

Personal perspective on microstructure of steels: 25th anniversary of *MST* and collection of papers in honour of Sir Robert Honeycombe

I was assigned the task in this silver jubilee of *Materials Science and Technology* to select and introduce some of its memorable papers which in my opinion have contributed significantly to the understanding of steel microstructures over the last 25 years. For those of you who know me, *MST* is my favourite journal by a large margin, so it is a pleasure to be able to celebrate its achievements. Its pedigree can be traced to the widely respected *Metal Science* and *Metals Technology* journals; I am gratified that the former is now incarnated as an electronic archive and hope that the latter soon will be. Indeed, I would go so far as to suggest that the *Journal of the Iron and Steel Institute* should be similarly honoured in this wonderful age of electronic information; after all, its knowledge content is awesome by any standard.

I also take this opportunity to place in context a collection of new scientific papers written in honour of the late Professor Sir Robert Honeycombe, whose general contributions to physical metallurgy and seminal work on microstructure pervades the literature on iron and its alloys. His Royal Society biography has just been published¹ and makes inspiring reading, covering an era where progress seemed relentless, driven by a pure passion for discovery.

It is appropriate to begin with a wonderful paper by Sellars,² which has the distinction of setting the scene for two subjects, the philosophy of mathematical modelling and the high temperature microstructure of rolled steels; there is an earlier paper which I am neglecting because it deals mostly with the science of hot rolling.³ I believe that the 1990 paper² is the first to spell out why it is necessary, when dealing with complexity, to create models founded in understanding but which unashamedly exploit numerical and empirical approaches in order to avoid losing the plot. Put this together with Ashby's contention that there is an optimum level of model development after which simplicity becomes a virtue,⁴ and we have the defining features of mathematical modelling in the context of materials.

To illustrate the Sellars philosophy, the temperature compensated dependence of strain rate $\dot{\epsilon}$ on stress σ is given by⁵

$$\dot{\epsilon} = A[\sinh(\alpha\sigma)]^n \exp(-Q/RT)$$

where apart from the gas constant R and temperature T , other parameters can be regarded as empirical. The term $Z = \dot{\epsilon} \exp(Q/RT)$ is often called the Zener-Holloman parameter, which is usually derived by fitting to deformation data, and used in expressing the time $t_{0.5}$ required for half the material to recrystallise; for example² $t_{0.5} = 1.06Z^{-0.6} \exp(Q/RT)$. This of course is dimensionally incorrect, but I do not think there is any basic recrystallisation theory that has in practice been demonstrated to be capable of achieving better.

The potential of mathematical modelling is often overstated⁶ but when it comes to the hot deformation of steel, the work of Sellars and his colleagues,^{2,3,7-9} has led the world in terms of the implementation of the science into the red heat of industry. But equally astonishing is the fact that the work to this day stimulates basic research,¹⁰ and is cited in fields diverse from the original intention, for example in the context of transformation texture¹¹ and friction stir welding!¹² Rolling deformation involves multiple processing stages and there may typically exist more than 100 variables which determine the outcome;¹³ overly simplified research then leads to rather dull outcomes. The paper² dealt also with aluminium, but I do recall Mike explaining that it is only steel which is forgiving in the sense that errors of calculation are not propagated as the different stages of rolling are modelled; nevertheless, significant success has recently been reported by him, for an aluminium alloy where static recrystallisation does not affect the constitutive behaviour of the material.¹⁴

