Dynamic measurement of recovery and recrystallisation in interstitial free steel using a high temperature electromagnetic sensor

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Interstitial free (IF) steel grades are used in many applications as they offer excellent formability and resistance to thinning, due to their favourable microstructures of uniform fine ferrite grains. IF steels are produced by hot then cold rolling, followed by an annealing heat treatment to soften the steel through the processes of recovery and recrystallisation. It is desirable to be able to monitor microstructural development during annealing to ensure full recrystallisation is achieved and to determine whether grain growth occurs. Introduction of an accurate in-situ dynamic monitoring system that directly assesses the microstructural state, allowing feedback control, would improve the steel manufacturing process, reducing costs and saving energy and time. This paper discusses the use of a laboratory based high temperature laboratory electromagnetic (EM) sensor to monitor recovery and recrystallisation in IF steels in-situ during annealing heat treatments. Interrupted heat treatments to reveal the microstructure and correlated to the change in EM sensor signal (inductance) showed that inductance is sensitive to the recovery and recrystallisation processes. It was found that the different rates of recovery and recrystallisation due to annealing at different temperatures (from 375 to 700°C) were clearly revealed by the in-situ inductance measurements.

KEYWORDS: INTERSTITIAL FREE STEEL - RECOVERY - RECRYSTALLISATION - ELECTROMAGNETIC - NON DESTRUCTIVE TESTING

INTRODUCTION

Interstitial (IF) steels are commonly used in the manufacturing industry across a wide range of applications due to their high formability. The main processes involved in IF steel sheet manufacture from cast steel are hot rolling, cold rolling and annealing. Each process affects the properties of the IF steel; this paper is concerned with the recovery and recrystallisation processes that take place during continuous annealing after cold rolling [1].

In order to obtain the necessary material properties during continuous annealing control systems are used, which employ mathematical models of the plant within fixed conditions for on-line control [2]. Final confirmation of material properties is achieved through traditional mechanical testing for strength, ductility and r-values. Current annealing control systems do not use direct dynamic feedback of the microstructural condition but infer microstructure from the models and temperature-time history in the line; the addition of such a direct measure of microstructure into the control process would represent an improvement for the manufacturing process through greater potential process efficiency and robustness during process changes. Cold rolling is used to reduce the IF steel to the required sheet thickness because of its high dimensional accuracy. Cold rolling causes severe deformation to the hot rolled microstructure, introducing dislocations, increasing hardness and decreasing ductility [3]. Annealing causes the IF steel to recover and recrystallise, reintroducing the material properties required for high degrees of formability [4]. It is known that changes to microstructure affect the magnetic properties of steel, with reports showing that the magnetic permeability increases as dislocations are annihilated and grains are reformed during recrystallisation [5, 6]. It has also been reported that the magnetic coercivity can be used to detect recovery and recrystallisation, with coercivity showing greater sensitivity to recovery than hardness measurements [7]. These reports deal with measurements of cold samples that have been heat treated to give different degrees of recovery and

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recrystallisation. Measurement of recovery and recrystallisation has not been limited to magnetic measurements, with previous studies showing that ultrasonic techniques can be used successfully [8, 9]. Dynamic measurement of recrystallisation has been shown to be effective using laser ultrasonics for different steels including IF steel, the tests used continuously heated samples of IF steel and recorded recrystallisation and phase change [10, 11]. Laser ultrasonics have also been shown to be sensitive to recovery in ultra-low carbon steels during annealing [12]. No dynamic measurement at temperature using EM sensing has been reported.

EM sensors have been used to monitor phase transformation during cooling after hot rolling of strip steel [13], detect differences in decarburisation on rod and rail samples [14] and to distinguish the effects of tempering and in-service exposure in power generation steels on relative permeability [15] EM sensors are sensitive to changes in the electrical conductivity and magnetic permeability (dominant effect) in the steel due to changes in microstructure. In previous work phase transformation has been assessed at high temperature in-situ during steel processing (on the run-out table after hot strip rolling), in that case the sensor was itself cooled but the steel strip being assessed was at temperatures up to the Curie temperature (approx. 770°C). Therefore the EM sensor technique has the potential for measuring recovery and recrystallization, due to the associated changes in magnetic permeability, with on-line measurements during annealing.

In this paper EM sensor measurements for monitoring recovery and recrystallisation in IF steels during annealing at a range of temperatures from 365 – 700°C is reported. An EM sensor capable of operating in a furnace at high temperatures has been used and in-situ inductance values have been correlated with microstructural changes using samples, which have undergone interrupted heat treatments..

