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Observation of the reversible stabilisation of liquid phase iron during nitriding

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ABSTRACT

Confocal scanning laser microscopy has been used to observe in-situ cooling of Grade 1 iron whilst under a nitrogen atmosphere. Liquid iron has been stabilised to temperatures below 1400 °C through nitriding at high temperatures. This has been achieved in two ways. Firstly by exposing the melt to nitrogen and observing the onset of solidification whilst cooling. Secondly, under isothermal conditions by re-melting of initially nitrogen free iron at 1400 °C through the introduction of a nitrogen atmosphere. The latter process has been shown to be reversible with liquid iron being unstable once reverting back to a helium atmosphere.

1. Main body

During processing of liquid steel there is tight control of atmospheric conditions. Contact with air is normally avoided by use of protective slag layers, sheathing and inert gas shrouding (usually argon) [1,2]. Both oxygen and nitrogen in the atmosphere can cause major issues later in the processing steps, for example through large/excessive inclusion formation, blow hole formation and embrittlement [3,4]. Due to this less research has been carried out on the potential benefits of adding nitrogen to steel.

After carbon, nitrogen has the next greatest influence on the liquidus temperature (per unit mass) of steel compared to the other more common steel alloying elements [5]. Whilst changing carbon content is a highly controlled method involving solid additions to the liquid steel, nitrogen offers a novel approach for liquidus control due to its gaseous form.

Although much work has been carried out on the benefits of nitriding in the solid state (particularly for stainless steel [6,7]), comparatively little research has been carried out for liquid metallurgy. For example Liapina [8] studied the influence a nitrogen atmosphere has on the stability of iron nitrides. The limited solubility of N in ferrite or austenite results in the formation of stabilizing phases that can tolerate the higher nitrogen content (such as the nitrides γ' and ε) [8].

The aim of this work is to look into the stabilisation of liquid iron through ultra-high temperature nitriding. In addition to this the reversible nature of the nitriding process has been studied (due to the implications down stream of high nitrogen contents). Being able to temporarily change composition at various points during solidification allows for curved/complex paths through the phase diagram on cooling, opening up more novel solutions for problems, as well as the additional benefits of lower melting points for applications such as joining.

For this study Grade 1 pure iron was used ($C<0.02$, $Mn<0.2$, $S<0.015$, $P<0.015$, all wt%). The samples were machined into small cubes of around 0.2 g. The pseudo-binary Fe-N equilibrium phase diagram can be seen in Fig. 1. The solubility limits of N in iron are well known and governed by Sievert"s law [9] and can be seen on the left side of Fig. 1.

A high temperature confocal scanning laser microscope was used in order to observe the in-situ microstructure during solidification of the iron. By focusing light inside a closed chamber onto a point around 1.5 mm diameter, heating rates of >10 °C/s for the small samples can be achieved. A laser is then used to scan the surface of the sample to give topological information (a more detailed description of this equipment has been given previously [10,11]). The advantage of the enclosed chamber is the ability to control the atmosphere; due to its small size (the sub chamber around the sample is around 100 ml) the chambers atmosphere can be changed in less than two minutes. Before each test the chamber is evacuated to an internal pressure of around 1.3 kPa and ventilated with grade N6 argon gas. The argon passes through three additional oxygen getters to obtain an oxygen concentration below 3 ppm as well as a drying chamber and particle filters. A flow rate of 0.4 L/min at 7 kPa is used (this remains constant for all gases in this study). This process was repeated three times before heating.

Two different thermal profiles were used in this study; the first used continuous cooling from the liquid phase. A calibration

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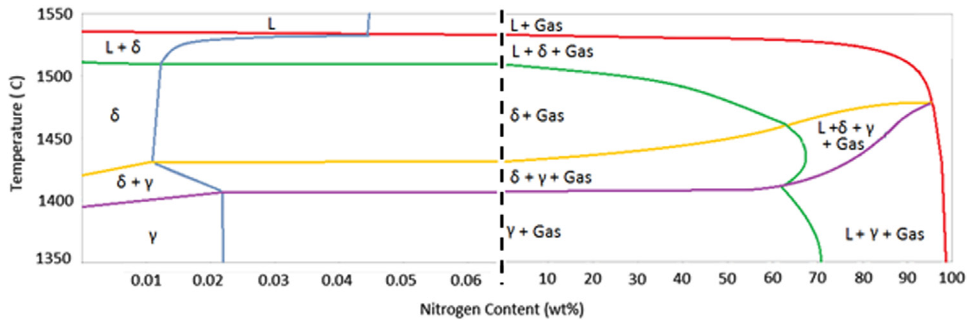

Fig. 1. Phase diagram showing the influence of N on the phase balance in Grade 1 pure iron (which contains low levels of C, Mn, S and P). The left hand side of the diagram shows a expanded section to highlight the equilibrium solubility of nitrogen in the steel.