Most of the work described thus far focuses on the size, shape and deformation state of the austenite (γ) grains before they transform into ferrite (α). And it is well known that the state of the austenite prior to transformation has major consequences on the ferrite that evolves.^{15,16} An important paper by Umemoto *et al.*¹⁷ recognised empirically that, for a particular steel, the ferrite grain size $\bar{L}_\alpha \approx 5.7\bar{T}^{-0.26}\bar{L}_\gamma^{0.46}$, where \bar{L} represents a mean lineal intercept and \bar{T} is the cooling rate. They went on to investigate the fundamental basis for this using overall transformation kinetics theory.¹⁸⁻²² The depth of this work is impressive – for example, even the concentration dependence of the diffusivity of carbon was accounted for. In this respect, Umemoto assumed that it is only the diffusion of carbon that matters, and later work²³ seemed

to support this contention by demonstrating that the time–temperature–transformation diagrams calculated assuming that the ratio of iron to substitutional solute atoms remained constant during transformation, are more reasonable than those which assume all elements partition during transformation. Subsequent work^{24–26} has followed this practice of assuming paraequilibrium^{27,28} to exist at the transformation interface.

A worrying twist to this story comes from the seminal work by Priestner and Hodgson²⁹ who discovered that when the number density of independent ferrite particles forming at the grain surfaces of deformed austenite is large, they may impinge and coalesce before they have a chance to develop into fully fledged grains. The consequence is a ‘coarsening’ process in which the number of ferrite grains which participate in the final microstructure is not established until about one-third of the austenite is consumed, and that this number is smaller than existed at even earlier stages of transformation. This means that ferrite grain refinement due to thermomechanical processing cannot in principle be modelled simply on the basis of the nucleation count. As far as I am aware, this work has not made it into mathematical models, presumably because the necessary theory is not established, or because the fitting parameters in such models hide the problem.

Turning now to creep resistance, there has been substantial international activity with the goal of designing steels which can survive 100 MPa of stress at 650°C over a service period of 25 years or more, bearing in mind the requirements of weldability, oxidation resistance and cost.^{30,31} These are demanding requirements and early theoretical work³² indicating new designs failed to live up to its predictions³³ because of the unexpectedly rapid formation of undesirable phases and failure to take account of oxidation. Similarly, large experimental programmes have systematically failed to achieve the targets for two reasons. The first is that the creep rupture strength deteriorated sharply during long term testing. In a brilliant piece of work, Strang and Vodarek³⁴ discovered the presence of a previously unknown complex nitride with a unit cell similar to Z-phase found in austenitic steels. There have been many papers published which show that the formation of this phase is associated with the dramatic decline in creep rupture strength that occurs during prolonged testing.³⁵ Of course, this gave rise to a problem for those involved in mathematical modelling, because until recently, there were no thermodynamic data available to feed the microstructure calculations.

The second problem that has held up the utilisation of advanced creep resistant steels is that of type IV cracking,³⁶ a pernicious form of failure where there is an enhanced rate of creep void formation in the fine grained and intercritically annealed heat affected zone of the weld. It appears that this problem can be ameliorated by reducing the nitrogen concentration and adding 10–20 wt-ppm of boron to the steel,^{37,38} but the mechanism involved is not clear and long term test data are awaited.

A number of vital papers have been published in *MST* on the influence of stress on the development of microstructure in steels. The original by Denis *et al.*³⁹ is a beautiful expression of transformation plasticity in complex stress states and its outcomes in terms of changes in transformation temperatures as a function of these states are simple and elegant to apply. Tamura’s 1982 paper⁴⁰ in *Metal Science* (the predecessor of *MST*) has stood the test of time. On the basis of studies on deformation induced martensite, he claimed that the main factor contributing to the formation of martensite is thermodynamic, i.e. a mechanical driving force rather than an enhanced heterogeneous nucleation rate due to defects generated in the austenite. Many modern studies support this with quantitative evidence.^{41–44} The term ‘strain induced’ transformation seems to be used too loosely in the literature and may not in many cases be justified.