EXPERIMENTAL PROCEDURES

A standard IF steel grade microalloyed with Ti was used to prepare samples measuring 110 mm x 19 mm x 1 mm. The samples were prepared from 1 mm thick cold rolled sheet supplied by TATA Steel UK Limited. All samples were taken in the rolling direction. Heat treatment was completed using a laboratory furnace; K-type thermocouples were used to ensure accurate sample temperatures were recorded and maintained.

A laboratory cylindrical EM sensor, capable of operating at temperatures up to 900°C, was used for the EM measurements; measurements were controlled and recorded using Solartron Analytical SMART software on a laptop computer connected to a Solartron 1260A frequency response analyser. Measurements were taken at a single frequency of 100Hz every two seconds, this frequency was selected as it is known that lower frequencies are more sensitive to changes in permeability than high frequencies [16]. The cylindrical sensor is composed of two coils of wire formed around a ceramic cylinder, the coils are wound concentrically such that one is on top of the other. The inner coil was used to detect the changes in the EM field induced using the sensor's outer coil. Consistency of sample position within the sensor is ensured by the inclusion of a ceramic plug at one end of the sensor. The sensor is placed inside the furnace and allowed to stabilise at the set temperature before the sample is placed inside the sensor. The time to reach temperature for each set temperature was determined and was typically less than two minutes.

In situ measurements were made on as-rolled samples (up to six hours hold at temperature) and annealed samples. Inductance measurements were taken every two seconds throughout the annealing period. Plots of the inductance signal were then used for data analysis. Interrupted tests with samples being removed and quenched after set periods were also carried out. Inductance measurements were recorded for an empty sensor to account for any change of inductance due to sensor heating, it was found that there was no change in inductance as the sensor temperature increased and that there was no requirement to correct in situ or interrupted test results for sensor heating. The standard error for samples stabilised at temperature after microstructural changes have happened for in-situ EM measurements is 8.0 x 10-8 H; this error is calculated using high temperature data at 700°C where the inductance signal scatter is greatest. The standard error between samples when both sample and EM sensor are at room temperature is 3.3 x 10-7 H.

An Indentec 5030 SKV Vickers hardness testing machine, with a 5kN load, was used to record hardness values for the as-received, fully annealed and interrupted heat treated samples. A Zeiss Axioskop 2 optical microscope was used to obtain microstructural images from samples mounted, ground, polished and etched in 5% Nital.

RESULTS AND DISCUSSION

Figure 1 shows in-situ inductance measurements for an as received IF sample and then the second run for the annealed sample (i.e. the same sample after the first run) at 700°C. Both tests began with the sample at room temperature being inserted into the hot sensor with the initial increase in inductance being governed by sample heating. It is known that the relative permeability, and hence EM sensor signal, for steel is affected by temperature, i.e. permeability (and inductance) increases with an increase in temperature [17]. Thermocouple measurements during a separate test showed the sample to reach the heat treatment temperature in 2 minutes, therefore after approximately two minutes the changes in inductance are expected to be due to any microstructural changes occurring in the sample. The end of the sample heating period is marked in figure 1 by a vertical line at 2 minutes. The difference between the initial inductance values is expected due to the different starting microstructures of the samples (i.e. as-received cold rolled microstructure compared to annealed recrystallised microstructure of the sample after the initial heat treatment). The data shows different behaviours for the inductance with time, after the samples have reached 700°C, between the as-received and annealed conditions. The annealed sample shows almost constant inductance with time, which would be expected for a fully annealed microstructure not exhibiting any further microstructural changes. In fact a small increase in inductance (from 8.5 x 10^{-5} H to 8.7 x 10^{-5} H) is seen, as shown in figure 1, which will be discussed later. For the as-received sample recovery and recrystallization are expected at this temperature which cause the increase in inductance values. The inductance value for the as-received sample reaches the same value as the annealed sample after approximately 10 minutes suggesting that recovery and recrystallization are complete at this stage. It can be seen from figure 1 that the initial increase in inductance during the heating part of the cycle is different for the two tests, this may be due to different responses in terms of inductance with temperature for different starting permeability values and/or because some recovery / recrystallisation occurs for the as-received material during heating. This will be discussed further later.

