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Combinatorial Development and High Throughput Materials Characterisation of Steels

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Abstract

A series of small iron specimens with minor additions of C, Si and Mn were manufactured via induction melting and characterised using a high-throughput methodology. The aim was to analyse the high-throughput approach itself, not the effects of minor additions to steel. Despite their small size the trends in measured standard mechanical properties were consistent with published data and target alloy compositions were achieved to a sufficient degree of accuracy. This is most encouraging as the experimental approaches described here delivered results in a very short time frame, with time per composition estimated to be less than 2 hours per sample. Such an approach would appear to be an excellent precursor to more traditional, expensive and time-consuming alloy development methods used by industry. Limitations of the methodology are described and key bottlenecks identified. However, the use of small specimens to quantify trends in properties of steels and identify possible new alloys is potentially a valuable addition to the development of new steels.

Keywords

Rapid Alloy Prototyping; Combinatorial Metallurgy Steels; High-throughput

1 Introduction

Combinatorial approaches to developing new alloys are not new, but have been attracting renewed interest in recent years through projects such as Accelerated Metallurgy in Europe, [1], the Materials Genome Initiative in the USA, see for example [2–4], and most recently through pan-European projects such as Metallurgy Europe, [5].

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Traditionally, experimental methods to establish phase diagrams for new alloys have consisted of a wide range of techniques, from diffusion based methods such as multiple point junctions, powder metallurgical routes such as hot isostatic pressing and mechanical blending, to melting with induction heating and lasers, and more recently chemical vapour deposition, [6,7]. Equilibrium based phase diagrams for binary and ternary systems are then established using a combination of differential calorimetry to establish the thermodynamics, optical and scanning electron microscopy to establish composition and microstructure, possibly accompanied by x-ray diffraction (XRD) analysis to verify phases and phase fractions.

Achieving equilibrium is a key part to the classical development of phase diagrams, and systems are typically held for long periods in vacuum furnaces to homogenise alloys. Whilst this provides understanding of systems and better matches to thermodynamic software, it does not bear resemblance to alloys in real manufacturing production which are rarely in equilibrium, have many more elemental components and higher levels of up to 1-2% of impurities. Considering the detrimental effect on the electrical conductivity of copper of very small impurities can only hint at the combined effect of multiple impurities.

Recent efforts at the Max Plank Institute have adopted combinatorial-type studies on Martensitic stainless steels, [8] and lightweight Transformation and Twinning Induced Plasticity (TRIP/TWIP) steels with high Mn and Al, ranging from low carbon compositions such as SIMPLEX, [9] (<0.3%wt.) to high carbon (0.7 to 1.2 wt. %C) variants such as TRIPLEX, [10]. These combinatorial studies have a limited range of composition intervals, possibly determined by the minimum sizes of the hot rolling or vacuum induction melting equipment which at 1kg per sample are not insignificant. However they have succeeded in examining the complex interactions between steel composition, microstructure and properties as affected by heat treatment and deformation processes such as hot rolling, [11].

In the work being described in this paper, a smaller sample size is being used and although in early stages of development, as-cast and heat treated samples can be screened using relatively high-throughput hardness testing and punch disk testing rigs. In the future, it is expected that hot and cold rolling will also be evaluated to work on the same size samples, which should greatly accelerate the traditional steel development route in which up to 80kg ingots are required.

2 Combinatorial Methods, Design of Experiments and Data Reduction

When adopting combinatorial methods it is necessary to consider the number of samples required for a particular testing campaign. Following work done for the synthesis of catalysts, such as [12], then for N_E elements chosen in N_M different ways in which order is irrelevant and there is no repetition, the number of combinations possible is given by:

$$\binom{\boldsymbol{N_E}}{\boldsymbol{N_M}} = \frac{\boldsymbol{N_E}!}{\boldsymbol{N_M}!(\boldsymbol{N_E}-\boldsymbol{N_M}!)} \qquad \textbf{Equation 1}$$

To experimentally look across the compositional space a discrete set of intervals is needed between 0% and 100%, labelling the number of intervals as N_c, the number of samples required to analyse the compositional space using a full factorial approach would be:

$$\binom{\boldsymbol{N_c}+\boldsymbol{N_M}-\mathbf{2}}{\boldsymbol{N_c}-\mathbf{1}} = \frac{(\boldsymbol{N_c}+\boldsymbol{N_M}-\mathbf{2})!}{(\boldsymbol{N_c}-\mathbf{1})!(\boldsymbol{N_M}-\mathbf{1}!)} \qquad \textbf{Equation 2}$$

The way in which the number of samples increases for a variety of weight percentage intervals and number of elements, is given in Table 1. It is clear even for a relatively small alloying range and coarse interval spacing that the full factorial experimental approach quickly becomes quite unwieldy in terms of the number of samples which would need to be screened. Even for applications such as catalyst, photo-voltaic and thermoelectric materials where an almost continuous range of compositions can be rapidly synthesised (e.g. by chemical vapour deposition) sometimes coupled with rapid property screening (e.g. IR thermography screening of catalysts), the quantity of data-handling would create a bottleneck at the analytical stage – as stated in [12,13].