In fact, to do the calculations of stress effects properly, it is necessary to know the transformation strain with which the stress interacts. In the case of displacive reactions the deformation due to transformation is an invariant-plane strain (a combination of shear and uniaxial dilatation normal to the habit plane), which for martensite has traditionally been measured using optical microscopy because of the coarse scales involved. Unfortunately, for bainite, the individual platelets are finer than the wavelength of visible light, so early measurements using similar techniques gave unrealistic values of the shear strain at about 0.13, far less than predicted theoretically. This is because the strain was measured over sheaves of bainite rather than individual platelets. The very first paper to resolve this discrepancy⁴⁵ was published in *MST* and used the then novel technique of atomic force microscopy to give a shear strain value of 0.26 ± 0.02 , consistent with crystallographic theory. At the same time, it revealed the plasticity that occurs in the austenite adjacent to the bainite platelet, and which is responsible for the sub-unit mechanism originally proposed by Hehemann,⁴⁶ with the quantitative closure being achieved quite recently.⁴⁷ It should be pointed out that an earlier paper⁴⁸ actually detected the anisotropic transformation strains due

to bainite by a different method, dilatometry, by making polycrystalline austenite behave macroscopically as something akin to a single crystal.

It is worth mentioning in this context a publication by Alberly and Jones⁴⁹ in *Metals Technology* (the predecessor of *MST*) which demonstrated with utter clarity how transformation plasticity of the type described above can mitigate the residual stresses^{50–52} that are an anathema in welded engineering structures. This paper is undoubtedly the basis of modern efforts to design low temperature welding alloys.^{49–51,53–65}

Four papers in this special issue are related in one way or another to stresses and transformation plasticity. The accommodation of external stresses and strains by phase transformations is now widely exploited in the automotive industry, and a relatively new concept in which austenite is retained for this purpose by extracting the carbon from martensite is described by Edmonds,⁶⁶ and the role of niobium in influencing austenite films by Furuhashi.⁶⁷ And, indeed, stresses can be created within a material by changes in configurations or anisotropy of physical properties, as described comprehensively in the paper by Greenwood.⁶⁸ Such anisotropy can of course manifest in polycrystalline materials that are textured.⁶⁹

The term ‘acicular ferrite’ has not been common in discussing the microstructures of wrought steels, but some decades ago it took on considerable prominence in the context of weld metals, which intrinsically have a higher concentration of non-metallic oxides and other compounds, and at the same time have a large austenite grain size. The latter makes the number density of grain boundary nucleation sites small in comparison with nucleation on the non-metallic particles. The resulting intragranularly nucleated plates emanate from point sites, forming an overall chaotic arrangement which is presumed to interfere with the propagation of plates. Bainite, in contrast, forms as parallel platelets, making it possible for cleavage to propagate across clusters of these plates without much deviation. The very first paper on the subject in *MST*, written by Thewlis,⁷⁰ has made a big impression on the subject. The substance of the paper features in three textbooks. My lasting memory of the work is their identification that it is possible to obtain a particularly small misfit between the lattices of ferrite and galaxite, assuming that these two crystals orient themselves in a particular relation. This paper stimulated considerable research on the subject with a focus on lattice misfit.

The advent of electron back-scattered diffraction (EBSD)^{71,72} made it possible to examine the hypothesis that the good toughness of an acicular ferrite microstructure has its origin in the crystallographic dispersal of plates of ferrite. A marvellous paper by Gourgues *et al.*⁷³ showed experimentally that highly misoriented plates formed from the same inclusion, so that the boundaries between plates represent formidable barriers to crack propagation. It is necessary therefore, to distinguish between the grain size as measured ordinary microscopy and the crystallographic grain size measured using the EBSD method. If the crystallographic size is coarser, then that does not bode well for toughness.

There have been a number of other papers by Thewlis and co-workers on acicular ferrite.^{74–79} I do not concur with the rather subjective classification schemes and interpretations of mechanisms, but the dedication to the subject has to be admired.

There is a problem with EBSD techniques which plagues the subject and permeates all journals. People so frequently ignore the inaccuracies associated with the measurements. To claim that the orientation relationship between (say) martensite and austenite is Kurdjumov–Sachs or Nishiyama–Wasserman is simply incorrect since neither would lead to the required invariant-line between the two lattices, a line which makes it possible for displacive transformation to occur.^{80,81} The true orientation must be irrational and the accuracy required to characterise this is much greater than has been reported in the papers I have seen. And why are the relationships between like crystals quoted using only angles? An orientation relationship of this kind has three degrees of freedom and hence requires an axis and an angle of rotation to be specified.