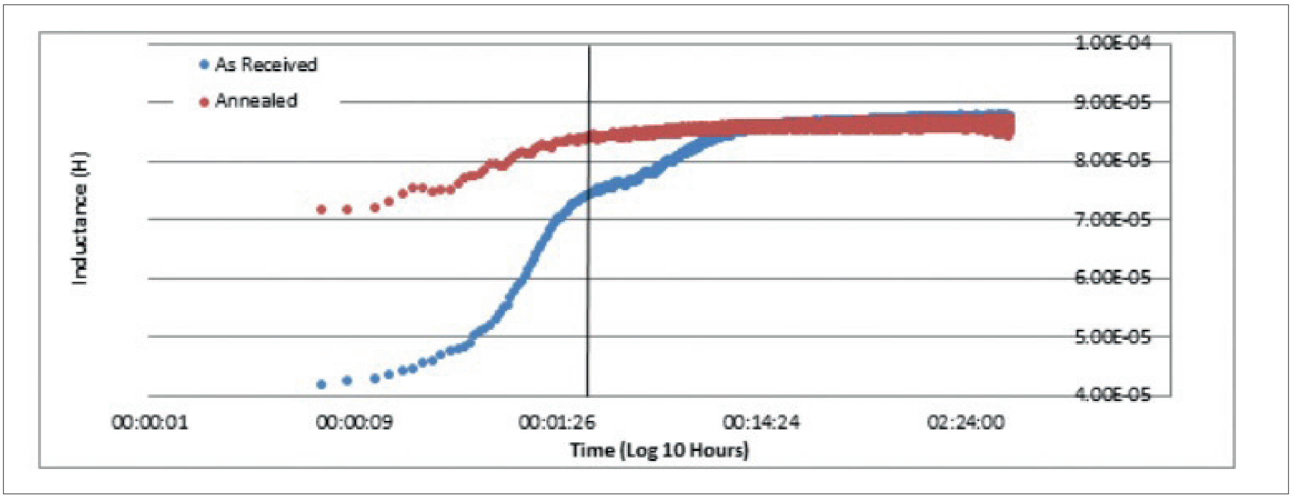

Fig. 1 - As received and annealed sample in-situ inductance measurements taken at 700°C, ime taken for the sample to reach furnace temperature is marked by a vertical line at 2 minutes.

Figure 2 shows in-situ inductance measurements for an as received IF sample and then the second run for the annealed sample at 420°C. The end of the sample heating period is marked at 2 minutes by a vertical line. Annealing at 420°C was expected to cause recovery only as it has been reported that only recovery is expected at temperature of less than 500°C for IF steels [18]. The initial inductance value for the as received sample is less than that of the annealed sample; this is expected as the annealed sample has undergone recovery (rearrangement and annihilation of dislocations) during the first heat treatment cycle, it has been reported that recovery increases the magnetic permeability in steel [6] and the EM sensor inductance values are directly related to the initial relative permeability [19]. Once the annealed sample has reached the heat treatment temperature the inductance value then shows no further significant change with time indicating that no further microstructural changes are taking place. The as-received sample continues to show an increase in inductance once it has reached the heat treatment temperature, which may be indicative of recovery taking place. The initial increase in inductance during the heating part of the cycle is different for the as-received and annealed samples, similar to that shown in figure 1 for the 700°C tests, which may be due to differences in the permeability-temperature response or due to some recovery occurring during the heating cycle, to be discussed later.