To this extent, Taguchi orthogonal arrays have been considered as a means of reducing the number of experiments for a variety of systems, such as magnesium alloys, [14], nickel superalloys, [15], high entropy alloys, [16], lead-free solders, [17] and thermo-electrics, [18]. If each composition is assumed be treated as an independent parameter an L25 array would reduce the number of samples required from 125 to 25, and an L9 array would reduce the same system to 9. However, the Taguchi methodology relies on the application of essentially linear statistical analysis to interpolate over wide parameter ranges in which the compositional effect is far from linear for properties such as hardness and tensile strength due to multiple phases that can form during solidification of many alloys.

3 Experimental Methods

Several experiments are now described including details of the different stages necessary and importantly, for high-throughput methodologies, the timings necessary to complete each stage.

3.1 Experiment 1: Effect of minor additions of Si and Mn on pure steel Fe-C

The purpose of this experiment was to use a combinatorial approach to select a compositional range of binary, ternary and quaternary compositions of high purity Fe-C alloyed with Mn and Si, and to determine the relative importance of the composition to properties which can be measured in a high-throughput manner, such as hardness and tensile properties. The following matrix of alloys was selected:

- Batch 1: Fe-C [0,0.1,0.5,0.75,1 wt%] – number of samples = 5^1 = 8 samples made
- Batch 2: Fe-C [0-1 wt%]-Si[0-1 wt%] – 5^2, 16 samples made + batch 1
- Batch 3: Fe-C [0-1 wt%]-Mn[0-1 wt%] – 5^2, 16 samples made + batch 1
- Batch 4: Fe-C (0-1 wt%)-Si(0-1 wt%)-Mn(0-1 wt%) – 5^3=16 samples made + batches 1-3

Throughout this work composition is presented in weight percentage and Fe is always determined by the balance with C, Si and Mn. Thus, a total of 60 samples were made as shown in Figure 1, which is much less than the 210 required for a full factorial experimental coverage of the compositions, but is more than would be advocated by a typical Taguchi orthogonal array. There were no repetitions of samples.

Stage 1 - Weighing the charges (10 hours)

Elemental metal powders of various purities (450 µm Fe 99%, 75 µm C 99.997%, 150 µm Si 97.5%, and 150 µm Mn 99.5%) were obtained from Goodfellows. The elemental powders were weighed out on a high precision balance (OHAUS Pioneer PA214CM) and blended to a total of 15g in the right stoichiometric mix. Based upon a 1mg accuracy of the balances, it was determined that the error in composition due to the weighing balance would only be of the order of 0.0067%, even for the lower 0.1 wt%. It was estimated that weighing the 60 samples took a total of 10 hours.

Stage 2 - Sample preparation and synthesis (15 hours)

The blended powders were dry compacted using a 25kN Tinius-Olson compression testing rig using a 20kN force, sufficient to make green pellets (12mm diameter by 20mm length) which could be manually handled, as shown in Figure 2 (b). The pellets were then loaded into a 30mmX12mm diameter alumina crucible as shown Figure 2 (c), and melted by induction in an argon atmosphere with a very low (1-10ppm) oxygen content to produce 12mm diameter solid samples of about 15mm height, as shown in Figure 2 (d). It was estimated that the samples took approximately 5 minutes to press and 10 minutes to melt, which meant that the total time for this stage of 15 hours.

Stage 3 - Sample homogenisation and metallographic preparation (29 hours)

The samples were cut in half and one half was homogenised in a vacuum furnace for 24 hours at 1200°C, and left to cool in the furnace, as suggested in [19,20].

Each half sample (the as-cast and the heat treated) was then sectioned into disks for hardness testing, punch disk tests and microscopy, as shown in Figure 2 (e) and (f). Porosity was, on the whole, not apparent except in some samples which had not melted properly and were discarded. The heat treatment took 24 hours for all 60 samples, and the sectioning took roughly 5 minutes per sample so this stage took 29 hours.

Stage 4 - Sample high-throughput hardness testing (10 hours)

Vickers hardness was obtained on a Buehler Wilson 3300 using 0.3kg load (0.3HV) which allows for automated testing of multiple mounted samples. Each sample (HV was taken as the average of a 4X4 matrix with 0.2mm spacing) took roughly 5 minutes to complete the testing. Ternary maps of hardness are shown in Figure 3, where it can be seen that the hardness increases with carbon content, as well as with the content of silicon and manganese, as would be expected.

