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Why do we need new inclusion experimental techniques?

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There is an ever increasing demand for cleaner steels, that is, those with low oxide inclusion content. This demand has highlighted the limitations of our current knowledge in controlling and predicting inclusion development during liquid steel processing.¹⁾ The formation of inclusions during liquid steel refining is an unavoidable consequence of the current industrial steelmaking process. Steel leaving a steelmaking converter contains anything from 400-1000 ppm of oxygen in solution [O].²⁾ The [O] concentration needs to be reduced to less than 30 ppm to be cast. If not, it will react on solidification producing gas and condensed oxides. If cast in such a condition the steel would be virtually worthless, containing significant porosity and oxide matter resulting in extremely poor mechanical properties.

To lower the [O] to a level so that the steel can be cast, the [O] is reacted with elements/compounds that have a strong affinity with oxygen, such as Al, Ca and Si. These are deoxidants and the process is referred to as deoxidation. The resulting oxides (inclusions) would then ideally be removed from the liquid steel by stirring it to promote particle reaction with a slag phase. Further, there are additions or steel processing specifically intended to modify the inclusions, either to enable their removal or to achieve a particular phase/morphology required for casting. In steel refining a classic example of particle modification would be Ca injection into the melt to modify solid Al₂O₃ inclusions formed by Al deoxidation. The Ca reacts with solid Al₂O₃ forming a liquid calcium aluminate that is easier to remove to the slag, less detrimental to the physical properties of the solidified steel and can also confer some local refining benefit by tying up [S] prior to casting.³⁾

Key knowledge gaps in this area are primarily a result of the complex nature of the problem. There is uncertainty with respect to the changes in phase and morphology of the inclusions with steel processing. In particular, there are difficulties predicting when an inclusion is liquid for a given set of conditions. To some extent this is due to the fact that industrial steel melts often are used in the inclusion studies. This is true whether these are on an industrial scale or simply the steel used in the laboratory experiments.^{1, 3-6)} There are other issues relating to classical metallography and SEM analysis techniques for inclusions in steel and laboratory scale experiments.

Industrial steel melts have a complex chemistry and potentially a complex array of inclusion types. This can often obfuscate any cause and effect analysis with respect to

the refining process. Further, particularly on full scale industrial processing studies, the melt composition is often necessarily transient. For example, it can be expected that soluble calcium [Ca] is lost from the steel melt during vacuum degassing to remove hydrogen or carbon as CO gas and to stir the melt to promote inclusion removal to the slag phase. The Ca oxide based or Ca sulphide based inclusions will be reduced to replace this lost [Ca] and thereby modify the inclusions.^{3,4)} Under such circumstances it is difficult to separate out the modifying effect of the change in steel melt composition on inclusion reactivity/removal to the slag and the stirring effects to promote slag-steel contact on inclusion removal. Thus, loss of [Ca] and resultant inclusion modification in industrial melts results in unwanted inclusions such as $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$ and $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$ that can cause rapid clogging of pouring tubes and reversion of S to the melt.

The transient nature of the melt can also result from steel-slag or steel-refractory interactions. For example in Al deoxidised steel the aluminium in solution [Al] can reduce MgO in the slag or refractory to magnesium in solution in the steel [Mg]. Once in the steel this [Mg] is able to react with the Al_2O_3 inclusions to form a spinel (MgAl_2O_3).^{4, 6)} Other mechanisms have been proposed for how the Mg enters solution from the refractories. Jansson et al⁷⁾ studied the reactions of carbon bonded MgO and Al deoxidised steel and proposed that Mg vapour formed via carbon reduction of the MgO and it was this vapour that reacted to form [Mg]. Spinel formation is generally considered a problem causing clogging of pouring tubes, limiting productivity and poor surface quality of final product due to the high hardness of the spinel. It is important to fully understand how and when this may form and how it could be modified to aid its removal to the slag phase.

Classical metallography and SEM analysis techniques for inclusions in steel have been widely used to analyse the morphology and composition of inclusions in liquid steel. Even though these techniques have provided significant information, it is qualitative and time consuming. In addition to being time consuming, the small area of the sample analysed raises questions about how representative the analysis is.^{1, 5, 6)} The number of inclusions being reported is usually low, particularly in clean steels. Recent studies by the authors⁸⁾ and others⁹⁾ have shown that there is segregation with respect to inclusion content in the standard steel button sampling techniques used in industry. This is also likely to be true of samples from laboratory experiments but the authors do not know of any reported work discussing this issue. It should be noted that the representative issues raised above relate to analysis of the sample. The SEM technique has become automated that is able to detect large numbers of inclusions (>5000) and assess with respect to size and composition, inclusions in steel. The ASPEXTM and ESPRIT SteelTM SEM automatic inclusion analysis techniques cut down inclusion analysis times to a few hours on something that would have previously taken weeks.^{1, 5, 6)} Judicious application of these new techniques in inclusion studies is likely to lessen the uncertainty in inclusion analysis.

