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A NOVEL LABORATORY TECHNIQUE FOR INTRODUCING INCLUSION TO LIQUID STEEL

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Abstract

Previous studies in steel cleanliness have been hampered by limited control of inclusions' phase and composition. This limited control is often a result of studying inclusion formation and development using industrial steel melts and the inclusions therein as starting material. This is an extremely complex situation and not always readily amenable to analysis. A new experimental laboratory technique has been developed that allows addition of inclusions of known, size, phase and composition to be added to liquid steel. These inclusions can then be tracked and changes in morphology with time can be assessed for different melt conditions. The technique involves the formation of a steel-oxide compact, where high-purity inclusions are sandwiched in steel foil at high pressure. This compact is added to a steel melt of controlled and known composition. The use of this sandwiching technique overcomes problems of passing small particles (inclusions <50 μm) through the gas-melt interface and inclusion agglomeration. Details of the technique are presented and discussed and results applying this new technique in a reactivity study using cerium oxide as a synthetic inclusion in steel are given.

Introduction

There is an ever increasing demand for cleaner steels, that is, those with low oxide inclusion content, and better control of the composition, morphology and size of the inclusions. This demand has highlighted the limitations of our current knowledge in controlling and predicting inclusion development during liquid steel processing [1]. 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 controlling the initial composition and phase of the inclusion in what are extremely complex reaction systems and predicting when an inclusion is liquid for a given set of conditions. This difficulty in control and prediction has given rise to uncertainty in detailing and understanding inclusion morphology changes in steel refining. This is true whether these are on an industrial scale or simply the steel used in the laboratory experiments [1-21].

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. To overcome this, there have been a number of studies that have used synthetic steel made from high purity reagents [11-20] (as opposed to steel from the industrial processes), then formed the inclusions in situ. Control of how the formation is achieved and what inclusions form is difficult experimentally.

Normally synthetic inclusions are not added to a steel melt because of the difficulty in getting inclusion past or through the melt-gas interface and doing this without significant agglomeration of the particles. Kang et al. [21] produced synthetic $\text{MgO} \cdot \text{Al}_2\text{O}_3$ spinel to study the formation mechanism of calcium aluminate inclusions from spinel. In their experiments, synthetic spinel pellets were placed on top of the melt sample. CaC_2 used as Ca source was positioned in a hole drilled into the melt sample. They analysed the interface between the steel and spinel pellets to determine the reaction phases formed. While this study gives useful information on the reaction chemistry it does not address important size (surface energy) effects and does not readily translate to changes in inclusion size and morphology in industrial processing.

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. While much has been learned using these techniques, they are time consuming and can be highly selective in the inclusion types found/studied that raise the issues of the representative nature of inclusion and its importance in steel processing. New techniques (ASCATTM, ASPEXTM and ESPRIT SteelTM) that allow automatic SEM inclusion analysis of large numbers of inclusions have been developed to address these issues. For example they are able to detect, size and analyse up to 5000 inclusions per hour, a process that would previously have taken weeks manually [1, 8, 22]. Judicious application of these new techniques in inclusion studies is likely to lessen the uncertainty in inclusion analysis.

A further problem in laboratory studies in steel cleanliness is that 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. This problem can be overcome by use of levitation techniques or doping of the steel with excess synthetic inclusions and using one of the aforementioned automatic inclusion analysis techniques.

In this paper a new laboratory experimental approach to the study of inclusion reactivity is presented. The new method uses controlled addition of inclusions of known size, phase and morphology to a steel melt and the use of automatic SEM inclusion analysis to improve the understating of inclusion reactivity steel. The primary focus of the results presented in this paper is to validate the inclusion addition technique.

Experimental materials and methods

The experimental work has focused on establishing the efficiency of the new experimental technique; specifically how effective the inclusion addition and sampling techniques were. A steel melt is heated to the experimental temperature of 1600°C under controlled atmosphere conditions then synthetic inclusions were added using the method described below. See figure 1 for a schematic diagram of the experimental set-up used. Suction samples ($\sim 5\text{g}$) using a quartz tube were taken periodically throughout the experiment. At the end of the experiment the melt plus crucible were rapidly cooled by lowering the crucible/melt assembly into the cooling chamber. Suction samples and the rapidly cooled melt were then analysed using classical SEM-EDAX, ESPRIT and ASPEX systems. In these experiments synthetic inclusions based on laboratory grade zirconia and cerium oxide were used as they were not present in the melt alloy and could therefore be used as doping agents/tracers to assess the efficiency of the inclusion addition technique. Zirconia and cerium oxide powders were supplied by Z-Tech and Sigma-Aldrich, respectively.