Traditionally in steels, particularly to ascertain the strength of welds where composition is important to the weld quality, [21], the components are lumped into a carbon equivalent (CE) content for which there are a number of variants depending on the primary carbon, as derived from regression analysis of empirical data. A traditional CE as advocated by the American Welding Society, [22], is given by:

$$\mathbf{CE} = \boldsymbol{C}\% + \left(\frac{\boldsymbol{Si}\%}{\mathbf{6}} + \frac{\boldsymbol{Mn}\%}{\mathbf{6}}\right) + \left(\frac{\boldsymbol{Cr}\%}{\mathbf{5}} + \frac{\boldsymbol{Mo}\%}{\mathbf{5}} + \frac{\boldsymbol{V}\%}{\mathbf{5}}\right) + \left(\frac{\boldsymbol{Ni}\%}{\mathbf{15}} + \frac{\boldsymbol{Cu}\%}{\mathbf{15}}\right) \qquad \textbf{Equation 3}$$

More sophisticated approaches, [21], take into account the cooling rates and the retained volume fractions of Bainite, Martensite and Ferrite-Pearlite in the microstructure, however, for the purpose of a quick comparison equation 3 was deemed good enough, and certainly, as can be seen in Figure 4 (a), the hardness data fits relatively well on the straight line defined by this carbon equivalent correlation, and it can be seen that net effect of the homogenisation heat treatment is to soften the samples, as would be expected. It was estimated that this stage took roughly 5 hours for each of the entire as-cast and heat treated batches.

Stage 5 - Sample high-throughput Punch Disk Testing (20 hours)

As mentioned previously, 1mm thick disks were sliced from both as-cast and heat treated samples using a precision diamond saw to obtain one sample from each half. Punch disk data was obtained for the disks using a jig similar to that previously described, [23]. A typical set of punch disk curves are shown in Figure 4 (b) for a variety of compositions (the same samples A-E for which SEM is done in the next stage), highlighting the differences between these curves and standard uniaxial tensile test, the curves show a clear set of regions, elastic bending, yield, plastic bending, membrane stretching and necking to failure. The punch disk test is convenient as it uses relatively little material and can be done quickly. The increasing ultimate force to failure and decreasing ductility is clear from Figure 4 (b).

From the ultimate force measured on the punch disk test, two equations were used to predict the upper tensile strength, [23], depending on whether the material broke in a ductile manner:

$$\boldsymbol{\sigma_{UTS}} = \frac{\boldsymbol{L_U}}{\boldsymbol{t(2.32D - 0.9Cl + 0.56)}} \qquad \textbf{Equation 4}$$

Or in a brittle manner:

$$\boldsymbol{\sigma_{UTS}} = \frac{\boldsymbol{L_U}}{\boldsymbol{t(0.14D - 0.28Cl + 2.17d_F + 0.6)}} \qquad \textbf{Equation 5}$$

Where, $\boldsymbol{\sigma_{UTS}}$ is the upper tensile strength in MNm^{-2}, L_u is the measured force in N, and the thickness t, punch diameter D and die clearance Cl are all in mm. The term d_F is the displacement at failure in mm, and relates to the reduced contact of the punch with the disk for brittle materials at the failure load.

For the heat treated samples, the UTS predicted from the punch disk test is plotted against the equivalent carbon content in Figure 4 (c), and follows a characteristic trend where the tensile strength with higher carbon content tapers off and the alloy becomes more brittle. In this figure the relative size of the markers is scaled by the Mn+Si content, so it is also worth noting that the relative effect on UTS of the Mn and Si additions is a lot more significant in the higher carbon samples.

The displacement of the punch was taken to be an indicator of elongation (as there is not a straight forward way of linking this to elongation measured in a tensile test). The "elongation" and UTS are plotted against each other for the heat treated samples as shown in Figure 4 (d), and it is clear that there are a group of low carbon, high Mn + Si samples which have the highest "elongation" and UTS, and these are circled on the figure.

It was estimated that each punch disk test took roughly 10 minutes, so the total time for the punch disk testing was 10 hours for each of the as-cast and heat treated batches.

Stage 6 - Sample microscopy and compositional analysis (5 hours)

Mounting, grinding/polishing and etching took about 20 minutes per sample, but SEM-EDS was done on a Jeol JSM-6010Plus-LV and a quick examination can take 40-60 minutes per sample. Thus, only 5 of the 60 samples were examined by SEM – but these were chosen to be representative samples with carbon content at (A) C=0%, (B) C=0.1%, (C) C=0.5%, (D) C=0.75% and (E) C=1%, all in wt.%, with a variety of manganese and silicon content as given in Figure 5 (F).