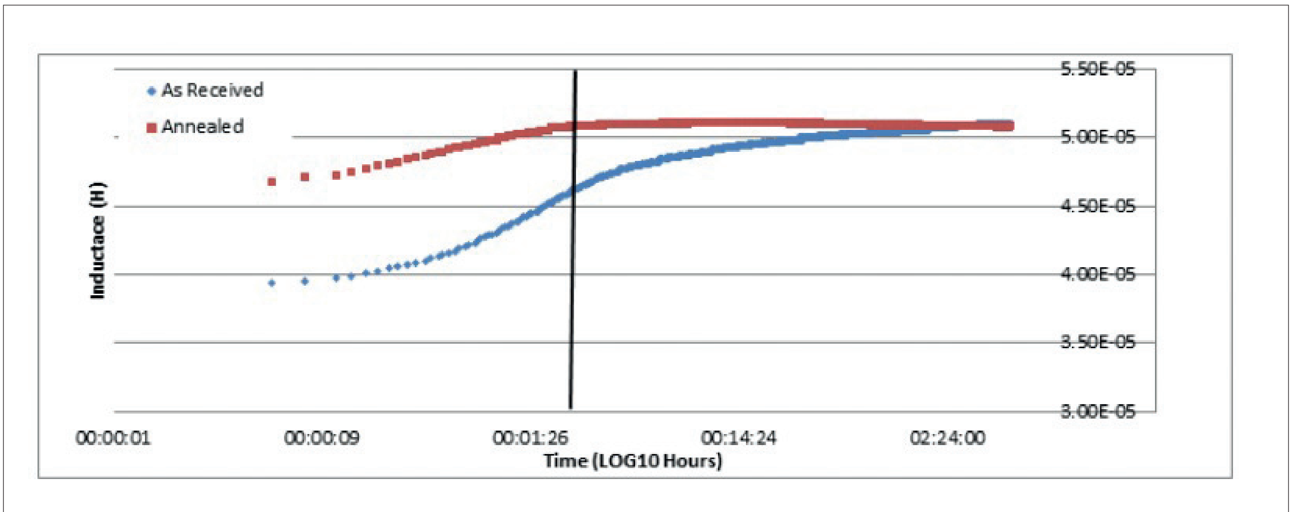

Fig. 2 - As received and annealed sample in-situ inductance measurements taken at 420°C, time taken for the sample to reach temperature is marked by a vertical line at 2 minutes.

Samples were annealed at 365°C, 420°C, 650°C and 700°C. The objective of the higher annealing temperatures (650°C and 700°C) was to establish whether different rates of recovery and recrystallisation could be measured, whereas the objective of the lower temperatures (365°C and 420°C) was to target the effects of recovery only. Figure 3 shows in-situ measurements for samples heated at 365°C, 420°C, 650°C and 700°C from the as-received condition.

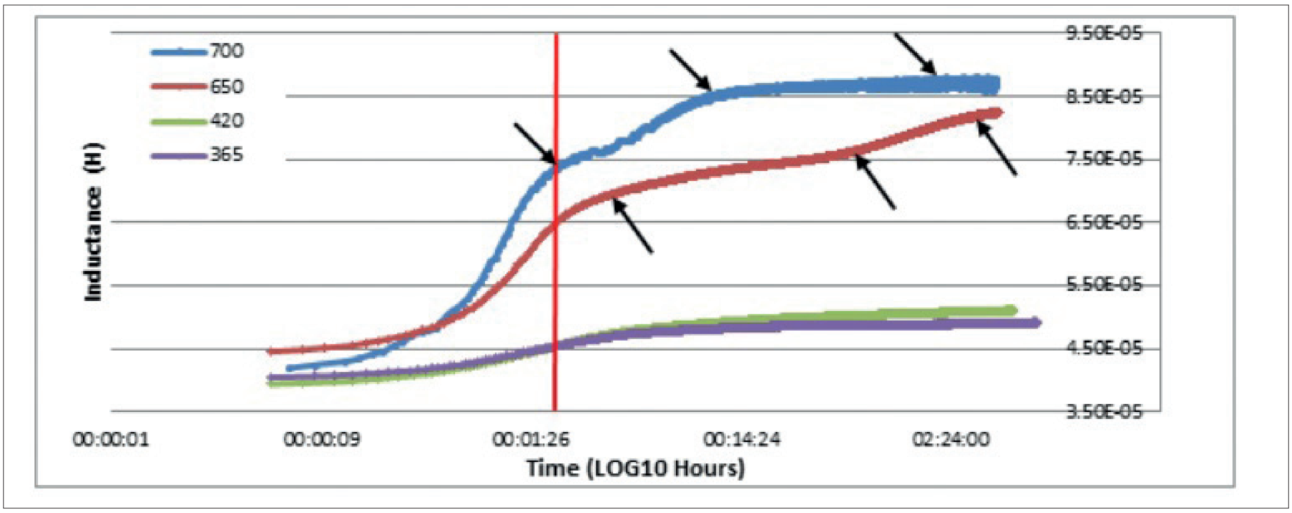

Fig. 3 - In-situ inductance data for 365°C, 420°C, 650°C and 700°C. Time taken for each sample to reach temperature is marked by a vertical line at 2 minutes. Arrows are used to mark gradient key points where different aspects of recovery and recrystallisation are considered to have taken place.