In this study the steel melt used was supplied by BlueScope Steel, the composition of which is given in Table 1. Inclusion compositions of this steel were in the Ca-Al-S-O system. The selection of this specific steel was based on ready availability and economy. In future studies the steel melt composition will be controlled and synthetic steel will be produced from laboratory grade materials.

Table 1. Chemical composition of steel used in this study (mass %).

C	Si	Mn	S	Ca	Al	O _{tot}
0.069	0.3	1.421	0.001	0.0009	0.033	0.0015

Inclusion addition

To add the synthetic inclusions to the steel, a technique was developed that overcomes problems of the passage of the inclusions through the gas-melt interface and inclusion agglomeration. It involves sandwiching the inclusions between two steel plates at high pressure (50 tonne). A schematic of the pressing set up is given in Figure 2. The result is

steel-inclusion sandwich or compact. It is this steel sandwich that is added to the steel melt. A view of the steel plates before and after pressing is given in Figure 3.

The initial size of the zirconia and cerium oxide additions before pressing was 15 and 50 μm , respectively. After pressing the size was approximately 5 μm for both inclusion types. The plates used in the pressing process were interstitial free steel.

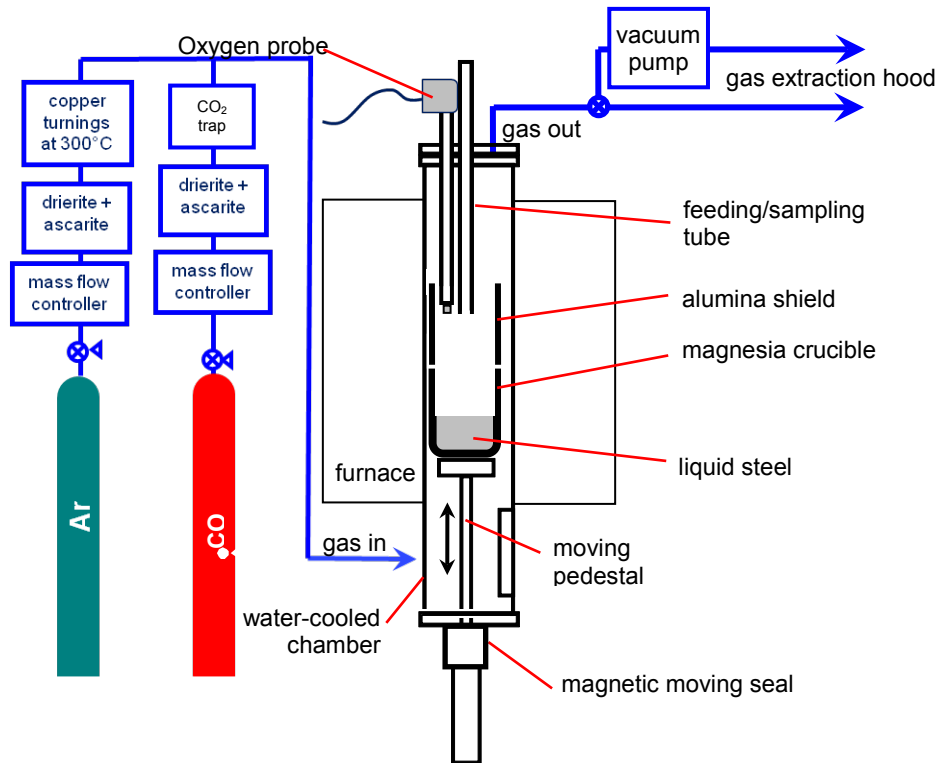


Figure 1. Schematic diagram of experimental set-up.

Experimental set-up

The experimental set-up is schematically presented in Figure 1. A resistance heated vertical tube furnace was used in this study. A 300g steel melt was held within a magnesia crucible. Additions of the inclusion containing compacts and metal sampling could be done through a central alumina tube, and the final melt could be rapidly cooled by lowering the crucible into a water cooled chamber.

In order to achieve low oxygen potentials, argon and carbon monoxide gases were used. The gases were passed through a gas cleaning system to remove moisture, CO₂ and oxygen before introducing them to the system. A partial pressure of oxygen of 8×10^{-10} atm was measured at the experimental temperature via an *in situ* zirconia based oxygen probe.