Without extensive characterisation, the microstructures presented in figure 5 provide useful information for the purposes of validation of the property – composition correlations. The trends in carbon content are reflected in the micrographs starting with no second phase, 100% ferrite matrix in sample A and increasing proportions of second phase pearlite at the expense of the matrix in samples C, D and E (which as a hypereutectoid alloy and hence contains primary cementite). The character of the matrix itself also appears to have been affected with martensitic matrix in the presence of elevated manganese and carbon levels as might reasonably be expected. This is reflected in increased strengthening at the expense of ductility where carbon levels are high.

The EDS measurements of Fe, Mn and Si are compared to the input elements, and within the known limitation of EDS precision of 0.1%, the trends are encouraging. The total time to prepare the 5 samples was about 5 hours.

3.2 Experiment 2: First trials on direct mixing of known steel compositions

In this second experiment on synthesising steels, 3 known steel compositions, namely an Ultra-Low Carbon ULC electrical steel (Sample 4074), a boron steel (sample 4950) and a high carbon steel (4928) were put together in a similar manner to that described in the previous section, but these samples were not homogenised. In addition to the elemental powder used in the previous section (Fe, C, Mn and Si), additional micro-alloying amounts of Al, B, Cr, Co, Cu, and Nb were added. The target compositions, the precursor elemental blend and the EDS characterisation are shown in Table 2. As with the previous experiment, EDS is not really capturing the lighter elements which are known to be in the alloys, so a more appropriate method of checking the composition is needed, however some of the added elements are in the target range. Nevertheless, the measured hardness in Table 3 follows reasonable trends, and the microstructures of the "synthetic steels" shown in Figure 6 appear very similar to those found in other publications.

4 Results and Discussion

In the case of the range of carbon content (0-1wt.%) selected for the first experiment which was done over the period of 2-3 weeks, it is obvious that the carbon content dominates the hardness for both the as-cast as well as the heat treated samples as would have been expected. The results in this paper are consistent with the fundamental relationship between properties and composition of steels, and indeed deeper analysis of the samples supported by XRD or TEM might be of interest due to the high purity of the elemental precursors.

The main outcome from the second experiment is that the addition of further micro-alloying elements presented no problem to the synthesis step, and could be done by directly mixing the elements without the need to resorting to master-alloys. This now leads to the application of the full process developed and described in the first experiment to a variety of specifically targeted full steel compositions.

Overall this study highlights some of the shortcomings of the combinatorial methodology adopted such as:

- The challenges associated in handling a large number of samples and properties data which can be generated over a short period of time and the visualisation and interpretation of the data generated.
- The compositional analysis is certainly one of the weakest links in the analysis, as the preparation and examination by EDS in the SEM is relatively slow. It is obvious that many of the light elements at small alloying percentages are a particular challenge, and the carbon content is known to be unreliable because of the metallurgical preparation. This might be less of a problem for alloys with wider and higher alloying ranges. A faster and more reliable way to quickly check the sample composition would be to use a multi-

sampler XRF, however a more precise method such as OES or wet analysis is needed to verify that the compositions are indeed as intended.

- The punch disk test also has a number of shortcomings which need to be addressed, not least of all that the force to stress conversion equations used here were developed for a completely different class of steels. However, the geometry of the test may in fact be a more suitable comparative test of formability than tensile testing due to the similarities with cupping and deep draw cup tests.
- The testing for this work would have benefited from a greater degree of repetition, particularly for the punch disk test, and this will be done in the future. The study needs to be extended into the applicability of Taguchi and Analysis of Variances (ANOVA) to the wider compositional ranges.
- Some experimental thermodynamic and XRD measurements have been undertaken on a small number of pure Fe-C samples but not reported in this paper. These show that the phase changes, liquidus and solidus are as expected from the phase diagram, but these results will be reported when a deeper study has been undertaken.

Whilst the study has yielded some interesting results, the emphasis has not been in verifying property data, but on the speed in which the compositional-property relationships can be established. The 2-3 week time frame for this analysis (60 compositions and one HT) works out at just under 2 hours per sample, and compares very favourably with 7h per sample estimated in [10] when looking at 5 compositions, albeit with rolling and 2-3 heat treatments. Both these accelerated compositional studies are in turn estimated to be 6-9 times faster than traditional methods of characterisation.

This work will pave the way for subsequent experimentation with a higher order of elemental additions in terms of understanding the limitations of compositional discretisation, handling of data and time bottlenecks when screening via high throughput property characterisation. One of the most important future steps to be addressed is how the hot rolling stage which plays such an important role in the properties of steel can be reproduced in the laboratory, and specifically in the context of a high-throughput methodology.