It is clear from figure 3 that the inductance response for each temperature shows different rates of change in inductance with time. The responses at the two lower temperatures show different behaviour to the two higher temperatures. At the lower temperatures only recovery is expected to take place, whilst at the higher temperatures a combined effect of recovery and recrystallisation is expected. Observation of the micrographs from the lower temperature samples, figure 4, shows no discernible change in microstructure after heat treatment from the as received condition, consistent with recovery only. Hardness results (HV5) were 191 and 192 for the 365°C and 420°C samples after annealing respectively, (standard error for measured hardness values is +/- 2.4), which compares to an initial (as-rolled) hardness of 200 HV, i.e. a relatively small change in hardness which is also consistent with recovery only [20]. The observed increase in inductance value with recovery is consistent with reported changes in magnetic measurements (magnetic coercivity and permeability) for samples that have undergone recovery when measured at room temperature [6].

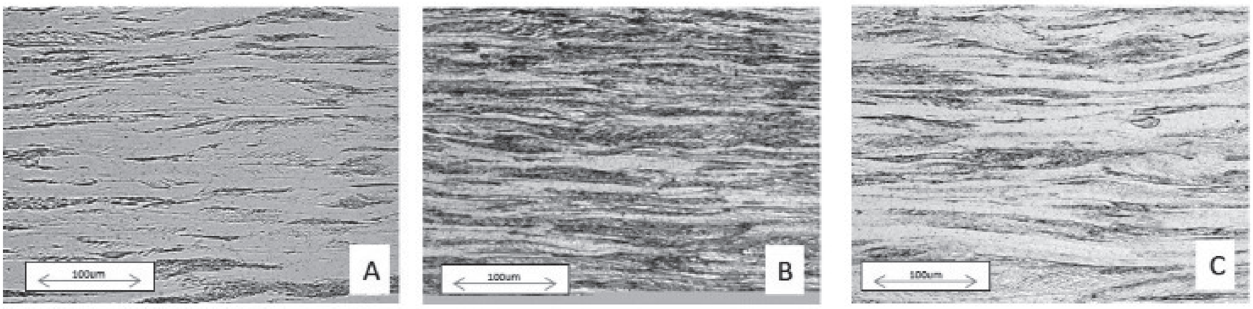

Fig. 4 - Optical micrographs for A: As received B: 365°C after 360 minutes and C: 420°C after 300 minutes. Samples were water quenched at the end of each annealing period. Rolling direction is left to right.

The inductance responses at higher temperatures (650°C and 700°C) in figure 3 display several gradient changes, whereas the samples annealed at lower temperatures (375°C and 420°C) show a steady increase in inductance during annealing, indicative of a single process. In order to determine if the gradient changes in the inductance responses for the higher temperature tests are related to a separation of the recovery and recrystallisation processes, similar to the behaviour associated with a general Avrami multistage process [21], interrupted tests at key points were undertaken. These points are marked in figure 3 for responses at 700°C and 650°C and were selected as transition points (changes in gradient) or final microstructural states. Samples were heat treated for 3 minutes, 40 minutes and 240 minutes at 650°C and for 2 minutes, 10 minutes and 150 minutes at 700°C. Micrographs for the interrupted test samples are shown in figures 5 and 6.

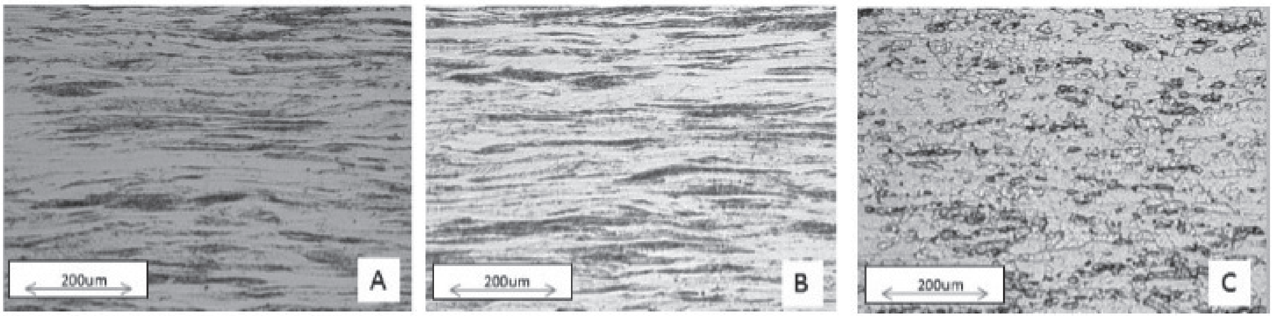

Fig. 5 - Optical micrographs for 650°C interrupted in-situ tests. A: 3 minutes, B: 40 minutes and C: 240 minutes.

