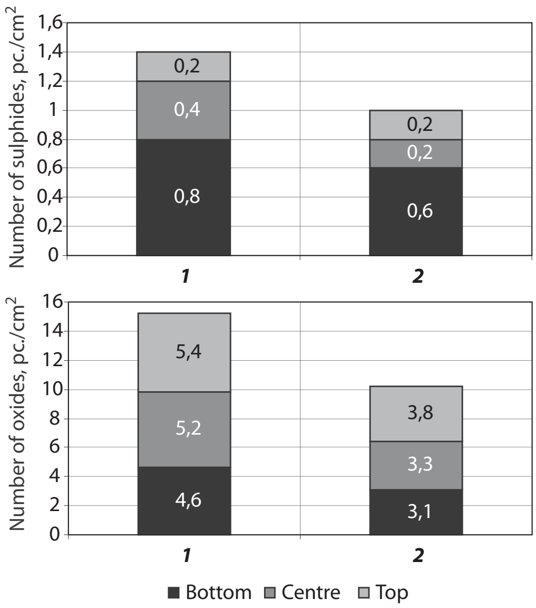

Fig. 4. Distribution of non-metallic inclusions along the specimens' height depending on the 38KhN3MFA steel deoxidation level:
1 — without deoxidizer; *2* — 0.25 SiCa kg/t

sulphides and oxides. No oxysulphides can be observed. Unlike the steel 45 specimens, the specimens from the non-deoxidized 38KhN3MFA steel contain significantly less sulphides along the height. This is also typical of the steel deoxidized with silicocalcium, but to a lesser degree. This can probably be attributed to the fact that this steel grade does not contain aluminium, so there is no effect that could make the non-metallic inclusions, including sulphides and oxides, move to the top of the specimens, as it happens in steel 45 following a mechanism described in the paper [13].

Hence, the effect of oxide inclusions on the formation of sulphides in a solidifying ingot, as described both in our findings and in the papers mentioned above, suggests that the instances of vacuum degassing affecting the steel quality are not of accidental nature. In a number of cases (primarily in the cases of relatively high concentrations of sulphur), fewer oxide inclusions and a changing distribution pattern result in the precipitation of bigger sulphides, which tend to locate at grain boundaries (**Fig. 5**) thus making the sulphide distribution even less uniform.

The same trend can be observed in the way the vacuum degassing process impacts the mechanical properties of steel. An effectively purer steel, in terms of oxygen and non-metallic inclusions, does not always imply enhanced mechanical properties, which are expected in vacuum degassed steel products.

Many authors in their papers [15-18] highlighted separate instances of the negative effect produced by vacuum degassing on the mechanical properties of steel. In particular, the paper [15] shows that a significant reduction in the concentration of oxygen (max. 0.0016%) and oxide inclusions (max. 0.0032%) achieved by DH degassing, dramatically affected the transverse impact toughness. It should be noted that the authors of the above mentioned papers relate the negative effect of vacuum degassing on the properties of deformed steel to the changing shape of sulphide inclusions and the higher ductility of sulphides as a result of hot deformation. With comparable concentrations of sulphur, vacuum degassed steel has a higher "sulphide index" than regular steel.

To understand how the oxidation level of the melt can affect the quality of steel, the authors looked at the effect produced by vacuum degassing and the type of steel (basic / acidic) on the mechanical properties of forgings meant for power engineering industry (**Fig. 6**). Standard disc specimens cut off the butt and crop ends of 15 forgings were analyzed per GOST 8479-70. The results of the analysis indicate that the vacuum degassed steel forgings may have lower ductility, especially in the case of acidic steel.

Since silicates constitute the main type of oxide inclusions in acidic steel, a decreased amount of silicates due to vacuum degassing would lead to a drop in the number of oxysulphides in steel and a corresponding rise in the number of "pure" sulphides located mainly at grain boundaries. Hence, vacuum degassing does not always benefit the quality of steel but may impact ductility in finished products, which is attributed to a specific morphology of oxide and sulphide inclusions in melted steel lacking in oxygen.

