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Nanostructural Engineering of Steel

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Abstract

The concept of microstructural engineering of steels is well established and is the basis for a wide range of processes and products. Recently, though, there has been increasing emphasis in developing microstructures that have submicron length scales and also in understanding in far greater detail the structures and solute effects that are occurring at the nanoscale. In this review it is proposed that we are heading towards the situation where we are applying nanostructural engineering concepts in the development of new steels. A range of examples are given as well as a discussion of the potential impact of new processing routes. Clearly the future advances will be driven through improved characterization methods, such as atom probe tomography, in combination with advanced modeling.

Keywords: *Advanced High Strength Steels, Ultrafine Ferrite, Precipitation, Atom Probe Tomography.*

INTRODUCTION

Steel is one of the most engineering materials in the World. Through alloy and process control it is possible to obtain strengths ranging for just over 100 MPa to over 2 GPa. The texture can be controlled to produce r -values of over 2.5 allowing the drawing and forming of a range of complex shapes. Also the toughness and weldability of structural steels have been remarkably improved over the past 3 decades opening up new oil and gas opportunities. Nearly all of these advances have occurred by the control of the microstructure evolution during processing; sometimes even from the start of solidification.

The most widely practised approach to microstructure control is Thermomechanical Controlled Processing (TMCP). This is based on microstructure control during hot rolling and subsequent cooling. Many of the

microstructure events are controlled at the micron level, though others such as precipitation hardening are clearly at the nanoscale level of control. Therefore, these and other processes are normally termed microstructural control, or engineering.

The hype associated with nanotechnology and the worldwide trend for government agencies to focus research funding into areas such as this has led to a quite significant change in the nature of research being pursued by many in the materials and metallurgy fields. Needless to say there has been a certain degree of scepticism as many feel steel and other metals are already using nanoscale structures; what indeed is pearlite if not a self assembled nanostructure! However, in reality most approaches to steel have largely been at the micron scale and this is largely to do with our ability to access information at the

nanoscale and then use this to further refine the alloy.

With improved characterisation methods and enhancements in modelling though, there is now greater scope to manipulate the microstructure at the nanoscale, observe the outcomes and then manipulate the composition and process to give the desired outcomes. This paper reviews some areas where we and others are exploring fine scale microstructure evolution. Much of this work is really between the microscale and nanoscale but there is no doubt that as characterisation techniques continue to improve, we will see a greater emphasis on manipulating grain boundaries, internal dislocation structures, fine scale precipitates and solutes as well as attaining greater understanding of very local composition profiles and their effects on properties.

An accompanying paper [1] also covers aspects of this work as it relates to the application of Atom Probe Tomography (APT) which has provided very unique insights into the nanostructure of steels.

ULTRAFINE GRAINED STEELS

Dynamic Strain Induced Transformation

Over the past decade this has been probably the research area with the greatest level of activity other than advanced high strength steels which will be discussed later. The impetus arose from a major initiative in Japan to develop step changes in strength, corrosion resistance and other properties in steels with as little use of alloying as possible. The initial objective in the strengthening of simple C-Mn steels was to reduce the ferrite grain size to 1 μm or lower as the Hall-Petch relationship for steels predicts that this could more than double the yield strength.

A number of groups [2–7], including some of the present authors [8–10], have achieved this by a range of processes from relatively simple thermomechanical processing to very complex multistage operations. The simplest and most effective method is to roll between the A_{e3} and A_{r3} and to activate transformation during deformation. This dynamic transformation process has been given a range of names. The present authors have clearly shown [10] that to obtain a uniformly fine grain size at room temperature requires a critical volume fraction to be formed during deformation (Figure 1). Some aspects of the process are still not completely resolved. For example, ferrite that is formed at lower strains does not appear to coarsen during further deformation; whereas if the deformation is stopped this ferrite will significantly coarsen. This suggests dynamic recrystallisation is occurring in the ferrite, although there is little evidence of nucleation [11–12].

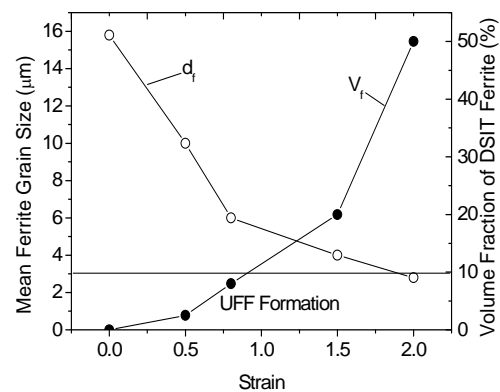


Figure 1: Grain size and volume fraction of DSIT ferrite as a function of strain for C-Mn steel

One of the more interesting results is that composition has relatively little effect on the ferrite grain size formed by strain induced transformation [13]. In fact even very high C steels can be made to form ferrite + cementite

as dispersed particles rather than pearlite (Figure 2). Here the ferrite grain size is much less than $1\mu\text{m}$ and the carbides are extremely fine. This type of microstructure is more typical of a spheroidised pearlitic steel and there has been some interest in using strain induced transformation in high C steels to remove the heat treatments required to form this type of structure in conventional processing.