Changing the subject, there was a certain kind of madness that pervaded science over the past ten years, where the adjective ‘nano’ became notorious. Huge numbers of papers were published on nanograined steels using exotic techniques which did not lead to a proper set of engineering properties; critical assessments have been published, among others, by Howe.^{82,83} Others working in the field of carbon nanotubes made premature claims about their potential for application as structural materials whose properties far exceed those of steels, so much so that *Materials World* reported some \$17 m being invested by NASA for a rope to make a space elevator. What was forgotten in all this publicity is the elementary fact that objects become weaker as they are made larger, because of the thermodynamically stabilised defects and also accidents of growth. This is fundamentally why the impressive mechanical properties of carbon nanotubes are not maintained (and indeed, should not be

expected to be maintained) when the tubes become long; it was in *MST* that this argument was first published,⁸⁴ with subsequent citations elsewhere.^{85–87}

However, there has been genuine excitement about an alloy system based on iron in which it has been possible to create a high density of interfaces by heat treatment alone. The resulting structure consists of a mixture of slender platelets of bainitic ferrite, just 20–40 nm in thickness, embedded in a matrix of carbon-enriched austenite. The rate at which this structure evolves is slow by conventional standards, but this permits components to be made which are large in all three dimensions, with uniform properties throughout. The first paper on this was published in *MST*,⁸⁸ the concept has been applied to the commercial production of high performance armour, and the work has stimulated research in many laboratories.^{89,90} The concept represents the world's first bulk nanostructured material.

Ferrite in the form of allotriomorphs and idiomorphs is the dominant phase in the vast majority of the 1.3 Gt of steel manufactured annually. In steels that are appropriately alloyed it becomes possible to observe a phenomenon known as interphase precipitation in which particles of a third phase precipitate at an advancing α/γ interface. This precipitate phase may be cementite, alloy carbides or other phases which have limited solubility in the majority phases at the transformation temperature. Examples include V_4C_3 (Refs. 91–94), NbC (Ref. 95), $Cr_{23}C_6$ (Ref. 96), and TiC (Ref. 97). There is a thoroughly readable review which formed the 29th Hatfield Memorial Lecture, delivered by Honeycombe;⁹⁸ the illustrations in this article are breathtaking in revealing the detailed mechanism by which interphase precipitation occurs, and this, of course, was the hallmark of the fine characterisation that he and his co-workers engaged in throughout their academic lives.

The characteristic feature of interphase precipitation is the fine dispersions observed as regular rows of particles, all of which usually have the same crystallographic orientation in any given ferrite grain. The process is for the most part associated with a step mechanism of α/γ interface motion, the particles precipitating on the stationary, immobile component of the interface⁹⁶ because the steps themselves move too rapidly to allow successful nuclei to develop.^{99,100}

So the idea of interphase precipitation has been around for four decades but, remarkably, it has just seen a new avatar.^{101,102} This is in the context of high strength, low alloy steels used in structural applications with a yield strength in the range 400–500 MPa. The process involves the introduction of minute particles of TiC or (Ti,Mo)C, which result in the yield strength being elevated to ~780 MPa while maintaining an elongation of some 20%. Typical compositions of such steels are in the range¹⁰¹ Fe–0.04C–0.2Si–1.5Mn–(0.02–0.2)Ti–(0–0.4)Mo (wt-%). Aspects of this are covered in Yang's paper in this issue¹⁰³ with further details in the article by Dunne.¹⁰⁴

Steel is a very big topic to cover and there are undoubtedly other aspects which should have been covered in this brief article. It is of course the future vitality of *Materials Science and Technology* that really matters and I can honestly say that I look forward to every new issue that is published. I congratulate the editors on this 25th anniversary and also thank the authors who wrote to respect one of the giants of our subject, Sir Robert Honeycombe.

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