5 Conclusions

Summarising the discussion in the previous section:

- A high-throughput synthesis route for the production of small steel samples has been presented and shown to be fast and a viable route for rapid prototyping of new steel compositions.
- Hardness and tensile properties are derived in a high-throughput manner and the property/composition relationships for steel compositions with small micro-alloying additions of 0-1% C, Mn and Si are shown to follow expected trends.
- Pros and cons have been discussed of the high-throughput methodology with respect to the traditional slower route, with the next significant step being the incorporation of representative process heat treatment and laboratory based hot and cold rolling.

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Table 1 – Number of samples required in a full factorial sampling of NM elements over NC interval points

Wt.% interval	N_c	$N_M = 2$	$N_M = 3$	$N_M = 4$	$N_M = 5$	$N_M = 6$
20%	6	6	21	56	126	252
10%	11	11	66	**286**	1001	3003
5%	21	21	231	1771	10626	53130
2%	51	51	1326	23426	316251	3478761
1%	101	101	5151	176851	4598126	96560646

Table 2 – Target compositions of selected TATA steels (full composition)

	4074 (EDS)	**4074 (Stoich.)**	**4074 (Target)**	**4950 (EDS)**	**4950 (Stoich.)**	**4950 (Target)**	**4928 (EDS)**	**4928 (Stoich.)**	**4928 (Target)**
Al	0.08	0.5	0.3-0.5	0.03	0.045	0.02-0.045		0.055	0-0.055
B				Nd	0.005	0.005			
Si	2.45	3.15	2.9-3.15	0.33	0.25	0.15-0.25	0.32	0.35	0.15-0.35
Cr	0.12	0.07	0.07	0.16	0.35	0.15-0.35	0.41	0.4	0-0.4
Mn	0.46	0.25	0.15-0.25	0.72	1.3	1.1-1.3	0.63	0.9	0.6-0.9
Fe	96.42	95.8	95.8-96.65	98.66	97.64	97.64-98.37	98.58	97.31	97.31-98.55
Co	Nd.	0.02	0	Nd.	0.02	0-0.02	Nd.	0.02	0-0.02
Cu	0.48	0.2	0-0.2	0.1	0.15	0-0.15	0.06	0.15	0-0.15
Nb	Nd.		0	0.01	0.01	0-0.01	Nd.	0.01	0-0.01
C					0.23	0.2-0.23		0.8	0.7-0.8

Table 3 – Hardness of the selected TATA steels (full composition)

Sample	Mean Hardness (HV0.3)	StDv	Max	Min	Range
4074	204.45	9.78	218.37	187.28	31.1
4590	147.36	7.8	158.51	127.28	31.23
4928	264.42	15.93	305.81	240.42	65.39

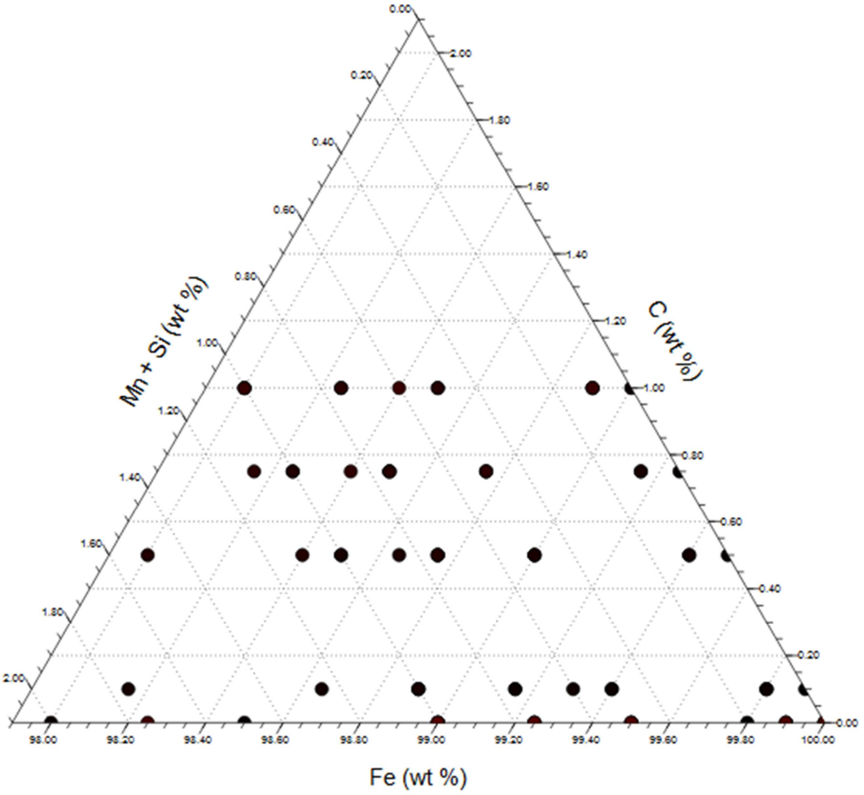


Figure 1 – 37 of the 60 compositional combinations synthesised for experiment 1

(a) (b) (c) (d) (e)