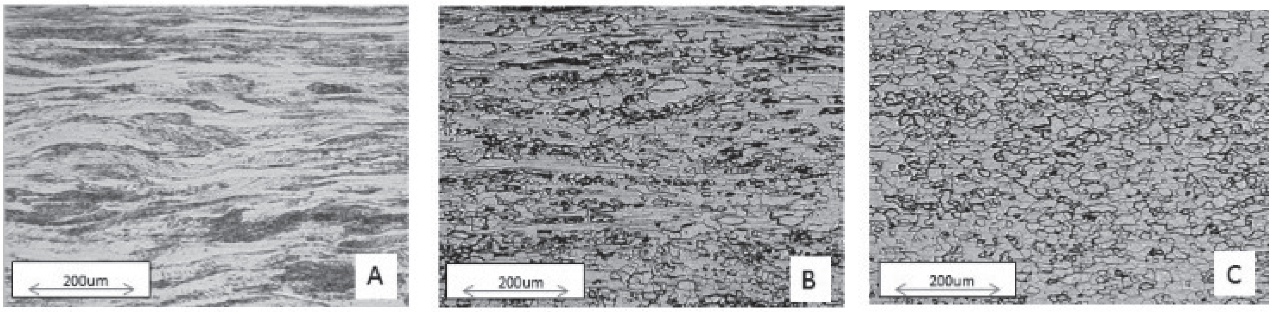

Fig. 6 - Optical micrographs for 700°C interrupted in-situ tests. A: 2 minutes, B: 10 minutes and C: 150 minutes.

Examination of the micrographs in figures 5 and 6 shows that there is no visual change to the as received deformed microstructure after 3 minutes at 650°C or 2 minutes at 700°C. There was very little visible evidence of recrystallisation after 40 minutes at 650°C, however after 10 minutes at 700°C significant recrystallisation can be observed. Visually after 240 minutes at 650°C and 150 minutes at 700°C both samples appear to be fully recrystallised.

Table 1 shows the inductance (room temperature and in-situ as appropriate) and hardness data for the interrupted tests and in-situ tests at 650°C and 700°C. Each of the interrupted in-situ tests shows that as recovery and recrystallisation take place there was a corresponding increase in inductance value and decrease in hardness value. Proportionally the biggest increases in inductance and the smallest decreases in hardness were seen after the shortest annealing times (3 minutes at 650°C and 2 minutes at 700°C). The inverse of this happened after the longest annealing times (240 minutes at 650°C and 150 minutes at 700°C).

Tab. 1 - Room temperature inductance and hardness values for samples heat treated for different times at 365°C, 420°C, 650°C and 700°C and the in-situ inductance values corresponding to the same times during annealing.

Temperature	365°C		420°C	
Time (minutes)	0	360	0	300
Microstructure	As-rolled	Partially Recovered	As-Rolled	Partially Recovered
Room temperature Inductance value (H)	3.8×10^{-5}	4.5×10^{-5}	3.8×10^{-5}	4.7×10^{-5}
In-situ Inductance value (H)	-	4.9×10^{-5}	-	5.1×10^{-5}
Hardness (HV5)	200	191	200	192

Temperature	650°C			
Time (minutes)	0	3	40	240
Microstructure	As-rolled	Partial recovery	Recovered	Recrystallised
Room temperature Inductance (H)	3.8×10^{-5}	4.7×10^{-5}	4.9×10^{-5}	5.2×10^{-5}
In-situ inductance value (H)	-	6.9×10^{-5}	7.6×10^{-5}	8.3×10^{-5}
Hardness (HV5)	200	181	168	84

Temperature	700°C			
Time (minutes)	0	2	10	150
Microstructure	As-rolled	Partial recovery	Partially recrystallised	Recrystallised
Room temperature Inductance value (H)	3.8×10^{-5}	4.7×10^{-5}	5.5×10^{-5}	5.8×10^{-5}
In-situ inductance value (H)	-	7.4×10^{-5}	8.5×10^{-5}	8.7×10^{-5}
Hardness (HV5)	200	181	90	82