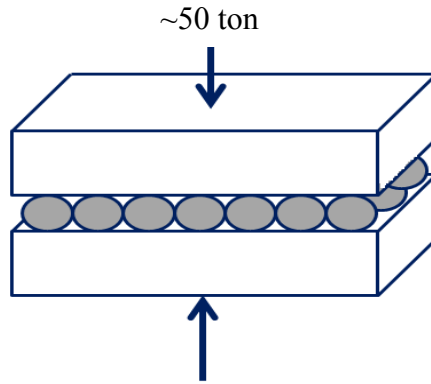


Figure 2. Schematic diagram of sandwiching technique.

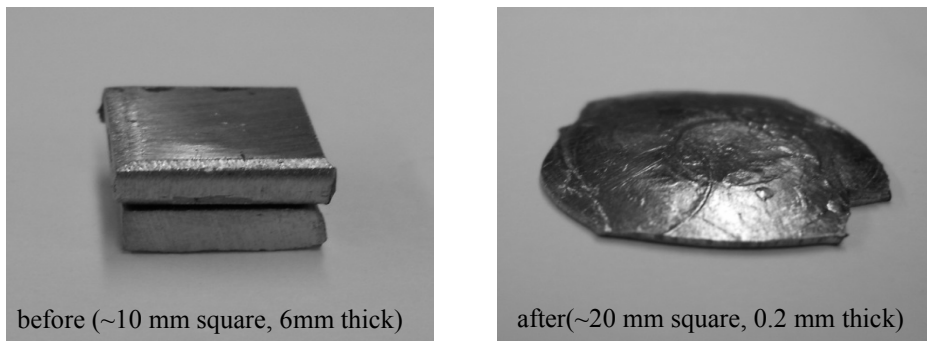


Figure 3. The comparison of the view of two steel disks before and after pressing.

Results and Discussion

Inclusions with ZrO_2

A typical micrograph and EDAX analysis of an inclusion containing zirconium from a rapidly cooled steel melt at the end of the experiment, taken using the ESPRIT automatic inclusion analysis system, is given in Figure 4. Inclusions containing Zr were found but were always associated with Al_2O_3 or calcium aluminate type inclusions. There was no evidence of discrete ZrO_2 inclusions. One possible reason for this could be that there was little contrast between ZrO_2 inclusions and the surrounding steel in the backscattered electron imaging used to locate the inclusions. The size of the typical size of zirconia containing inclusions was $\sim 1\mu m$ which may have been due to the break-up of the inclusions under experimental conditions or by reaction with the Al_2O_3 inclusions. For these reasons, CeO_2 was proposed as a more appropriate tracer to confirm the efficiency of the addition technique.

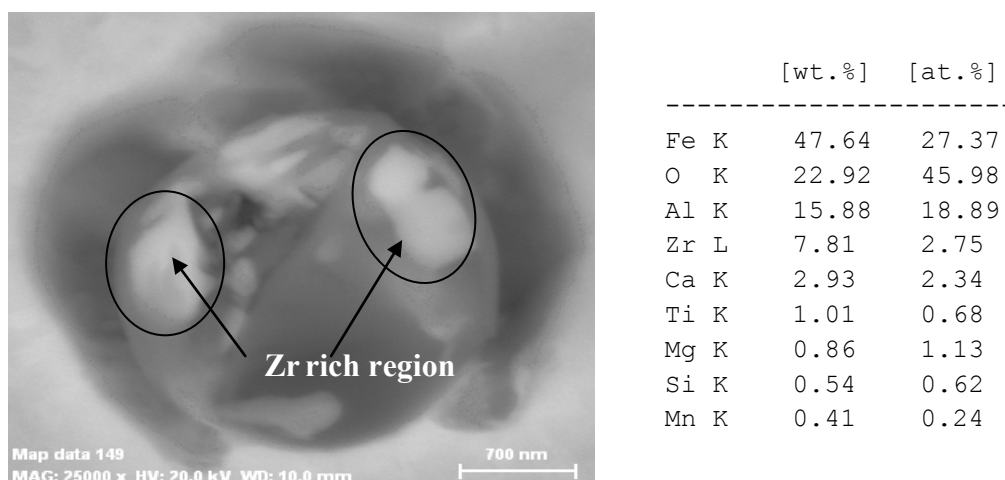


Figure 4. Image of a calcium aluminate inclusion in the remaining steel sample with Zr rich areas highlighted under FEGSEM.