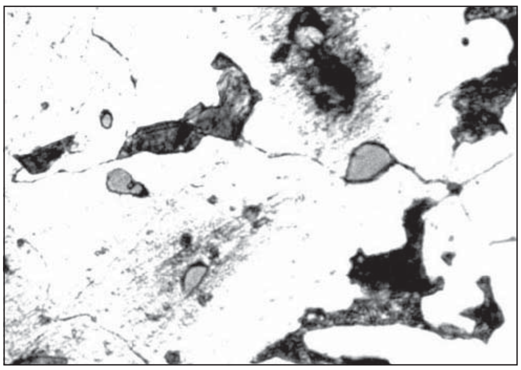

Fig. 5. Sulphide inclusions located at grain boundaries in 38KhN3MFA steel, ×500

Conclusions

Forming of sulphide inclusions is a complex process influenced by the level of deoxidation, the type and the amount of the deoxidizer used, as well as the initial concentration of sulphur.

A greater amount of deoxidizer in steel 45 is associated with less oxide and sulphide inclusions and the unchanged number of oxysulphides.

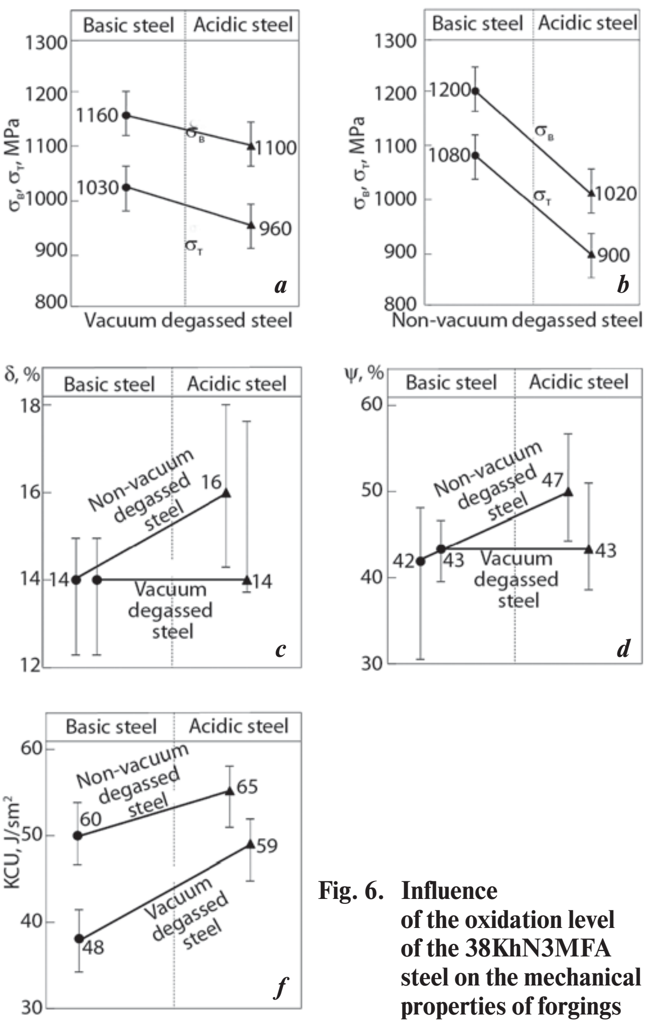

Fig. 6. Influence of the oxidation level of the 38KhN3MFA steel on the mechanical properties of forgings

The key factor contributing to a possible negative effect of sulphides on final products' ductility is the presence of sulphides at grain boundaries. This is governed by the amount of aluminium used as a deoxidizer, as well as the sufficient amount of oxysulphides, which prevent the sulphide phase from precipitating in the intergranular areas.

Without resorting to costly processes of secondary metallurgy, the negative effect of sulphur can be mitigated by controlling the amount, the shape and the arrangement of sulphide inclusions.

When deciding on the deoxidizer, one should account for its deoxidizing capacity but also for how it would change the arrangement of non-metallic inclusions in relation to the grain boundaries. In spite of its prevailing use, aluminium would be the least favourable option.

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