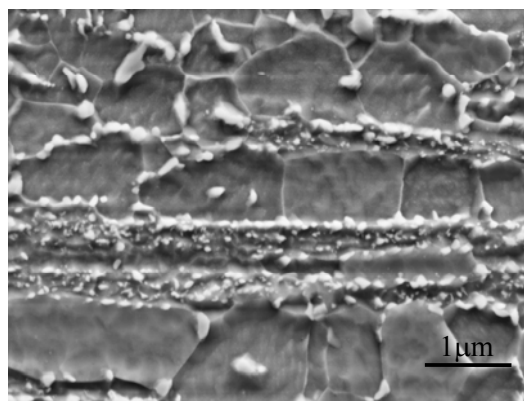


Figure 2: UFF structure formed in 0.4 wt% C steel

The difficulty with all of these processes is that they require a large strain and accurate control of the final deformation temperature. There have been some attempts to develop multipass processes where the strain is built up over a number of deformations but it still requires quite high levels of deformation [14]. Japanese researchers have recently been modifying rolling processes so that they are designed to promote this type of transformation; a key aspect of this is also to have intense cooling immediately after the last pass so that the steel is rapidly cooled before ferrite coarsening can occur and then the high level of undercooling will promote further austenite to ferrite transformation with a refined structure [15].

Other Approaches

Much higher levels of grain refinement have been achieved in steels using accumulative roll bonding [16], ball milling [17], high pressure torsion [18] and equal angle channel extrusion [19]. These are all termed severe plastic deformation (SPD) approaches and can give exceptionally high strength levels. The grain size can be reduced to 100–200 nm. All of these processes are extremely difficult to implement commercially but they have provided interesting starting points for more fundamental studies into deformation and strengthening mechanisms in ultrafine microstructures.

Two other approaches are being considered in our group. One is based on the cold deformation of an initially martensitic microstructure and the other is transformation to very fine bainite. The latter is based on the pioneering research of Bhadeshia and co-workers at Cambridge University [20].

The cold rolling and annealing of martensite has already been used to generate nanostructured steels [21]. In the present work a low C Nb microalloyed steel was used and after the formation of martensite different tempering conditions were used prior to 50% cold rolling. This was then followed by a low temperature anneal at 550°C for various times. One of the difficulties when cold rolling martensite is that there is a large degree of heterogeneity in the deformation of individual grains and this is then reflected in the subsequent recovery and recrystallisation behaviour. Some regions will show strong cell formation or even recrystallisation nuclei while other regions still have the original cold worked structure. The strength levels and total elongation were from 850 to 950 MPa and 12–13%, respectively, while the microstructure

showed a wide range of deformation and annealing features (Figure 3).

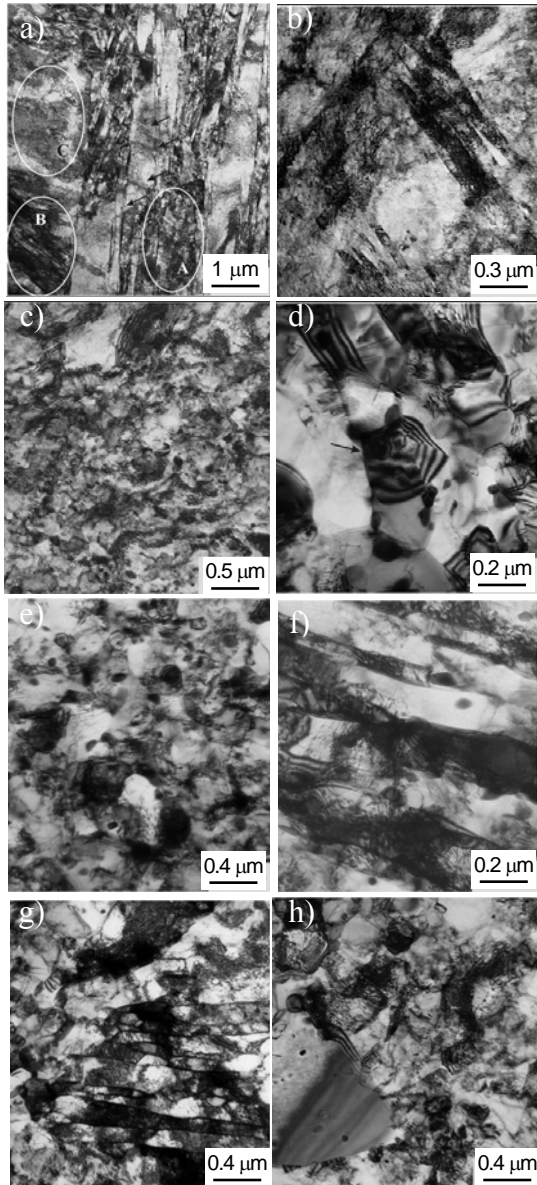


Figure 3: TEM micrographs of the 350 steel: a, b) cold rolled martensite; c, d) cold rolled martensite after annealing at 550°C for 300s; e, f) cold rolled martensite after annealing at 550°C for 30 min; g, h) cold rolled martensite after annealing at 550°C for 2h

The as deformed structure showed three different types of microstructure: very fine lamellar structure (with an average thickness of $0.14 \pm 0.05 \mu\text{m}$) elongated mainly along rolling direction (marked as A on Figure 3(a)), areas of extensive micro shear banding (marked as B on Figure 3(a)), and areas of martensite with high dislocation density (marked as C on Figure 3(a)). The observed micro shear bands are inclined at 40–60° with respect to the rolling direction (Figure 3(b)).