After heat treatments of 2 minutes at 700°C and 3 minutes at 650°C the increases in inductance and decreases in hardness are similar, the hardness changes are small and are indicative of the recovery process but not recrystallisation [20], this corresponds with micrographs which show no evidence of recrystallization, Figures 5 and 6. These hardness and inductance value changes for these samples suggest that recovery starts very quickly when the sample is placed into the furnace and, for both samples heat treated at 700°C, some recovery takes place during sample heating as an inductance increase and hardness decrease is seen after 2 minutes when thermocouple readings indicate the sample has reached the furnace temperature. It can be reasonably expected that some recovery may take place during heating for the 650°C tests as well. Inductance values at room temperature for the samples annealed at 365°C and 420°C where only recovery has occurred are 4.5×10^{-5} H and 4.7×10^{-5} H respectively, these are consistent with the inductance values from the recovered samples at 2 or 3 minutes at 700°C and 650°C respectively in the interrupted tests, giving confidence that recovery is being measured. The hardness change for the in-situ recovery samples (365°C and 420°C) are lower than those of the recovered samples in the interrupted tests, this would be expected if the in-situ samples (365°C and 420°C) were only partially recovered. These results indicate that the initial changes in inductance shown in figures 1 and 2 during heating cannot be compared between the as-received and annealed samples for the effect of temperature on inductance as the as-received samples are undergoing some recovery during the heating process.

After 40 minutes at 650°C there is a further increase in inductance and a corresponding small drop in hardness, indicating further recovery has occurred - examination of the corresponding micrograph (figure 5B) suggests that recrystallisation has not occurred. After 10 minutes at 700°C there is a significant decrease in hardness and increase in inductance, the larger decrease in hardness is associated with recrystallisation [20] and correlates well with the significant recrystallisation visible in the related micrograph (figure 6B). For both temperatures, after the longest annealing times (240 minutes at 650°C and 150 minutes at 700°C) recrystallisation is complete, corresponding hardness values are lowest (and similar at 84 and 82 respectively) and inductance values are highest. The fully recrystallized room temperature inductance value for the 700°C sample is greater than that at 650°C (5.8×10^{-5} compared to 5.2×10^{-5} H) and may be due to a larger average grain size in that sample as grain size is also known to affect the permeability values of steel [22]; the average grain diameter for the sample heat treated at 650°C for 240 minutes is 11 µm and the average grain diameter for the sample heat treated at 700°C for 150 minutes is 14 µm. It is also possible that there may be some unrecrystallised areas in the 650°C sample after 240 minutes as figure 5C shows some elongated grains still present in the microstructure, which, whilst not affecting the HV significantly may be having an effect on the inductance value.

After 2 minutes at 650°C and 3 minutes at 700°C both samples show a drop in hardness from 200 HV to 181 HV, with a corresponding increase in inductance at room temperature of 0.9×10^{-5} H. The room temperature inductance difference for the interrupted measurements from 3 minutes to 240 minutes at 650°C is 0.7×10^{-5} H and from 2 minutes to 150 minutes at 700°C is 1.0×10^{-5} H. The inductance differences for the in-situ high temperature measurements, with samples stabilised at furnace temperature, for the same time periods are 1.4×10^{-5} H for 650°C and 1.3×10^{-5} H for 700°C, showing that the inductance response for in-situ measurements is more sensitive to recrystallisation than at room temperature. This sensitivity would be particularly appropriate for sensors operating in a continuous annealing line where both sensor and steel strip are at high temperature.

It can be noted from Table 2 that the in-situ inductance values for the 700°C samples are consistently higher than those at 650°C with a corresponding microstructural state e.g. fully recrystallized or partially recovered. This is due to the higher permeability, and hence higher inductance, expected at a higher temperature. To explore the effect of temperature on inductance for IF steel used in these tests a separate experiment was conducted which recorded temperature and inductance for an already annealed IF steel sample during slow heating at approximately 6°C/minute, Figure 7. Whilst the absolute inductance values cannot be compared because of small differences in the sensor and sample sizes, the effect of temperature on inductance can be clearly seen, and its effect on inductance measurements for monitoring recovery and recrystallisation in situ can be considered. The rate of change of inductance with temperature becomes greater as T_c is neared, then there is a drop off in inductance once the measurement temperature exceeds T_c with the trend shown in Figure. 7 being consistent with that reported for permeability versus temperature in low magnetic fields for pure iron [17]. The percentage change in inductance from start temperature (23°C) to the T_c is 46% with a percentage change in inductance between 650°C and 700°C of 5%. The inductance changes shown for the recrystallization process (after recovery is complete) at 650°C is approximately 8% with the change due to recovery and recrystallization being approximately 20%.