Figure 2 - Manufacturing of steel samples (a) 15g of blended elemental powder, (b) 20mmx12mm diameter green compact (c) 15mmx12mm diameter steel ingot (d) 1mmx12mm slice for Punch Disk Test (e) mounted sample for SEM microscopy

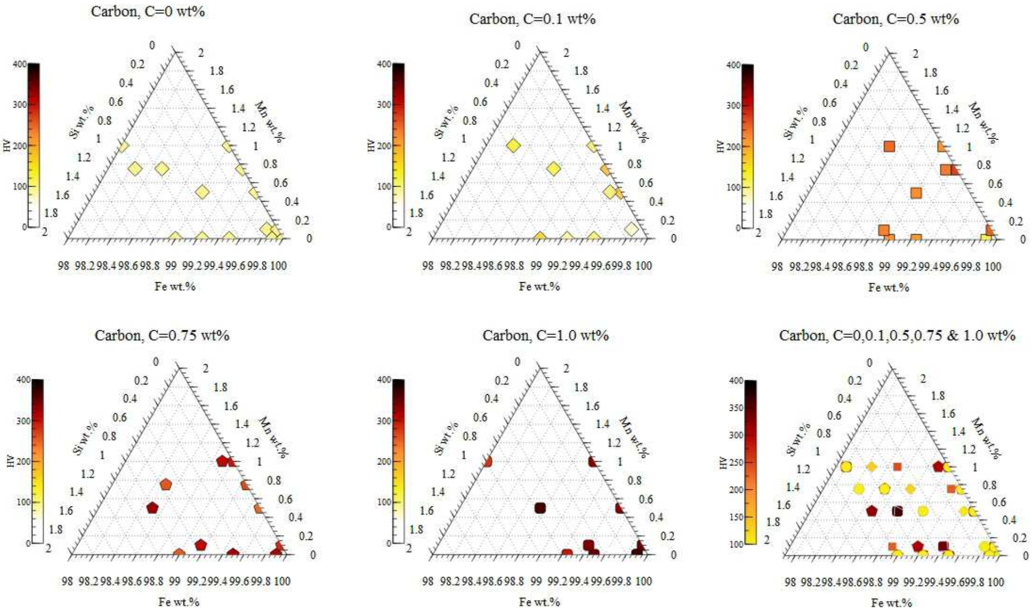
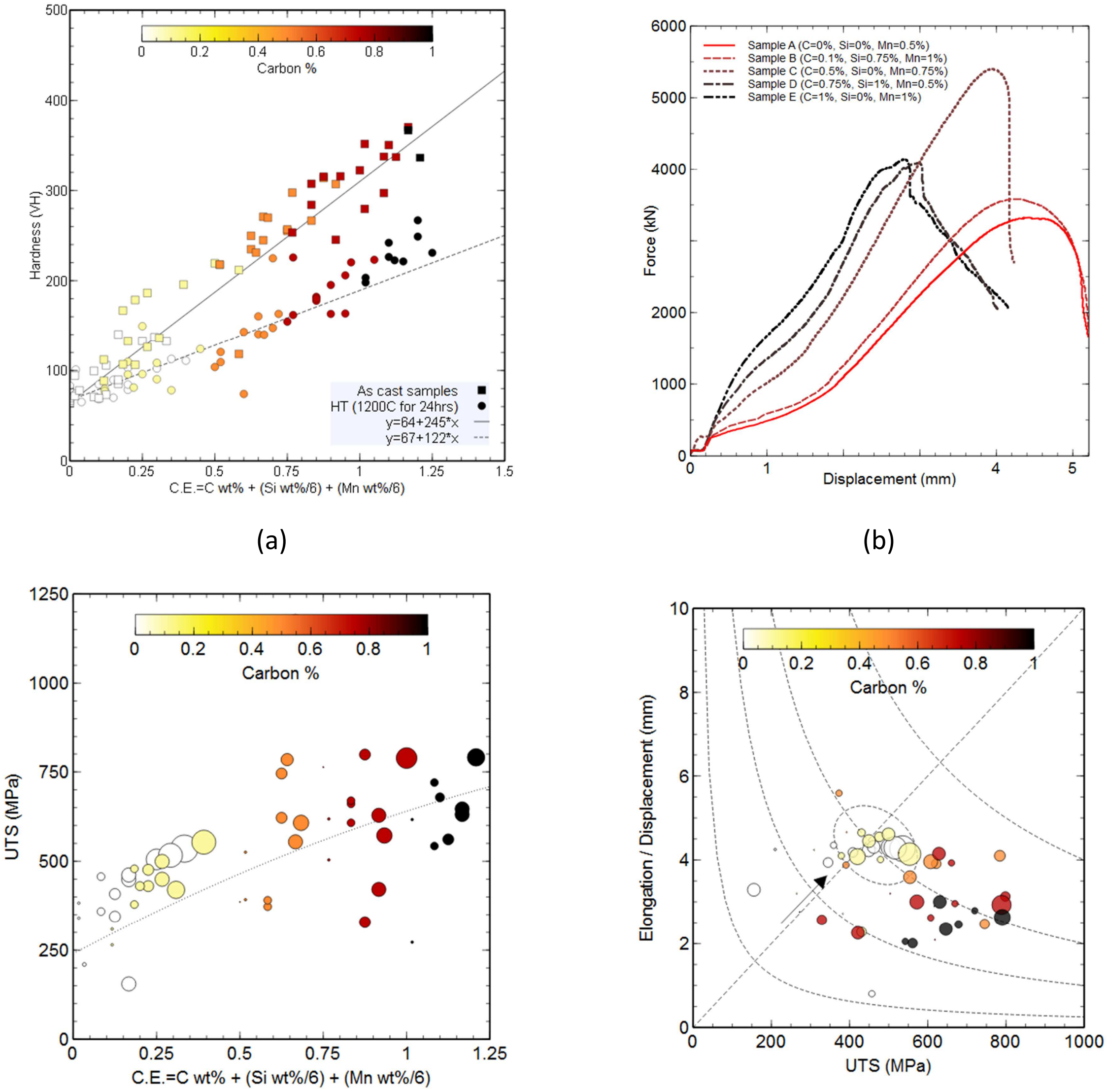