After annealing for 300s recovery occurred (Figure 3(c), (d)) with low levels of misorientations (2–3°) and an average cell diameter of $0.3 \pm 0.1 \mu\text{m}$ (Figure 3(c)). There were numerous carbide particles located at grain/subgrain boundaries and triple junctions. The carbide particles along with micro shear bands act as nucleation sites for recrystallisation (marked by an arrow in Figure 3(d)). The nuclei had average diameter of $0.15 \pm 0.08 \mu\text{m}$ and were characterized by high levels of mis-orientation ($>15^\circ$) with respect to the matrix (Figure 3(c)).

The formation of a cell structure with complex dislocation tangles in the cell walls and subgrains with lower dislocation density in their interiors is observed in the samples annealed for 30 min (Figure 3(e), (f)). The average size of the cells and subgrains was $0.3 \pm 0.08 \mu\text{m}$. The preferential sites for recrystallised nuclei formation were deformation bands and particles (Figures 3(e), (f)). However, deformation bands were still present in some areas (Figure 3(e)). Formation of dislocation walls across these bands was observed, leading to the formation of a cell structure in these regions (Figure 3(f)). Recrystallised grains and growth of individual grains was observed after annealing for 2h (Figure 3(g), (h)). The average size of coarse grains is $1.6 \pm 0.3 \mu\text{m}$ but their volume

fraction is low. A high volume fraction of subgrains was still present (Figure 3(g)).

The development of the ultrafine bainites arose from thermodynamic calculations by Bhadeshia and co-workers [20]. This led to an alloy design approach where nanostructured bainite is formed through the conventional isothermal heat treatment at relatively low temperatures (i.e. $< 200^{\circ}\text{C}$). A new generation of nanostructured bainitic steels appears to offer notable strength (2.3 GPa), toughness ($30 \text{ MPa m}^{1/2}$) and ductility (30%) and a very high strain rate sensitivity. The nanostructured bainite consists of very fine bainitic ferrite laths with an average thickness of $\sim 50 \text{ nm}$ and retained austenite films between these laths (Figure 4).

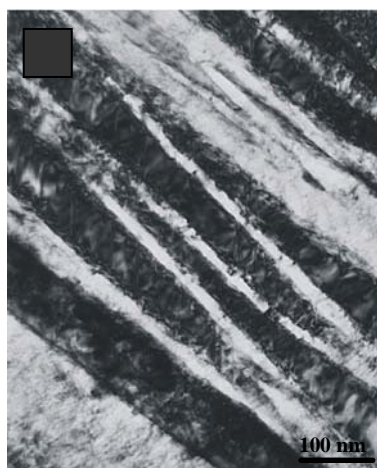


Figure 4: TEM image of nano-structured bainite transformed at 200°C

Recent work by our group revealed that the isothermal transformation temperature appears to have a significant effect on the retained austenite characteristics (i.e. size, morphology and composition) and variant selection of the bainitic ferrite laths [22]. In general, a decrease in the transformation temperature refined the bainitic structure (i.e. lath, block

and packet size) and weakened the variant selection. Interestingly, the variant selection mechanism at a temperature less than 250°C is similar to a martensitic structure [23] where the misorientation across adjacent packets is mainly greater than 40° (Figure 5). This can partly explain the notable combination of mechanical properties in nanostructured steels since high-angle bainite packet boundaries can efficiently stop the brittle cleavage microcracks propagation [24].

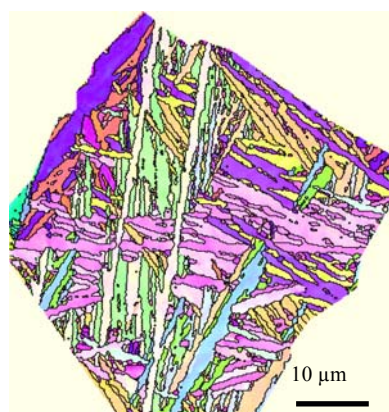


Figure 5: Inverse pole figure of an austenite grain transformed to nanostructured bainite at temperature of 200°C , respectively

COMPLEX MULTIPHASE STEELS

While the above dealt with potential steels of the future there is significant interest at present in the new advanced high strength steels for the automotive industry. These steels can have a range of phases, including ferrite, bainite, martensite and retained austenite. The focus of our work has been to understand the evolution of these microstructures and how the arrangements of different phases influence subsequent phase transformations and the final properties. This mostly involves microstructural engineering, but with the application of APT we are also

starting to consider local submicron compositional effects [25]. In the conventional annealing approach the phases tend to be simply distributed. So for example in TRIP steels the hot rolling process is designed to form a significant fraction of ferrite with a pearlite/bainite second phase after hot rolling and coiling. During the annealing process this second phase and some of the primary ferrite transform to austenite and the subsequent heat treatments then control the volume fraction of carbide free bainite and the nature and distribution of austenite.