Inclusions with CeO₂

Figure 5 shows the positions of inclusions detected within a sample area of 42 mm² using the ASPEX system. There were 1460 inclusions in total of which 9% contained Ce. The size classification of inclusions with Ce with other inclusions such as Al₂O₃ and CaO.Al₂O₃ is given in Figure 6. The inclusions are divided into 4 size classes ranging between 1 to 20 µm. Inclusions were distributed randomly across the sample area, and a majority were smaller than 5 µm. The majority of Ce containing inclusions were again attached to other inclusions. Only two discrete CeO₂ inclusions were detected in this field of view.

The attachment of the Ce containing inclusions to the inclusions originally in the steel is not considered a major problem. The steel used in this study is an industrial steel. It is envisaged that in future studies a synthetic steel will be used prepared from laboratory grade materials. This synthetic steel should have minimal inclusion content and will be of a controlled composition.

More in depth elemental analysis of Ce containing inclusions was conducted using SEM-EDAX. Figure 7 shows the mapped images of a typical Ce containing inclusion in a rapidly cooled melt sample. The elemental analysis clearly shows that this inclusion is a discrete CeO₂ particle. The CeO₂ particle was brighter than the surrounding steel and thus could be easily distinguished in backscattered mode in the SEM. The results indicate CeO₂ inclusions added to the system were successfully incorporated into the steel melt and the sandwiching technique has high potential to be used in inclusion studies.

Steel samples taken from the melt using quartz tubes were also analysed. One or two inclusions including CeO₂ were found in each sample. The distribution, size and composition of the inclusions in these samples were similar to the rapidly cooled final melt. Additionally, the interface between steel sample and crucible wall was

investigated. There was no evidence of cerium at the interface between the crucible and melt. This indicates that the added inclusions were predominantly within the steel melt.

The presence of Zr and Ce containing inclusions within the steels indicate that the addition of the inclusion containing compacts successfully introduced inclusions in to the molten steel baths. This is considered a validation of the inclusion addition technique. Application of this inclusion addition technique, should allow better control and delineation of the inclusion reactivity in the study of steel cleanliness.

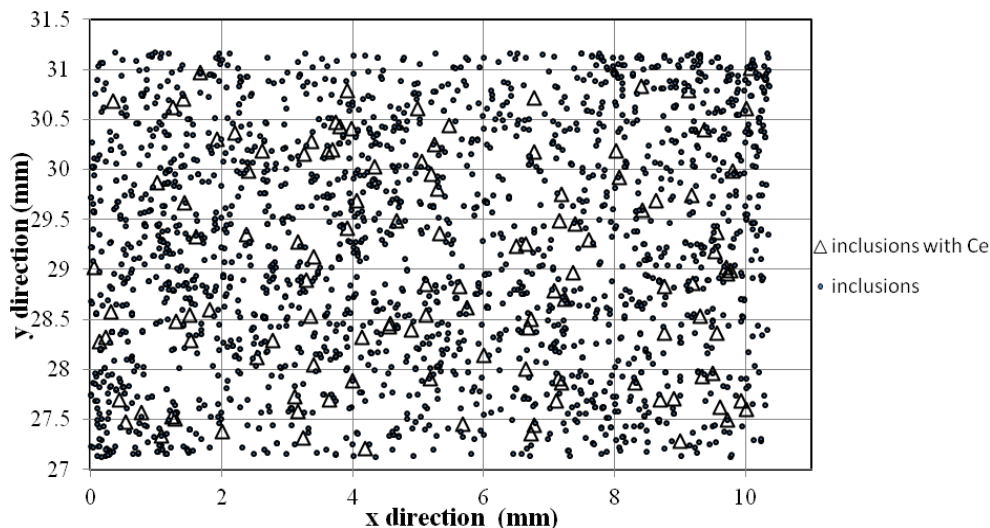


Figure 5. The position of the inclusions found in steel sample remained in the crucible (measured by ASPEX).

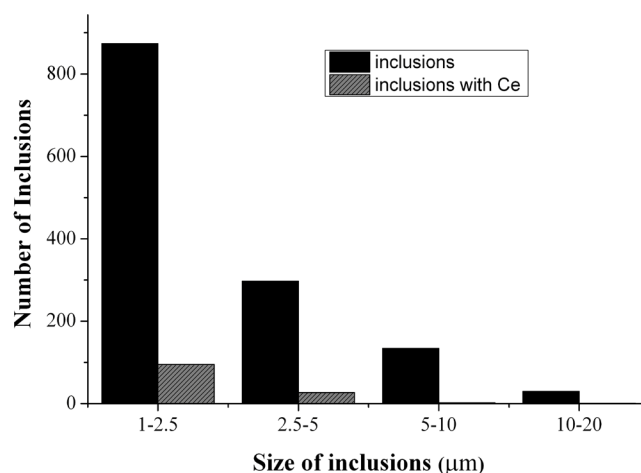


Figure 6. Size distribution of inclusions in steel sample remained in the crucible (measured by ASPEX).