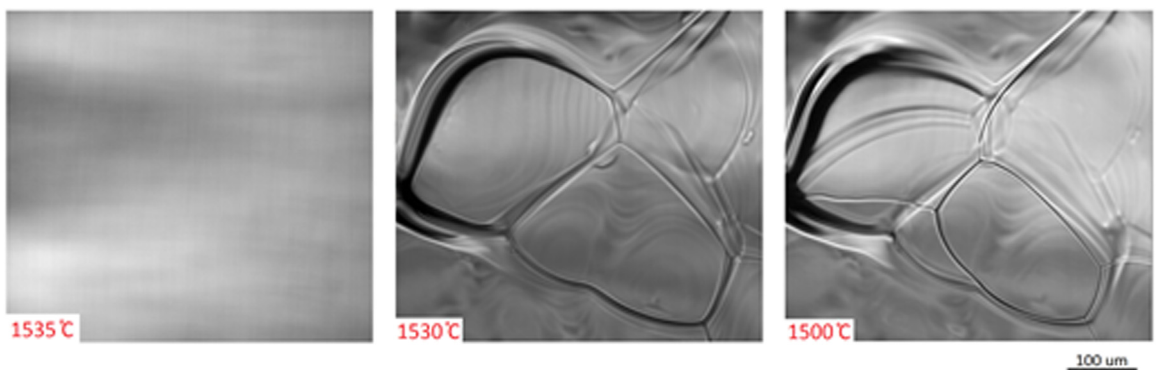

Fig. 2. Time lapse images of Grade 1 pure iron cooled from liquid phase at a rate of 0.5 °C/s in argon.

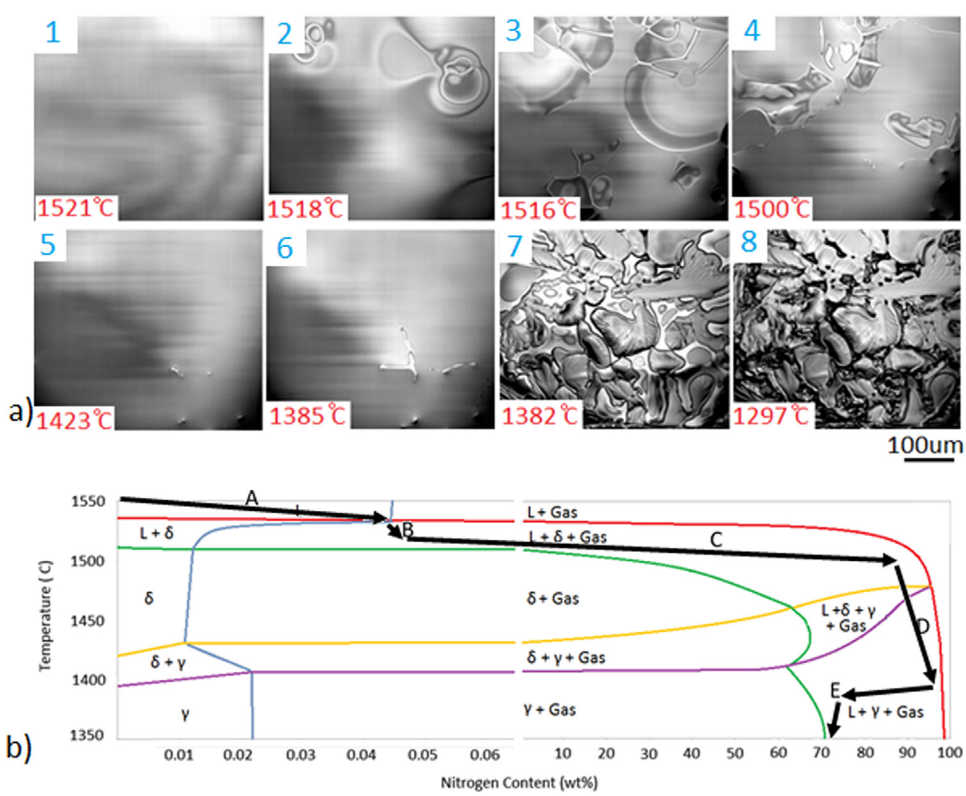

Fig. 3. a) Time lapse images showing liquid iron being cooling in a nitrogen atmosphere b) the composition path taken during cooling.

experiment was carried out for melting and solidification of the Grade 1 pure iron in an argon atmosphere, as argons solubility in liquid steel is minimal (<1 ppb [12]). The sample was heated at 500 °C/min to 1590 °C to ensure that the sample is completely molten and held for 1 min, the sample was then cooled at 0.5 °C/s until 1200 °C. A video with recording rate of 10 Hz was used during the cooling period. A series of time lapse images during solidification can be seen in Fig. 2, where the transformation from