In thermomechanically processed TRIP steels the process is more complex as the phases all need to be produced in a short controlled period of time. This complexity also provides the opportunity to distribute the various phases in quite different arrangements. For example, the addition of Nb allows the austenite to be preconditioned which then alters both the rate of ferrite transformation as well as the grain size and phase distribution [26]. Also the compositional profile within the phases is more complex as it is influenced by the order and temperature at which the phases form. In these steels the critical aspect is the location and composition of the retained austenite. This is the phase that provides the high levels of work hardening, but it depends on both the chemical and mechanical stability of this phase. For example, the study on the retained austenite stability in the TRIP steels showed that the retained austenite in the TRIP steels could be in five main morphologies: (i) islands in granular bainite (Figure 6(a)) (ii) thin films or layers between acicular ferrite laths (Figure 6(b)) (iii) islands between polygonal ferrite grains (Figure 6(c)), (iv) islands at the polygonal ferrite /bainite interface and retained austenite/martensite constituent (Figure 6(d)).

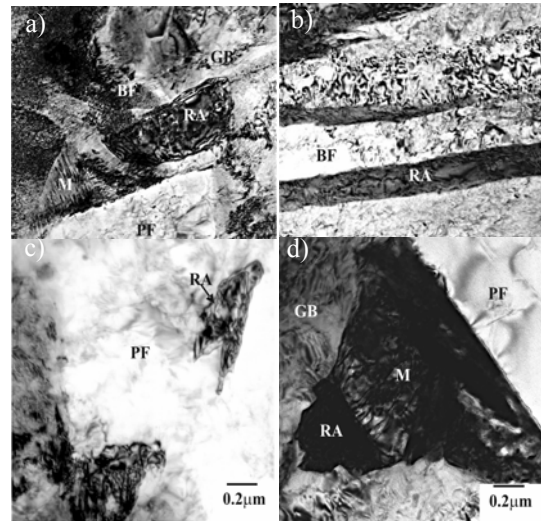


Figure 6: Thin foils TEM microstructures of TRIP steels a) island of retained austenite in granular bainite, b) acicular ferrite with interlayers of retained austenite, c) retained austenite in polygonal ferrite, d) martensite/retained austenite constituent, PF is polygonal ferrite, BF is bainitic ferrite, GB is granular bainite, RA is retained austenite and M is martensite.

Examination of the microstructures of the TRIP steels during straining revealed that the rate of retained austenite transformation during straining was affected by its location within the microstructure. The retained austenite crystals between the polygonal ferrite grains and at the bainite/polygonal ferrite interface transformed to martensite at early straining (Table 1).

However, TEM demonstrated some of the untransformed, coarse blocks of retained austenite in polygonal ferrite and at the polygonal ferrite/bainite interface in the microstructure after 0.14 strain (Figure 7(a)). Furthermore, some of the retained austenite crystals at the polygonal ferrite/bainite interface showed partial transformation to martensite (Figure 7(b)).

Table 1: Distribution of Retained Austenite (in Vol%) within the Microstructure of TRIP Steel at Different Amounts of Strain

Location of RA	Strain, (ϵ)			
	0-0.06	0.1%	0.14%	0.2%
Between BF	~70%	~90%	~95%	~99%
At PF/Bainite interface	~25%	~10%	~5%	<1%
Between PF	~5%	<1%	<1%	0%
Total amount of RA	11%	10%	8%	5%
Total amount of martensite	13%	14%	15%	18%

BF is bainitic ferrite, PF is polygonal ferrite, RA is retained austenite

It was also found that the kinetics of the retained austenite transformation during testing was affected by the morphology of the bainitic ferrite. The retained austenite formed between coarse, long, parallel bainitic ferrite laths transformed to martensite at an earlier stage of deformation than those located within granular bainite. Moreover, the regions of retained austenite crystals located in close proximity to the bainitic ferrite laths transformed faster than the areas in the middle of the austenite crystal (Figure 7(c)). The retained austenite between the refined bainitic ferrite laths appeared to be over-stabilised and was observed in the microstructure of the Nb-steel even after fracture (Figure 7(d)). The retained austenite between the bainitic ferrite grains in the granular bainite demonstrated the optimum stability with gradual transformation to martensite during deformation.

Hence, the mechanical properties of the complex multiphase microstructures are determined not only by the strain-induced

transformation of retained austenite, but also depend on the characteristics and interactions of all phase present in the microstructure. Both polygonal ferrite and bainite are responsible for a good combination of strength and ductility in the TRIP steels.