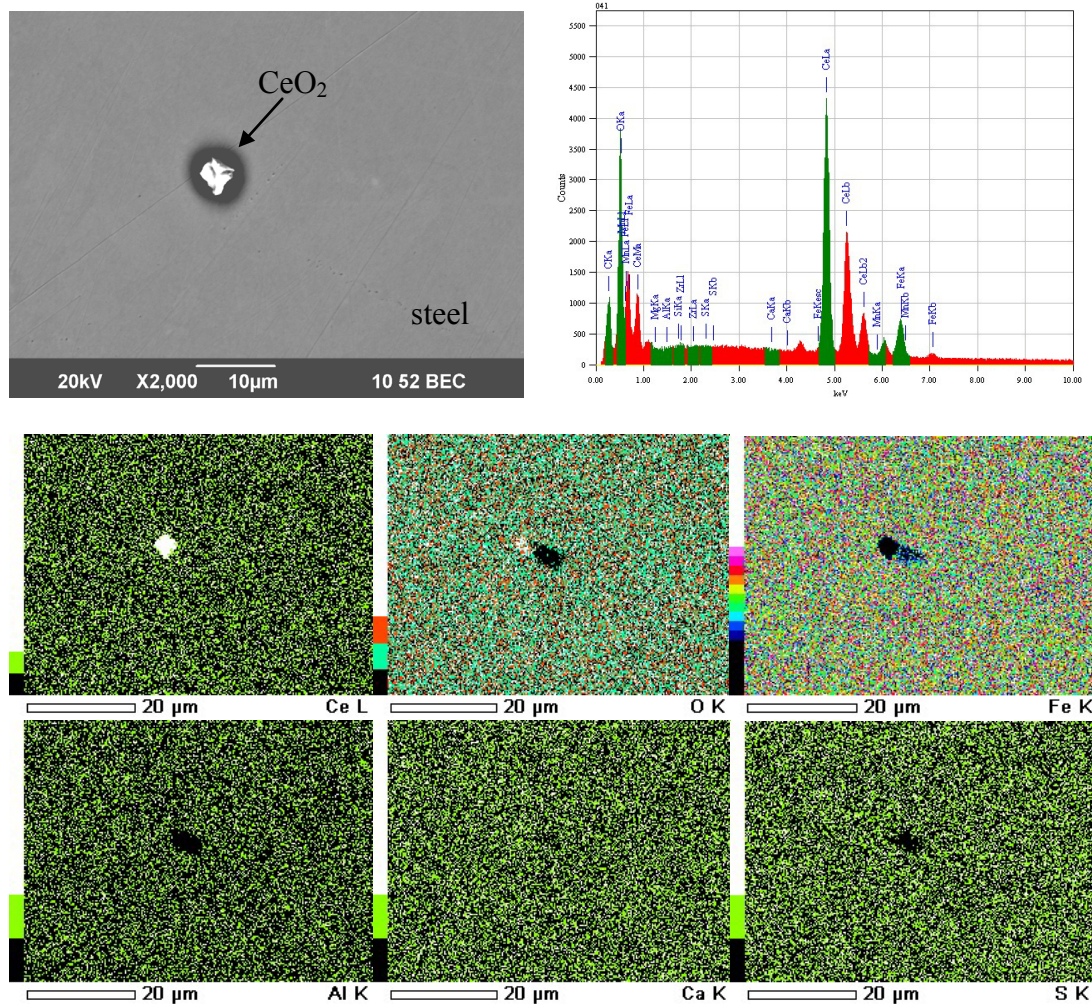


Figure 7. Mapping images of CeO_2 inclusion in liquid steel taken from remaining steel sample by SEM-EDAX

Conclusion

A technique was developed to enable the addition of inclusions of known size, morphology and composition to liquid steel by sandwiching inclusions between two steel plates prior to addition to the melt. This sandwich was added to a steel melt of known composition. The effectiveness of this addition technique was evaluated by examining the chemistry, morphology and distribution of inclusions in samples taken from the melt with time and in the residual melted steel.

Zr and Ce containing inclusions were detected and their composition, size and distribution in liquid steel were measured. The results indicated that the synthetic inclusions produced in the laboratory scale can be successfully added and detected in liquid steel.

Acknowledgements

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