Figure 3 – Ternary hardness maps for the as-cast samples



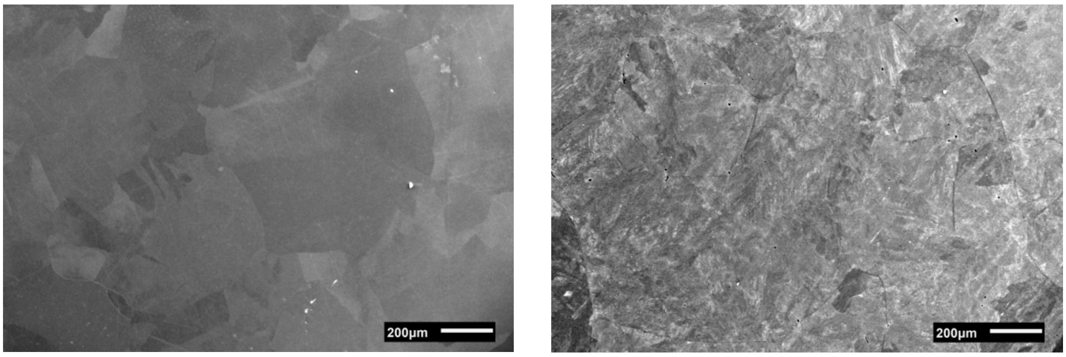
(a)

(b)

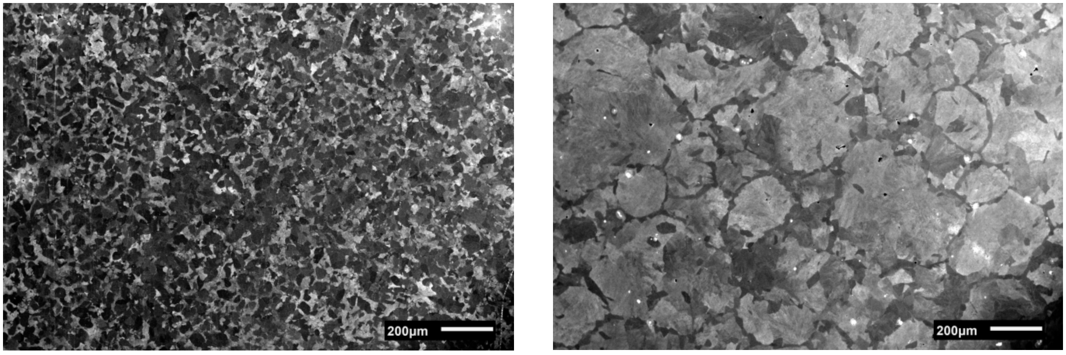
(c)

(d)