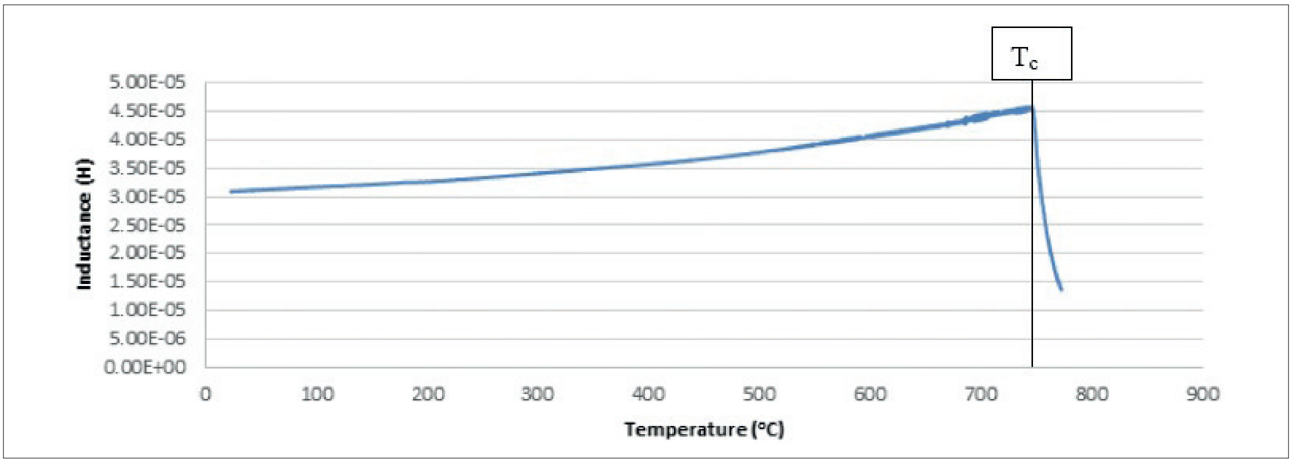

Fig. 7 - Plot showing how inductance increases with temperature for an annealed IF steel sample up to and beyond T_c.

In summary, the in-situ and interrupted measurements both show an increase in inductance for recovery and recrystallisation. The interrupted measurements show smaller changes in inductance for recrystallisation compared to comparatively large changes for recovery, which is different from the hardness response where larger hardness changes are seen for recrystallization compared to recovery. The results indicate that if a number of EM sensors were placed along a continuous annealing line then the progress of recovery and recrystallisation could be mapped and, for an EM sensor located close to the end of the annealing zone confirmation of full recrystallisation could be given. The effect of temperature on inductance measurement should not be ignored as it has been shown that the effect of temperature on inductance measurement is great (Figure 7), therefore calibration and correction for known temperature along the annealing line or measurement at constant temperatures would be required. The presented work shows the potential of the technique for use in an industrial application, but the complexities of the industrial environment mean that issues such as sensor ruggedisation, appropriate sensor design, accounting for different strip types, varying distances from strip to sensor and strip tension would need to be addressed.

CONCLUSIONS

A laboratory based cylindrical EM sensor has been used to detect changes in microstructural state caused by recovery and recrystallisation for IF steel samples for both in-situ measurements at temperatures between 365 – 750 °C and for room temperature tests on heat treated samples. The changes in EM sensor signal (inductance) have been correlated with hardness measurements and optical micrographs. The main conclusions are as follows:

- The inductance values are affected by temperature (as the relative permeability of steel increases as temperature increases) therefore inductance values measured as different temperatures cannot be directly compared as a measure of microstructural state (fraction recovered or recrystallized). However, changes in inductance at a given temperature can be used to follow microstructure changes.
- In-situ inductance measurements allowed the recovery and recrystallization processes to be followed. Clear differences in the rates of recovery and recrystallization could be seen for in-situ measurements at 365°C and 420°C (recovery only) and 650°C and 700°C (recovery and recrystallization). Confirmation of the relationship between inductance changes and recovery and recrystallization was made using optical micrographs for interrupted heat treated samples.
- The change in inductance attributed to recovery is larger than that attributed to recrystallisation for both in-situ and interrupted measurements.

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