Laboratory scale inclusion experiments using liquid steel are extremely difficult. There is a tendency for the crucible material (usually Al_2O_3 or MgO) to react with the inclusions thereby cleaning the steel. The principle problem when this happens is

finding any inclusions in the steel. In addition to this, the observed changes in the inclusions may be dominated by crucible reactivity. This problem can be overcome by use of levitation techniques or doping of the steel with synthetic inclusions and using one of the aforementioned automatic inclusion analysis techniques.

There have been many excellent and worthwhile works that have used synthetic steel made from high purity reagents^{10, 11)} (as opposed to steel from the industrial processes). Generally when using this approach there is an attempt to form inclusions in-situ. Even though these studies offer better control over the steel melt compositions, control of the inclusion formation and composition is extremely difficult. Normally synthetic inclusions are not added to a steel melt because of the difficulty in getting inclusion past the melt-gas interface and doing this without significant agglomeration of the particles.

In this study a new experimental technique has been developed to assess inclusion changes with time during processing. It involves the production and use of synthetic inclusions that are reacted in a synthetic steel melt composition, where both the inclusion and melt properties are closely controlled. It is believed that this approach will overcome the inherent reproducibility and uncertainty problems associated with the use of industrial steel melts. To add the synthetic inclusions to the steel a technique has had to be developed that overcomes problems of passage of the inclusions through the gas-melt interface and inclusion agglomeration. It involves sandwiching the particles between two steel disks at high pressure, prior to adding the particles to the steel melt. The view of steel disks before and after pressing is given in Figure 1. This solution offers research possibilities with respect to inclusion reactivity that hitherto were either problematic or impossible.

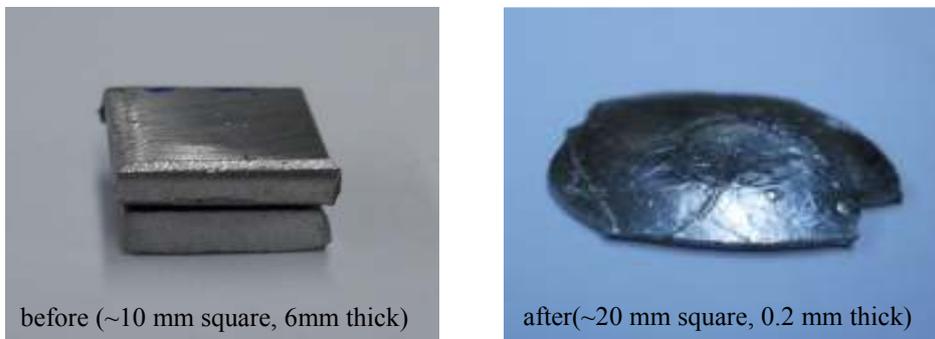
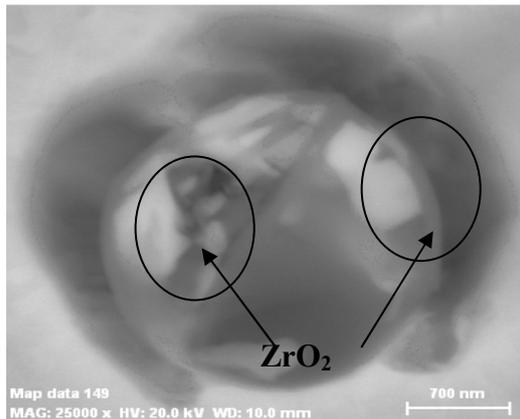


Figure 1. The comparison of the view of two steel disks before and after pressing

Further, in addition to the development of the experimental technique, the use of the analytical automatic SEM techniques such as ESPRIT and ASPEX will be used in characterising the experimental samples. It is believed this will address many of the issues researchers and industrialists have had regarding the representative nature of the particles and steel studied to real industrial melts. Preliminary work has focused on establishing the efficiency of the new experimental technique; specifically how effective the inclusion addition and sampling techniques were. To do this zirconia and cerium

oxide were added to a steel melt as tracers, using the sandwiching technique. At this point no attempt to control the starting melt composition and steel supplied by BlueScope Steel was used. This steel contained no zirconia and cerium oxide, therefore any of these compounds found in the melt would confirm successful inclusion addition. The microanalyses of an inclusion of the industrial melt using the ESPRIT Steel technique is given in Figure 2. The initial size of the zirconia additions was 30 μ but was reduced down to approximately 5 μ after pressing the steel disks. In Figure 2, it can be seen that a zirconia inclusion was attached to an Al₂O₃ inclusion. It was found that the size of the zirconia inclusion was around 1 μ which may be due to the break-up of the inclusion under experimental conditions or reaction with the Al₂O₃ inclusion.



| | [wt.%] | [at.%] |
|------|--------|--------|
| Fe K | 47.64 | 27.37 |
| O K | 22.92 | 45.98 |
| Al K | 15.88 | 18.89 |
| Zr L | 7.81 | 2.75 |
| Ca K | 2.93 | 2.34 |
| Ti K | 1.01 | 0.68 |
| Mg K | 0.86 | 1.13 |
| Si K | 0.54 | 0.62 |
| Mn K | 0.41 | 0.24 |

Figure 2. Observation of an inclusion including zirconia in industrial melt using the ESPRIT Steel technique

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