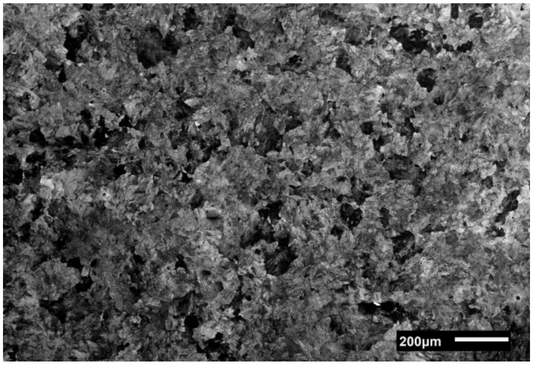
Figure 4 – For homogenised samples (a) Hardness as a function of carbon equivalent content, (b) UTS for as a function of carbon equivalent content, (c) UTS vs "elongation" and (d) typical punch disk test curves for a number of different compositions



A (Carbon = 0%, Si=0%, Mn=0.5%) B (Carbon = 0.1%, Si=0.75%, Mn=1.0%)



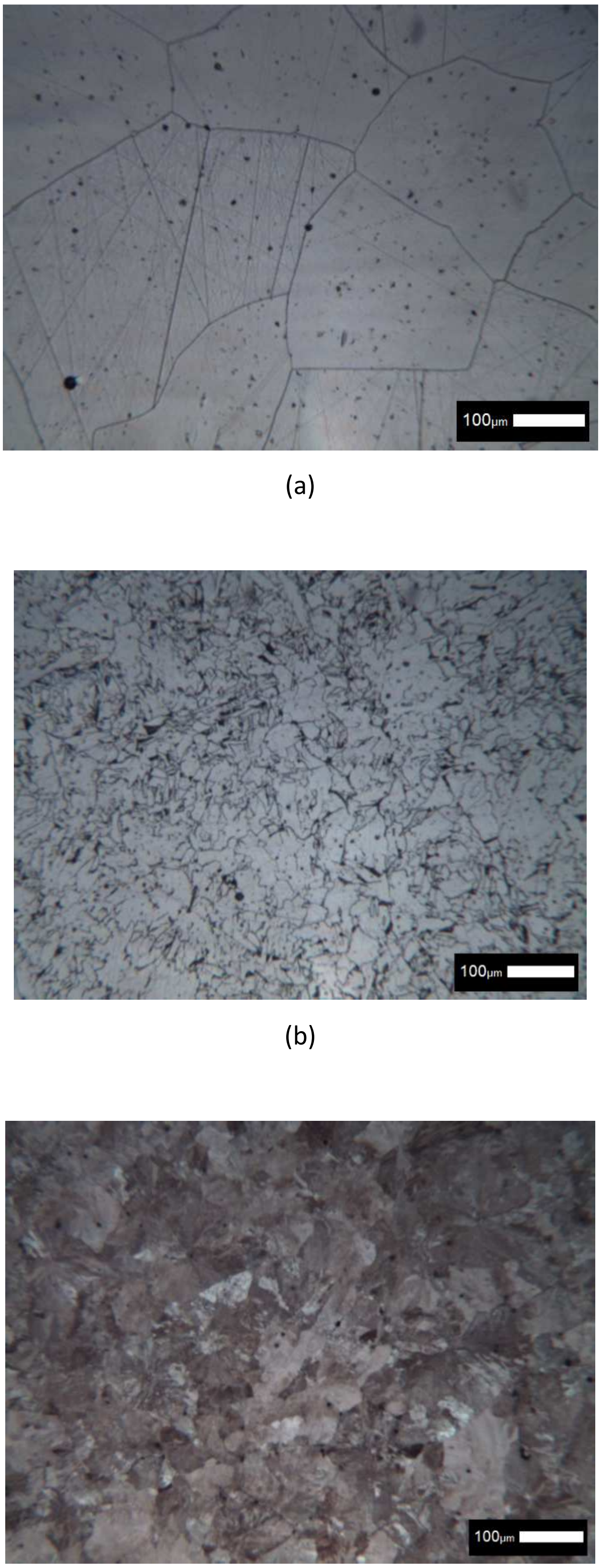
C (Carbon = 0.5%, Si=0%, Mn=0.75%) D (Carbon = 0.75%, Si=1%, Mn=0.5%)



	Fe	Fe (EDS)	Si	Si (EDS)	Mn	Mn (EDS)
A	99.5	99.8	0	0	0.5	0.2
B	98.15	99.69	0.75	0.02	1.0	0.29
C	98.75	99.16	0	0.02	0.75	0.84
D	97.75	98.60	1	0.72	0.5	0.68
E	98	99.01	0	0.11	1	0.88

E (Carbon = 1.0%, Si=0%, Mn=1.0%) F Compositions of samples A-E

Figure 5 – Microscopy of selected compositions spanning the carbon range C=0 (A), 0.1(B), 0.5(C), 0.75(D) and 1 (E) wt%



(a)

(b)

(c)

Figure 6 – Synthetic as-cast samples of the (a) ULC Electrical Steel (4074), (b) Boron Steel (4950) and (c) High Carbon Steel (4928) compositions.