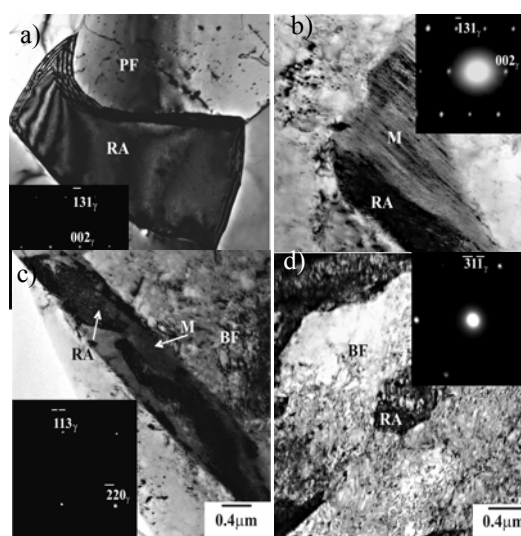


Figure 7: Thin foils TEM microstructures of TRIP steels a) retained austenite in polygonal ferrite after 0.14 strain (zone axis is $[310]_{\gamma}$), b) retained austenite/martensite constituent after 0.1 strain (zone axis is $[332]_{\gamma}$), c) retained austenite after 0.2 strain (zone axis is $[114]_{\gamma}$) and d) partial transformation of retained austenite (zone axis is $[310]_{\gamma}$).

Moreover, the morphology of the bainitic ferrite plays a vital role in the retained austenite stabilization. It has been demonstrated that the coarse blocks of retained austenite located in the polygonal ferrite or at the polygonal ferrite/bainite interface transform to martensite at an early stage of deformation, while the retained austenite present between the bainitic ferrite laths is more stable and can remain in the microstructure even after the testing to failure.

PRECIPITATION HARDENING

Precipitation hardening of the final product is an area that has been studied intensively for high strength steels. Until recently the level of strength that could be achieved was simply predicted by simple equations and design rules. However, recent work by JFE in Japan has challenged many of the established concepts in precipitation hardening and they have developed new combinations of alloys that appear to provide much greater levels of precipitation strengthening. The aim in this work is to utilise combined additions of different elements (eg Ti and Mo) to optimise the driving force for precipitation.

Conventional TEM has been widely used to study the different precipitates in micro-alloyed steels. However, there are a number of limitations. Firstly, due to the coherent or semi-coherent nature of these precipitates it is necessary to use foils rather than replicas. The precipitates also have defined crystallographic relationships with the parent phase and sometimes imaging can require large rotations of the specimen which due to the magnetic nature of ferrite can be an issue in many TEMs. This often leads to the appearance of precipitates in some grains and not in others. While this may actually be the case it is not always possible to rule out the limitations of the technique.

We have used APT to study both a conventional HSLA steel as well as one of the advanced steels developed by JFE (Figures 8 and 9). The advantage of this technique is that it clearly shows the nanoscale morphology of the precipitates as well as their composition and crystal structure. The disadvantage is that it still represents a very small sampling size and a number of samples would be required to develop a quantitative understanding of what

is occurring as well as whether this is occurring in all grains.

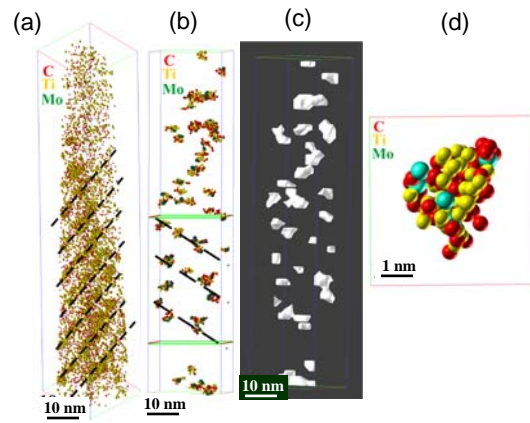


Figure 8: APT characterisation of particles in the advanced steel: (a) C, Ti, Mo atom map; (b) C, Ti, Mo particles. Dashed lines show suggested interface lines; (c) corresponding 1.75 atom% C + Ti + Mo iso-concentration surface; (d) selected atom map of $Ti_{0.98}Mo_{0.02}C_{0.6}$ particle. The matrix atoms for (b) were suppressed with the maximum separation method with $d_{max} = 1$ nm. Total number of atoms analysed in (a) is 3455274 [27]

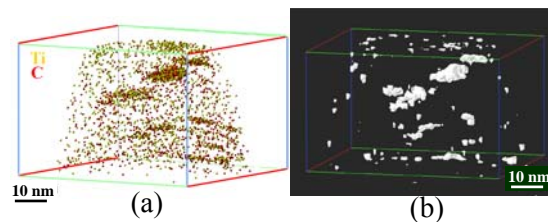


Figure 9: APT characterisation of particles in conventional HSLA steel: (a) C-Ti atom map; (b) Corresponding 1.7 atom% C + Ti iso-concentration surface. Total number of atoms analysed in (a) is 5225129 [27]

In our work the new approach to precipitate engineering by JFE is clear, with exceptionally fine, regularly spaced precipitates being formed by interphase precipitation, as evidenced by the row-like arrangement of the precipitates. In this case the average Guinier radius of particles was 2.2 ± 0.5 nm with

regular spacing of 7 ± 0.4 nm between these lines and with a distance between the particles in the row of 5.2 ± 0.2 nm (Figure 8).

In our current work, we are attempting to understand the role of thermomechanical processing and composition on the size and distribution of precipitates in complex microalloyed steels. We are also interested in the ability to provide precipitation hardening at lower temperatures and in more complex microstructures. One of the interesting aspects is clearly the interplay between transformation and precipitation and how this can also be manipulated to maximise both effects.

SOLUTE EFFECTS

One of the more surprising issues in the use of advanced high strength steels has been the additional strengthening from the bake hardening process. It has been observed [28] that the bake hardening response can vary significantly between various steel grades and even within grades (Figure 10). This is an interesting case where microstructural engineering is establishing conditions that can be used in nanostructural engineering. Our work has shown that one of the major differences between different advanced high strength steels lies in the carbon content of the ferrite. Dual Phase steels undergo a more severe quench to lower temperatures and therefore the supersaturation of C is higher than in TRIP steels where the coiling (or holding depending upon the process) and final transformations occur at a much higher temperature. This led to the segregation of carbon to linear features, such as dislocations formed during processing and after pre-straining (PS) in ferrite of the DP steel after bake-hardening (BH) treatment (Figure 11). Hence, the high concentration of interstitial

carbon in the ferrite matrix of DP steel and formation of high density of mobile dislocations around martensite islands entailed the segregation of carbon and the dislocation pinning during bake-hardening.

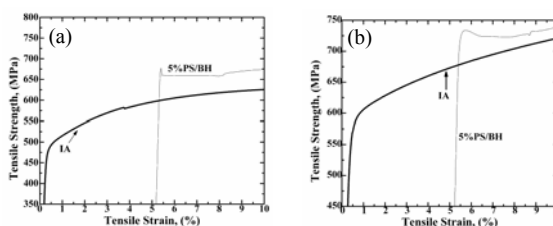


Figure 10: Representative stress-strain and strain-hardening rate curves after processing and PS/BH treatment: (a) DP steel and (b) TRIP steel

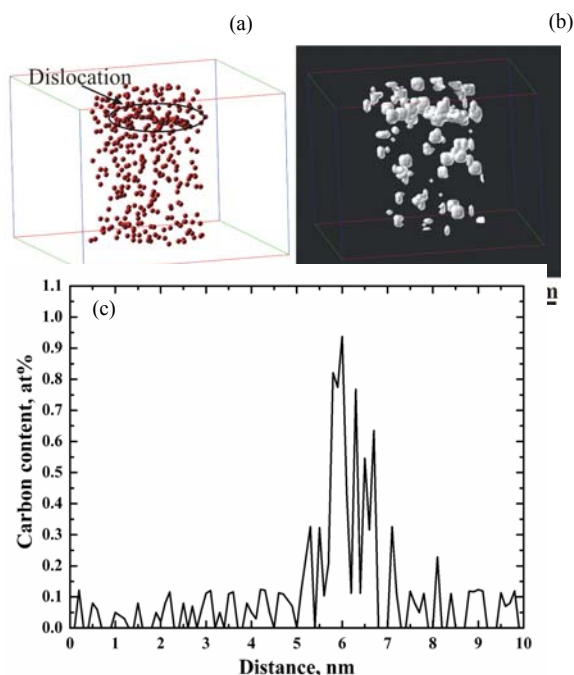


Figure 11: (a) Representative carbon atom map, (b) 1 atom% carbon iso-concentration surface, (c) corresponding carbon concentration profile across the dislocation observed in the polygonal ferrite of DP steel after PS/BH treatment

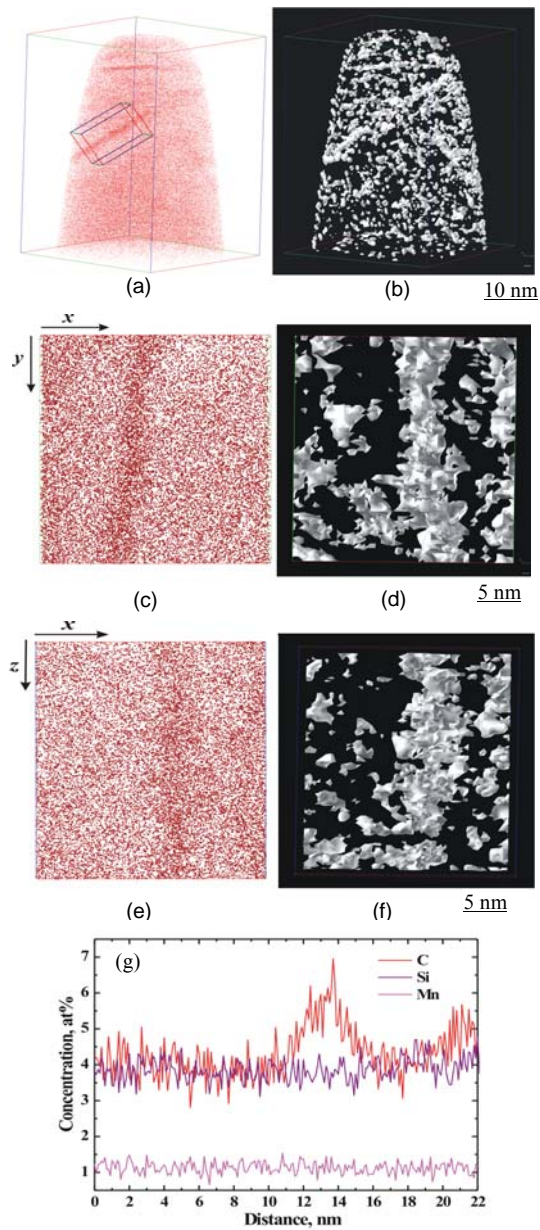


Figure 12: (a) Representative carbon atom map and (b) 7.5 atom% C iso-concentration surface of coarse retained austenite island in TRIP steel after PS/BH treatment, (c, e) selected carbon atom maps in two perpendicular directions shown in (a) and (d, f) corresponding 7.5 atom% C iso-concentration surfaces, and (g) compositional profile across retained austenite island

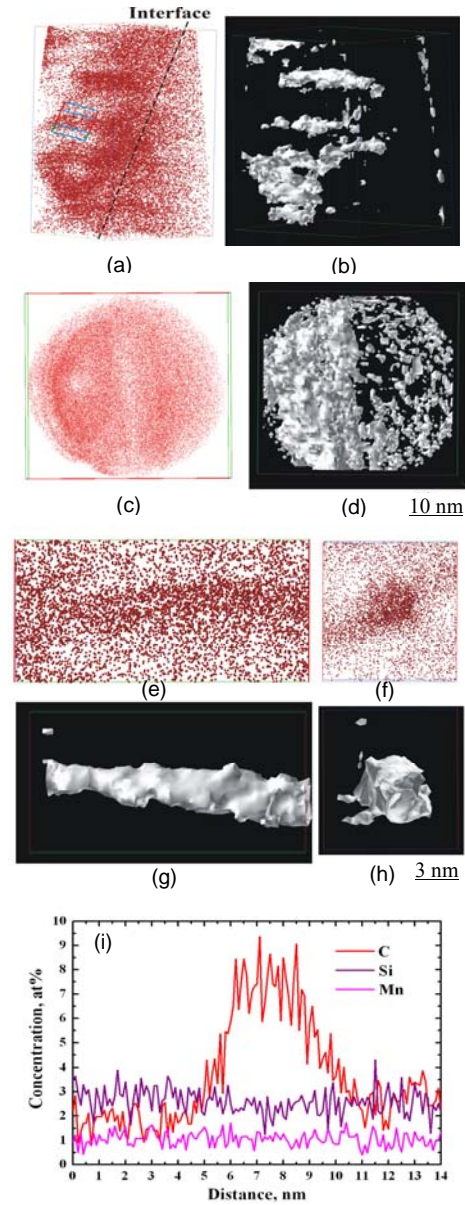


Figure 13: (a, c) Carbon atom map and (b, d) 8 atom% carbon iso-concentration surface of martensite/austenite crystals after PS/BH treatment in DP steel, (e, f) selected carbon atom maps and (g, h) 8 atom% selected carbon iso-concentration surfaces in two perpendicular directions of carbide in martensite and (i) corresponding carbon concentration profile across the carbide after PS/BH

The use of APT has also shown segregation of carbon to the particular crystallographic planes in retained austenite in TRIP steels (Figure 12) through to clear evidence of iron carbide precipitation in the Dual Phase steels (Figure 13). The differences in the behaviours suggests other ways of providing this additional strength increase (or in some cases this may want to be avoided) by altering aspects of the thermal cycle and microstructure evolution to control the supersaturation of C. Also other alloying elements will control the activity of C in these steels.

In the future, the study of solute effects will be extended to the engineering of grain boundaries either in the final microstructure to control the strength or at intermediate stages to control the transformation reactions.

NEW PROCESSING ROUTES

The above discussion has largely been related to conventional processing. However, increasingly the industry is moving to more near net shape processing. This includes thin slab casting, strip casting and a range of more novel spray casting approaches. Early work on hot direct rolling and thin slab casting simulations highlighted significant changes in the precipitate evolution. For example in Ti microalloyed steels some of the typical benefits from low Ti contents were lost.

It appears that the development of the fine nanoscale precipitates that are responsible for pinning the austenite are formed in conventional slab processing during the austenite to ferrite to austenite cycle. When this does not occur then the Ti is in solution (there are some coarse Ti present from the solidification process but these are ineffective for grain size control) and may precipitate during the rolling or in the product. Whether

these are effective in controlling the heat affected zone coarsening was not studied in that work.

Another example in the thin slab casting research was the behaviour of S, especially in the presence of Cu. Rather than the conventional coarse MnS, the S was retained in solution once a certain cooling rate was exceeded. Very fine nanoscale sulphides then formed during rolling. These had the potential to reduce the austenite recrystallisation and/or provide precipitation hardening of the product. More recently, researchers at NIMS in Japan have also been studying precipitate formation in strip cast carbon steels and again have found a range of nanoscale oxides and sulphides.

The above only relates to the development of novel precipitates but it is also expected that there will be other fine scale solute segregation and interactions that will alter the properties of the steel products.

MODELLING

The above has dealt with the physical evolution of the microstructure and the effect on properties. To allow the effects to be more generalised there is still great interest in combining experiments with modelling. However, there are a number of issues and limitations at present as you attempt to go below micron scale modelling; in fact even micron scale modelling still has some way to go. So-called multiscale modelling is attempting to combine models that work at different dimensional and time scales. Molecular dynamics approaches are needed to really understand atomistic effects, but their application is still restricted to a limited number of well defined problems. Our own multiscale modelling is still largely restricted

to combining macroscale (eg finite element) models with mesoscale (eg cellular automata) and microscale (eg realistic digital microstructures) to solve problems ranging from shear band formation [29] to recrystallisation [30] to the deformation behaviour of complex multiphase steels [31]. In the latter case we have demonstrated the advantage of using realistic digital microstructures for complex multiphase steels over conventional idealised microstructures. In Figure 14 the deformation of a TRIP steel is shown. Here the strain partitioning between the various phases and the interactions between the phases can be clearly seen.

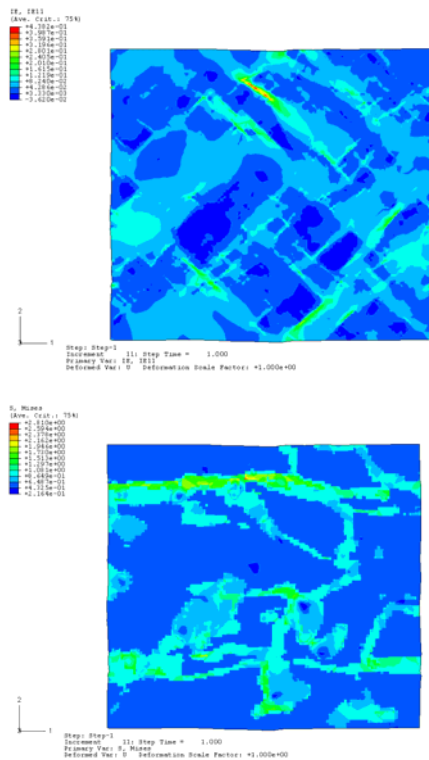


Figure 14: Strain partitioning (top) and stress partitioning (bottom) in a realistic microstructural unit cell of a three phase (ferrite, austenite and martensite) steel

In other work related to Cu, we have constructed finite element model for a mixed microstructure consisting of nanosized grains with larger grains embedded within it (Figure 15) [32]. In this work we are attempting to develop an understanding of how the ductility can be improved for these heavily refined microstructures. The role of the large grain in this case is to slow the development of shear bands which are responsible for failure in these materials.

In the future we will combine the two above approaches to consider the design of ultrafine microstructures. However, one of the barriers at present is in the development of the realistic digital microstructures where can place different phases with different properties and sizes in the microstructure.

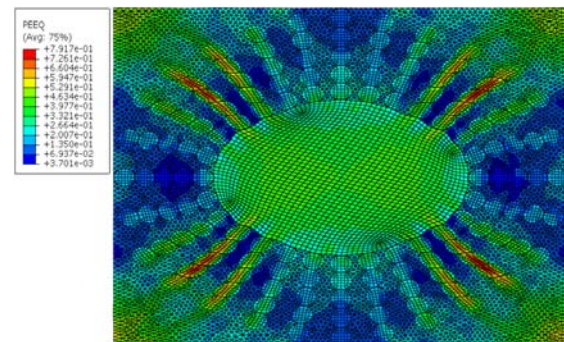


Figure 15: Von Mises strain contours in the deformed domain of unit cell models subject to prescribed displacement boundary condition at master node (Cu with multimodal microstructure (grain sizes: 10 μm , 1 μm , 100 nm deformed to 25%))

SUMMARY

Steel is a highly engineered material with a wide range of properties that can be tailored through control of the microstructure. Up until now the concept of microstructural engineering has been widely applied in steels and is

the basis for thermomechanical controlled processing, advanced high strength steels and other approaches. With the recent interest in developing ultrafine grained steels, new advanced complex multiphase steels, different design approaches to precipitation hardening and new near net shaped processing methods there is now interest in understanding and manipulating processes at the nanoscale. While in reality we are still probably operating across the micro and nano length scales there is no doubt that much of the future research in steel will be driven more from the characterization and manipulation of the nanoscale structures.

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